



D6.1 : Report on video and audio tape deterioration mechanisms and considerations about implementation of a collection condition assessment method (D6.2)

Period of work: February 2004 – June 2005-06-30

DOCUMENT IDENTIFIER PS_WP6__CRCDG_D6.1
DATE 2006-09-01
ABSTRACT This report is deliverable D6.1 of the WP06 Preservation WA. Known or suspected deterioration mechanisms are presented with their relations to deterioration symptoms as experienced while playing back some archival magnetic tapes. Related condition assessment methods are described. The on-site transfer of these methods are evaluated. A proposal for a “condition assessment method” based on knowledge database and knowledge management system is proposed in a second part.
KEYWORDS Magnetic tape, Deterioration mechanisms, Assessment methods, Knowledge database, KM systems
WORKPACKAGE / TASK WP06
AUTHOR, COMPANY Benoît THIEBAUT, Léon-Bavi VILMONT, Bertrand LAVEDRINE
 Centre de Recherches sur la Conservation des Documents Graphiques, CRCDG (CNRS-French Ministry of Culture, MNHN)
NATURE Report
INTERNAL REVIEWERS Frédéric Dumas (INA), Didier Giraud (INA)

DOCUMENT HISTORY

Release	Date	Reason of change	Status	Distribution
0.1	2005-06-15	Created	Living	Confidential
0.2	2005-10-19	French draft version	Living	Confidential
0.3	2005-12-15	Bibliographic notes added	Living	Confidential
0.4	2006-01-13	BBC proof	Living	Confidential
0.5	2006-03-20	INA reading	Living	Confidential
0.6	2006-09-01	Final report	Published	Public

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Report introduction

The tasks for “Media Condition Assessment” Work Package (WP06) are:

- to understand the way video tapes degrade over time and become unplayable
- to develop a method to measure the deterioration level in order to anticipate playback problems.

These objectives are particularly important considering the financial and time impact of mass transfer operations of audiovisual (A/V) tapes for preservation and access purposes. Optimisation of the preservation workflow and prioritisation of the media according to technical considerations are highly required.

The magnetic tapes deterioration is a difficult notion to define because numerous parameters are involved: operator tape handling, tape player, original media quality, materials formulations, and chemical decay. As a consequence, the magnetic tapes deterioration study requires multidisciplinary fields of investigation from mechanical player considerations to organic chemistry analysis. The CRCDG used a comprehensive study strategy involving all aspects of the problem. This report reflects this approach.

It is expected that anyone involved in any aspect of the preservation process of magnetic tape find information related to their own specific problems.

The first part (**Part A**) intends to provide an overview of the reasons why a tape becomes unplayable and to identify specific chemical deterioration issues. Then, all relevant deterioration mechanisms leading to playability problems are reviewed. This review allows us to establish a first list of potential “deterioration markers” that are evaluated on the basis of measurement methods considerations.

The second part of the report (**Part B**) is based on the results and data obtained from our laboratory investigations in order to propose a condition assessment method for archival magnetic tape collections based on a statistical approach. A knowledge database and its management system and applications are presented in detail. This presentation is accompanied with requirements for a software tool and demonstration software.

A TECHNICAL REPORT

A.1 INTRODUCTION TO MAGNETIC TAPES

A.1.1 HISTORICAL BACKGROUND OF MAGNETIC RECORDING

Valdemar Poulsen discovered the magnetic recording principle in 1884 and patented the Telegraphone in 1898. Since this first magnetic recorder using wire or steel ribbon, the evolution of magnetic tapes technology is bound to the development of recorders.

Audio recorders were first developed in the 1920s in Germany for steel tapes; paper strips with iron particles (Fritz Pfleumer, 1928) were also used as recording media. Finally the AEG company developed the Magnetophon (Berlin, 1935) a recorder that used the preferable cellulose acetate as tape substrate

Earlier audio magnetic recordings on wire, steel ribbons, paper and earlier acetate tapes with low coercivity constitute a very small part of the actual A/V archive collections. They can be considered as historical media.

Conversely, recordings on oxide coated plastic tapes (3M Company, 1947) became popular with the audio tape recorder (Ampex, 1948) and that technology has been widely used and adopted as the reference technology with no major modifications for decades except for the materials used.

A search of better magnetic particles plastic tape was conducted aiming for 3 properties: to increase the coercivity, to increase the recording density, and to increase the mechanical properties required by tape players under constant evolution.

The early longitudinal recording technology (tape scanning speed = tape speed) requires the most simple tape transport system. The small audio signal bandwidth (about 20 000 kHz) implies rather slow tape speed and as consequence, no high tape mechanical performance.

In order to increase the scanning rate, without increasing the tape speed which leads to accurate tape transport design problems, rotary heads were introduced in 1950 along with transverse tape recording. This recording technology allowed bandwidth compatible with television video signals. The first rotating head video recorder (Quadruplex, Ampex Corporation, 1956) was developed for 50.8 mm (2 inches) wide tapes with slow tape-speed and high scanning rate (38m/s). The high head rotation

frequency implied the development of new tape materials with high tribological performance to withstand important stresses induced by head friction.

A new level was reached with the introduction of helical scan recording in 1960 and developed as a compromise to overcome both the rotating-head speed limitation and difficulties inherent to high speed tape transport.

Tape chemical composition also was improved in order to fit the new equipment specifications, to improve the quality of the retrieved signal and to allow a higher density of recorded information.

Magnetic layers tended to have higher density of “active magnetic” material. The materials used range from the oxides to metal particles (MP) dispersed in a binder and finally, the higher magnetic density is achieved for thin metallic films that form evaporated metal (ME) systems without binder.

Further historical background on magnetic recording technology is presented in [1,2].

A.1.2 MAGNETIC TAPES TYPOLOGY

Exhaustive reviews on the magnetic formulations are available in [3,4].

Tape housing

Magnetic tapes can be packaged in open-reels, cartridges or cassettes, in many size formats. Design of the housing is important for tape protection, handling and play an active role in the tape-transport system. Cassette and to a lesser extent, cartridge and open-reel, include some complex mechanical parts that have no direct utility in information recording but are part of the whole tape transport system.

Tape structure

Magnetic media can be discriminated between **particulate media** (oxides or metal) and **thin film of magnetic material** (metal evaporated). In both cases, magnetic tapes present a typical multi-layered cross section structure [5]:

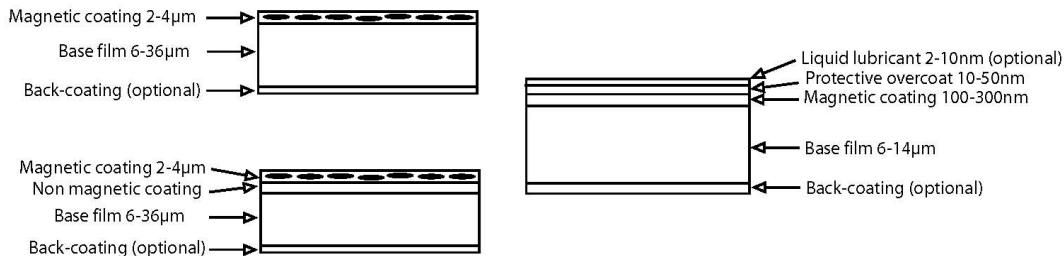


Figure 1 Conventional particulate media (upper left), Dual Coat particulate media (left bottom), Thin-film magnetic tape (right).

Tape composition: magnetic material

The most common particles used are: chromium dioxide (CrO_2), gamma Ferric Oxide ($\gamma\text{Fe}_2\text{O}_3$), and cobalt-doped $\gamma\text{Fe}_2\text{O}_3$.

MP is made from pure metal particles: Barium Ferrite particles, iron alloyed with cobalt or nickel. MP Technique is used for high density recording applications. Its higher concentration of magnetic material in a given binder volume allows higher coercivity and magnetization than metal oxide.

