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Research on Methods to Remove Mold From Cellulose Acetate Audiotape

I. INTRODUCTION

Mold on audiotape presents challenges that are different than mold on books and manuscripts. This difference is due to the materials used in the manufacture of the media and, perhaps more importantly, in the manner in which the media are used. When an audiotape is played, and especially when it is rewound, the tape is subjected to high rates of speed, and friction develops between the audiotape and the different components of the tape player, such as the play and record heads as well as the metal capstans. As a result, high potential exists for mold spores to become airborne where they can be a health hazard and where they can spread to other parts of the collection.

During the winter of 2005, my colleague, Rick Taylor, formerly a fellow student at the Kilgarlin Center for Preservation of the Cultural Record at the University of Texas at Austin, proposed research to investigate methods suitable for removing mold from cellulose acetate audiotape. We began research in February 2005. Such research seems justified since mold on audiotape is a fairly common problem and no standard method exists for removing the mold. For example, Allan McConnel (2005), of the Motion Picture, Broadcasting, and Recorded Sound Division at the Library of Congress, indicates that the Library encounters mold on audiotape periodically, but has not found a completely satisfactory method to remove the mold. Thus, mold on audiotape remains problematic for custodians of recorded sound collections.

2. RESEARCH GOALS

The goal of the research described in this paper was to develop a mechanical cleaning method and a solvent-facilitated cleaning method for removing mold from cellulose acetate audiotape. Both methods would allow cleaning an entire real of tape in a reasonable amount of time without damaging the tape. Methods to assess cleaning effectiveness and potential damage to audiotape would be used to determine whether the research goals were achieved.

3. DESCRIPTION OF AUDIOTAPES

Seven audiotapes, slated for disposal, were salvaged and used as test samples for this research project. The audiotapes are one-quarter inch, cellulose acetate audiotapes on 7-inch open-face reels. The tapes contain discussions of a political nature from State of Texas government proceedings. The tapes, now owned by Rick Taylor, are considered of low artifactual value and, therefore, could be subjected to various tests during our research. Although care was provided to protect the tapes as much as possible, some destructive analysis was conducted. Six of the audiotapes were used in the mechanical cleaning research, while the remaining tape, Tape No. 4, was used for the solvent-facilitated cleaning research.

Tape No. 4 appeared typical of the entire group of seven audiotapes. The tape had been housed in a 3M Company cardboard box. The manufacturer's label on the front of the box indicates the contents as "Scotch Brand Magnetic Tape." In the lower left corner, the tape product number is shown as "111" along with the descriptors, "Plastic" and "All Purpose." In the lower right corner, the "3M" logo is printed. Assuming the seven tapes were in their original containers, and based on package labeling, all seven tapes were manufactured by the 3M Company.

A graphite inscription on the top edge of the box indicates the tape was recorded on January 7, 1959, and the subject is described as "Sam Kimberlin". The bottom edge of the box states the tape product number as "111-12," along with the descriptions, "1200 feet" and "Plastic." Additional printed information on the inside of the cover indicates the tape contains a "silicone lubrication" to reduce recorder head wear.

3M first introduced audiotape carrying a product number of 111 in 1948 (Eilers 2000). This type of tape was reportedly very common among recording professionals. "The ubiquitous 111 tape became one of the most widely used tapes for professional applications in the 1950s" (Daniel et al. 1999, 75).

Delos Eilers (2000), formerly a Senior Technical Service Specialist at 3M Company, created a table containing data about open reel audiotapes introduced by 3M from 1947 to 1993. The table is provided in Appendix A of this report. According to the table, 3M Tape 111 contains a binder represented as Type B, a brown-colored oxide coating, and a backing made of acetate. The thickness of the backing layer is 1.42 mils and the thickness of the oxide coating is 0.5 mils for a total tape thickness of 1.92 mils. Thus, the backing layer is approximately three times as thick as the binder layer. The chart indicates that this type of audiotape was not back-coated. Tape No. 4 has a thickness of 0.002 inch (or 2 mils), as measured with a caliper, which is about the same thickness as that specified in the table. As Taylor (2005) notes, a comparison of the physical characteristics of the audiotape with 3M audiotape specifications published by Eilers (2000) reveals that the audiotape used in the research is consistent with the package labeling and product number in that the tape was manufactured with a cellulose acetate backing, is about two mils thick, and has a brown oxide coating.

An illustration of the cross section of this type of audiotape is provided in Figure 1 (Van Bogart 1995; Eilers 2000).

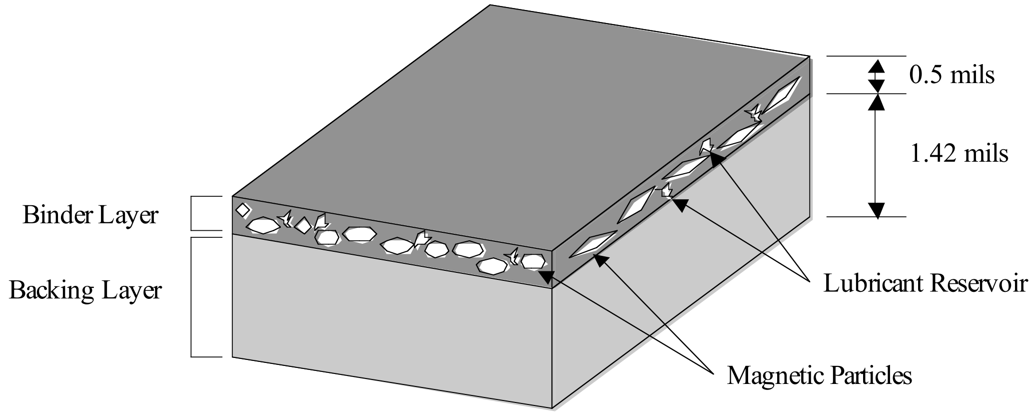


Figure 1. *Cross section of 3M Tape 111.*

4. CONDITION OF AUDIOTAPES

4.1 MOLD

4.1.1 Extent of Mold Coverage

All seven of the audiotapes were in similar condition in terms of extent of mold coverage. The tapes contained a moderate (approximately 40-50% coverage) amount of mold on both sides of the tape packs. The heaviest infestation of mold was located in the central portion of the tape packs. Photographs of Tape No. 4, typical of the seven audiotapes, are provided in Figures 2 and 3.

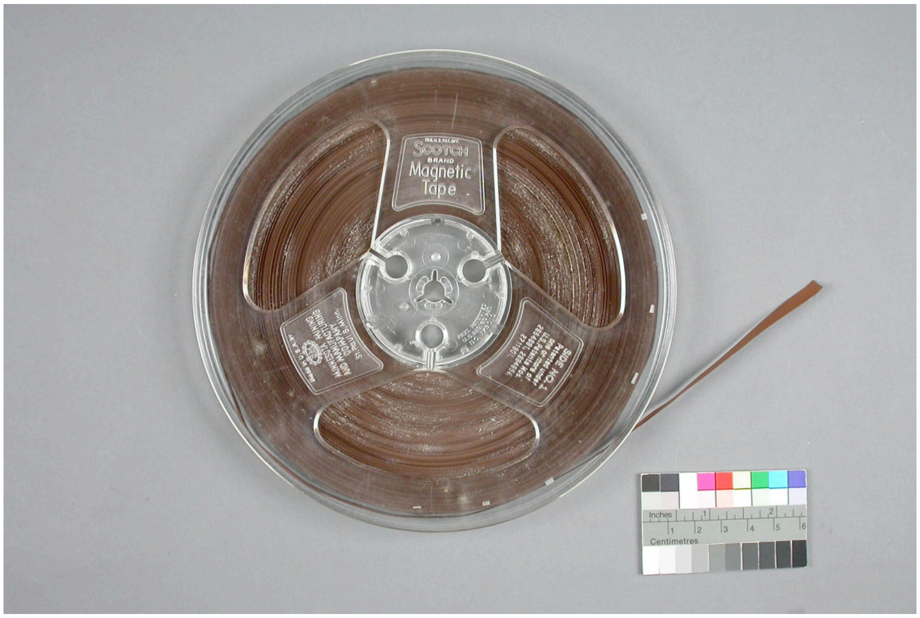


Figure 2. *Before-treatment photograph of Tape No. 4*

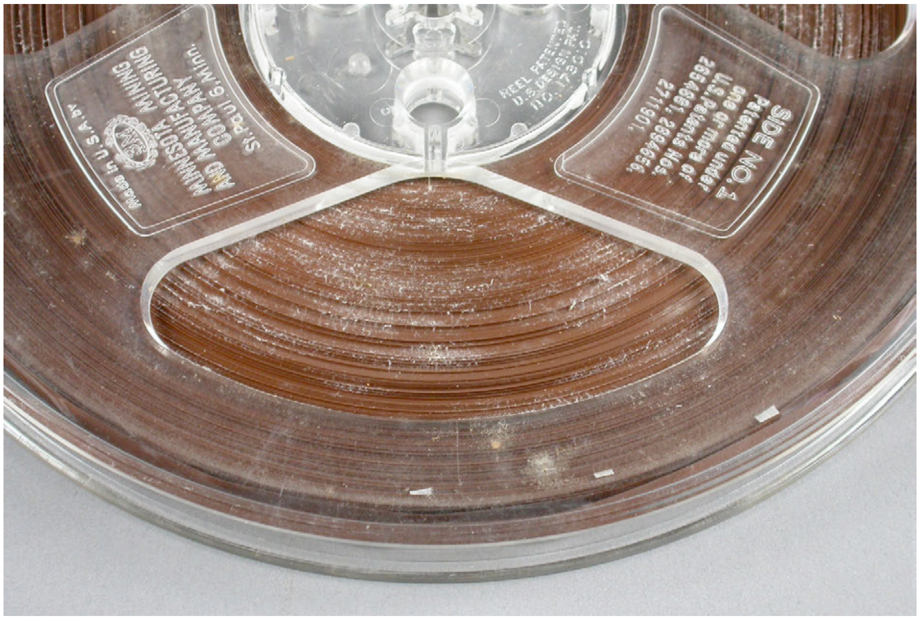


Figure 3. *Before-treatment photograph of Tape No. 4*

Where mold was present, the mold usually extended across the binder layer of the tape and, to a much lesser extent, across the backing layer.

4.1.2 Scanning Electron Microscopy

A Philips 515 scanning electron microscope (SEM) was used to collect high magnification images of both the backing and binder sides of the audiotape. Images were taken prior to cleaning of the audiotape to allow views of the undisturbed mold. The microscopy required destructive sampling since two pieces of tape (each measuring approximately 1 cm long) had to be covered with a metallic coating and mounted on a Cambridge-type aluminum mount with a carbon adhesive tab between the samples and the mount. A gold-palladium coating of approximately 10 nm thickness was applied to the two samples. The purpose of the coating is to make the mold more conductive to electricity and produce an electric signal closer to the outer surfaces of the mold, thereby increasing detail in the images. SEM images of the audiotape and mold are provided Figures 4, 5, 6, and 7.

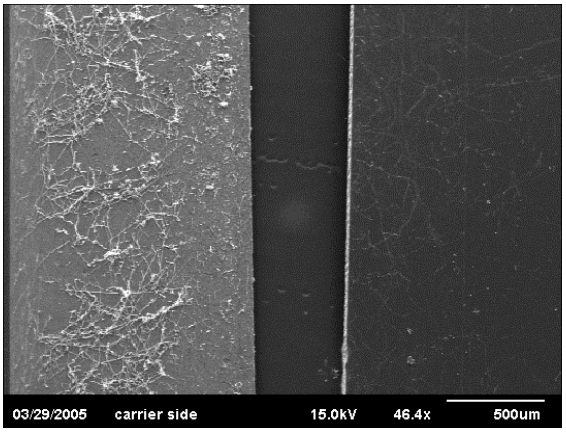


Figure 4. *Binder side is shown on left; backing side on right. Note heavier mold infestation on binder side.*
Photo by John Mendenhall.

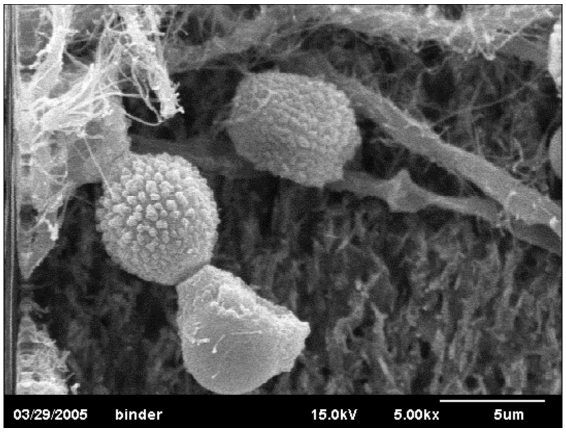


Figure 5. *Close-up of mold spores.*
Photo by John Mendenhall.

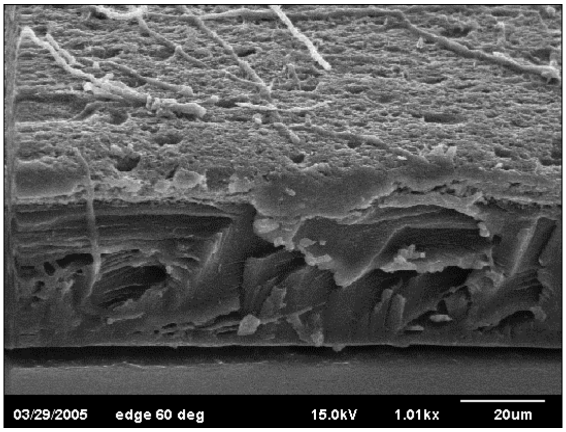


Figure 6. *Cross section of audiotape with binder side on top. Note the thinner binder in relation to the backing.*
Photo by John Mendenhall.

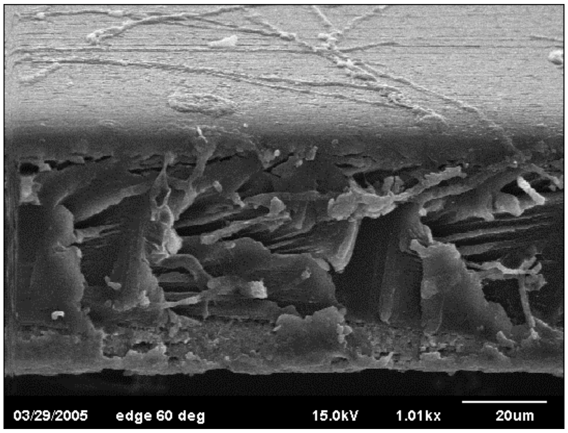


Figure 7. *Cross section of audiotape with backing side on top. Note the thicker backing in relation to the binder.*
Photo by John Mendenhall.

4.1.3 Sampling of Mold

As explained by Taylor (2005), prior to cleaning of the audiotapes, a sample of mold was collected from one of the audiotapes using a cotton swab and sent to an off-site laboratory for mold identification. The testing laboratory attempted to culture the mold on a malt extract agar for approximately one week to enable the mold to multiply under controlled conditions and to allow identification of the mold. The laboratory was unable to make the mold grow, possibly because the mold was dead. Mold is able to live in a dormant mode, but since the culturing process resulted in no growth, dormancy seems less likely.

4.2 EVALUATION OF PREVIOUS DEGRADATION

As described by Mills and White (1994), the primary cause of deterioration of cellulose acetate is hydrolysis of acetate groups, leading to deacetylation and liberation of acetic acid. The release of acid catalyzes additional hydrolysis of acetate groups and promotes hydrolysis and scission of the cellulose chain. As Ballany et al. (2001), explain, if hydrolysis results in scission of enough cellulose acetate chains, the polymer is weakened. Odor emanating from acetic acid, signifying the condition known as vinegar syndrome, was not noticed, but even small amounts of acid, including acids used in the production process, are sufficient to catalyze hydrolysis of the C-O bonds in the cellulose acetate backbone. Whether the tape has undergone hydrolysis is not known, but major hydrolysis is not evident because the tape was eventually cleaned under tension with no breakage.

Loss of plasticizer may lead to deterioration of cellulose acetate materials (Ballany et al. 2001). Sticky surfaces and brittleness are both indicative of plasticizer loss, but the audiotape exhibits neither characteristic. Cellulose acetate may also undergo oxidation at room temperature, but indicators of oxidation - brittleness and weakness, are not pronounced in this audiotape (Horie 1987).

Mold may also cause degradation of audiotape as described by Florian (2002). For example, enzymatic degradation can result in loss of structural polymers and, therefore, a loss in strength and increase in porosity. Mold can also cause pitting and localized surface losses. The extent of mold damage is unknown, but the tensile strength of the tape does not seem to have been seriously compromised, as described previously.

5. COMPOSITION OF AUDIOTAPES

5.1 BACKING LAYER

5.1.1 Cellulose acetate

The backing layer of 3M Tape 111 is reported as cellulose acetate (Daniel et al. 1999). According to Ballany et al. (2001), cellulose acetate is a generic term used to describe any of the following four different cellulose esters:

- Cellulose diacetate
- Cellulose triacetate
- Cellulose acetate butyrate
- Cellulose acetate propionate

Cellulose acetate propionate was manufactured only as a specialty polymer and apparently was not used frequently.

5.1.2 Plasticizers

Stannett (1950), writing around the time 3M Tape 111 was produced, indicates that plasticizers were almost always incorporated into cellulose acetate. Unplasticized cellulose acetate films are too brittle to serve most functions. Thus, an important reason for adding plasticizers is to increase flexibility of the cellulose acetate, but they serve other purposes too. Specific plasticizers may be added to increase water resistance and fire resistance. “Plasticizers also help in the manufacture of the plastic base by causing the mass to gel to a homogeneous whole, and so aiding in the working up of the plastic mix, prior to the sheet or moulding powder formation” (Stannett 1950, 55).

At the time of her writing, Stannett (1950) stated that the most common plasticizers used in cellulose acetate were the following:

- Dimethyl phthalate
- Diethyl phthalate
- Triphenyl phosphate
- Tricresyl phosphate
- Dimethoxyethyl phthalate
- Trichloroethyl phosphate
- Triacetin
- o- and p- Toluene ethyl sulphonamides
- Butyl phthalyl butyl glycollate
- Ethyl phthalyl ethyl glycollate
- Methyl phthalyl ethyl glycollate

Brydson (1989) writes that of the many plasticizers suggested for cellulose acetate, very few have been widely used. Historically, the most common plasticizers used in cellulose acetate were:

- Dimethyl phthalate
- Triphenyl phosphate
- Triacetin

5.2 BINDER LAYER

The exact composition of the binder is unknown since binder formulations were proprietary information (McKnight 2005). Despite these uncertainties, the binder includes metal oxide particles and a polymeric material to hold the metal oxide particles in place. Other compounds are also likely present, most notably plasticizers. According to St-Laurent (1996, 14), “Tape binder is somewhat susceptible to fungi growth though less so with modern tapes as fungicides are presently incorporated into the binder.”

5.2.1 Polymeric material

Over the course of many years, different polymers were used by 3M in the manufacture of binder layers for audiotapes (Eilers 2000). Tapes manufactured prior to the 1970's did not contain polyester polyurethane binders, regardless of manufacturer (Magnetic Reference Laboratory, Inc 1997). According to the chart prepared by Eilers (2000), the binder in Type 111 tape is denoted in a generic manner as Type B. Eilers (2005) states, "Our in house jargon for this type of binder was a vinyl binder." Pickett and Lemcoe (1959), state chlorine-bearing vinyls were used frequently at the time their report was published, the same year as the recording date of Tape No. 4.