Metal particulates and Chromium dioxide provide a higher signal output and permit higher recording frequencies than iron oxide. However, if untreated, these latter result in less stable magnetic coatings. Metal particles are subject to corrosion, and chromium dioxide is harmful to the magnetic layer binding polymer due to its natural oxidative behaviour.

Tape composition: polymeric binder

Binder is present only in particulate media tapes (oxides, MP) and is used to bind particles within a compact layer onto the base film. Higher magnetic particles density requires lower binder quantity but sufficient binding properties. The majority of tapes use polyester-polyurethane (PUR-ES) as binders polyurethane polymers are extremely diverse and complex molecules produced by combining a large number of

simpler molecules called monomers. The quasi infinite formulations and blending allow the possibility to obtain a great variety of binders matching specific mechanical properties. Unfortunately, depending on the starting monomers used, some PUR-ES will undergo hydrolysis reaction leading to the polymer breakdown.

Tape composition: additives

Additives for magnetic tapes include manufacturing agents (dispersive products) ensuring good homogeneity of the magnetic particle dispersion, fillers (abrasives) also called “head cleaning agent”, carbon black for anti-static effect, lubricating agents and stabilizers (anti-oxidant, anti-hydrolytic etc).

Tape composition: substrate

Cellulose acetate was used in the 1940s and 1950s as tape backing for analogue audio tapes. Cellulose acetates are susceptible to decompose over time generating acetic acid. (This is known under the name of vinegar syndrome). **PolyVinylChloride PVC** was used less frequently.

PolyEthylene Terephthalate (PET) and **PolyEthylene Naphtalate (PEN)** are by far more stable chemically in archival environmental conditions. Since the 1960s, oriented PET film was used as tape substrate material for both audio tapes and video tapes.

A.1.3 U-MATIC FORMAT: A MODEL FOR MAGNETIC TAPE STUDY

U-matic tapes were widely used all over the world for broadcasting purpose, from the 1970s to the late 1990s and are finally present in numerous A/V archives even if they were not primarily designed for storage purpose.

The U-matic tapes represent a good model for studying magnetic tapes: 1) for tape composition reasons, 2) tape transport design considerations and 3) it exists numerous feedback from users reporting on preservation issues.

1) Tape composition

Magnetic particles are dispersed in a PUR-ES binder coated on a PET substrate. Being part of the mass production for a long period of time, it can be reasoned that major PUR-ES and PET formulations have been used at a given period for U-matic manufacturing.

As a consequence, all observations made regarding this format may be extended to the majority of the A/V magnetic tapes.

2) *Tape-transport technology*

The recording technology for U-matic tapes is helical. The tape transport design related to this technology is very sensitive to tape deterioration or tape manufacturing quality variations. This transport design can serve as an early warning system allowing to enhance the observation of the failure mechanisms.

3) *Preservation issues reporting*

U-matic tapes were manufactured on a large scale by well known companies that all had specific quality control management insuring certain homogeneity in terms of product specifications among batches. Preservation issues experienced by the archive community have largely been observed and described. As a consequence, identifying main deterioration issues and related manufacturing technologies is easier with U-matic tape format.

A.2 TAPE DETERIORATION : SYMPTOMATOLOGY

An “alteration symptom” can be defined as the signs regarded as evidence of the existence of an alteration process. These symptoms can be experienced either when playing a tape, either by quick tape examination. If the alteration leads to loss of performance of the magnetic tape, this alteration is a deterioration.

Different types of symptoms can be categorized according to the effect of the alteration processes on the fundamental role of an archival magnetic tape:

- Symptoms that are connected to the recording playback (access) (A2.1)
- Symptoms related to the preservation of the recorded information (A2.2)
- Symptoms not related so far to the information preservation or access (A2.3).

A.2.1 ACCESS-RELATED SYMPTOMS

Definition

Access related symptoms are dealing with alteration processes that affect **the capacity for reading a tape** (setting in mechanical motion and access to magnetic tracks). They are the most commonly encountered symptoms.

By definition, access related symptoms are experienced only while reading a tape. Even if in most cases, they do not lead to the loss of the document, they represent an important issue for large scale migration projects.

Origins and examples

These symptoms have a manifold origin and different parameters that can modulate them (strengthen or reduce) and that are dependent and independent on the magnetic media itself. The table below give examples of access related symptoms, origins and significant parameters.

Origin	Parameters	Examples of symptoms
Tape player and/or operator	<ul style="list-style-type: none"> → reading machine settings → tape mishandling → highly frequent tape reading (wear, lubricant consumption) → environmental conditions (humidity, temperature, dust etc) while tape playing. 	<ul style="list-style-type: none"> → tape-transport instability → no tape-transport → many dropouts → signal strength decrease → loss of signal
Tape packaging	<ul style="list-style-type: none"> → integrity of cassette/cartridge/hubs parts. 	
Magnetic recording	<ul style="list-style-type: none"> → initial quality of the recording → signal processing options 	
Magnetic tape	<ul style="list-style-type: none"> → alteration of dimensional properties → alteration of tribological properties → lubricant evaporation/conversion → alteration of the head to tape gap by deterioration products or deposited materials (mould, dust etc) → head-clogging by deterioration products. → alteration of particle magnetic properties 	

Considering the diversity of the origins and parameters, these symptoms cannot be attributed solely to the deterioration of the materials.

Impact on preservation process by migration

The alteration process resulting in these symptoms are more or less problematic depending on the playback quality requirements. If only some parts of the recording are affected, if only loss of quality is experienced or if time is not a financial issue, these symptoms can be overcome.

However, in some cases, technologies or financial resources may not be available for mass treatment of the tapes presenting such symptoms that, in theory, do not lead to a definitive loss of the information. This is the reason why some recordings are occasionally destroyed when they are not read on time.

In addition, attempting to play tapes showing these symptoms without monitoring or by unskilled persons, may lead to more serious magnetic tape deterioration. For instance, excessive tape wear or the tape jammed inside the playing deck may occur.

A.2.2 RECORDING PRESERVATION RELATED SYMPTOMS

Definition

These symptoms are related to alteration processes that affect the **ability for magnetic media to keep the recording safe**. In the most serious cases, they can be observed from the tapes in the storage area. Tapes can also occasionally show these symptoms while they are played back due to additional mechanical stress.

Origins and Examples

Origin	Parameters	Examples of symptoms
Structural integrity of the magnetic coating	<ul style="list-style-type: none"> → wear → mishandling or storage disaster → Important playback stress → deterioration of the physical and chemical properties upon ageing under some environmental conditions 	<ul style="list-style-type: none"> → particles shedding → base film breakage → magnetization loss
Base film	<ul style="list-style-type: none"> → mishandling → important playback stress → deterioration of the physical and chemical properties upon ageing under some environmental conditions 	
Magnetic properties	<ul style="list-style-type: none"> → demagnetization (external field, temperature etc) → deterioration of the physical and chemical properties of the particles upon ageing under some environmental conditions 	

Impact on preservation process by migration

These symptoms are extremely severe because they lead to a definitive loss of the recorded information.

If only parts of the recording are affected, some recording technology may overcome very small loss (for instance redundant systems).

A.2.3 OTHER SYMPTOMS

Definition

The relationship between the alteration processes leading to these symptoms and access or preservation issues haven't been so far demonstrated. They cannot be associated systematically to a "deterioration".

Origins and examples

Origin	Parameters	Examples of symptoms
Chemical formulation	<ul style="list-style-type: none"> → Storage conditions → Tape storage in open air 	<ul style="list-style-type: none"> → Evaporation of manufacture residual products (solvents, un-reacted products) → Consumption and conversion of stabilizers (antioxidants, anti-hydrolysis agents)
Unknown	Unknown	→ odours

Impact on tape migration preservation

Even if these alteration processes have no demonstrated effects on the preservation process, they are all related to materials changes and may lead to mechanical properties modification. Especially, high additives consumption or important chemicals decay producing odouring compounds may result in further playability problems as those already described in A2.1 and A2.2.

A.2.4 SCOPE OF THIS STUDY

Because relationships between deterioration processes and symptoms are numerous, various and complex, a considerable effort of multidisciplinary research and technological development is required in order to identify an early deterioration marker.

For the purpose of our study, it will take into account only the alteration mechanisms that appear at a significant frequency and have a direct effect on document mass transfer workflow or financial charges.

Some of the preservation issues presented earlier are solved by common preservation practices such as housing replacement, tape cleaning or signal restoration. These methods are well known, commonly applied and already integrated to the workflow of preservation projects. Therefore, we only focus on alterations that are not overcome by these techniques.

We exclude from these techniques: baking [6], relubrication [7], chemical cleaning [8] or tape back-coat removal [9]. These techniques are currently much debated among the audiovisual professionals community and not used for magnetic tape mass-treatment.

As a consequence, we will limit our investigation to the following preservation issues:

- tape damage that have consequences on **tape tribological behaviour**
- tape damage causing **head-clogging**
- tape damage causing **cohesion loss of the magnetic coating**

Study restricted to the magnetic layer

We restrict our research on tape deterioration to the study of the magnetic coating deterioration for the following reasons:

- polyester substrate has no essential function in recording and outlast the magnetic layer in archival conditions.
- the magnetic coating is much more complex in binder formulation, in magnetic particles, lubricants and additives and as consequence, subject to various alteration mechanisms
- during playing or storage, it is this upper layer that is the most exposed to environment and mechanical stresses (friction)
- the preservation issues selected earlier (tribological behaviour, head clogging and loss of cohesion) all concerned the tape surface or the magnetic layer.
- backcoating and magnetic coating have a similar chemical composition. They only differ from their surface roughness and from their mineral or organic fillers and additives.

A.2.5 SYMPTOM TESTING APPARATUS

Preservation issues selected in paragraph A2.4 are exclusively due to changes in magnetic coating properties.

Tape mechanical characteristics will be studied in conjunction with the tools that have been developed at CRCDG. These tools will not only allow us to identify with precision the factors responsible for the symptoms but also to quantify their impact on the capability to read magnetic tapes.

These devices are:

- tape friction measurement system,
- tape wear assessment system,
- head-clogging characterisation system.

Damage (A2.4)	Test device developed
Magnetic coating tribology change	Friction tester
Head-Clogging	High Speed reading heads photography
Magnetic coating cohesion Change	Wear tester

The tools developed at CRCDG are primarily intended for research. However they can be fitted in order to be used as diagnostic tools which are able to anticipate problems that could appear during tape reading. Such tools allow just a 'rejection test' for damaged tapes before or during the reading and they cannot be fully considered as assessment tools for tape condition.

A2.5.1. Friction Testing (see Appendix 11)

Symptoms of tribological nature cause some difficulties to start a magnetic tape in motion in a reading machine. These symptoms can be assessed by friction measurement.

The nature of friction is composite and heterogeneous. The subcomponents of adhesional and deformational friction and the related physical and chemical properties are shown in the following schematic adapted from [10]:

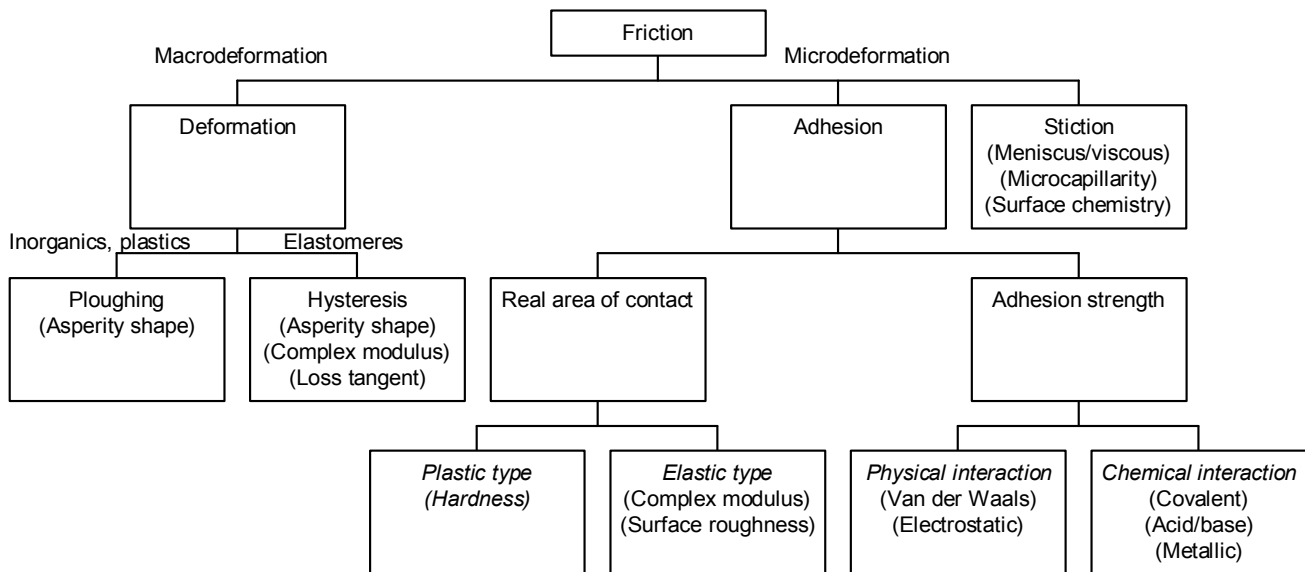


Figure 2 Schematic of sources of polymer friction.

Research tool

Friction measurement allows the study of most of physico-chemical phenomena involved in material deterioration reactions and that have an effect on tribological behaviour of a tape all along the tape path.

Diagnostic tool (rejection test before tape playing)

Apart from its origin, it is known that an extreme friction is generated as a direct consequence due to a difficulty in reading a tape.

Therefore, a tape rejection test for reading can be developed from a friction measurement for which a threshold has to be determined.

Measuring Device

The device developed at CRCDG for research purpose allows the indirect measurement of friction on a given area on a magnetic tape loop using tension sensors. The device design has been adapted from the apparatus used by some authors [11]. More details of the experimental set up are given in the appendix 11.