5.2.2 Iron oxide particles

Audiotape contains magnetic metal oxide particles embedded in the binder layer allowing sound to be stored and later replayed. Labeling on the box containing Tape No. 4 indicates the product as Type 111-12. According to Daniel et al. (1999), Type 111 audiotape incorporated gamma ferric oxide (γ-Fe_2O_3) particles as the recording medium, which improved the erasure and re-recording functions of a tape. Gamma ferric oxide is a reddish-brown iron oxide. Although this type of oxide was first introduced as a recording medium in 1939, 3M did not incorporate this oxide into its tapes until 1948. Gamma ferric oxide remained the dominant type in audiotapes until the introduction of chromium dioxide in 1971.

5.2.3 Plasticizers

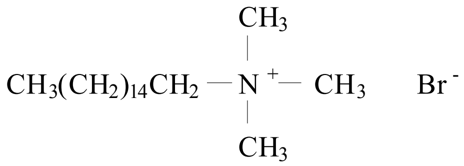
Plasticizers may have been added to the binder material of audiotapes (Pickett and Lemcoe 1959). Whether plasticizers were added to the binder of the audiotapes used in this research is unknown due to the proprietary nature of the manufacturer formulations and lack of historical data. Some polymers may warrant the inclusion of plasticizers to aid in the manufacturing process or to provide the requisite flexibility in the finished product. For example, polyvinyl chloride (PVC) is a very hard, rigid polymer. In order to make the polymer flexible, plasticizers are added to the compound during manufacture (Streitweiser and Heathcock 1985). Some polymers are plasticized internally by incorporating side chains onto the polymers, eliminating the need to add separate, external plasticizers.

6. CONSULTATIONS AND LITERATURE REVIEW

Several people with audiotape experience were consulted and literature was reviewed during the planning stages of this research to learn of potential cleaning methods and solvents. The following provides a summary of what was learned.

6.1 CANADIAN CONSERVATION INSTITUTE

Joe Iraci (2005), a Senior Conservation Scientist at the Canadian Conservation Institute, indicated that mold spores can migrate easily into the different layers of audiotape and are difficult to remove completely; so chemical treatment of the audiotape may be required. He stated that 1,1,1-trichloroethane, cetrimonium bromide, and ozone could be considered for cleaning audiotape, but cautioned that an acetate backing layer may be destroyed by these compounds. 1,1,1-trichloroethane and cetrimonium bromide were both considered for use as cleaning solvents in this research. 1,1,1-trichloroethane is a chlorinated alkane and, historically, has been a very common solvent for various purposes. Cetrimonium bromide, a quaternary ammonium compound, is a white powder, soluble in about 10 parts water, with a chemical composition as shown in Figure 8 (Frear 1969; Budavari 1996; Pouchert, C.J. 1997; Mallinckrodt Baker, Inc. 2004):



$$CH_3(CH_2)_{14}CH_2 - \overset{CH_3}{\underset{CH_3}{N^+}} - CH_3 \qquad Br^-$$

Figure 8. Cetrimonium bromide

Cetrimonium bromide has traditionally been used as a cationic detergent, antiseptic, and as a laboratory reagent (Budavari 1996).

Ozone, a gas, was not considered for this research due to its special handling and equipment requirements, as well as health hazards. Iraci (2005) recommended against the use of 2-propanol for cleaning of cellulose acetate audiotape due to the likelihood of damage to the backing layer.

6.2 3M COMPANY

Eilers (2005), provided guidance stating, “About the strongest ‘solvent’ I’d consider is IPA. We also used to clean tape using Freon TF, which is now not an acceptable solvent in the environment.” He suggested dry wiping the tape using a tape-cleaning fabric and stressed the importance of testing cleaning approaches on non-valuable tape of the same type and problem.

The term “IPA” in the above quote is likely an acronym for isopropyl alcohol. Isopropyl alcohol is synonymous with 2-propanol. Based on rules of nomenclature established by the International Union of Pure and Applied Chemistry (IUPAC), the solvent is designated as 2-propanol, so that name is used in this paper (Streitwieser and Heathcock 1985).

6.3 IMATION COMPANY

Richard Jewett of Imation Co. (2005) was involved in cleaning of Ampex 797 tape from an instrumentation recorder recovered from the Columbia shuttle crash. He stated, "We used DI water when we cleaned up the NASA tapes from the Columbia, dried them with clean lab cloths followed by a N2 gas blow off after which we let the media sit overnight to reach ambient environmental conditions before rewinding."

Jewett (2005) recommended the following procedure for cleaning mold from the 3M cellulose acetate audiotapes:

1) Use the transport as a cleaning station (Most audio recorders ran at 1-7/8, 3-3/4, and 7-1/2 " per second. I would use the slowest speed possible. Go slow and be careful)
2) Place a wiping station on your transport composed of four small rolls of lab cloth, two for the front and two for the back. Wet the leading one with DI water and leave the other dry. Mount these in a way so that the front and back gets wiped.
3) Mount the tape on the transport and thread it in such a way that you avoid the heads (or cover the heads with a piece of the wiping cloth)
4) Prevent running the tape over the heads until you have the mold removed. After the tape has been wiped clean end to end, then rewind it using the normal tape path but tape a small piece of lab cloth over the heads. If no mold is observed in this step you are good to use the tape. Watch for the telltale green.
5) Clean up the transport with alcohol, especially the capstan and rubber pinch roller. Wipe it very clean.
6) Play the tape.

6.4 VidiPax

Abbey Newsletter (2001) describes the process VidiPax recommends for removing mold from audiotape. VidiPax recommends vacuuming dry mold from a tape pack using a HPEA vacuum equipped with a soft brush attachment. A fiberglass screen is placed over the tape pack through which mold can be removed by the vacuum. After vacuuming, the tape is given a final cleaning using a buffer-winder cleaning machine. This machine is described as a proprietary process whereby the tape is moved at high speed across rolls of dust-free fabric. A vacuum is used to remove mold from the fabric during the cleaning process.

6.5 SPECS BROS., LLC

Peter Brothers, President of SPECS BROS., LLC, (1997) recommends first driving active mold into dormancy by storing the moldy tape in a low humidity environment. He states the tape can then be cleaned using a number of different methods, but that chemical decontamination is necessary to kill the mold.

6.6 UNESCO

The United Nations Educational, Scientific and Cultural Organization (UNESCO 1997) provides similar recommendations as those provided by VidiPax. UNESCO suggests moving moldy audiotape to an environment having a relative humidity of less than 50% and a temperature of less than 75^{o}F. The tape pack is vacuumed, avoiding direct contact. Remaining debris is removed from the tape pack using a brush. The audiotape is then run through a winder-cleaner with an appropriate fungicide. No further detail is provided on the winder-cleaner nor the fungicide.

7. MECHANICAL CLEANING METHOD

As explained by my colleague (Taylor 2005), prior to mechanical cleaning of the audiotapes, the audiotapes were placed in a desiccant chamber for several weeks. Drying of the mold was conducted to facilitate removal of the mold.

As Taylor (2005) explains, the mechanical method of mold removal consisted of first vacuuming the sides of the tape packs using a vacuum equipped with a high efficiency particulate air (HEPA) filter. Vacuuming was followed by a more aggressive cleaning effort consisting of running both sides of the tape past polyester/rayon cleaning fabric on a commercial tape player (TEAC 3300-10) in a semi-automated process. A commercial reel-to-reel audiotape player was used to wind the tape as the tape was cleaned. The tape player was retrofitted with a brass plate to which two fabric winders and two posts are mounted. The fabric winders allow the operator to continually advance two lengths of cleaning fabric during the cleaning operation, exposing clean fabric to the upper and lower sides of the audiotape. The two posts create a path around which the lengths of cleaning fabric are advanced, and around which the audiotape is run. A schematic diagram showing the conceptual design of the mechanical cleaning apparatus is provided in Appendix B. The completed mechanical cleaning apparatus, based on this design, is shown in Figure 9.

Figure 9. *Mechanical audiotape cleaning apparatus. Photo by Rick Taylor.*

The fume hood in the laboratory at the Kilgarlin Center is not equipped with a filter designed to capture biological matter, so the audiotape cleaning operation was conducted outside. During the cleaning process, the operator wore an air purifying respirator.

8. SOLVENT-FACILITATED CLEANING METHOD

8.1 SELECTION OF CLEANING SOLVENT

As often occurs in the conservation field, a balance is necessary between removing harmful or damaging material from an artifact while preventing or minimizing adverse effects of the treatment process. Several factors were considered during the solvent selection process consisting of the following:

- Solubility of audiotape
- Solvent evaporation rate
- Toxicity of the solvent to humans
- Toxicity of the solvent to mold
- Availability and cost of the solvent

8.1.1 Solubility of Audiotape

A solvent used to clean mold from an audiotape must be compatible with the audiotape material. The chosen solvent should not dissolve or react in other ways with components of the audiotape. This presents a challenge because of the proprietary nature of the audiotape materials; the exact composition of the tape at the time of manufacture is unknown. A compounding factor is the chemical reactions that may have occurred within the tape over the past 45+ years, possibly causing changes in the original tape composition. Less is known about the composition of the binder layer than the backing layer, but more stable vinyl binder systems were used during the 1940's and 1950's for cellulose acetate tapes than for subsequent polyester tapes (Van Bogart 1995). The backing of Tape No. 4 is known to consist of a form of cellulose acetate (most likely containing additives), so solubility considerations will focus on the cellulose acetate backing material.

Cellulose acetate is produced by heating a mixture of cellulose and acetic anhydride along with a catalyst such as sulfuric acid (Horie 1987). The reaction is illustrated in Figure 10 and is simplified to show the substitution of hydroxyl groups by acetate groups on one glucose unit of a cellulose chain. In this example, each of the three hydroxyl groups on the glucose unit is replaced by an acetate group forming a fully acetylated, triacetate product (Stannett 1950; Streitwieser and Heathcock 1985; Heinze and Liebert 2004).

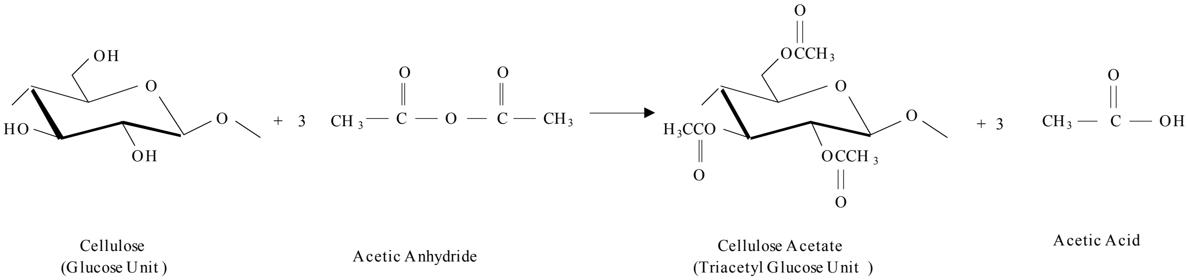


Figure 10. *Complete acetylation of cellulose to produce cellulose triacetate.*

As Horie (1987) states, the degree of substitution (DS), or degree of acetylation, of the hydroxyl groups along the cellulose chain affects the chemical properties of the polymer, including solubility. Brydson (1989) reports that partial acetylation of hydroxyl groups has the following effects on the polymer:

- A reduction of intermolecular hydrogen bonding
- An increase in intermolecular separation
- A reduction in the polarity of the polymer

Thus, as the degree of substitution of cellulose acetate increases, the polymer becomes less polar (Brydson 1989).

Horie (1987, 583) writes, "Because of these influences cellulose acetate can be dissolved in a variety of media, although a liquid suitable as a solvent for cellulose acetate with a degree of substitution of two would be unlikely to be a solvent for acetates with degrees of substitution of either one or three." The explanation of this property is corroborated by Ballany et al. (2001) who indicate that the four forms of cellulose acetate exhibit different solubilities. For example, cellulose diacetate is soluble in acetone, and cellulose triacetate is soluble in dichloromethane. Cellulose acetate butyrate is insoluble in either solvent.

Heinze and Liebert (2004, 204) state, "It is obvious that a correlation between DS and the polarity of the solvents exists. The solubility shifts to more non-polar solvents with increasing DS." Cellulose acetate with very low DS values (i.e., 0.5 to 1.1) has been reported to be soluble in water. Brydson (1989) writes that increasing the degree of substitution beyond that of cellulose diacetate increases resistance to water. According to Horie (1987), at a degree of substitution of 2.4, the polymer is soluble in acetone and similar polar solvents. At a degree of substitution of 3, less polar solvents such as chlorinated solvents are required to dissolve the polymer.

Horie (1987) lists the following compounds as non-solvents for cellulose acetate having a degree of substitution of 2.4:

- Heptane
- Cyclohexane
- Toluene
- Xylene
- Tetrachloromethane
- Diethyl ether
- 2-Ethoxyethanol
- 2-Butoxyethanol
- Isopropyl acetate
- n-Butyl acetate
- Ethane-1,2-diol
- Ethanol
- Propan-2-ol [Note: This is synonymous with 2-propanol]
- Butanol
- Dimethyl sulphoxide
- Water

This list was used as a starting point for consideration of potential solvents for cleaning mold from the audiotape, but not all of the listed solvents were considered. Of the compounds listed above, ethanol and 2-propanol were given the most consideration based on toxicity to humans and on its availability. Based on information received from Iraci (2005), 1,1,1-trichloroethane and cetrimonium bromide were also considered. A separate subsection is devoted to the consideration of water as a solvent.

8.1.2 Solvent Evaporation Rate

A solvent with a rapid evaporation rate was desired for the cleaning process for the following two reasons:

- To minimize the amount of time the solvent was in contact with the audiotape, thereby minimizing potential damage to the tape, and
- To allow the tape to dry completely before the tape is re-wound onto the take-up reel.

Other factors being equal, a solvent with a rapid evaporation is less likely to dissolve the surface of the audiotape than a solvent with a slower evaporation rate. Miller-Chou and Koenig (2003, 1223) state, "Unlike non-polymeric materials, polymers do not dissolve instantaneously, and the dissolution is controlled by either the disentanglement of the polymer chains or by the diffusion of the chains through a boundary layer adjacent to the polymer-solvent interface."

An aqueous solution of cetrimonium bromide would likely have an evaporation rate similar to water, so the evaporation rate of cetrimonium bromide was approximated based on that of water. Thus, the relative evaporation rates of 1,1,1-trichloroethane, 2-propanol, and cetrimonium bromide can be described by the following expression (Horie 1987):

1,1,1-Trichloroethane > 2-Propanol > Cetrimonium bromide

Based on relative evaporation rates given by Horie (1987), 2-propanol evaporates approximately 8 times faster than an aqueous solution of cetrimonium bromide. 1,1,1-trichloroethane evaporates approximately 17 times faster than an aqueous solution of cetrimonium bromide.

Thus, from an evaporation rate perspective, 1,1,1-trichloroethane and 2-propanol appear more suitable than cetrimonium bromide for an audiotape cleaning process. The slow evaporation rate of an aqueous solution of cetrimonium bromide could potentially be overcome by mixing the cetrimonium bromide in a non-aqueous solution.

8.1.3 Solvent Toxicity to Humans

The low toxicity of ethanol to humans, compared with other solvents, makes ethanol appealing from a toxicity perspective. Although 1,1,1-trichloroethane presents toxicity concerns, the solvent was eliminated from further consideration based on Clean Air Act restrictions as described below in the subsection titled "Availability and cost of solvent."

A review of the Material Safety Data Sheet for cetrimonium bromide (Mallinckrodt Baker, Inc. 2004) reveals that the material, if inhaled, may irritate the respiratory tract. Dermal contact may cause redness, itching, and pain. Absorption through the skin is possible, resulting in potential systemic effects. Ingestion may result in irritation of the gastrointestinal tract.

Aliphatic alcohols such as 2-propanol are considered of low acute toxicity (EPA 1995). Based on the Material Safety Data Sheet for 2-propanol (Fisher Scientific Corp. 2004), the solvent may cause adverse health effects in humans if safety precautions are not followed. Although dermal absorption is considered toxicologically insignificant, chronic exposure may cause defatting and dermatitis. Inhalation of high concentrations of vapors may affect the central nervous system resulting in nausea, headache, dizziness, unconsciousness, or coma. Ingestion may cause serious health effects, but this exposure route is not considered probable during an audiotape cleaning operation.

8.1.4 Solvent Toxicity to Mold

Historically, both 2-propanol and cetrimonium bromide have been used as general purpose fungicides (Frear 1969). 2-Propanol is registered by the Environmental Protection Agency as a fungicide (EPA 1995). The mode of action of alcohols on microbes is through disruption of cell membranes, dissolution of lipids, and denaturation of proteins by acting on the sulfur-hydrogen functional groups (University of Colorado at Boulder 2005). Quaternary ammonium compounds such as cetrimonium bromide act on microbes through inactivation of energy-producing enzymes, denaturation of cell proteins, and disruption of cell membranes, but are usually ineffective against spores (University of Colorado at Boulder 2005).

A product named *Microban X-580 Institutional Spray Plus* was used by personnel for washing of moldy artifacts salvaged from areas impacted by Hurricane Katrina (Striegel 2006). This product contains 2-propanol and a nonionic surfactant at concentrations of 10-30% and 1-5% by volume, respectively (II Rep-Z, Inc. 2004).

One must exercise caution in relying on historical information regarding fungicides. As Lee (1988, 39) states, “Of the fungicides recommended in the literature, most have proved ineffective in terms of long term protection or deleterious to the materials themselves.” Some solvents can actually encourage the growth of mold. For example, ethanol reportedly may activate conidia, a common type of mold spore (Florian 2002). Thus, ethanol has traditionally not been used as a fungicide (Wiswesser 1976). For this reason, ethanol was eliminated from further consideration in this research.

The toxicity of a solvent to mold is determined not only by the chemical characteristics of the solvent, but also upon factors such as level of microbial contamination and the duration of time that the solvent is in contact with the mold (University of Colorado at Boulder 2005). A given solvent may be toxic to mold at one time duration, but may have little effect at shorter durations. Therefore, a need exists to balance cleaning speed and evaporation rate with toxicity.

8.1.5 Availability and Cost of Solvent

According to the U.S. Environmental Protection Agency (1996), the use and production of both 1,1,1-trichloroethane (also known as methyl chloroform) and CFC-113 (also known as Freon 113 and Freon TF) have been banned in the United States under the Clean Air Act phase-out of ozone-depleting chemicals. As of January 1, 1996, these solvents cannot be produced or used in this country unless the solvent is obtained from an existing inventory or recycled. Seven exemptions are allowed for CFC-113, including for use in document preservation sprays (limited to books or paper in tightly bound documents) and for lubricants, coatings, and cleaning fluids for electronic equipment. Whether the latter exemption would apply to cleaning of audiotapes is unknown, so consultation with the U.S. Environmental Protection Agency is warranted.

In contrast, 2-propanol is easily obtained. A 1-liter bottle of reagent grade 2-propanol is available from Fisher Scientific for under $25.

8.2 SOLUBILITY TESTING

Prior to solubility testing, a dry cotton swab was rubbed along both sides of a short (approximately one centimeter long) piece of audiotape from Tape No. 4. No offset occurred from either side of the audiotape using a dry swab.

Solubility testing was conducted on a short (approximately one centimeter long) piece of audiotape from Tape No. 4. Separate solubility tests were conducted using water and 2-propanol. The tests were completed by swabbing the tape with a cotton swab saturated with either water or 2-propanol and noting whether offset occurred on the cotton. Both water and 2-propanol caused a rust-colored offset during swabbing of the binder layer, but no offset occurred from the backing layer.