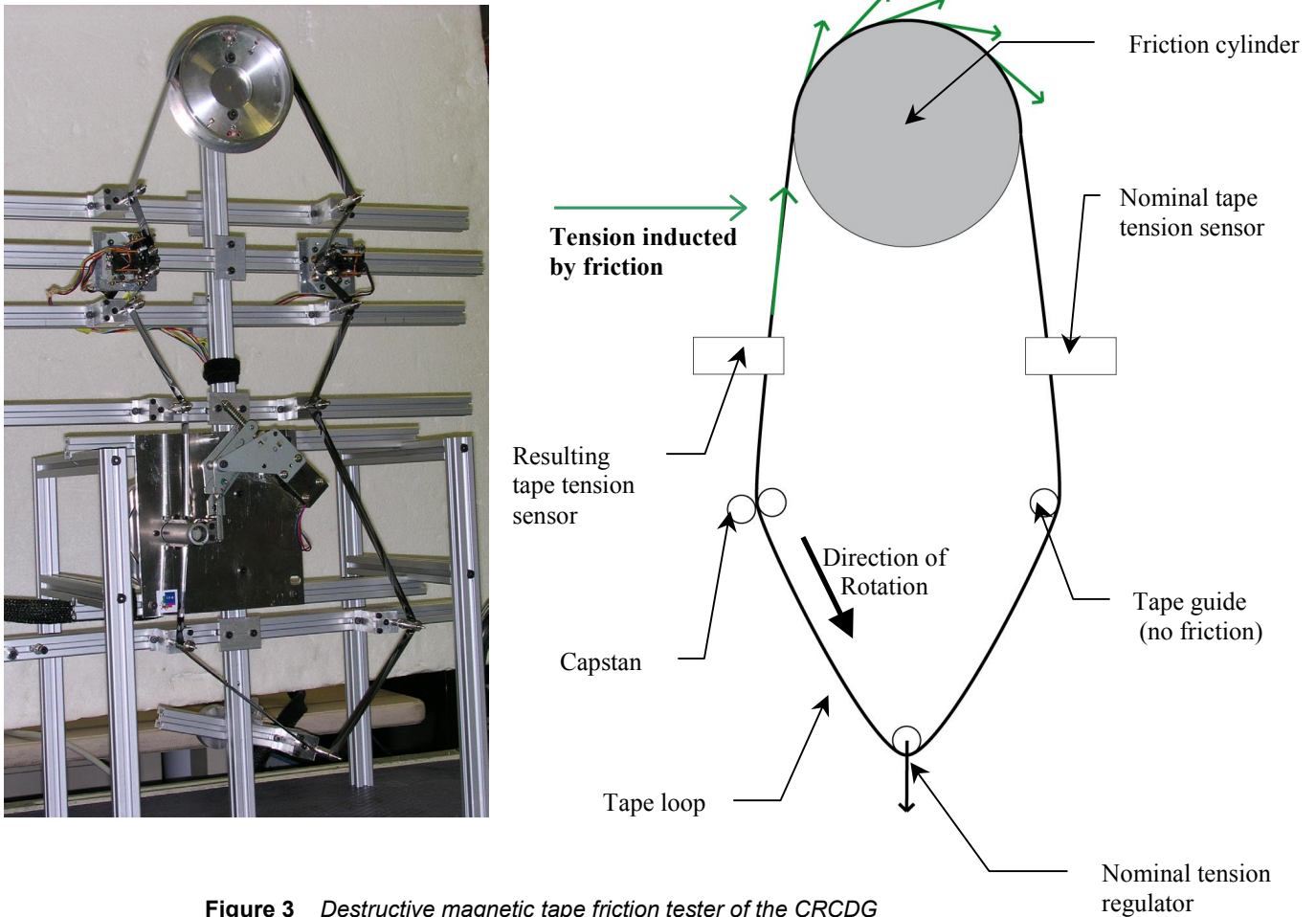


Figure 3 Destructive magnetic tape friction tester of the CRCDG

Further developments

The experimental set-up is not fully operational at the present time. A higher sensitivity to friction variations has to be achieved to get more reliable results. The objectives are to make correlations between chemical decay of tapes, tribological behaviour and tape exploitation symptoms.

A2.5.2. Wear Resistance Tester

The apparatus designed for friction testing as described above can also be used for tape durability testing (wear). Tests over long periods and in different environmental conditions constitute a means for assessing tape durability and wear modes.

A2.5.3. High speed Head-Clogging Imaging (see Appendix 10)

A significant force is applied mechanically on the magnetic layer as a result of high pressure of the heads exerted onto the tape surface and of the relative head/tape speed [12]. As a consequence, the point of contact between tape and head is where most of playback problems occur.

Head-clogging or and an increase of the head to tape distance are the two problems most encountered and of direct importance towards magnetic signal retrieval. Any phenomenon that tends to increase the head/tape gap results inevitably in a loss of quality or in a total loss of head signal [13, 14]. This loss (L) is expressed as (Wallace equation):

$$L(Db) = \frac{d}{\lambda}$$

where d is the change in the head to tape distance and λ is the wavelength of the recorded signal.

A device allowing micro-observation in real time of the phenomenon occurring at the head to tape surface while playing a magnetic tape is useful to investigate head-clogging and recurrent dropout problems.

Research Tool

The heads of a conventional U-Matic tape can be observed continuously by a digital camera equipped with a macro-lens using a stroboscope lighting system synchronized with the drum rotation. The images of the head surfaces are recorded and can be replayed.

This tool developed at CRCDG and adapted from Osaki et al's previous work [15] allows us to associate the quality loss of a signal to estimation and identification of wear debris and materials piled up on the heads surface.

The results of this observation can be connected to other measurements and tests in order to clarify relationships between material deterioration and head-clogging.

Qualitative evaluation of restoration methods, cleaning procedure can also be made.

Diagnostic Tool

A head-clogging detection tool used before any damaging consequence on the reading quality, would permit the optimisation and actuation of a cleaning device. More generally, before reading a tape, such a system can assess the ability for a tape to shed debris onto the heads.

Measuring Device

The device developed at CRCDG for research purposes allows us to observe the forming shed of debris onto the magnetic heads until it becomes impossible to read the tape. A sample movie is available on the enclosed CD.

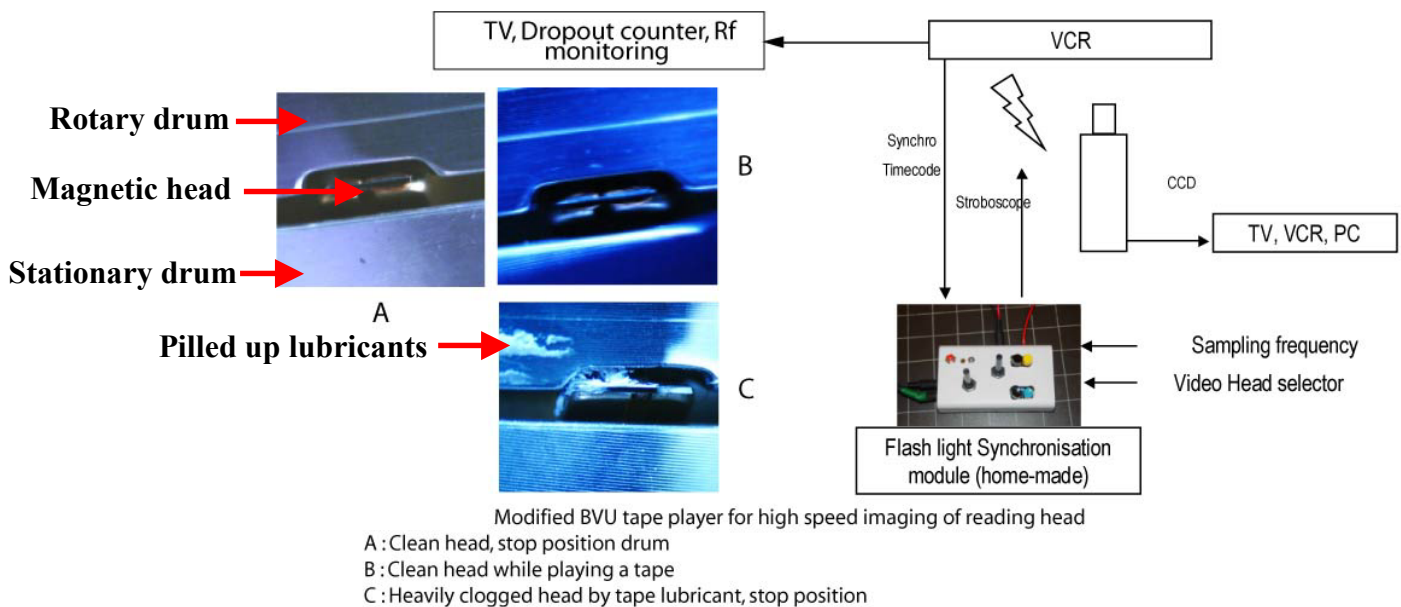


Figure 4 Schematic diagram of the experimental apparatus

In addition to this apparatus, a particles collecting system has been added to the tape transport. Particles present on the tape surface can be samples for further analysis.