8.3 WATER AS A SOLVENT

Based on its low toxicity to humans, low cost, and convenient availability, water was the first solvent considered for cleaning the audiotape. But several disadvantages resulted in quickly eliminating water from further consideration. The disadvantages consist of the following:

- Water is slow to evaporate, so the audiotape may not dry before being re-wound onto the take-up reel.
- Water is not only non-toxic to mold, but essential for its growth, so water may encourage growth of mold or reactivate dormant mold.
- Water could promote hydrolysis of the ester groups in cellulose acetate. Although the hydrolysis reaction is slow, it is catalyzed strongly by the presence of acids (Streitwieser and Heathcock 1985). Small amounts of such acids may be present in cellulose acetate audiotape, either as remnants of the manufacturing process, which utilized acid reagents, or as degradation products formed within the tape since the time of manufacture over 45 years ago.

8.4 SOLVENT SELECTED

After consideration of potential cleaning solvents, 2-propanol was selected because the solvent:

- Evaporates rapidly
- May have fungicidal properties
- Has low toxicity to humans
- Is inexpensive and readily available

Although Iraci (2005) advised against its use, and solubility testing performed with a swab saturated with 2-propanol resulted in some rust-colored offset, the decision was made to proceed with cleaning the audiotape with undiluted 2-propanol. Potential changes in the composition of the tape would provide an opportunity to test the ability of FT-IR to detect the changes as well as an opportunity to test the statistical method used in this research for assessing such changes.

9. APPLICATION OF SOLVENT TO AUDIOTAPE

9.1 CLEANING APPARATUS

In order to clean the audiotape in an efficient, cost effective manner, a cleaning apparatus is necessary to allow cleaning while the tape moves from the supply reel to the take-up reel. For this research, a TEAC 3300-10, commercial tape player was employed in a semi-automated process. The tape player winds the tape, allowing the operator to concentrate on application of solvent. A third reel is provided to lengthen the path of the audiotape between the supply and take-up reels, which allows room for a cleaning platform and additional time for the audiotape to dry before winding onto the take-up reel. A schematic diagram showing the conceptual design of the solvent-facilitated cleaning apparatus is provided in Appendix C. In practice, the tape player was moved completely onto its side as shown in Figure 11 to maximize the distance between the forward-moving and backward-moving lengths of audiotape. The extra space was helpful in positioning the cleaning platform between the two lengths of audiotape.

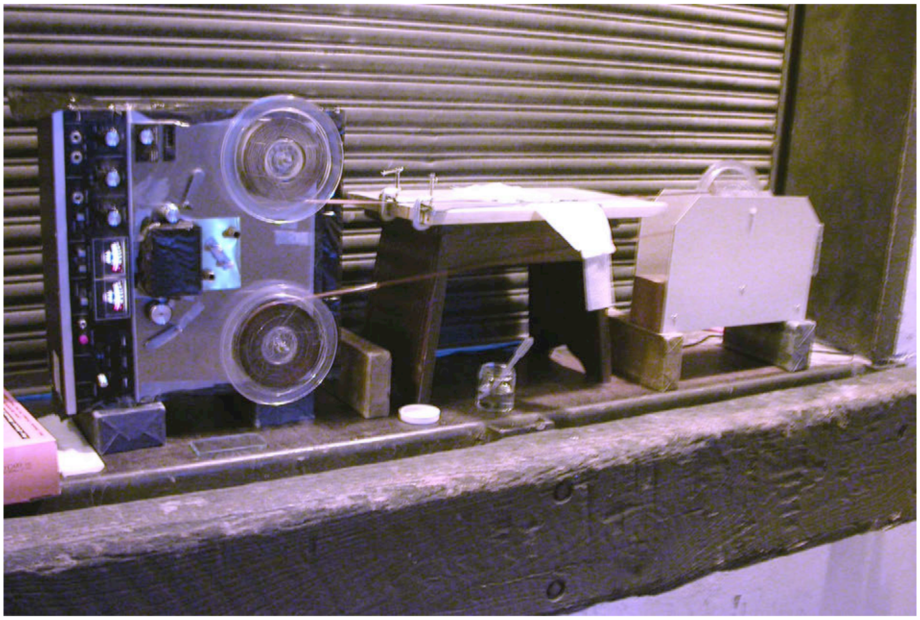


Figure 11. *Solvent-facilitated audiotape cleaning apparatus.*

9.2 CLEANING PLATFORM

The cleaning platform allows application of the solvent to the audiotape and consists of a ¾-inch plywood board with a piece of white paper attached to one side of the wood. The paper and edges of the board are covered with a sheet of polyester. White paper is used to increase visibility of potential color changes in the cleaning tissue during the cleaning process. The polyester sheet prevents solvent from soaking into the wood board.

9.3 CLEANING PADS

The cleaning pads consist of *Kimwipes* laboratory tissue wrapped lengthwise around a strip of *Hollitex*. The *Hollitex* provides added support to the tissue, allowing the pads to be shifted manually during the cleaning process. A total of four cleaning pads are used. Each pad consists of two layers of *Kimwipes* measuring 21.6 centimeters long by 11.4 centimeters wide, folded once lengthwise around a strip of *Hollitex* measuring 30.5 centimeters long by 5.1 centimeters wide. Two of the pads, one positioned above and one positioned below the audiotape, are saturated with 2-propanol. After the tape passes between the solvent-saturated pads, the tape moves between the second two pads used for drying the tape. As the saturated pads become dirty, the operator shifts the pads slightly so that clean portions of the pads come in contact with the audiotape. Similarly, as the drying pads become damp, the pads are shifted toward dry portions of the pads. Thus, the operator must pay attention to the condition of the pads during the cleaning process to determine when the pads should be shifted. To avoid causing lateral movement in the audiotape during the cleaning process, the upper and lower pads are shifted in opposite directions. In this way, shear forces introduced by shifting the upper and lower pads are counteracted.

In order to achieve adequate contact between the cleaning pads and the audiotape, one glass weight measuring 7.6 centimeters x 15.2 centimeters and weighing 165 grams is placed on the solvent-saturated pads. Two glass weights, one stacked on top of the other, are placed on the drying pads. All three glass weights are held in place using metal shelving brackets and c-clamps. A close-up image of the cleaning platform, cleaning pads, glass weights, and metal brackets is provided in Figure 12.

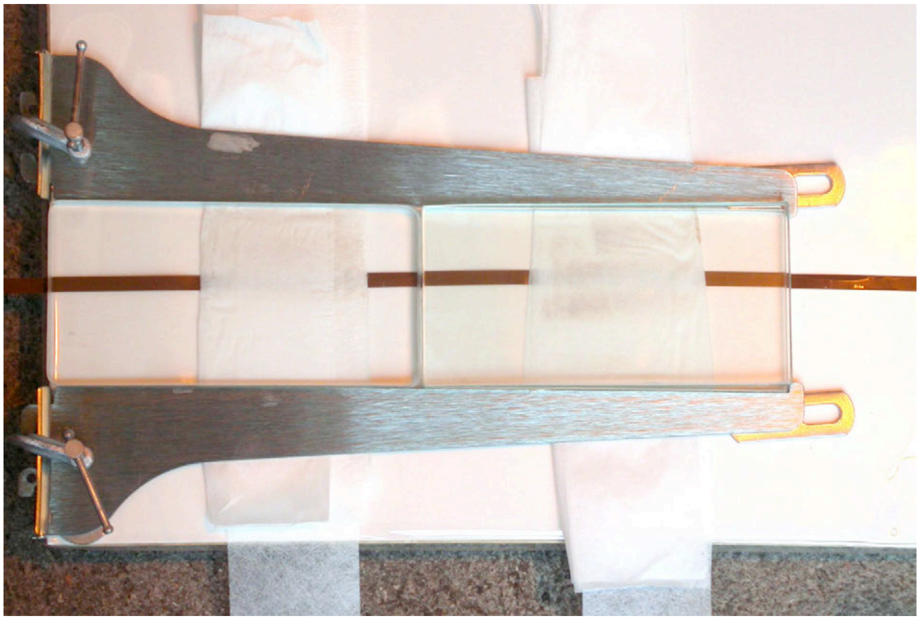


Figure 12. *Top of cleaning platform showing solvent-saturated pads (left) and drying pads (right) beneath glass weights. The direction of audiotape travel is from left to right.*

Images of the solvent-saturated pads and drying pads, after the audiotape were cleaned, are shown in Figures 13 and 14, respectively. This was the appearance of the cleaning pads after the entire reel of audiotape (approximately 366 meters) was cleaned. Note more rust-colored offset on the drying pads than on the solvent-saturated pads. This may be the result of a lubricating effect provided by the 2-propanol on the solvent-saturated pads, and twice as much weight placed on the drying pads as on the solvent-saturated pads.

Figure 13. *Solvent-saturated cleaning pads following audiotape cleaning.*

Figure 14. *Drying pads following audiotape cleaning. Note heavier rust-colored offset on drying pads than on solvent-saturated pads.*

As with mechanical cleaning, the solvent-facilitated cleaning operation was conducted outside. During the cleaning process, the operator wore an air-purifying respirator.

10. EVALUATION OF CLEANING EFFECTIVENESS

10.1 PHOTOGRAPHIC DOCUMENTATION

Digital photographs were taken of the backing and binder sides of the audiotape before and after cleaning. The photography equipment consisted of a Nikon CoolPix digital camera attached to an Olympus BX41 System Microscope with a 10x objective lens. The photographic documentation was a non-destructive process whereby the audiotape was placed between a glass slide and cover slip, and then placed on the platform of the microscope. In order to keep the audiotape flat during photography, two sides of the cover slip were weighted down using two square glass weights. The audiotape was top-lit using a Fiber-Lite Model 190 Fiber Optic Illuminator set on the “high” setting.

Photographed areas were first marked by placing two pieces of Filmoplast P on the backing layer of the audiotape approximately one inch apart. This allowed the same areas of the audiotape to be centered on the microscope platform and photographed before and after cleaning. Photographs of the audiotape backing side and binder side, after Filmoplast P markers were applied, are provided in Figures 15 and 16, respectively.

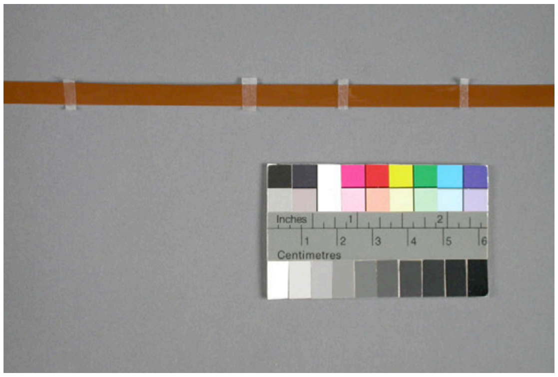


Figure 15. *Audiotape and Filmoplast markers with backing side up.*

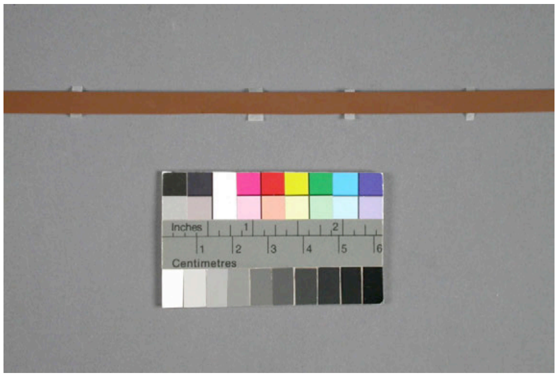


Figure 16. *Audiotape and Filmoplast markers with binder side up.*

Images of the microscope and fiber optic light used to document the audiotape before and after cleaning are shown in Figures 17 and 18.

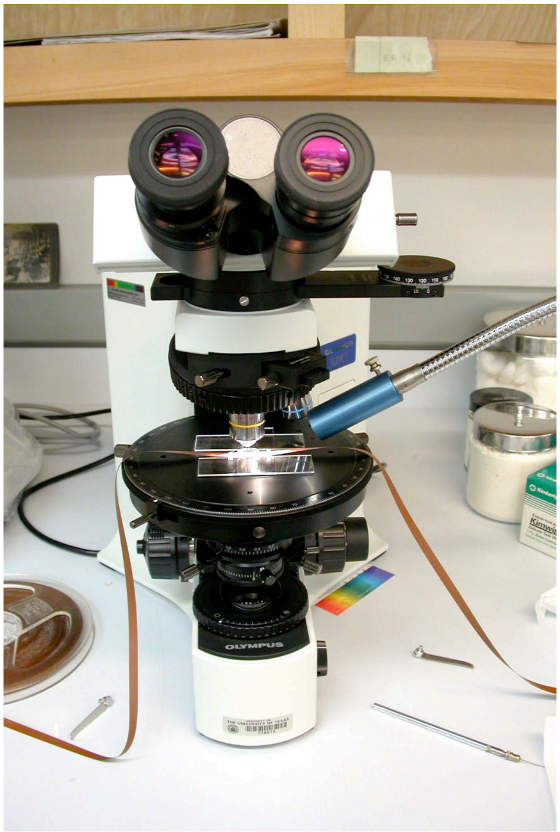


Figure 17. *Microscope and audiotape top-lit by fiber optic light. Camera not shown.*

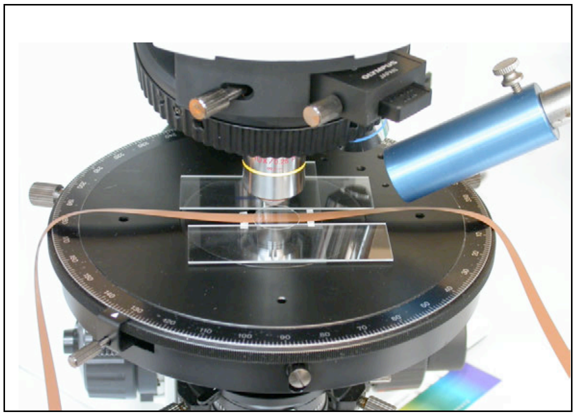


Figure 18. *Close-up of microscope platform. Note the two Filmoplast markers centered on the platform.*

10.2 CLEANING EFFECTIVENESS OF MECHANICAL METHOD

Before- and after-treatment photographs were compared to evaluate the cleaning effectiveness of the mechanical cleaning method. Photographs (10x magnification) of the audiotape binder side are shown in Figures 19 and 20.

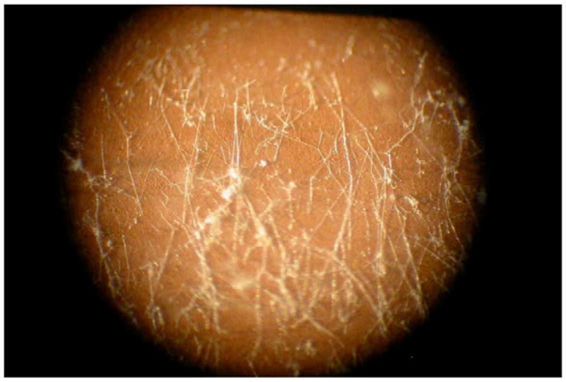


Figure 19. *Before-treatment photograph of audiotape binder side. Photo by Rick Taylor.*

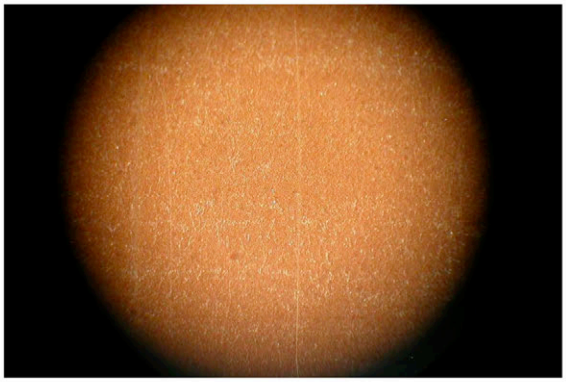


Figure 20. *After-treatment photograph of audiotape binder side. Photo by Rick Taylor.*

A comparison of the before- and after-treatment photographs shows that the cleaning process removed most, but not all, of the mold. Some mold remains on the audiotape following cleaning, particularly on the binder side.

10.3 CLEANING EFFECTIVENESS OF SOLVENT-FACILITATED METHOD

As with the mechanical cleaning method, before- and after-treatment photographs were also compared to evaluate the cleaning effectiveness of the solvent-facilitated cleaning method. Photographs (10x magnification) of the audiotape binder and backing sides are shown in Figures 21, 22, 23, and 24. The edge of the audiotape is visible at the top of the photographs.

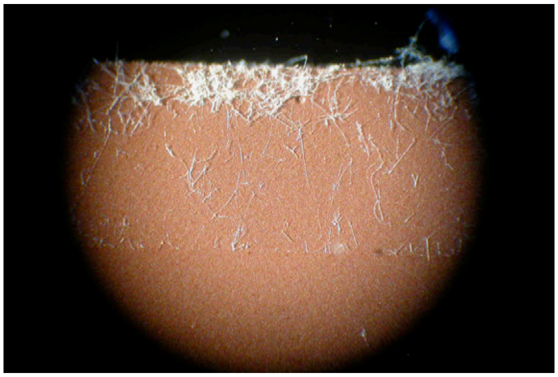


Figure 21. *Before-treatment photograph of audiotape binder side.*

Figure 22. *Before-treatment photograph of audiotape backing side.*

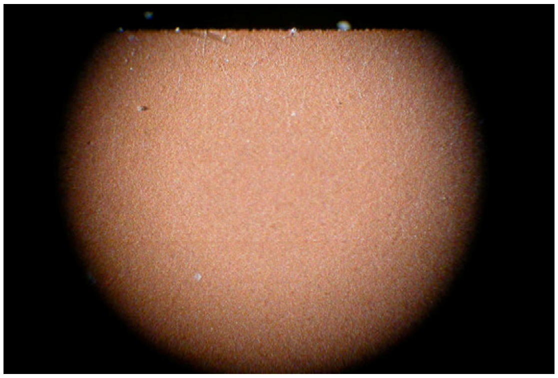


Figure 23. *After-treatment photograph of audiotape binder side. Although not obvious in the image, some mold remains.*

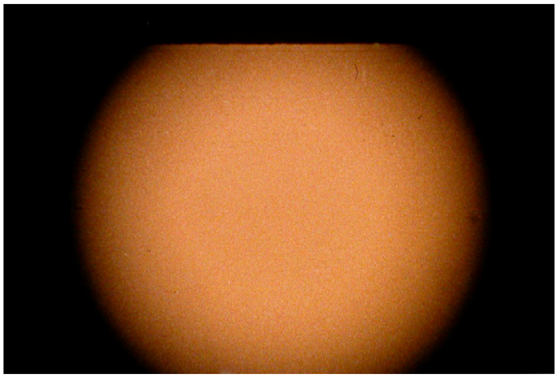


Figure 24. *After-treatment photograph of audiotape backing side.*

As was observed with the mechanical cleaning method, a comparison of the before- and after-treatment photographs shows that the cleaning process removed most, but not all, of the mold from both sides of the audiotape. Some mold remains on the audiotape following cleaning, particularly on the binder side.

11. EVALUATION OF POTENTIAL CHEMICAL CHANGES IN AUDIOTAPE CAUSED BY SOLVENT APPLICATION

11.1 FOURIER TRANSFORM INFRARED SPECTROSCOPY

Infrared spectroscopy traditionally has been used to identify functional groups in organic compounds and to help determine the structure of such compounds (Sorrell 1988). This type of spectroscopy is versatile. For example, researchers have used infrared spectroscopy to monitor differences in the extent of interaction between biopolymers and certain chemicals (Feo et al. 2003). The method has been used by scientists to provide a quantitative measurement of the amount of bound acetic acid and the distribution of primary and secondary hydroxyl groups in highly acetylated samples of cellulose acetate (Heinze and Liebert 2004). Infrared spectroscopy has also been used to detect changes in polymers caused by mold activity (Florian 2002).