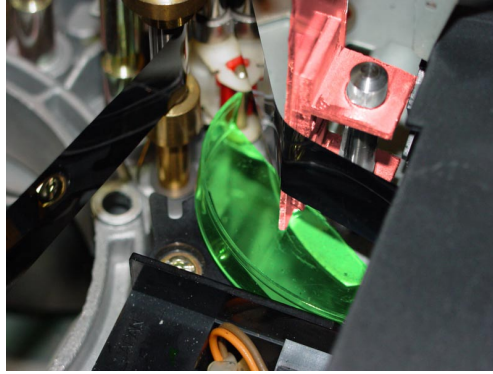


Figure 5 *Particles sampling system assembled in a U-Matic player on a guide before the tension regulation arm. In red: glass microscope slide with an edge in contact with the tape surface In green: Petri dish for collecting the particles.*

The U-matic player modified with the high speed imaging system and the particles collector also allow for the monitoring of playback quality or mechanical parameters.

These parameters are:

- Rf signal
- Dropout counts
- Tape tension
- Drum speed

Further developments

For future work, the integrated quality reporting tape player developed within the previous PRESTO project may be used to greatly improve the quality reporting [16].

Improving the optical system would also enhance the detection of deposits.

A.3 DETERIORATION MECHANISMS OF THE MAGNETIC LAYER

The identification of the deterioration mechanisms requires a good understanding of the deterioration symptoms, the tribological phenomenon of tape-transportation systems, the tape structure and chemicals formulation and finally, the archival storage conditions.

Almost all these issues have been at one time or another studied individually for manufacturing optimisation purposes or materials archival stability. This abundance of research work answered specific questions, not always relevant to archival magnetic tapes, but failed to unify the deterioration mechanisms and to clarify their links with playback problems as experienced by the users.

The following diagram shows synthetically the mechanisms of material deterioration that are responsible for the symptoms selected for this study.

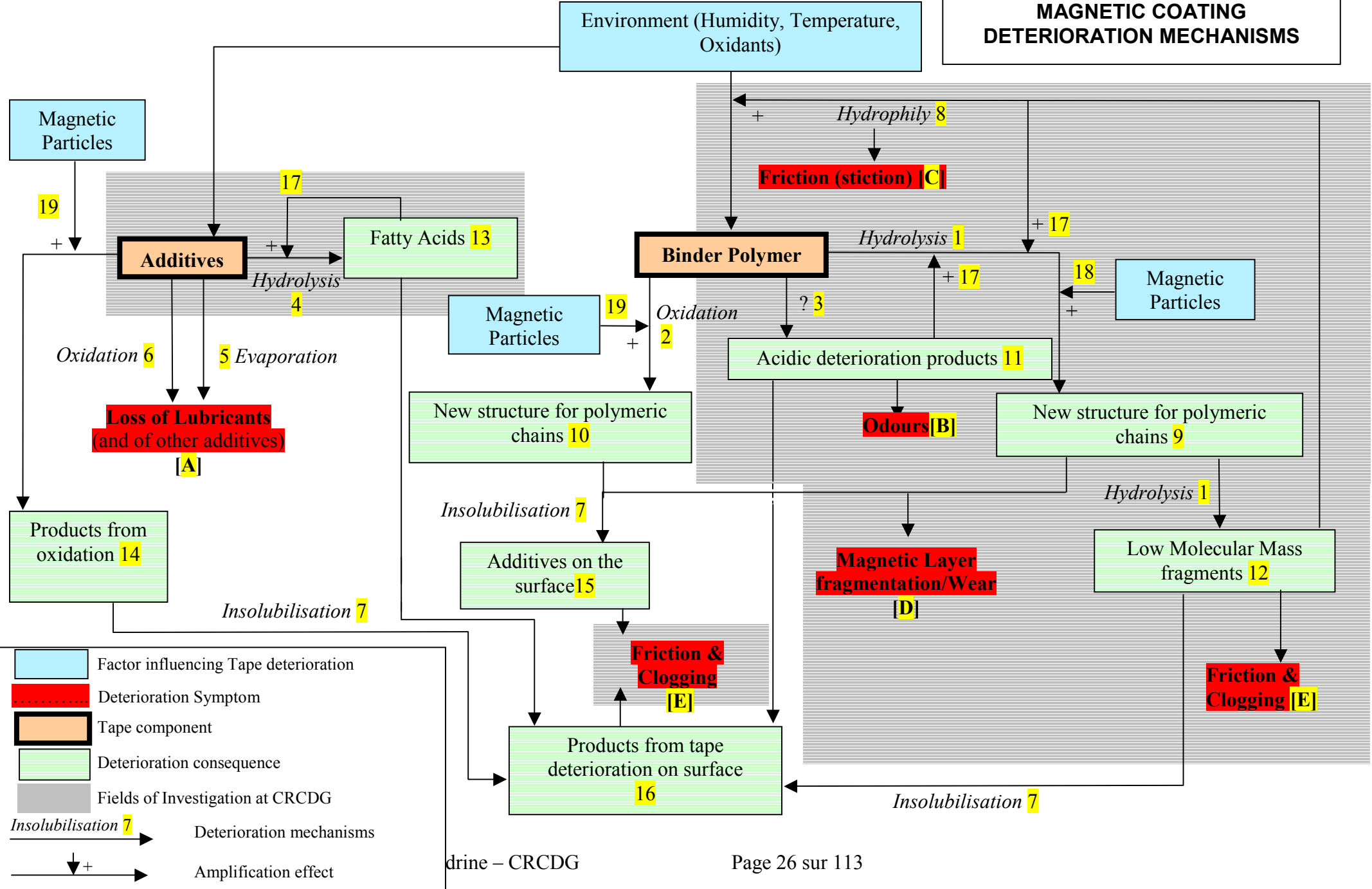
For some reasons mentioned previously in chapter A2.4, only mechanisms and symptoms related to the magnetic layer's composition, in particular, the polyester-urethane binder and the ester lubricants, will be taken in account in this diagram.

In addition to establishing a brief review of deterioration mechanisms, some relationships have been studied at the CRCDG and will hopefully be studied in much more details in further research projects.

The objectives of this diagram are to present all deterioration mechanisms and their relationships to each other, to storage conditions and to playback symptoms.

Interactions and constituents shown in this diagram refer to an attached table with related short explanations and literature sources.

**SYNTHETIC PRESENTATION OF
MAGNETIC COATING
DETERIORATION MECHANISMS**



 	Factor influencing Tape deterioration
 	Deterioration Symptom
 	Tape component
 	Deterioration consequence
 	Fields of Investigation at CRCDG
<i>Insolubilisation</i> 7 →	Deterioration mechanisms
↓ + →	Amplification effect

Link	Indications	Ref :
1	Polyurethane-Polyesters are magnetic coat polymers for most of tapes. Polyester chain breakdown occurs in reaction with humidity from the air (hydrolysis). Hydrolytic breakdown can lead to oligomers that were involved in PUR-ES manufacturing.	[17], [18], [19], [20], [21]
2	Oxidative deterioration of a polymer leads to chain-scission resulting in smaller molecules that will recombine very rapidly through radical mechanism to give a new macromolecular matrix. The newly formed polymer has different mechanical and chemical properties than the parent polymer.	[22], [23], [24], [61]
3	Ageing generates acidic products as it was demonstrated at the CRCDG. Deterioration mechanism is not totally known even if it may be an action of hydrolysis and/or oxidation.	Appendix 8 Appendix 18
4	Fatty ester Lubricants are susceptible to breakdown by hydrolysis. Products resulting from hydrolysis are fatty acids. Their lubricating property is degraded.	[25], [26]
5	Additives are mostly small organic molecules and are prone to evaporation. Exposed to abnormal high temperatures, they are lost quickly. At room temperature, evaporation may proceed slower by transfer to the ambient air.	[27], [28]
6	The deterioration of additives and lubricants by an oxidative environment (oxygen, oxidative pollution) is probably a mechanism of additive consumption and of deterioration of the initial formulation.	[28], [25]
7	Producing a synthetic polymer and adding on purpose selected additives is a complex science based on solubility properties. The homogeneity and physical stability of the end product is dependant on a complex balance. Any modification of the physical or chemical properties of the chemicals or sometimes only variations in environmental conditions may lead to loss of stability of the overall formulation. This causes fractionation and exudation at the polymer surface of some chemicals.	[29], [3], [30]
8	Acid functionalization of the polymer surface upon hydrolysis of the PUR-ES and its consequences 9, 12 leads to surface polarity and acid/base interaction increase. In addition to increase bonding to any metal oxide surfaces (particles, player components), this means higher affinity with water molecules (hygroscopicity). As a consequence, the uptake of water within the magnetic coating is facilitated and hydrolysis is accelerated.	[30], [31], [32]
9	The reduction in molecular weight of the macromolecules composing the polymer occurs upon hydrolytic breakdown of the ester chemical bonds of the PUR-ES. This breakdown leads to a loss of cohesion of the polymer and if hydrolysis proceeds longer 12 occurs.	[17], [33], [34], [35], [36]
10	Oxidative deterioration of the polymer leads to a modification of the mechanical properties of the resulting polymer (in general more brittle) and to changes in molecular weight of the macromolecules.	[37], [38]
11	If the volatility of the acidic products formed is sufficient they can be emitted from the tape as odorous compounds. In a hermetically sealed environment, acidic products have a catalytic effect on hydrolysis.	Appendix 8, [39]
12	In some cases, the hydrolysis reaches an end point corresponding to the production of end-fragments. These fragments are not affected by further breakdown. These small compounds have a high mobility and a high tackiness compared to parent macromolecules and can migrate to the surface onto the player heads. These fragments are easily extractable by organic solvents and have acids or alcohol organic functions.	Appendix 18 Appendix 7
13	Products formed upon hydrolysis of lubricant fatty esters are the related fatty acids. They do not have the same lubricating properties and, due to their acidity may have catalytic effect on their production.	[28], [27]
14	Products formed upon oxidation of the additives do not have in most cases, the properties of the parent products. However, some antioxidants maintain their properties for multiple oxidations.	[28], [40]
15 & 16	Chemical compounds exuded from the magnetic layer are present on the surface and are submitted to environmental conditions. If chemically pure, these compounds can form crystals. In most cases the uptake of water or the complex mixture of products leave these products on the surface in a thin film viscous and sticky film.	[13], [41], [42]

17	Acidic compounds have an important polarity and viscosity, this leads to an increase of the water uptake. As consequence, they have a catalytic effect on the hydrolysis of the magnetic layer binder.	[30], [43], [44], [45]
18	Some studies show a catalytic effect of the chromium dioxide particles on the hydrolysis of the PUR-ES.	[20], Appendix 7
19	Most of the oxidative chemical reaction pathways involve highly reactive radical compounds. The formation of these radicals are enhanced by the presence of metal oxide particles.	[22], [25], [27], [46]
[A]	The loss of the lubricants by various mechanisms is sometimes referred as LOL syndrome (LOSS OF LUBRICANT). Tribological (and friction) behaviour of the tape on the tape path is greatly affected by this loss.	/
[B]	Odours are often associated to degrading magnetic tapes. For polyester based tapes, the symptom is different than for acetate tape which develops the “vinegar syndrome” characterized by a strong vinegar smell emitted upon deterioration of the cellulose acetate base film. Some PUR-ES composing the magnetic layer emits odorous chemicals identified at the CRCDCG as organic acids and lactones. (Appendix 8).	Appendix 8
[C]	Surface uptake of water may lead to the formation of thin film of water on the surface when tape is played. High stiction may occur between tape surface and tape-path components altering the tape transport mechanism. This symptom is part of the Sticky Shed Syndrome (SSS)	[47], [48],[49]
[D]	Embrittlement or breakdown of the magnetic coating by 10 or 9 lead to tribological changes. Wear is increased and may lead in the most severe cases to fragmentation of the magnetic layer. Wear also leads to smoothing the tapes and thus increasing area of contact and friction. Fragmentation of the magnetic layer can result from handling (for most severe cases) or playback mechanical stress. The debris can be either loose (powdery) or adherent (sticky). This symptom is part of the Sticky Shed Syndrome (SSS)	[50], [51]
[E]	Clogging of the reading heads leads to the loss of the access to the document. This clogging may also occur on the components of the tape path. Important cleaning of the tape player is sometimes required to recover all the player functions. Modification of the condition of contact lead to increased friction. This symptom is part of the Sticky Shed Syndrome (SSS)	[13], [47], [52]

A.4 DETERIORATION MARKERS

A “Deterioration Marker” is a given chemical compound or a physical/chemical property of which the quantification or measurement allows to assess the level of a process regarded as a deterioration.

The assessment method can be based on a detection system with a binary answer according to a given threshold value, or a quantitative linear system over a range of measured values.

A.4.1 REQUIREMENTS FOR A SUITABLE DETERIORATION MARKER

Here are summarised the requirements for considering a given marker as a suitable parameter for condition assessment. All the following properties should be first evaluated from a theoretical side, then from an experimental side (artificial ageing) and at last, on naturally aged objects.

Monotonic evolution

Deterioration assessment is based on the quantification of a marker. Thus a relation of proportionality has to be established between both. This leads to the need of monotonic evolution.

Consistent with deterioration symptoms time-scale

In order to be valid on larger set of media and various deterioration states and ages, the evolution time-scale of the marker has to be strongly related to the time-scale of the experienced deterioration symptoms.

Related only to deterioration

Unless the past storage conditions or the history of tape handling is known with precision, the deterioration mechanism (monitored through the marker measurement) has to be dependent solely on natural ageing parameters.

For instance if friction is used as a marker, excessive wear due to numerous playbacks should not interfere in the marker evolution nor analysis unless to be able to correct the assessment.

Initial / Final state known or threshold levels known

Knowing the initial level of a given marker enables the “dating” function of the assessment. However, knowing significant thresholds may be enough to predict the consequences of the deterioration. Especially the last threshold of interest the End-of-Life level.

A.4.2 POTENTIAL TAPE DETERIORATION MARKERS

On the basis of each deterioration mechanism identified in the diagram presented in the section A3, some deterioration markers can be defined. These markers are presented in the following table:

	Marker	Diagram relations (chapter A3.)	Measurement (Chapter A5.X)	
CHEMICAL Marker	Polymeric binder molecules weight	[9-10] ; [12]	A5.1.1	Any deterioration of the polymeric chains leads to a modification of the molecular weight distribution towards higher or lower length.
	Solvent extractables	[9-12]-[5]	A5.1.2	Slight modification of the chemical properties of the chemicals composing the magnetic layer (including the polymeric chains) leads to modification of the solubility parameter (loss or gain) in given solvent. This affects the total amount of solvent-extractables. Loss of products by evaporation has the same consequences.
	Tape Surface acidity	[9] ; [11-13] ;	A5.1.4	Functionalisation of the tape surface by environmental deteriorations (and in particular alcohol and acid functionalisation by hydrolysis) leads to increased surface acidity. If the newly formed acids are extractable in water, a pH drop occurs.
	Hydrophilicity / surface tension	[9-10] ; [16] ; [15]	A5.1.3 & A5.1.4	Any modification of the surface chemistry (chemical deterioration) or of surface composition (chemicals exudation) leads to modification of the free surface energy which is measured as the « surface tension ». This physical quantity, when considering surface-water interactions, is strongly related to the hydrophily.
	Loss of Tape additives	[4-7]	A5.1.5	These markers are based on the loss of products present in the initial tape formulation.
	Products of hydrolysis / oxidation / chemical deterioration	[11-14]	A5.1.6	These markers are based on the production of products absent from the initial tape chemical formulation.
MECHANICAL Marker	Loss of cohesion / embrittlement	[9-10]	A5.2.1	Physical or chemical deteriorations that affect the heart of materials lead to progressive modification of the macroscopic mechanical properties.
	1. Friction and adhesivity	Direct measurement of the symptoms [A], [C], [D], [E]	A5.2.2	Physical or chemical deteriorations (or modification by chemicals exudation) that affect the surface of the magnetic layer lead to progressive modification of the tribological properties of the surface.