Sorrell (1988) describes the region of the infrared spectrum extending from 1300 to 4000 cm-1 as the functional group region, which is where most interpretation is required. The region extending from 600 to 1300 cm-1 is known as the fingerprint region. This region is typically the most crowded portion of the infrared spectrum (Field et al. 2002). The fingerprint region is unique for each compound (Sorrell 1988).

Fourier transform infrared spectroscopy (FT-IR) equipped with an attenuated total reflectance (ATR) device was used in this research as a means to quantitatively measure potential changes in the composition of the audiotape caused by application of solvent. All of the FT-IR/ATR measurements were collected in the absorbance mode. Absorbance values, unlike transmittance values, are directly proportional to concentration, in accordance with Beer's law (Smith 1996). Although results of the FT-IR/ATR analysis were evaluated to help identify the chemical composition of the backing and binder layers of the audiotape, the primary purpose of the analysis was to measure potential changes in chemical composition of the two layers. The test results helped determine whether the solvent is compatible with the backing and binder layers, and whether the solvent may be suitable as a cleaning fluid.

The depth penetration of an infrared beam emitted from an FT-IR equipped with an ATR can range from 0.004 μm to 7 μm (Urban 1993). Converting the thicknesses of the backing and binder layers of the audiotape to the same units yields values of 36.1 μm and 12.7 μm, respectively. Thus, little risk exists of the infrared beam passing entirely through one layer and into the next layer, complicating the results. As a result, analyses of the two layers were conducted separately. Iron oxides absorb in the far infrared region, outside the range of the ATR accessory used in this research, but the iron oxides reportedly will not cause interference in measuring absorbance values in the polymeric binder material (Bradley 2006).

11.1.1 Procedure

A Nexus 470 FT-IR E.S.P. and SMART MIRACLE ATR, both manufactured by Thermo Nicolet Corporation, were used in this research. The FT-IR/ATR is pictured in Figures 25 and 26. Testing was performed on the audiotape before and after tape cleaning. For each of the before and after tests, the FT-IR was set to collect 16 scans over a spectral range of 4000 to 600 cm^{-1}. The resolution was set at 4 cm^{-1} and the data spacing at 1.928 cm^{-1}. Background readings were scanned with the pressure clamp in the "up" position as recommended by Gillespie (2005).

Figure 25. *FT-IR/ATR instrument and computer.*

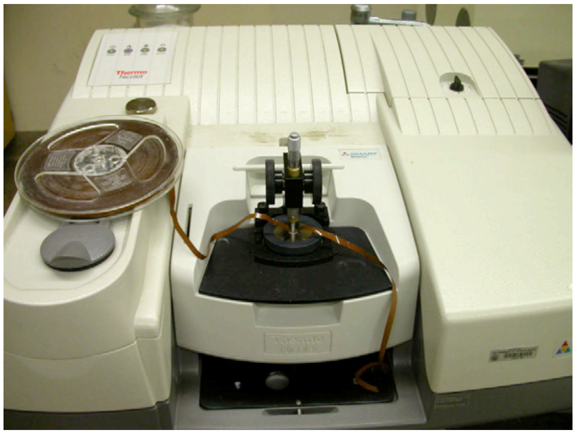


Figure 26. *Close-up of the ATR attachment and pressure clamp holding the audiotape in place.*

The spectrum of a tested sample is compared by the application software to spectra of known compounds contained in the software library. The software then displays the closest matches to help identify the sample material. Since the spectral library contains only spectra of pure compounds, infrared spectroscopy is best suited to identification of pure materials rather than mixtures of materials. If a sample is composed of a mixture of materials, more interpretation and/or testing with additional analytical methods may be necessary for proper identification of a sample.

FT-IR/ATR testing was conducted at 40 locations on the backing layer and at 40 locations on the binder layer, both before and after cleaning. Each test requires positioning a portion of the audiotape beneath the ATR pressure clamp. This clamping action causes a crimp in the audiotape, so the test is considered semi-destructive. The audiotape can still be played on a tape player, but the crimps would likely cause some flutter sound as the crimps pass the record head.

Infrared absorbance values were collected at 40 locations to allow large-sample statistical tests on the data collected. Test locations were separated by approximately 2.5 centimeters. The before-cleaning tests were conducted along the first six to eight feet of tape. This portion of the tape was cut off from the rest of the tape and was never cleaned. The after-cleaning tests were conducted along the next six to eight feet of tape, still on the reel. This portion of the tape and all of the remaining tape on the reel was cleaned with solvent prior to testing. The two groups of tests were performed on different portions of tape to allow all of the before- and after-cleaning FT-IR tests to be completed on the same day, thereby minimizing any influence of fluctuations of audiotape moisture content on infrared absorption measurements. This precaution also eliminated possible changes in FT-IR settings since the instrument was also used periodically by students in an instrumental analysis course. All of the before- and after-cleaning FT-IR tests were administered on portions of the tape that appeared free of mold growth to avoid the influence of mold on infrared absorbance measurements.

Each FT-IR/ATR test provides a display of ten of the closest matches ranked in order from the closest match to the furthest match as compared to compounds in the spectral library. Each of the ten compounds is assigned a "Match" number on a scale of 0 to 100%. According to a Thermo Electron Corporation Technical Support Specialist (2005), "If you are searching pure materials a good match would be > 90%. If you are testing a product which can vary a little in impurities a good match might be > 80%. Anything under 75% would not be considered good under normal conditions. The best you can do with low match numbers is to look at the spectra to make some educated guesses as to what the material might be."

11.1.2 Results of FT-IR Analysis

Results of FT-IR/ATR analysis on the backing layer of the audiotape showed good matches, probably because the backing layer is composed chiefly of a form of cellulose acetate with much lower amounts of other additives. The matches for one test on the backing layer, typical of most results for this side of the tape, are shown in Table 1 below.

TABLE 1

Typical FT-IR/ATR Results
Audio Tape Backing Layer

	Index	Match	Compound Name
1	2358	90.34	Cellulose triacetate
2	2694	87.57	Triacetyl-b-cyclodextrin
3	2860	83.14	3,4-Di-O-acetyl-6-deoxy-L-glucal
4	672	77.97	Allyl acetate
5	647	77.27	Butyl acetate
6	293	77.09	Triacetin
7	1007	76.12	Tetrahydrofurfuryl acetate
8	650	75.49	Amyl acetate
9	2366	74.71	2-Ethylbutyl acetate
10	651	74.48	Isoamyl acetate

Although the comparison between the sample spectrum and the cellulose triacetate library spectrum resulted in a high match value, this may not rule out the other three forms of cellulose acetate because the spectral library did not contain spectra for the other three forms. According to Ballany et al. (2001), all four forms of cellulose acetate have similar, but distinguishable FT-IR spectra. Literature FT-IR spectra for cellulose acetate, cellulose acetate butyrate, cellulose acetate propionate, and cellulose triacetate are provided in Appendix G (Pouchert 1997).

A typical before-treatment spectrum of the backing layer, along with the three closest matches, are shown in Figure 27.

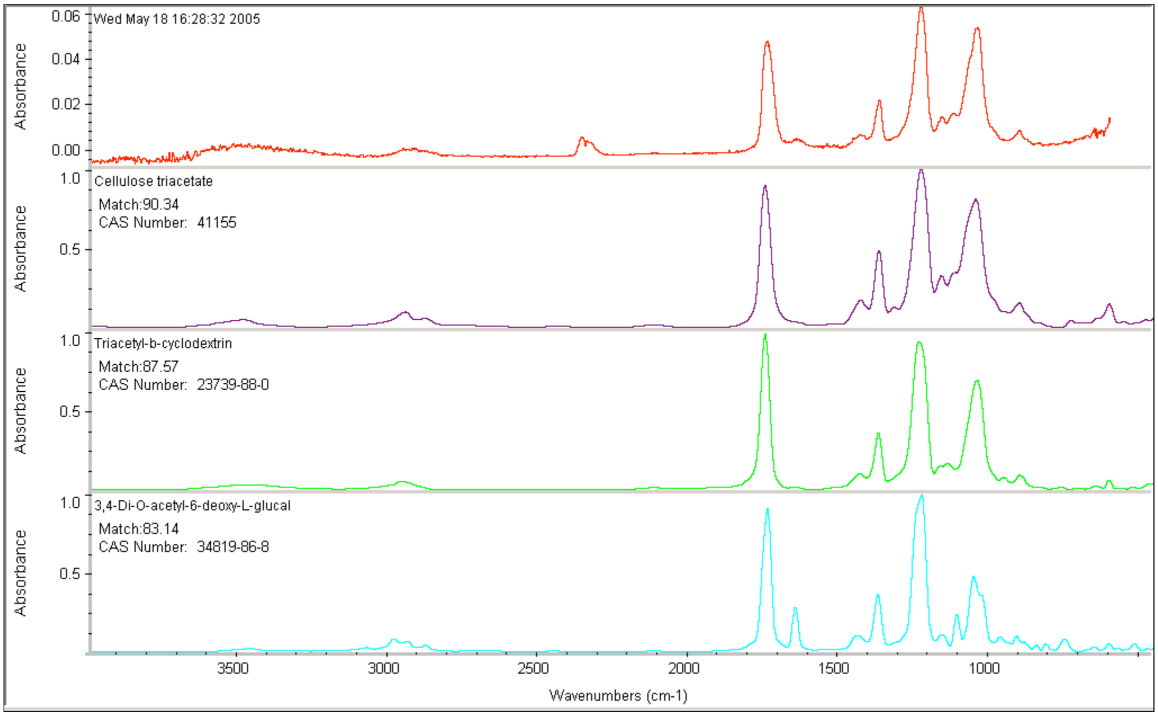


Figure 27. *Typical before-treatment spectrum of the audiotape backing layer.*

A typical after-treatment spectrum of the backing layer, along with the three closest matches, are shown in Figure 28.

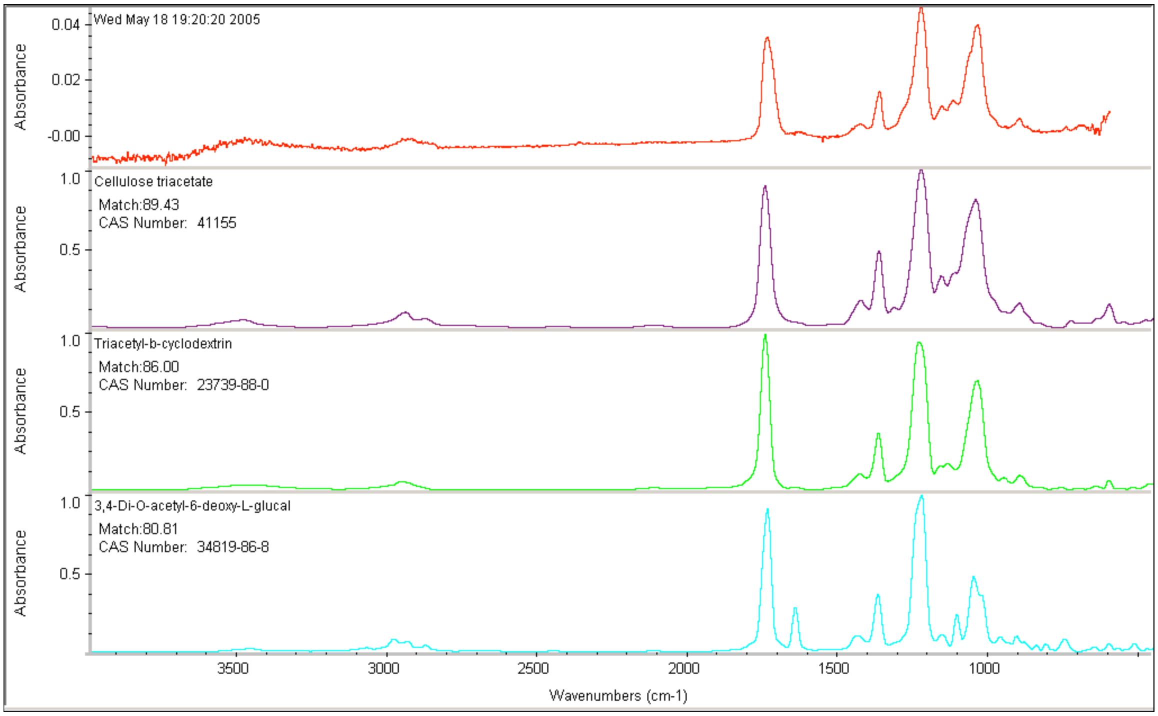


Figure 28. *Typical after-treatment spectrum of the audiotape backing layer.*

Much less definitive matches were obtained for the binder layer. Typical results for the binder layer are shown below in Table 2.

TABLE 2

Typical FT-IR/ATR Results
Audio Tape Binder Layer

	Index	Match	Compound Name
1	31	48.03	Poly(Vinyl Acetate:Ethylene) 3:1
2	29	47.63	Poly(Vinyl Acetate:Ethylene) 4:1
3	1363	40.93	3-Aminorhodanine
4	2333	40.68	Poly(vinyl acetate)
5	2991	40.65	Ethyl 2-thiopheneacetate
6	2398	40.26	Diethyl dodecanedioate
7	1543	40.24	Diethyl suberate
8	940	40.01	Cyclohexyl acetate
9	16	39.93	Poly (Vinyl Propionate/Acetate)
10	298	39.66	2-Phenylethyl acetate; Phenethyl acetate

A typical before-treatment spectrum of the binder layer, along with the three closest matches, are shown in Figure 29.

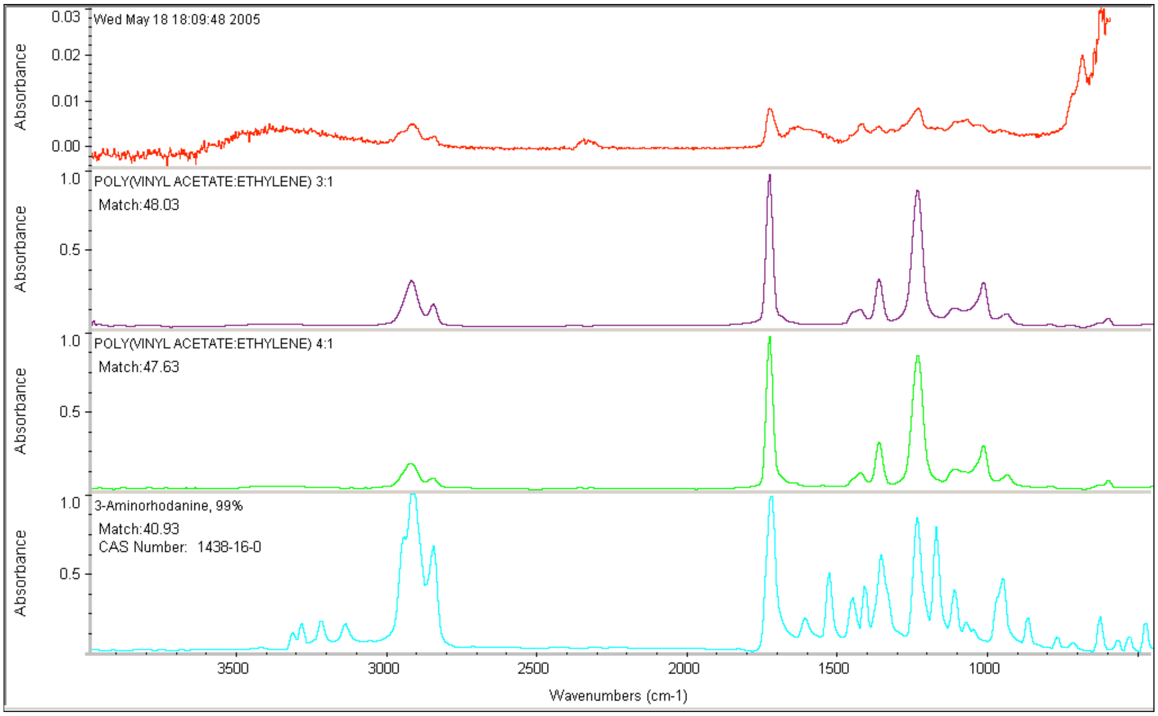


Figure 29. *Typical before-treatment spectrum of the audiotape binder layer.*

A typical after-treatment spectrum of the binder layer, along with the three closest matches, are shown in Figure 30.

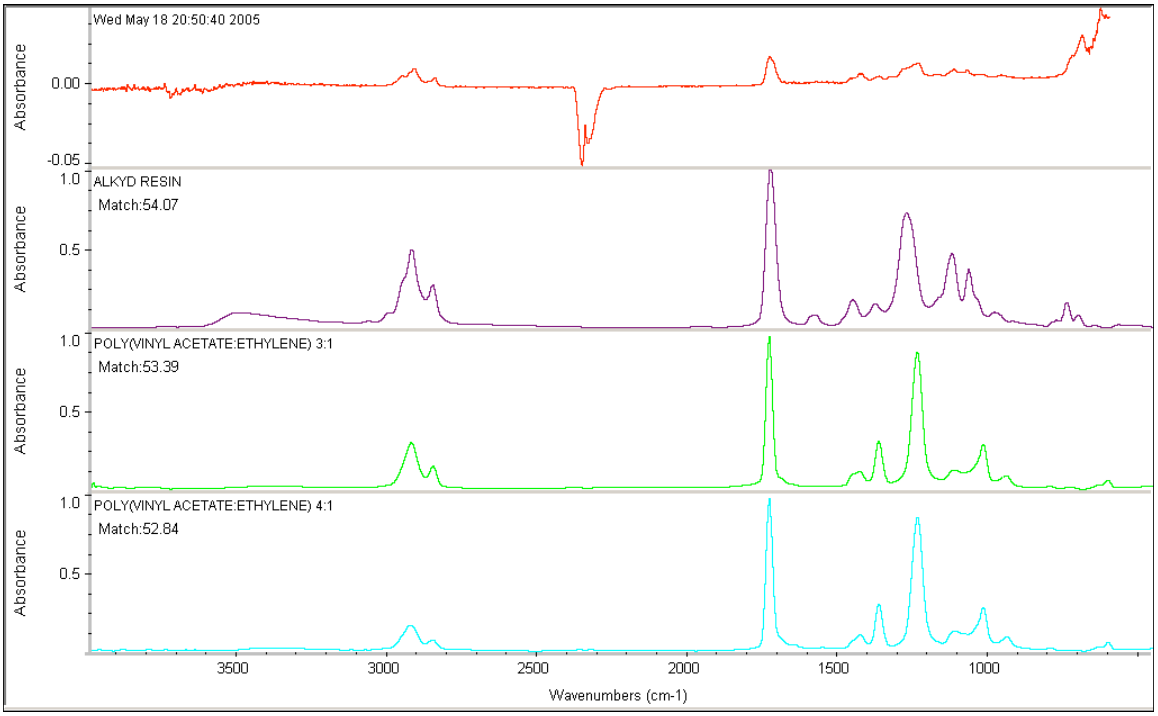


Figure 30. *Typical after-treatment spectrum of the audiotape binder layer.*

The low match values for the binder layer are probably the result of a binder layer composed of a mixture of compounds. Although testing on the binder did not result in exact matches, the top two matches shown in Table 2 are consistent with a "vinyl binder" as reported by Eilers (2005). Determination of the exact composition of the backing and binder layers was not achieved through infrared spectroscopy, but the primary reason for performing infrared spectroscopy in this research was to detect potential changes in the composition of the tape caused by application of solvent.

12. STATISTICAL ANALYSES

12.1 METHODOLOGY

A large-sample test to detect whether a statistically significant difference exists between two populations is possible if the sample sizes are greater than or equal to 30 (Mendenhall et al. 2003). This type of test was conducted on before- and after-cleaning infrared absorbance values for both the backing layer and the binder layer.

Each FT-IR/ATR measurement results in a quantitative infrared absorption value for each of 1,764 wavelengths within the infrared spectrum ranging from 4000 to

600 cm^{-1}. Thus, for each of the 1,764 wavelengths, 40 absorbance values were collected before and after tape cleaning. A statistical test was then conducted, with the aid of Microsoft Excel, on absorbance values for each wavelength. In this way, one can ascertain whether chemical changes occurred to the tape. Since a statistical test is performed on each wavelength, and because absorbance in specific regions of the infrared spectrum are indicative of specific functional groups, one may be able to deduce what chemical changes, if any, have taken place.

The hypotheses tested in this research are stated in Table 3.

TABLE 3
Statement of Hypotheses

Null hypothesis, H_0	$(\mu_1 - \mu_2) = D_0$
Alternative hypothesis, H_a	$(\mu_1 - \mu_2) \neq D_0$

Where: μ_1 represents the population mean of before-treatment absorbance values.
μ_2 represents the population mean of after-treatment absorbance values.
D_0 represents a specified difference between the two population means.

For the statistical tests conducted as part of this research, $D_0 = 0$ because the null hypothesis will be tested to determine if no (i.e., zero) difference exists between the two population means.

According to Mendenhall et al. (2003), the statistical test of hypothesis for the difference between two population means requires the calculation of a value called the "z test statistic." This value is estimated with the following formula:

$$z \approx \frac{(x_1 - x_2) - D_0}{\text{SE}} = \frac{(x_1 - x_2) - D_0}{\sqrt{[(s_1^2/n_1) + (s_2^2/n_2)]}}$$

where: x_1 = average of before-cleaning absorbance values
x_2 = average of after-cleaning absorbance values
D_0 = hypothesized difference between before- and after-cleaning absorbance values (i.e., 0)
SE = standard error
s_1^2 = variance of before-cleaning absorbance values
s_2^2 = variance of after-cleaning absorbance values
n_1 = number of before-cleaning absorbance values (i.e., 40)
n_2 = number of after-cleaning absorbance values (i.e., 40)

A statistically significant difference could result if the after-cleaning absorbance values (μ_2) are either too large or too small as compared to the before-cleaning absorbance values (μ_1). In other words, no directional difference exists. Therefore, a two-tailed test is appropriate and is illustrated in Figure 31. For this research, a 1% level of significance (i.e., $\alpha = 0.01$) was chosen. Thus, a 1% probability exists of rejecting the null hypothesis when the null hypothesis is true. A two-tailed test requires that the level of significance be divided by two, and is represented as $\alpha/2$ or 0.005. This value is used to establish the null hypothesis rejection regions. For $\alpha/2 = 0.005$, the rejection region, as obtained from a table of critical values, is $z_{a/2} = 2.58$ and $-z_{a/2} = -2.58$.

A z test statistic was calculated for infrared absorbance values at each wavelength. If the value of the test statistic was greater than -2.58 and less than 2.58, the null hypothesis was accepted (i.e., no statistically significant difference between before- and after-cleaning absorbance values). In contrast, if the value of the test statistic was less than -2.58 or greater than 2.58, the null hypothesis was rejected (i.e, a statistically significant difference between before- and after-cleaning absorbance values). The statistical analysis was conducted using a Microsoft Excel spreadsheet.

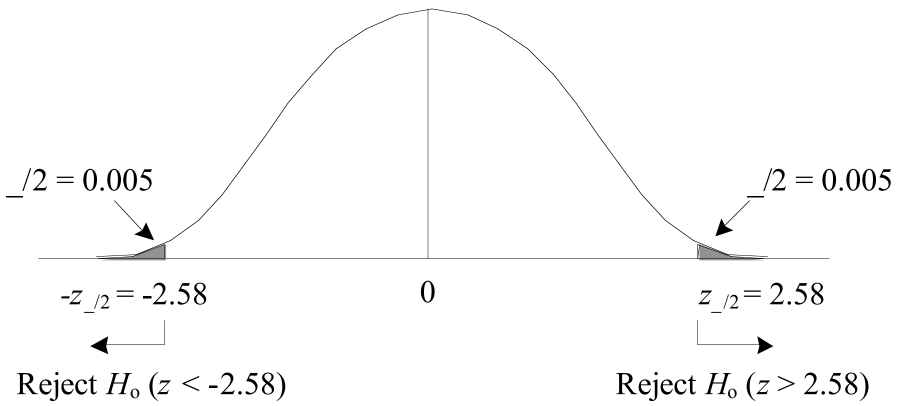


Figure 31. *Illustration of the two-tailed test showing the null hypothesis rejection regions.*

12.2 RESULTS OF STATISTICAL ANALYSES

Results of the statistical analyses show statistically significant differences between before- and after-treatment infrared absorbance values for a majority of the wavelengths at which absorbance values were measured. Such differences are observed in both the backing and binding layers. The results are summarized in Table 4.

TABLE 4
Summary of Statistical Analyses

	Tape Backing	**Tape Binder**
Total number of wavelengths at which absorbance values were collected and statistical tests conducted.	1,764	1,764
Total number of wavelengths showing statistical difference in before- and after-cleaning infrared absorbance values.	1,491	1,481
Percentage of wavelengths showing statistical difference in before- and after-cleaning infrared absorbance values.	85%	84%

Although the tape backing and tape binder show a similar percentage of wavelengths with statistically different absorbance values, based on evaluation of the wavelength-specific results, the magnitude of the individual differences appeared greater in the tape binder. Thus, the tape cleaning appears to have caused greater changes, at most wavelengths, on the tape binder than on the tape backing.

Appendix D contains results of the statistical analyses for the backing layer and binder layer. These results are summarized visually in Figure 32. Figure 32 does not provide as much detail as the tables in Appendix D due to space constraints, but is more effective in conveying the ranges of wavenumbers where changes occurred within the infrared spectrum, as well as the direction (i.e., increase or decrease of absorbance values) of the changes.

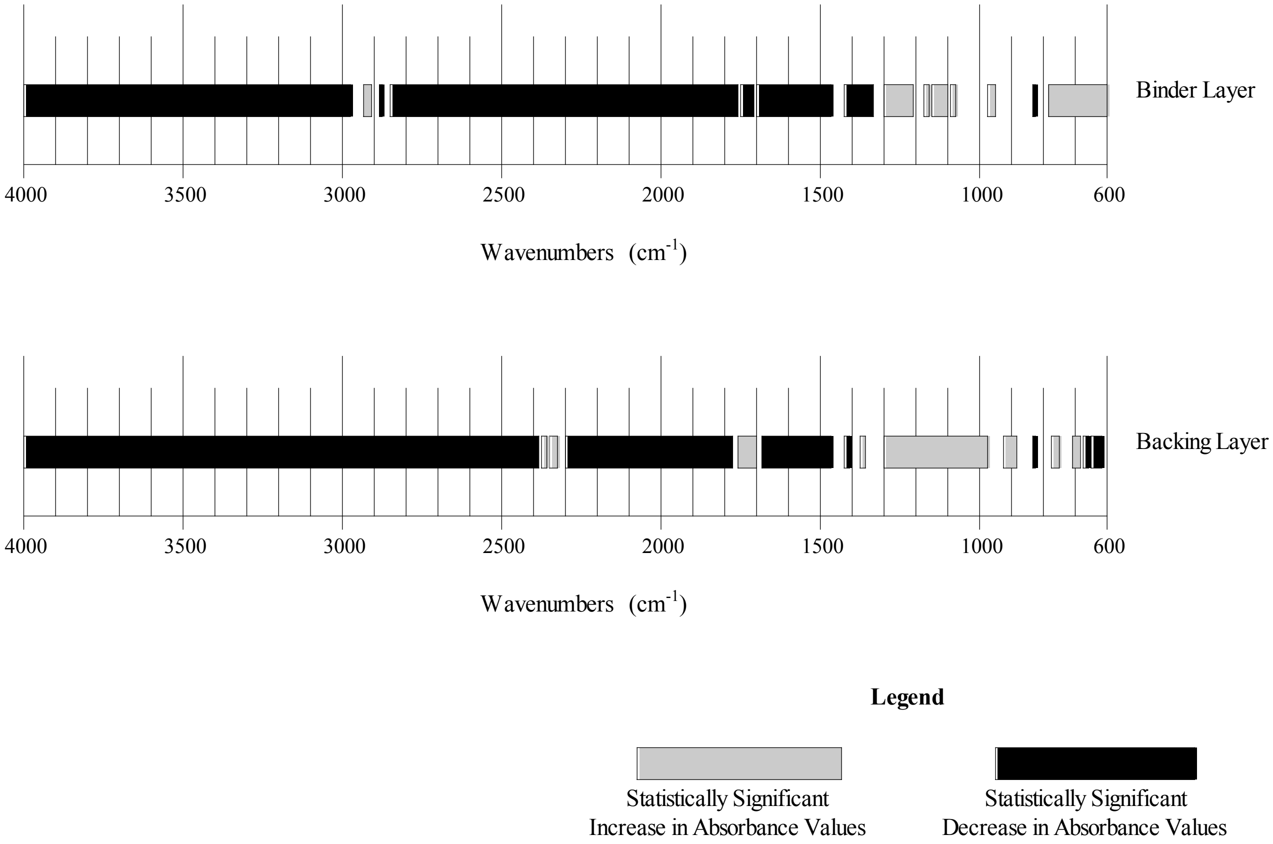


Figure 32. Visual representation of results of statistical analyses

13. AUDIO QUALITY

The audiotapes were not played and digitized prior to cleaning due to concerns of spreading mold spores throughout the audio digitization laboratory. Thus, a before- and after-cleaning evaluation of audio quality was not possible. However, all seven audiotapes were digitized after they were cleaned. Once digitized, the audio files were played to evaluate the after-cleaning audio quality. The information recorded on the tapes was easily understandable and seemed of good quality, especially for tapes recorded in 1959. The audio quality of the tapes cleaned with the mechanical method was compared with the audio quality of the tape cleaned with the solvent-facilitated method. No obvious difference in the audio quality was noticed, although the tapes had been recorded in different settings (e.g., a conference room with several people being recorded versus an office with only one person being recorded), so a comparison of the audio quality of tapes cleaned using the two cleaning methods was made more difficult.

14. DISCUSSION OF RESULTS

14.1 MOLD REMOVAL EFFECTIVENESS

Comparison of photographs taken before and after cleaning of the audiotape shows that both the mechanical method and the solvent-facilitated method removed most but not all of the mold. Since some mold remains, the fungicidal potential of the solvent in the solvent-facilitated method is of increased importance. Mold on all seven audiotapes used in this research is believed to have been dead prior to cleaning, but other audiotapes may be encountered supporting active mold growth.

14.2 POTENTIAL CAUSES OF CHEMICAL CHANGES

A review of the two tables in Appendix D and the summary of those results in Figure 32 shows statistically significant chemical changes occurred to both the backing and binder layers of the audiotape due to cleaning. These changes may have been caused by a combination of factors, most notably the removal of water from the audiotape. Application of 2-propanol may also have caused selective dissolution and removal of specific components (e.g., plasticizers and lubricants) from the audiotape.

Much of the statistically significant changes observed in both the backing layer and the binder layer of the audiotape could be due to removal of water from the tape caused by application of 2-propanol. As seen in the FT-IR reference spectrum for water in Appendix E, water absorbs infrared energy in the regions 3800 cm^{-1} to 2900 cm^{-1}, 2200 cm^{-1} to 1900 cm^{-1}, 1750 cm^{-1} to 1500 cm^{-1}, and 800 cm^{-1} to 600 cm^{-1} (Pouchert 1997). As seen in Figure 32, broad regions where statistically significant decreases in absorption were observed in both the backing and binder layers correspond to regions where the FT-IR reference spectrum for water show strong absorption. An exception is the region extending from 800 cm^{-1} to 600 cm^{-1}, particularly for the binder layer. Since poly(vinyl chloride) compounds also absorb strongly in that region, the polymer may have obscured the effect of water removal.

FT-IR testing was conducted the day after solvent-facilitated audiotape cleaning, so the audiotape may not have yet regained its equilibrium moisture content. Adelstein et al. (1997) researched moisture relationships for photographic film, including 35 mm motion picture film with a backing layer of cellulose triacetate. The film was conditioned, with no housing, at 21° C and 20% relative humidity. The film was then exposed to a 21° C, 50% relative humidity environment. Twelve days were required for the film to reach 90% of its equilibrium moisture content.

If the concentration of water in the audiotape is reduced, one would expect to observe a corresponding increase in the concentration of remaining components (i.e., the vinyl compound in the binder layer and cellulose acetate in the backing layer). The FT-IR reference spectra for poly(vinyl chloride) and poly(vinyl chloride-co-vinyl acetate) compounds shown in Appendix F reveal these compounds absorb strongly in the regions 3000 cm^{-1} to 2900 cm^{-1}, 1450 cm^{-1} to 1200 cm^{-1}, 950 cm^{-1}, and 700 cm^{-1} to 600 cm^{-1} (Pouchert 1997). These regions correspond closely to regions where statistically significant increases in binder layer absorbance values were observed, as seen in Figure 32.

Similarly, the FT-IR reference spectra for cellulose acetate compounds shown in Appendix G reveal these compounds absorb strongly in the regions 1750 cm^{-1}, 1400 cm^{-1} to 1000 cm^{-1}, 900 cm^{-1}, and 600 cm^{-1} (Pouchert 1997). With the exception of the absorption at 600 cm^{-1}, these regions correspond to regions where statistically significant increases in backing layer absorbance values were observed, as seen in Figure 32. A statistically significant increase in absorption was observed in a narrow band at about 2350 cm^{-1}. This was unexpected because the FT-IR reference spectra for cellulose acetate compounds show little absorption in this region. Similarly, the reference spectra for the three most common plasticizers used in cellulose acetate show little absorption in this region. So, the reason for the statistically significant increase in absorption in the area of 2350 cm^{-1} is unknown.

15. CONCLUSION

The statistical analyses seem to have been successful in determining whether 2-propanol altered the composition of the audiotape. Thus, the same type of analysis could be performed if additional research is conducted with a different solvent. An alternative solvent, or a diluted solution of 2-propanol, should be investigated to determine its drying effect on the audiotape and its fungicidal properties.

The mechanical cleaning method has the advantage of eliminating potential chemical incompatibilities between the audiotape and the solvent. Based on this research, the mechanical cleaning method appeared to be at least as effective in removing mold as the solvent-facilitated method. So, for institutions that routinely encounter mold on audiotape, an alternative to the use of solvents would be to clean audiotape using the mechanical cleaning method. However, because mechanical cleaning will not kill mold spores, the cleaning apparatus should be set up within a fume hood equipped with a biological filter to prevent exposure of the operator to airborne mold and to prevent the spread of mold to the surrounding area. The operator must follow proper health and safety precautions during the cleaning process. The cleaning should be performed using a tape player or custom-built cleaning device dedicated to cleaning moldy audiotape to avoid cross-contamination of clean audiotape.

The apparatus used to apply solvent to audiotape in this research was successful, but the cleaning would be more effective using a custom-built cleaning apparatus with the same general configuration but with a more powerful, variable-speed motor. A more powerful motor would prevent the tape speed from slowing down toward the end of the audiotape as occurred in this research. A variable-speed motor would allow the use of a solvent with a slower evaporation rate and ensure the audiotape is dry before winding onto the take-up reel.

Solvent-facilitated cleaning of audiotape requires consideration of many factors. The cleaning of cellulose acetate audiotape presents a particularly challenging task due to the susceptibility of the tape to chemical change when exposed to solvent. Additional research is recommended.

16. RECOMMENDATIONS FOR FUTURE RESEARCH

The configurations of the mechanical and solvent-facilitated cleaning apparatuses, the use of FT-IR/ATR for detection of potential changes in tape composition, and the statistical analyses all seemed to serve their intended functions. Therefore, the methods used in this research could be used in a similar manner in future research.

For solvent-facilitated cleaning, the use of a 70% 2-propanol / 30% water mixture is recommended. This mixture would cause less drying of the audiotape and would likely increase the fungicidal effects of 2-propanol since this proportion of water has been reported to facilitate the entry of alcohol into the mold structure, allowing the alcohol to act on the mold (Lewincamp 2005).

Recommendations for future work include the following:

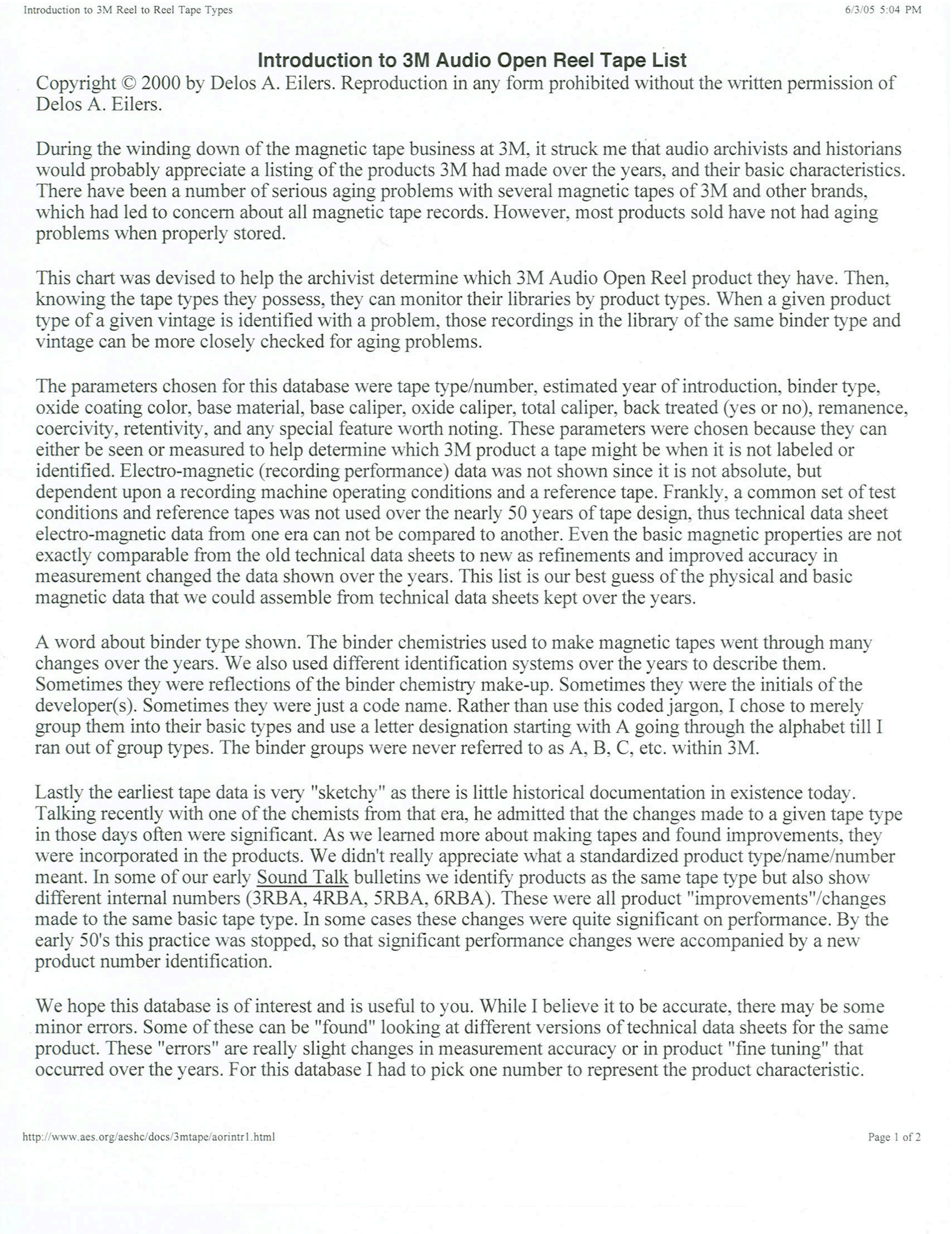
- Experiment with a 2-propanol/water mixture and other solvents to determine audiotape compatibility. Solubility testing using swabs is recommended as a quick, initial test.
- Determine effectiveness of mold removal using higher power magnification than was used in this research.
- Determine fungicidal effects of the chosen cleaning solution on any remaining mold using laboratory tests such as culturing.