A.5 MEASURING METHODS FOR POTENTIAL MARKERS

Analytical and measurement methods have been developed for every marker identified and presented in chapter A4.2. The technologies we used are part of R&D laboratory protocols.

The following sections present the evaluation of these potential markers on the basis of their specific measurement method. Each time, we propose a way to transfer from laboratory method to a simple “on-site” measurement and recommendations for further developments.

We intentionally excluded experimental considerations in the following presentations to not complicate the understanding of the potential of each marker and measurement method for pragmatic condition assessment method.

More details of the laboratory achievements are given in the appendix section.

All analytical developments, artificial ageing tests and analysis have been carried out on a collection of U-Matic tapes provided by the INA audiovisual archive department. The sampling procedure (Appendix 19) included all major manufacturers and manufacturing years from 1975 to 1995. In addition some audio, 1” video and 2” video tapes have been tested and compared.

A.5.1 CHEMICAL MARKERS

A5.1.1 Polymeric Binder Molecule Size

<p>Introduction</p> <p>Methods for molecular-weight measurement usually require the polymer to be in solution and the use of laboratory instrumental methods (viscosimetry, Matrix-assisted desorption MALDI, Light scattering MALS, Size Exclusion Chromatography SEC). Attenuated Total Reflection Infrared spectroscopy, that is easy and affordable, has sometimes been applied to magnetic coating to quantify the number of organic functions specific of the polymer chain extremities. This quantification allows a kind of deterioration assessment [37, 45, 53, 61]. Unfortunately this method is not precise and reliable enough. More over, it requires a good knowledge of the complex organic formulation that is different from tape to tape.</p>
<p>Analytical method developed at CRCDG (Appendix 7 and 12)</p> <p>On the basis of previous works [20, 52], an analytical protocol by Size Exclusion Chromatography (SEC) was developed by CRCDG for magnetic coating in order to identify the molecular weight distribution.</p>
<p>Main analytical results</p> <p>Tetrahydrofuran extractables were studied for most of the U-Matic samples. As a marker of polymer breakdown, molecular weight can be monitored by this method. Significant differences exist between tapes having different ageing times (lower weights for older tapes). Unfortunately, when comparing tapes from various manufacturers or with obvious distinct manufacturing processes, molecular weight distribution can not be solely related to hydrolytic degree of conversion. The initial weight distribution, at manufacturing date is dependant of numerous parameters and is cannot be retrieve today. However, whatever the causes are, the presence of low molecular weights or/and important amount of extractables still remains a good indicator of binder weakness. (due to ageing or due to poor manufacturing practices) Artificial ageing tests (heat/humidity) evaluated by SEC analysis, allow clear evaluation of the stability disregard the initial molecular weight distribution. The catalytic effect of chromium dioxide on binder breakdown has been confirmed.</p>
<p>On-site method transfer</p> <p>No « on-site » analytical method can be implemented using this analytical technique. But the SEC analysis is the major method for binder study in a R&D laboratory.</p>
<p>Further developments</p> <p>At the CRCDG, this technique is fully operational and systematic analysis of tapes can be undertaken. SEC analysis are mandatory to evaluate any other binder breakdown marker (intercalibration purpose) and to investigate the deterioration symptoms / materials decay relations.</p>

A5.1.2 Solvent Extractables

<p>Introduction</p> <p>Since the initial study on hydrolytic magnetic tape deterioration by Cuddihy in 1980 [17], solvent extractable weighting was considered as a method for determining the tape binder breakdown and was widely used [54, 61]. This technique is affordable and does not require a complex equipment. Unfortunately, this method is not accurate and is highly dependent on the initial magnetic tape formulation. In addition, it is unlikely to form significant solvent extractable products until the binder is near exhaustive hydrolysis stage [55]. Despite these experimental difficulties, the extractables weighting remains an interesting marker but only on the condition that concurrent characterization and precise quantification can be made. As a consequence, extractables measurements have to be made in a well equipped laboratory and with alternative to classic gravimetric methods.</p>
<p>Analytical method developed at CRCDG. (Appendix 7 and 12)</p> <p>The Size Exclusion Chromatography (SEC) (A5.1.1) allows an accurate quantification of extractables and their characterization as a function of their molecular weights. This latter is sufficient to measure the part of extractables produced upon deterioration and extractables initially present in the tape formulation (lubricants...).</p>
<p>Main results</p> <p>Results show that according to the brand or manufacturing process:</p> <ol style="list-style-type: none"> 1) Tapes have different amounts of extractables 2) Responses of this marker vary differently when tape samples are submitted to artificial ageing (heat/humidity) ranging from sharp increase of the extractable amount with decreased molecular weight to amount conservation with molecular weight decrease. For a minority of tapes, artificial ageing tests have no consequence on the amount of either extractables or the molecular weight distribution.
<p>On-site method transfer</p> <p>“On-site” analytical method cannot be implemented using this analytical technique and the simple gravimetric method alone does not allow reliable condition assessment. Only a better understanding of the hydrolytic effects on each major magnetic tape formulations and development concurrent micro-sampling and micro-weighting method development would permit to transfer this marker « on-site » for condition assessment.</p>
<p>Further developments</p> <p>As it was told previously, results confirm that “solvent extractables” quantification remains a relevant marker. Suitable measurement method could be developed with more advanced study of PUR-ES. Solutions could be found in improving methods for weighting more accurately the extractables, e.g. with quartz crystal microbalance which have a sensitivity down to the nanograms.</p>

A5.1.3 Hydrophilicity / Surface Tension

<p>Introduction</p> <p>Tribological Interactions as well as chemical affinity of the tape surface with the environment are mainly defined by surface properties such as surface tension [56]. The “hydrogen-bonds” physical term entering in the definition of the surface tension is closely related to the affinity of the surface with water (hydrophilicity).</p> <p>These quantities are often mentioned in the patents related to tape manufacturing and surface tension is strongly related to the work of adhesion of the friction.</p>
<p>Analytical method developed at CRCDG (Appendix 9)</p> <p>If a drop of liquid is placed on a solid surface, the liquid and solid surfaces will come together at a characteristic angle called contact angle or wetting angle. This angle can be related to the surface energy by mathematical representation. The CRCDG developed a device to accurately measure contact angle for different liquids (test-liquids) and different surfaces. If water is used, hydrophilicity of tape surface can be assess. The experimental laboratory set-up allows depositing 5µL drops with an angle reading error of less than 0.5%.</p>
<p>Main results</p> <p>Up to now only instrumental development has been carried out. Preliminary results of hydrophilicity measurements tend to show a great variability from tape to tape and a significant modification of this parameter with artificial ageing.</p>
<p>On-site method transfer</p> <p>The contact angle measurement method is widely used by industrials. It exists numerous affordable fields apparatus allowing precise measurements.</p> <p>The term « total wetting » implies a value of contact angle of 0° and that the liquid spreads over the surface. This case occurs when the liquid internal cohesion is weaker than the adhesion work of the surface.</p> <p>Sets of calibrated and especially formulated inks allow to quickly and easily measure the surface tension by “total wetting” experiments. (Appendix 17)</p>
<p>Further developments</p> <p>In order to complete the development of a test based on surface tension measurement, efforts have to be made on the fundamental understanding of tape surface chemistry and on the relationships between surface tension and friction. Among all the markers presented here, this marker is the most promising because it can be easily implemented and the resulting “device” easy to use.</p>