- Perform FT-IR testing of audiotape at least two weeks after the audiotape is cleaned to ensure the tape has regained its equilibrium moisture content.
- Develop a custom-built, electric device to wind the tape from one reel to another during the cleaning process. A small motor equipped with a rheostat is recommended to allow speed adjustment. This would allow reduction of tape speed to accommodate slower-drying solvents.
- Employ commercially available audio engineering software to evaluate potential changes in the audio quality of the audiotape. This portion of research could be conducted on mold-free audiotape to avoid spreading mold spores before the tape is cleaned.

APPENDIX A

3M Audio Tape Specifications and Historical Data

From Eilers, D.A. (2000)



Introduction to 3M Audio Open Reel Tape List

During the winding down of the magnetic tape business at 3M, it struck me that audio archivists and historians would probably appreciate a listing of the products 3M had made over the years, and their basic characteristics. There have been a number of serious aging problems with several magnetic tapes of 3M and other brands, which had led to concern about all magnetic tape records. However, most products sold have not had aging problems when properly stored.

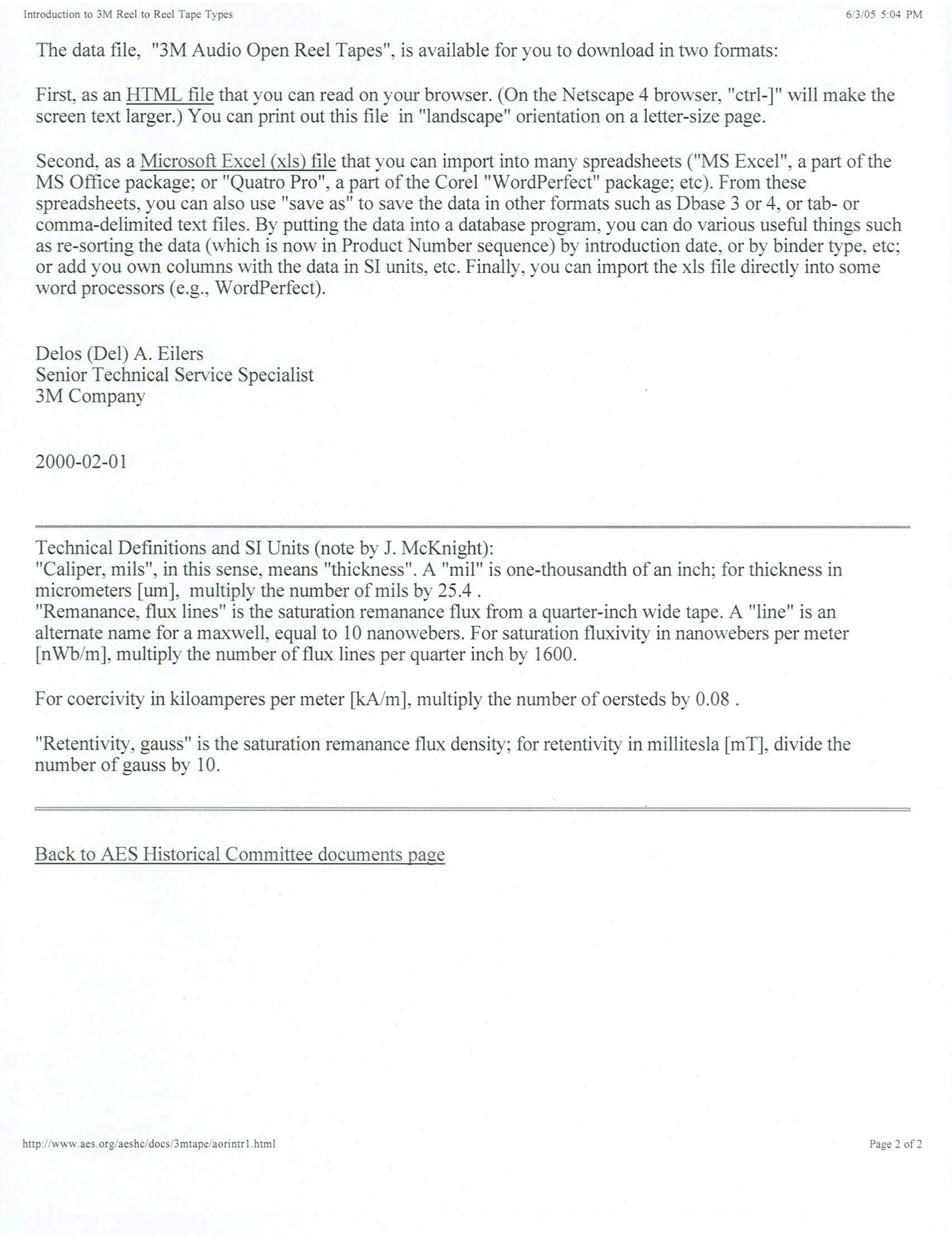
This chart was devised to help the archivist determine which 3M Audio Open Reel product they have. Then, knowing the tape types they possess, they can monitor their libraries by product types. When a given product type of a given vintage is identified with a problem, those recordings in the library of the same binder type and vintage can be more closely checked for aging problems.

The parameters chosen for this database were tape type/number, estimated year of introduction, binder type, oxide coating color, base material, base caliper, oxide caliper, total caliper, back treated (yes or no), remanence, coercivity, retentivity, and any special feature worth noting. These parameters were chosen because they can either be seen or measured to help determine which 3M product a tape might be when it is not labeled or identified. Electro-magnetic (recording performance) data was not shown since it is not absolute, but dependent upon a recording machine operating conditions and a reference tape. Frankly, a common set of test conditions and reference tapes was not used over the nearly 50 years of tape design, thus technical data sheet electro-magnetic data from one era can not be compared to another. Even the basic magnetic properties are not exactly comparable from the old technical data sheets to new as refinements and improved accuracy in measurement changed the data shown over the years. This list is our best guess of the physical and basic magnetic data that we could assemble from technical data sheets kept over the years.

A word about binder type shown. The binder chemistries used to make magnetic tapes went through many changes over the years. We also used different identification systems over the years to describe them. Sometimes they were reflections of the binder chemistry make-up. Sometimes they were the initials of the developer(s). Sometimes they were just a code name. Rather than use this coded jargon, I chose to merely group them into their basic types and use a letter designation starting with A going through the alphabet till I ran out of group types. The binder groups were never referred to as A, B, C, etc. within 3M.

Lastly the earliest tape data is very "sketchy" as there is little historical documentation in existence today. Talking recently with one of the chemists from that era, he admitted that the changes made to a given tape type in those days often were significant. As we learned more about making tapes and found improvements, they were incorporated in the products. We didn't really appreciate what a standardized product type/name/number meant. In some of our early Sound Talk bulletins we identify products as the same tape type but also show different internal numbers (3RBA, 4RBA, 5RBA, 6RBA). These were all product "improvements"/changes made to the same basic tape type. In some cases these changes were quite significant on performance. By the early 50's this practice was stopped, so that significant performance changes were accompanied by a new product number identification.

We hope this database is of interest and is useful to you. While I believe it to be accurate, there may be some minor errors. Some of these can be "found" looking at different versions of technical data sheets for the same product. These "errors" are really slight changes in measurement accuracy or in product "fine tuning" that occurred over the years. For this database I had to pick one number to represent the product characteristic.



Introduction to 3M Reel to Reel Tape Types 6/3/05 5:04 PM

The data file, "3M Audio Open Reel Tapes", is available for you to download in two formats:

First, as an HTML file that you can read on your browser. (On the Netscape 4 browser, "ctrl-]" will make the screen text larger.) You can print out this file in "landscape" orientation on a letter-size page.

Second, as a Microsoft Excel (xls) file that you can import into many spreadsheets ("MS Excel", a part of the MS Office package; or "Quatro Pro", a part of the Corel "WordPerfect" package; etc). From these spreadsheets, you can also use "save as" to save the data in other formats such as Dbase 3 or 4, or tab- or comma-delimited text files. By putting the data into a database program, you can do various useful things such as re-sorting the data (which is now in Product Number sequence) by introduction date, or by binder type, etc; or add you own columns with the data in SI units, etc. Finally, you can import the xls file directly into some word processors (e.g., WordPerfect).

Delos (Del) A. Eilers
Senior Technical Service Specialist
3M Company

2000-02-01

Technical Definitions and SI Units (note by J. McKnight):
"Caliper, mils", in this sense, means "thickness". A "mil" is one-thousandth of an inch; for thickness in micrometers [um], multiply the number of mils by 25.4 .
"Remanance, flux lines" is the saturation remanance flux from a quarter-inch wide tape. A "line" is an alternate name for a maxwell, equal to 10 nanowebers. For saturation fluxivity in nanowebers per meter [nWb/m], multiply the number of flux lines per quarter inch by 1600.

For coercivity in kiloamperes per meter [kA/m], multiply the number of oersteds by 0.08 .

"Retentivity, gauss" is the saturation remanance flux density; for retentivity in millitesla [mT], divide the number of gauss by 10.

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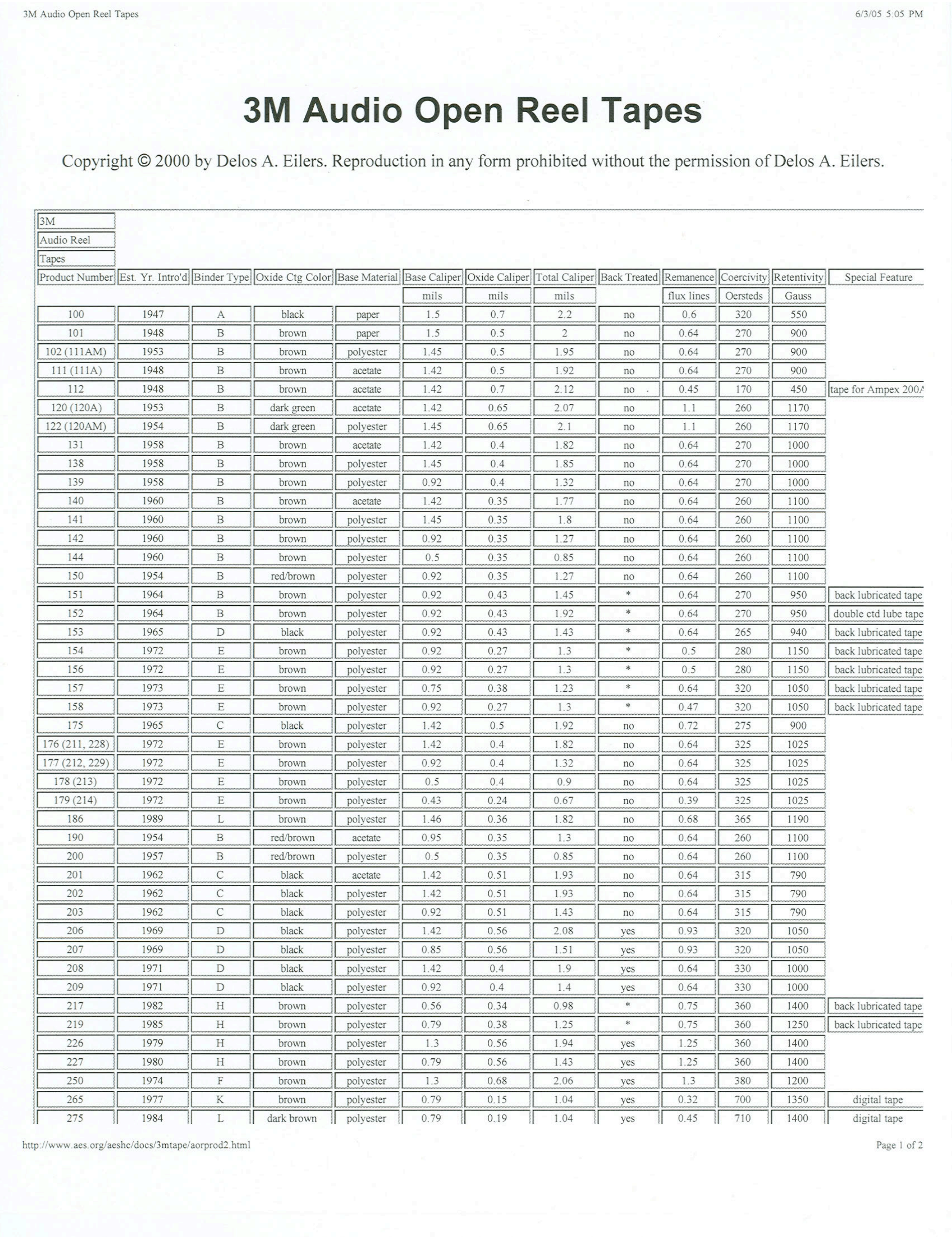
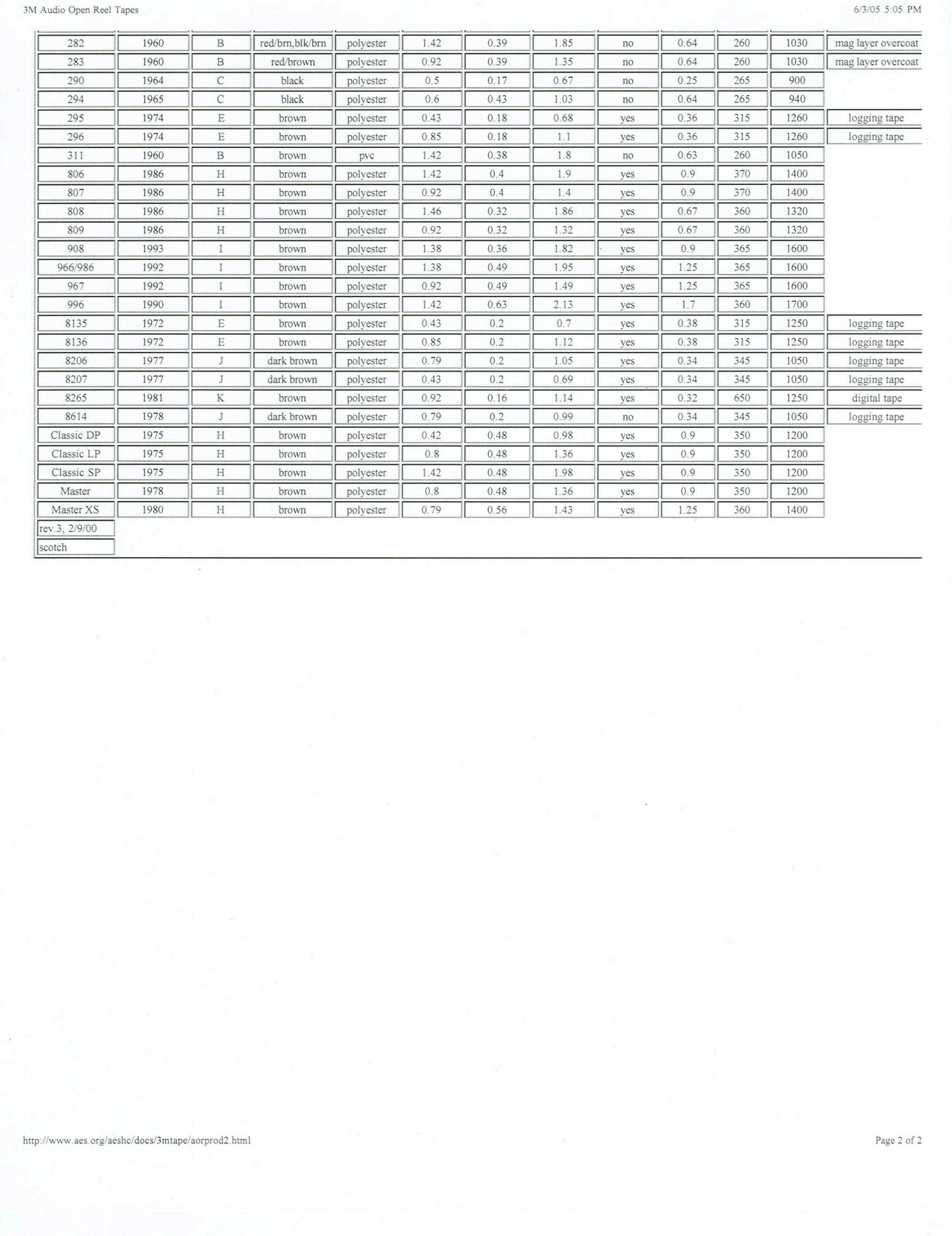


3M Audio Open Reel Tapes

3M
Audio Reel
Tapes

Product Number	Est. Yr. Intro'd	Binder Type	Oxide Ctg Color	Base Material	Base Caliper	Oxide Caliper	Total Caliper	Back Treated	Remanence	Coercivity	Retentivity	Special Feature
					mils	mils	mils		flux lines	Oersteds	Gauss	
100	1947	A	black	paper	1.5	0.7	2.2	no	0.6	320	550	
101	1948	B	brown	paper	1.5	0.5	2	no	0.64	270	900	
102 (111AM)	1953	B	brown	polyester	1.45	0.5	1.95	no	0.64	270	900	
111 (111A)	1948	B	brown	acetate	1.42	0.5	1.92	no	0.64	270	900	
112	1948	B	brown	acetate	1.42	0.7	2.12	no	0.45	170	450	tape for Ampex 200
120 (120A)	1953	B	dark green	acetate	1.42	0.65	2.07	no	1.1	260	1170	
122 (120AM)	1954	B	dark green	polyester	1.45	0.65	2.1	no	1.1	260	1170	
131	1958	B	brown	acetate	1.42	0.4	1.82	no	0.64	270	1000	
138	1958	B	brown	polyester	1.45	0.4	1.85	no	0.64	270	1000	
139	1958	B	brown	polyester	0.92	0.4	1.32	no	0.64	270	1000	
140	1960	B	brown	acetate	1.42	0.35	1.77	no	0.64	260	1100	
141	1960	B	brown	polyester	1.45	0.35	1.8	no	0.64	260	1100	
142	1960	B	brown	polyester	0.92	0.35	1.27	no	0.64	260	1100	
144	1960	B	brown	polyester	0.5	0.35	0.85	no	0.64	260	1100	
150	1954	B	red/brown	polyester	0.92	0.35	1.27	no	0.64	260	1100	
151	1964	B	brown	polyester	0.92	0.43	1.45	*	0.64	270	950	back lubricated tape
152	1964	B	brown	polyester	0.92	0.43	1.92	*	0.64	270	950	double ctd lube tape
153	1965	D	black	polyester	0.92	0.43	1.43	*	0.64	265	940	back lubricated tape
154	1972	E	brown	polyester	0.92	0.27	1.3	*	0.5	280	1150	back lubricated tape
156	1972	E	brown	polyester	0.92	0.27	1.3	*	0.5	280	1150	back lubricated tape
157	1973	E	brown	polyester	0.75	0.38	1.23	*	0.64	320	1050	back lubricated tape
158	1973	E	brown	polyester	0.92	0.27	1.3	*	0.47	320	1050	back lubricated tape
175	1965	C	black	polyester	1.42	0.5	1.92	no	0.72	275	900	
176 (211, 228)	1972	E	brown	polyester	1.42	0.4	1.82	no	0.64	325	1025	
177 (212, 229)	1972	E	brown	polyester	0.92	0.4	1.32	no	0.64	325	1025	
178 (213)	1972	E	brown	polyester	0.5	0.4	0.9	no	0.64	325	1025	
179 (214)	1972	E	brown	polyester	0.43	0.24	0.67	no	0.39	325	1025	
186	1989	L	brown	polyester	1.46	0.36	1.82	no	0.68	365	1190	
190	1954	B	red/brown	acetate	0.95	0.35	1.3	no	0.64	260	1100	
200	1957	B	red/brown	polyester	0.5	0.35	0.85	no	0.64	260	1100	
201	1962	C	black	acetate	1.42	0.51	1.93	no	0.64	315	790	
202	1962	C	black	polyester	1.42	0.51	1.93	no	0.64	315	790	
203	1962	C	black	polyester	0.92	0.51	1.43	no	0.64	315	790	
206	1969	D	black	polyester	1.42	0.56	2.08	yes	0.93	320	1050	
207	1969	D	black	polyester	0.85	0.56	1.51	yes	0.93	320	1050	
208	1971	D	black	polyester	1.42	0.4	1.9	yes	0.64	330	1000	
209	1971	D	black	polyester	0.92	0.4	1.4	yes	0.64	330	1000	
217	1982	H	brown	polyester	0.56	0.34	0.98	*	0.75	360	1400	back lubricated tape
219	1985	H	brown	polyester	0.79	0.38	1.25	*	0.75	360	1250	back lubricated tape
226	1979	H	brown	polyester	1.3	0.56	1.94	yes	1.25	360	1400	
227	1980	H	brown	polyester	0.79	0.56	1.43	yes	1.25	360	1400	
250	1974	F	brown	polyester	1.3	0.68	2.06	yes	1.3	380	1200	
265	1977	K	brown	polyester	0.79	0.15	1.04	yes	0.32	700	1350	digital tape
275	1984	L	dark brown	polyester	0.79	0.19	1.04	yes	0.45	710	1400	digital tape



3M Audio Open Reel Tapes 6/3/05 5:05 PM

282	1960	B	red/brn,blk/brn	polyester	1.42	0.39	1.85	no	0.64	260	1030	mag layer overcoat
283	1960	B	red/brown	polyester	0.92	0.39	1.35	no	0.64	260	1030	mag layer overcoat
290	1964	C	black	polyester	0.5	0.17	0.67	no	0.25	265	900	
294	1965	C	black	polyester	0.6	0.43	1.03	no	0.64	265	940	
295	1974	E	brown	polyester	0.43	0.18	0.68	yes	0.36	315	1260	logging tape
296	1974	E	brown	polyester	0.85	0.18	1.1	yes	0.36	315	1260	logging tape
311	1960	B	brown	pvc	1.42	0.38	1.8	no	0.63	260	1050	
806	1986	H	brown	polyester	1.42	0.4	1.9	yes	0.9	370	1400	
807	1986	H	brown	polyester	0.92	0.4	1.4	yes	0.9	370	1400	
808	1986	H	brown	polyester	1.46	0.32	1.86	yes	0.67	360	1320	
809	1986	H	brown	polyester	0.92	0.32	1.32	yes	0.67	360	1320	
908	1993	I	brown	polyester	1.38	0.36	1.82	yes	0.9	365	1600	
966/986	1992	I	brown	polyester	1.38	0.49	1.95	yes	1.25	365	1600	
967	1992	I	brown	polyester	0.92	0.49	1.49	yes	1.25	365	1600	
996	1990	I	brown	polyester	1.42	0.63	2.13	yes	1.7	360	1700	
8135	1972	E	brown	polyester	0.43	0.2	0.7	yes	0.38	315	1250	logging tape
8136	1972	E	brown	polyester	0.85	0.2	1.12	yes	0.38	315	1250	logging tape
8206	1977	J	dark brown	polyester	0.79	0.2	1.05	yes	0.34	345	1050	logging tape
8207	1977	J	dark brown	polyester	0.43	0.2	0.69	yes	0.34	345	1050	logging tape
8265	1981	K	brown	polyester	0.92	0.16	1.14	yes	0.32	650	1250	digital tape
8614	1978	J	dark brown	polyester	0.79	0.2	0.99	no	0.34	345	1050	logging tape
Classic DP	1975	H	brown	polyester	0.42	0.48	0.98	yes	0.9	350	1200	
Classic LP	1975	H	brown	polyester	0.8	0.48	1.36	yes	0.9	350	1200	
Classic SP	1975	H	brown	polyester	1.42	0.48	1.98	yes	0.9	350	1200	
Master	1978	H	brown	polyester	0.8	0.48	1.36	yes	0.9	350	1200	
Master XS	1980	H	brown	polyester	0.79	0.56	1.43	yes	1.25	360	1400	
rev.3, 2/9/00												
scotch												

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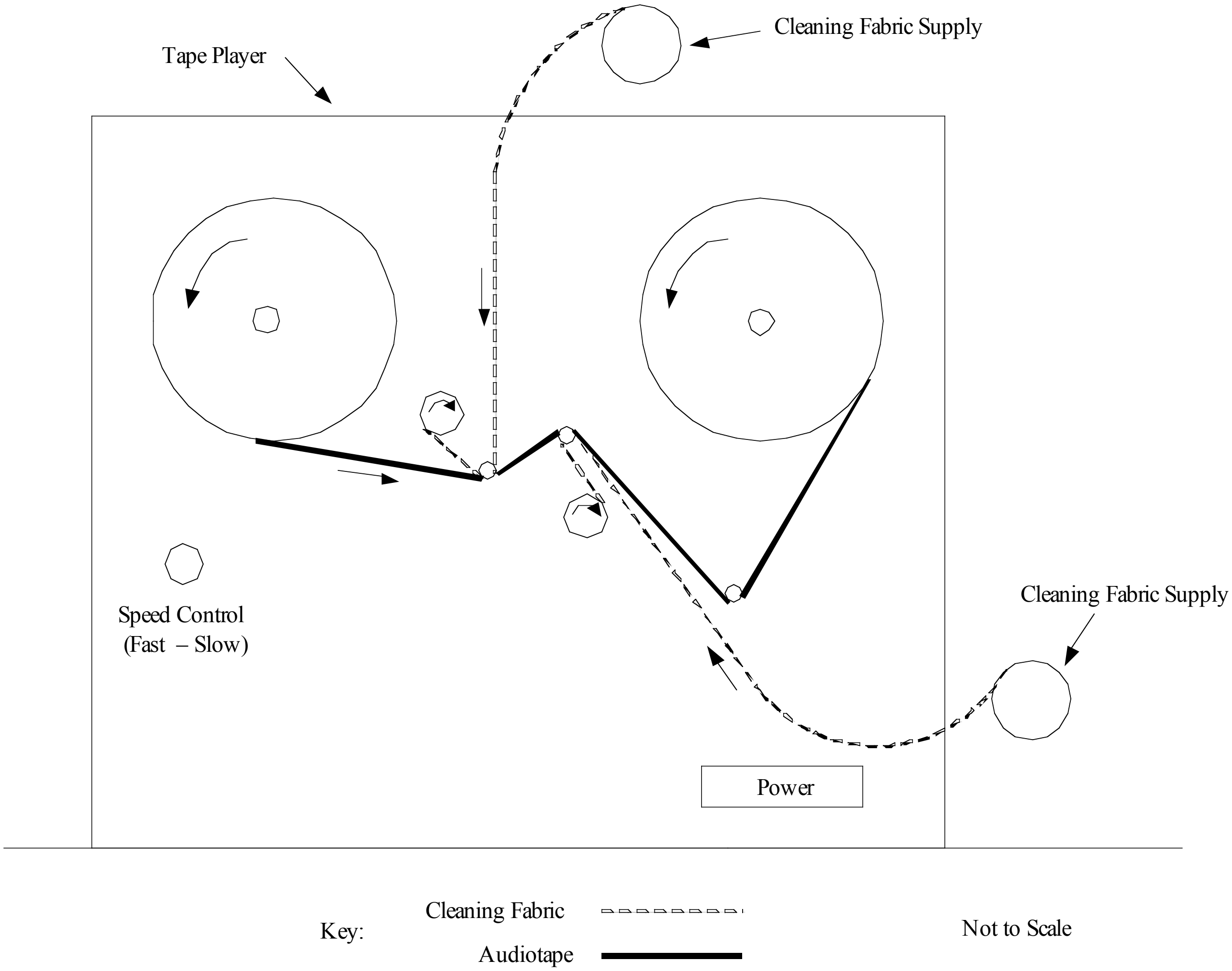
APPENDIX B

Conceptual Design of Audiotape Cleaning Apparatus (Mechanical Method)

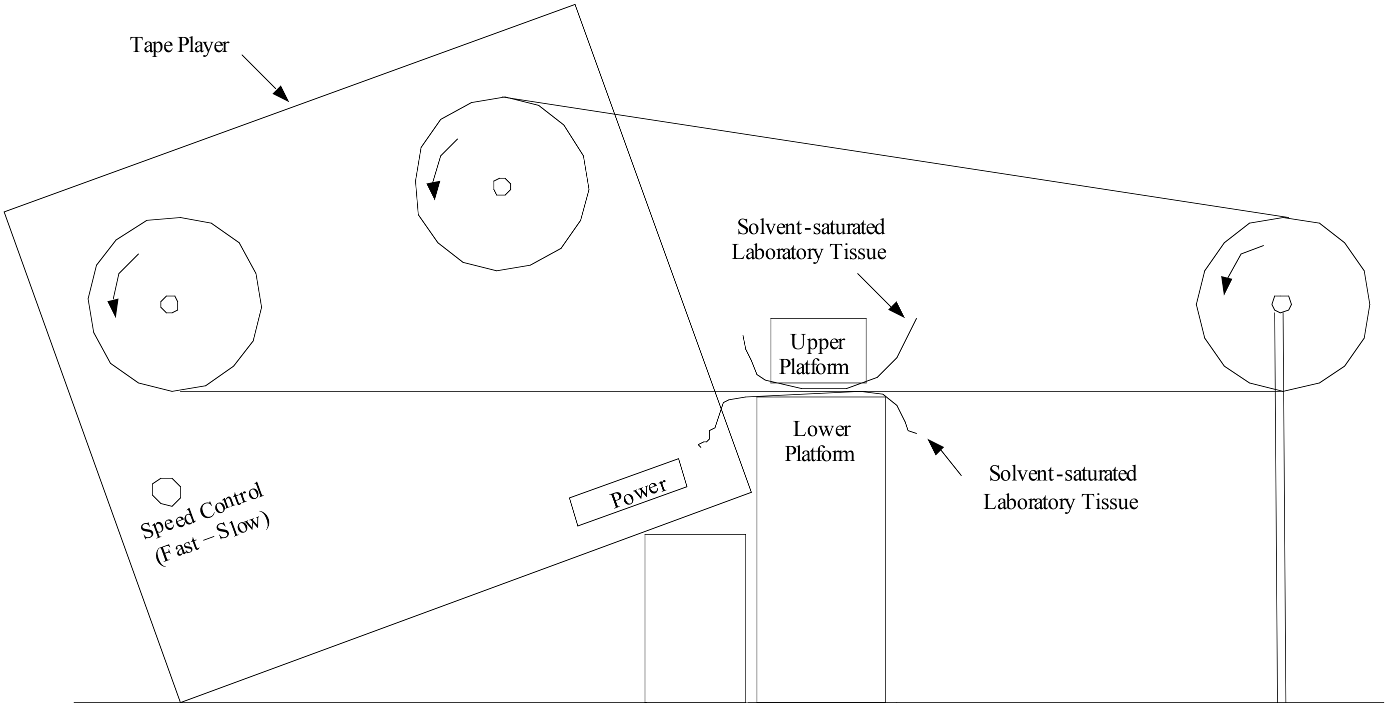
Conceptual Design of Audiotape Cleaning Apparatus (Mechanical Method)



APPENDIX C

Conceptual Design of Audiotape Cleaning Apparatus
(Solvent-Facilitated Method)

Conceptual Design of Audiotape Cleaning Apparatus
(Solvent-Facilitated Method)



Not to Scale

APPENDIX D

Results of Statistical Analyses

TABLE A.1
Results of Statistical Analyses
Backing layer

Wavenumbers showing no statistically significant change in absorbance	Wavenumbers showing a statistically significant change in absorbance	Direction of statistically significant change *
599.8 – 622.9	624.8	+
626.8 – 628.7	630.6 – 651.8	+
653.8 – 657.6	659.5	+
661.5	663.4 – 665.3	+
667.3 – 669.2	671.1	+
673.0 – 684.6	686.5	-
688.5 – 692.3	694.3	-
696.2 – 702.0	703.9	-
705.8 – 744.4	746.3 – 748.3	-
750.2 – 754.0	756.0 – 757.9	-
759.8 – 823.5	825.4	+
827.3 – 891.0	892.9 – 904.5	-
906.4 – 910.3	912.2 – 914.1	-
916.0	918.0 – 919.9	-
921.8 – 925.7	927.6	-
929.5 – 981.6	983.5 – 1305.6	-
1307.5 – 1359.6	1361.5 – 1375.0	-
1376.9 – 1407.8	1409.7	+
1411.7 – 1465.7	1467.6 – 1469.5	+
1471.4 – 1473.4	1475.3 – 1538.9	+
1540.9	1542.8 – 1556.3	+
1558.2	1560.2 – 1683.6	+
1685.5	1687.4 – 1693.2	+
1695.2 – 1704.8	1706.7 – 1764.6	-
1766.5 – 1778.1	1780.0 – 2304.6	+
2306.5 – 2325.8	2327.7 – 2345.1	-
2347.0 – 2350.8	2352.8 – 2368.2	-
2370.1 – 2377.8	2379.8 – 3999.7	+

* Note: Statistically significant changes denoted by (+) represent *decreases* in before- to after-cleaning absorbance values. Statistically significant changes denoted by (-) represent *increases* in before to after-cleaning absorbance values. Although counterintuitive, this occurs because in the statistical analysis calculations, the average after-cleaning absorbance values were subtracted from the average before-cleaning absorbance values. This affects only the sign (i.e, + or -) of the difference, not the magnitude of the difference. Only the magnitude of the difference is used to determine whether a statistically significant change occurred.

TABLE A.2
Results of Statistical Analyses
Binder layer

Wavenumbers showing no statistically significant change in absorbance	Wavenumbers showing a statistically significant change in absorbance	Direction of statistically significant change *
	599.8 – 757.9	-
759.8 – 761.8	763.7 – 773.3	-
775.3 – 779.1	781.0	-
783.0 – 823.5	825.4 – 827.3	+
829.3 – 960.4	962.3 – 964.2	-
966.2 – 997.0	999.0	+
1000.9 – 1068.4	1070.3 – 1080.0	-
1081.9 – 1099.2	1101.2 – 1145.5	-
1147.5 – 1164.8	1166.7 – 1176.4	-
1178.3 – 1218.8	1220.7 – 1303.7	-
1305.6 – 1332.6	1334.5 – 1421.3	+
1423.2 – 1469.5	1471.4 – 1700.9	+
1702.9 – 1708.7	1710.6 – 1747.2	+
1749.1 – 1753.0	1754.9 – 2844.5	+
2846.5 – 2861.9	2863.8	+
2865.7 – 2873.5	2875.4 – 2887.0	+
2888.9 – 2910.1	2912.0 – 2937.1	-
2939.0 – 2973.7	2975.7 – 3999.7	+

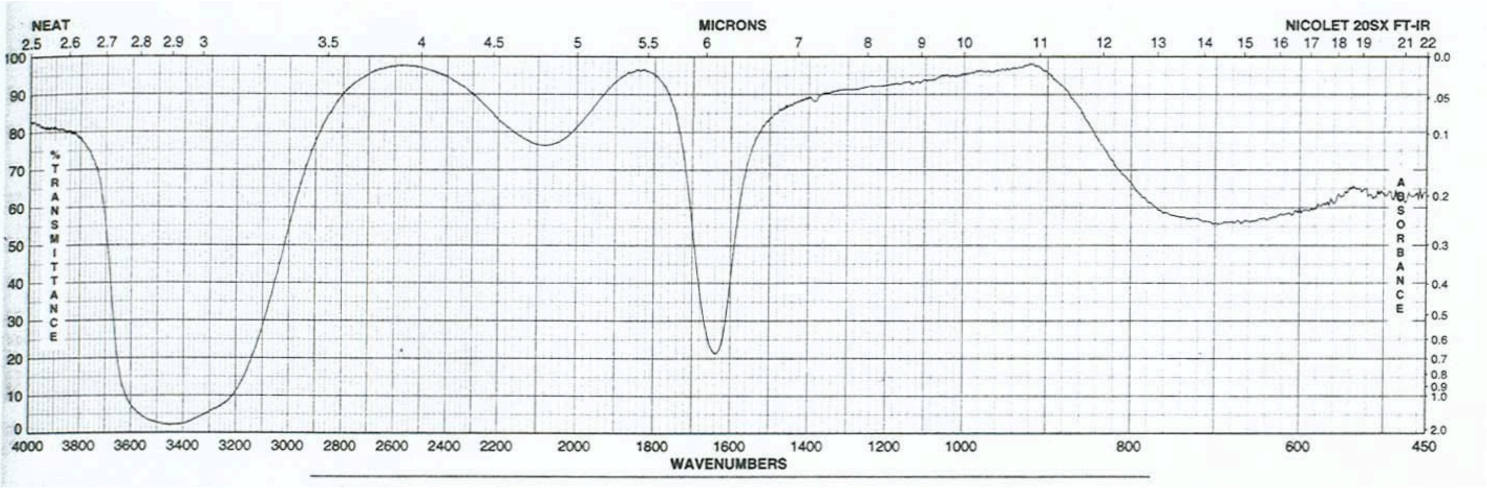
* Note: Statistically significant changes denoted by (+) represent *decreases* in before- to after-cleaning absorbance values. Statistically significant changes denoted by (-) represent *increases* in before to after-cleaning absorbance values. Although counterintuitive, this occurs because in the statistical analysis calculations, the average after-cleaning absorbance values were subtracted from the average before-cleaning absorbance values. This affects only the sign (i.e, + or -) of the difference, not the magnitude of the difference. Only the magnitude of the difference is used to determine whether a statistically significant change occurred.

APPENDIX E

FT-IR Reference Spectrum
For
Water

From Pouchert, C.J. (1997)

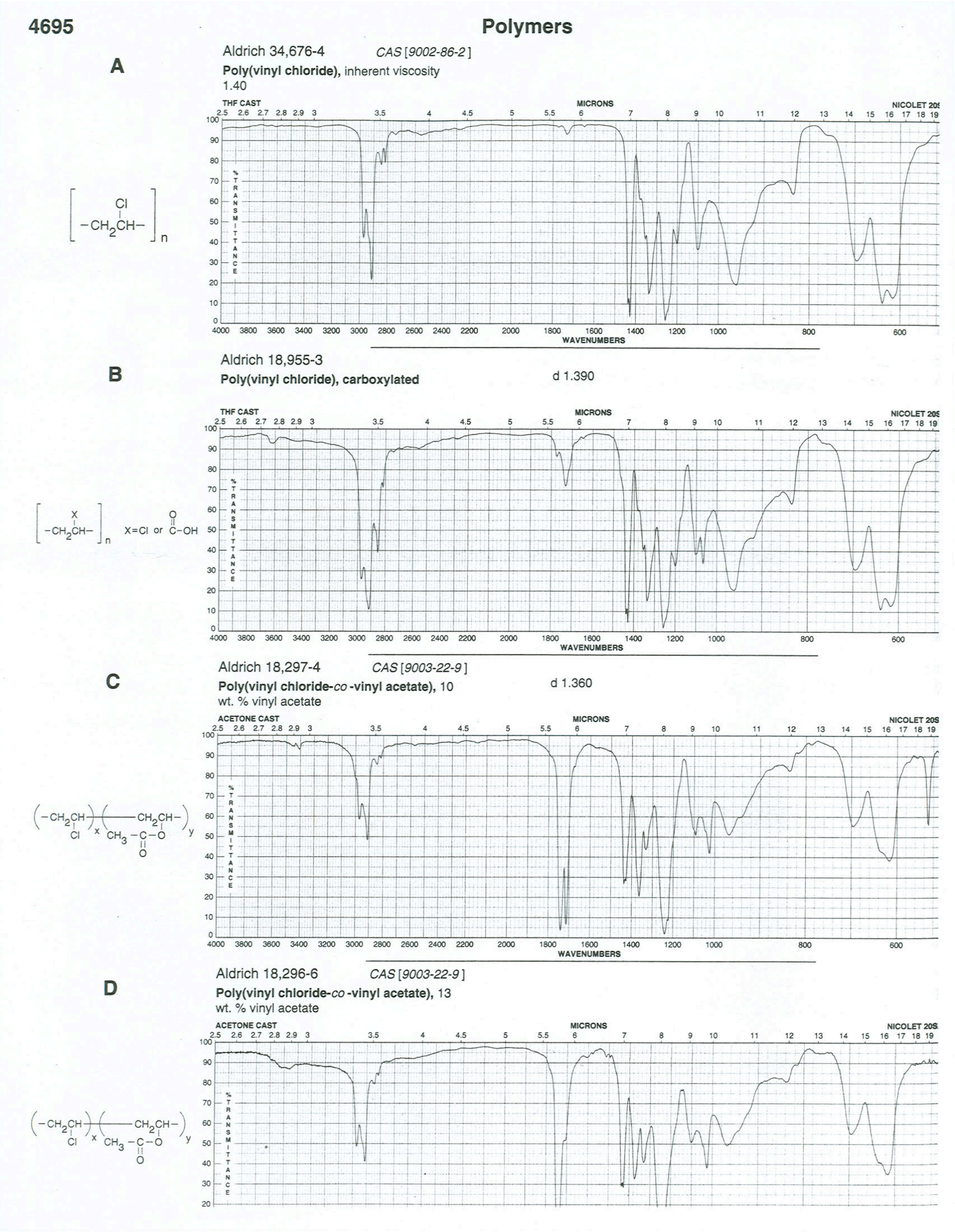
FT-IR Reference Spectrum
Water, deuterium-depleted



APPENDIX F

FT-IR Reference Spectra
For
Poly(vinyl chloride) Compounds

From Pouchert, C.J. (1997)



4695 **Polymers**

A

Aldrich 34,676-4 *CAS [9002-86-2]*

Poly(vinyl chloride), inherent viscosity 1.40

$[-CH_2CH(Cl)-]_n$

B

Aldrich 18,955-3

Poly(vinyl chloride), carboxylated d 1.390

$[-CH_2CH(X)-]_n$ X=Cl or C(=O)-OH

C

Aldrich 18,297-4 *CAS [9003-22-9]*

Poly(vinyl chloride-*co*-vinyl acetate), 10 wt. % vinyl acetate d 1.360

$(-CH_2CH(Cl)-)_x(-CH_2CH(O-C(=O)-CH_3)-)_y$

D

Aldrich 18,296-6 *CAS [9003-22-9]*

Poly(vinyl chloride-*co*-vinyl acetate), 13 wt. % vinyl acetate

$(-CH_2CH(Cl)-)_x(-CH_2CH(O-C(=O)-CH_3)-)_y$

APPENDIX G

FT-IR Reference Spectra
For
Cellulose Acetate Compounds

From Pouchert, C.J. (1997)

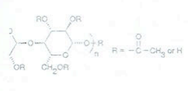
Polymers

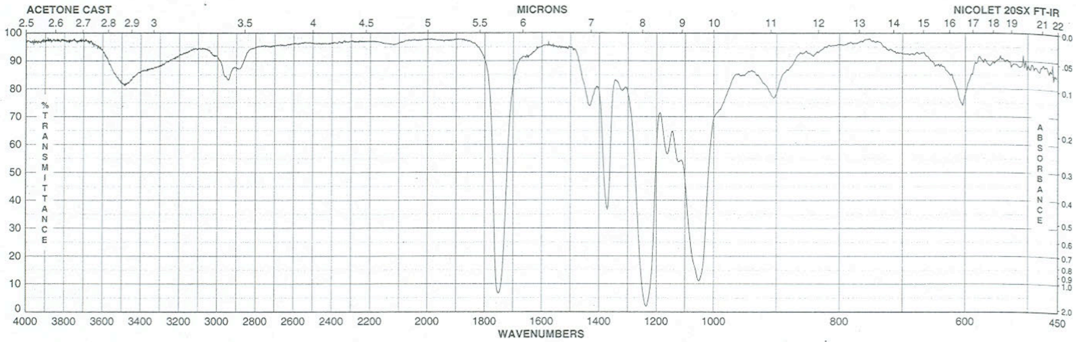
A

Aldrich 18,095-5 *CAS* [*9004-35-7*]

Cellulose acetate

d 1.300
n_D^{20}1.4750

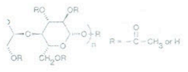


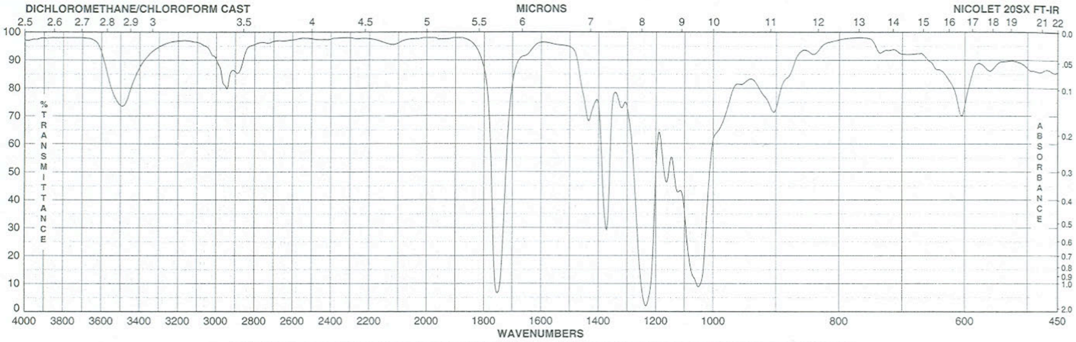


B

Aldrich 41,902-8 *CAS* [*9004-35-7*]

Cellulose acetate

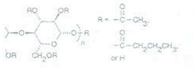


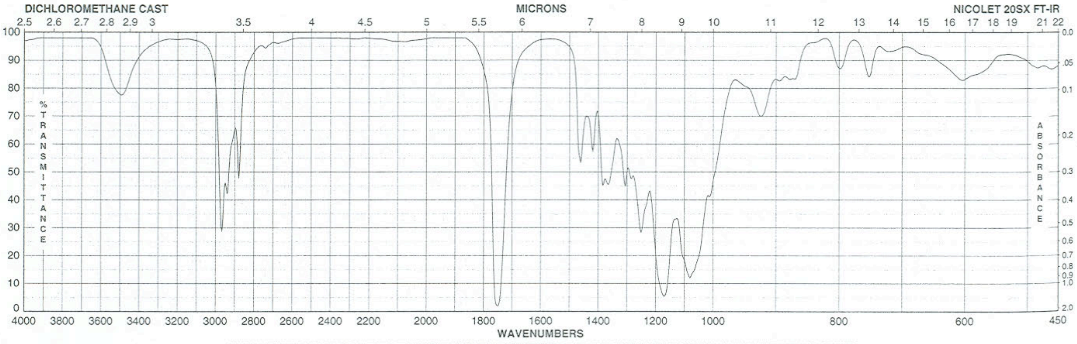


C

Aldrich 41,906-0 *CAS* [*9004-36-8*]

Cellulose acetate butyrate

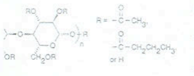


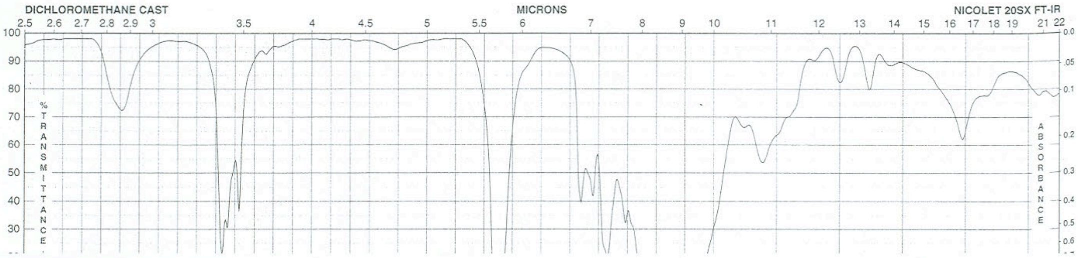


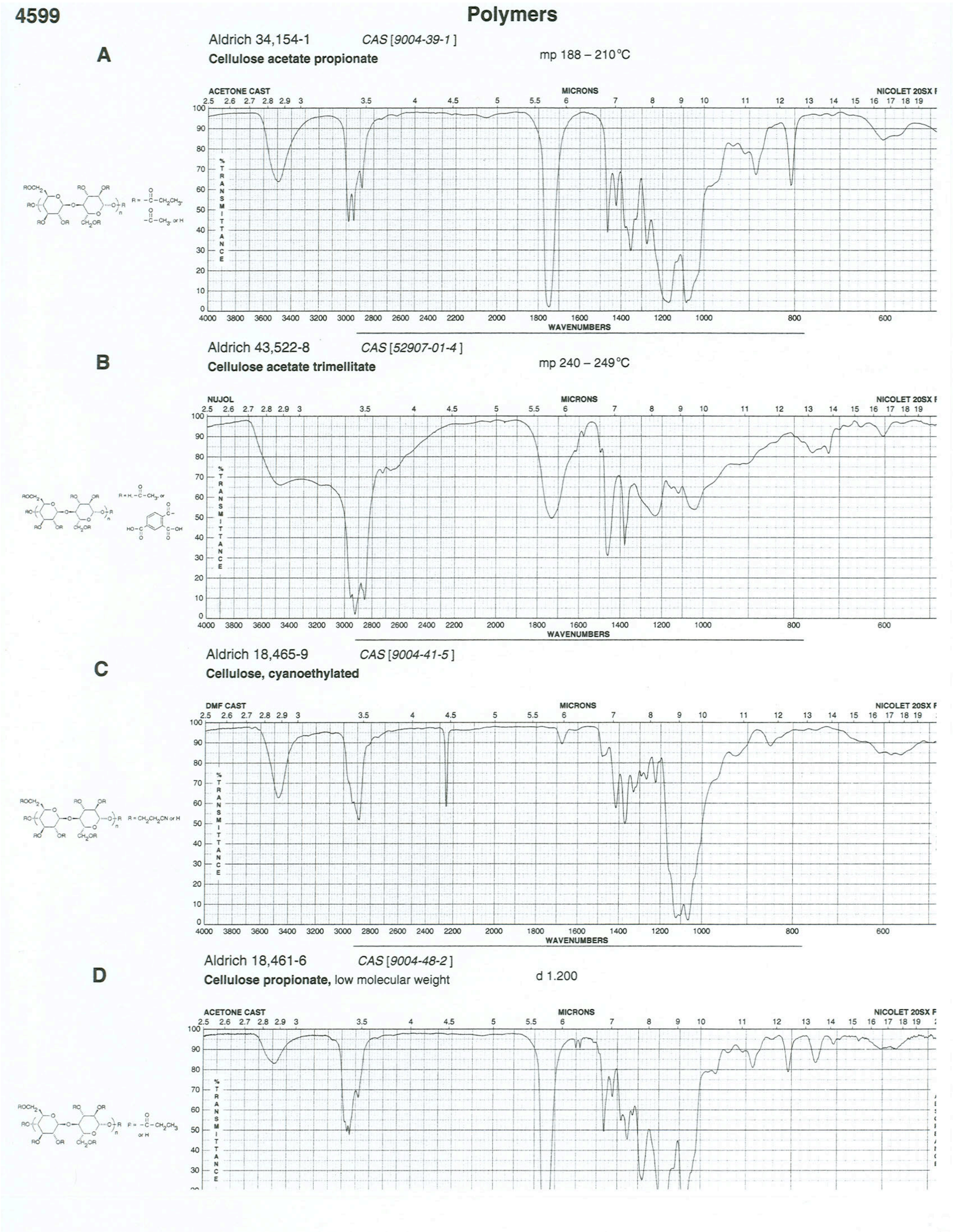
D

Aldrich 41,905-2 *CAS* [*9004-36-8*]

Cellulose acetate butyrate







4599 **Polymers**

A

Aldrich 34,154-1 *CAS [9004-39-1]*

Cellulose acetate propionate mp 188 – 210°C

B

Aldrich 43,522-8 *CAS [52907-01-4]*

Cellulose acetate trimellitate mp 240 – 249°C

C

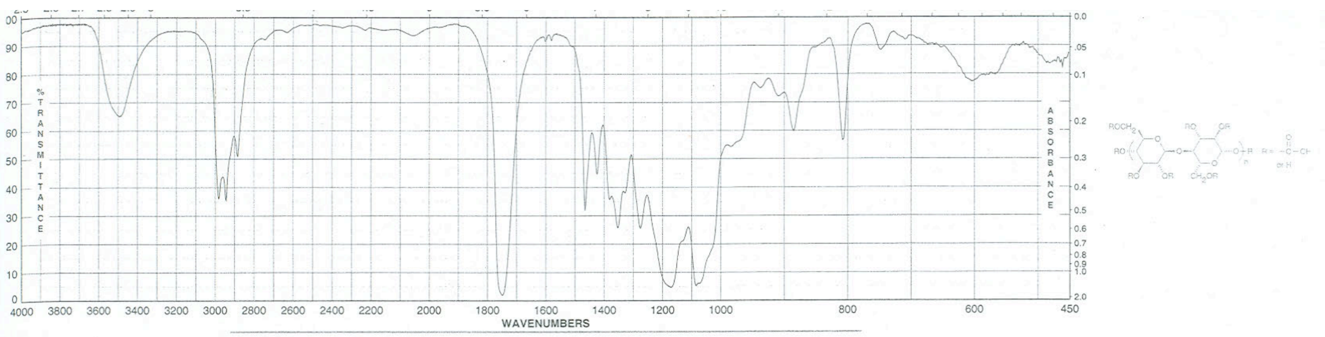
Aldrich 18,465-9 *CAS [9004-41-5]*

Cellulose, cyanoethylated

D

Aldrich 18,461-6 *CAS [9004-48-2]*

Cellulose propionate, low molecular weight d 1.200

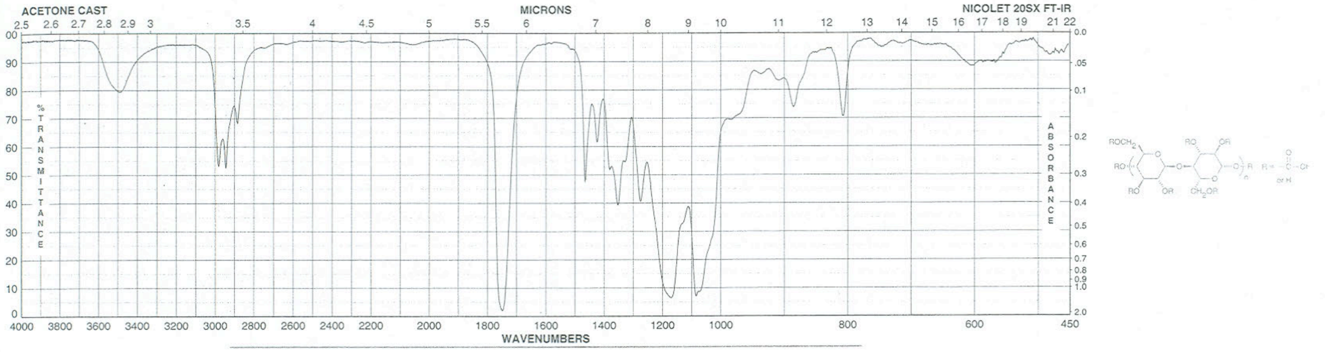


Aldrich 18,462-4 *CAS [9004-48-2]*

Cellulose propionate

d 1.230

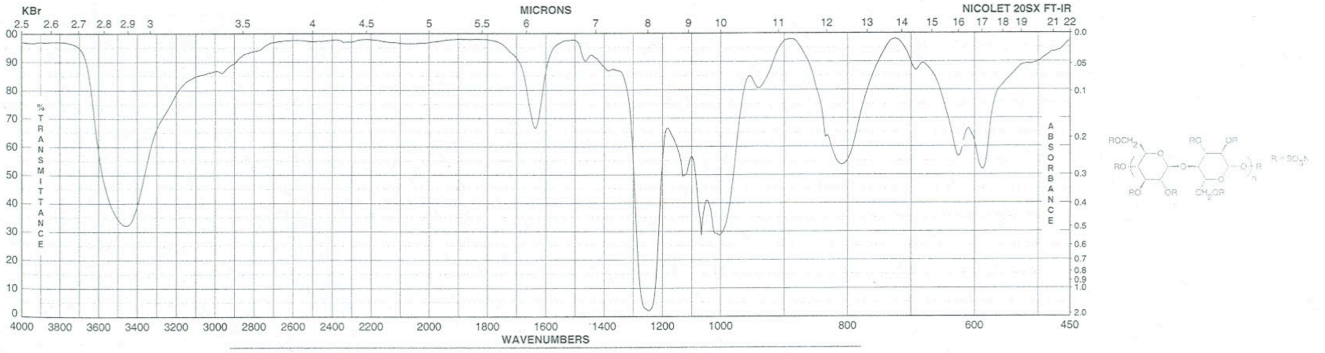
B



Aldrich 18,099-8 *CAS [9005-22-5]*

Cellulose sulfate, sodium salt

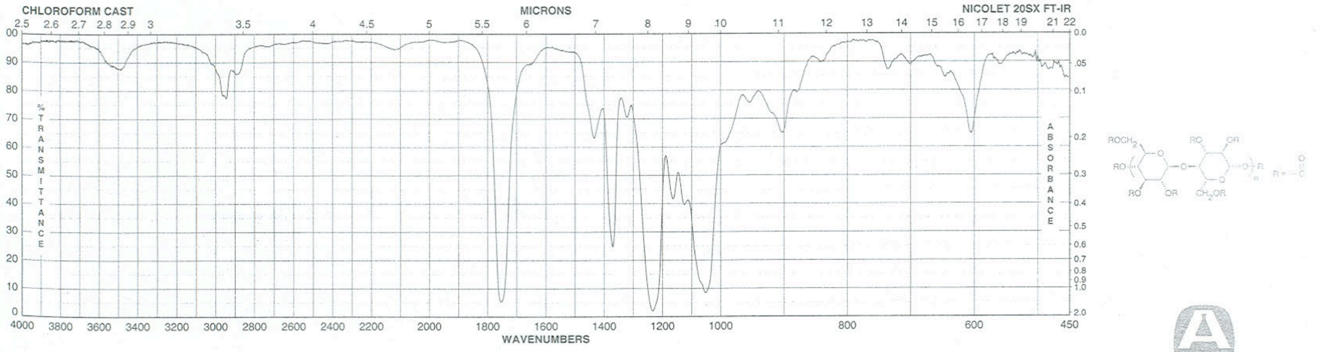
C



Aldrich 18,100-5 *CAS [9012-09-3]*

Cellulose triacetate

D



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