A5.1.4 Tape Surface Acidity

<p>Introduction</p> <p>Acidity of the magnetic tapes is the second parameter after solvent extractable weighting that is widely used in order to assess the condition of archival magnetic tapes [54]. The hydrolytic deterioration pathway results in creating acidic organic functions on the PUR-ES chains, thus monitoring the acidity is a valuable deterioration marker. Unfortunately, due to measurement difficulties it does not exist so far, accurate surface acidity assessment methods.</p>
<p>Analytical method developed at CRCDG</p> <p>Acidity is usually determined by measuring the pH of water extract solutions with a glass electrode. Unfortunately, only “free” acidity can be measured and not the organic acids of polymeric chains. Surface measurements thanks to especially designed glass electrode are similar to the extraction measurement and does not represent a real alternative. In addition, production by hydrolysis of water extractable acids implies a high level of deterioration of the polymer for beyond the “archival” definition of a degraded tape. Measurement of the “acid number” [57, [58] is more suitable for acidity assessment but this technique usually requires to dissolve the polymer in an organic solvent. This cannot be achieved for most of the resin-like polymers as PUR-ES. The CRCDG developed an original non-invasive approach of indirect titration of surface acidity of magnetic layer surface. This method is based on contact angle measurement of droplets of water at different pH [59]. A sudden drop of angle value can be observed at a pH where the acid content of the droplet is equal to the quantity of acidic functions on the polymer surface. The experimental set-up dedicated to that measurement is the same as the one for tension surface measurement.</p>
<p>Main results (Appendix 9)</p> <p>Preliminary tests have been carried out to develop a reliable experimental protocol and to evaluate the method feasibility. The equivalent contact angle drop as function of the pH, if proven theoretically and experimentally on simple laboratory samples [59], had to be demonstrated on real samples. This has been observed for magnetic tapes.</p>
<p>On-site method transfer</p> <p>Using this method based on contact angles measurement, the conclusions are the same as for the surface tension measurement (A5.1.3). These methods represent the best opportunities to develop practical testing methods for condition assessment of magnetic tapes.</p>
<p>Further developments</p> <p>More research is required to practically implement a test for condition assessment. The key issues are: 1) understanding the relationships between surface acidity and equivalent pH measurement by contact angle. (intercalibration required with other measurement techniques) 2) understanding the relationship between surface acidity and tribological behaviour (friction) 3) calibration of the test response as function of the playback symptoms and tape manufacturing.</p>

A5.1.5 Chemical Analysis I: Loss of Additives & Manufacturing By-products

<p>Introduction</p> <p>This section focuses on the quantification over time of all the products initially present in the magnetic coating except for the polymer matrix and the particles. These chemicals may or may not be an additive added to the tape formulation for a specific purpose. We refer to additives (lubricants, stabilizers, manufacturing agents etc) or production by-products (unreacted prepolymers, solvents, or chemical reaction byproducts)</p> <p>All these compounds are lost by evaporation, migration and deposition on tape path, deterioration and are subject to conversion by deterioration or consumption.</p>
<p>Analytical method developed at CRCDG</p> <p>Quantification of the products present in the magnetic layer formulation is based on complex techniques and laboratory protocols. The techniques used at the CRCDG are mainly gas chromatography/mass spectrometry and infrared spectroscopy.</p> <p>These techniques allow characterization and quantification of products with various chemical natures.</p>
<p>Main results</p> <p>The following products have been found on tapes with different levels and are suspected to be additives or products by-products: lubricants (palmitic acid butyl ester, myristic acid butyl ester, capric acid butyl ester, and paraffinic oils), antioxidants (phenolic), polyester manufacturing by-products (cyclic oligoesters, unreacted diisocyanate), solvents (cyclohexanone, toluene, and styrene). This knowledge has been added to the “demonstration knowledge database” presented in the part B on this report.</p>
<p>On-site method transfer</p> <p>The powerful chromatographic methods, cannot be transferred on site for easy and affordable testing method. Only field micro-sampling such as “solid phase microextraction” (SPME) (Appendix 8) and further off-site analysis by skilled analytical laboratory would allow such development. Conversely, Infrared spectrometry is affordable and easy to use when a reflective mode is used, but does not allow reliable quantification and only poor characterization of complex mixtures.</p>
<p>Further developments</p> <p>Advanced knowledge of the magnetic tape chemical formulation is mandatory for deterioration understanding, marker identification and for tape technology identification among manufacturers and manufacturing practices.</p> <p>Markers based on consumption of initially present products will unlikely give birth to testing method because initial levels are not known and are highly variable from tape to tape even among tapes produced by the same manufacturers.</p>

A5.1.6. Chemical Analysis II: Deterioration products

Introduction
Deterioration products represent the traditional and the best candidates for deterioration markers. Indeed, at the time of tape manufacturing, these chemicals are not present or are present at lower levels. Their quantity increases with environmental factors at a given rate.
Analytical method developed at CRCDG
Techniques used are the same than techniques presented in the section A5.1.5. In the case of deterioration products and compared to analysis of initially present products, the detection threshold has to be far lower.
Main results (Appendix 8 and 18)
Some products have been identified as deterioration products: <ul style="list-style-type: none"> - carboxylic acids (odorous compounds). - lactones (odour causing compounds) - polyurethane basic building block (adipic acid)
On-site method transfer
<p>The powerful chromatographic methods cannot be transferred on site for an easy and affordable testing method. But on site micro-sampling such as “solid phase microextraction” (SPME) (Appendix 8) followed by a further off-site analysis by skilled analytical laboratory would allow such development. Infrared spectroscopy is affordable and easy to use when a reflective mode is used, but does not allow reliable and sensitive quantification and only poor characterization of complex mixtures.</p> <p>Chromatography is nevertheless mandatory to identify relevant marker and to calibrate other testing methods designed for a given compounds.</p> <p>Depending on the chemical nature of the marker, sensor based technologies can be developed for detection rather than quantification:</p> <ul style="list-style-type: none"> - electronic nose for VOC (semiconductors or quartz microbalance) - colorimetric methods (reacting strip, liquids...) - electrochemical micro sensors (coulometric microelectrodes...)
Further developments
Good understanding of the production of the VOC is required to undertake calibration to alteration symptoms, technological development and intercalibration with laboratory instruments. Chromatographic studies and artificial ageing testing have to be carried on.

A.5.2 MECHANICAL MARKERS

A5.2.1 Loss of Cohesion/ Embrittlement

<p>Introduction</p> <p>Internal cohesion of the binder has directly been studied by tape durability testing [60, 61] or nano-indentation study [62] (hardness mano-testing).</p> <p>Some semi-quantitative tests based on simple tests (“time to rub” and “rolls of binders”) sometimes have been used to characterize magnetic coating cohesion. [17, 45]</p>
<p>Analytical method developed at CRCDG</p> <p>At the CRCDG, only wear testing with the friction tester already presented in this report (A2.5.1) allows the estimation of the tape surface durability and the related loss of cohesion.</p>
<p>Main results</p> <p>So far, only preliminary tests have been carried out along experimental development and only reproductions of already known results have been done (smoothing of the tape by wear).</p>
<p>On-site method transfer</p> <p>The implementation of a micro-abrasivity tester is possible through miniaturization of existing laboratory apparatus. If the device is assembled on a cleaning machine could be used as a “reject test” before playback.</p>
<p>Further developments</p> <p>Loss of cohesion has to be related to the play back symptoms and to the chemical changes occurring upon ageing. All the laboratory work presented in this report will help to gain good understanding of this mechanical parameter and would allow to calibrate and develop a testing method.</p>

A5.2.2 Friction and Adhesivity

<p>Introduction</p> <p>Friction and its physical and chemical components were already been introduced in section A2.5.1.</p>
<p>Analytical method developed at CRCDG</p> <p>The friction tester developed by the CRCDG is presented in section A2.5.1. No method of adhesivity measurement has been developed due to important technical difficulties to measure very weak adhesion forces.</p>
<p>Main results</p> <p>Preliminary results show significant differences in the frictional behaviour of magnetic tapes according to brands. Artificial ageing have been conducted but heat alter the physical properties of the base film and it is impossible so far to investigate the frictional changes of the magnetic coating upon artificial ageing.</p> <p>Even if tendencies can be observed, the lack of repeatability of the friction tester, does not allow to quantify precisely friction. The friction tester is still under development at the CRCDG.</p>
<p>On-site method transfer</p> <p>The implementation of a micro-friction tester is possible through miniaturization of existing laboratory apparatus. Such test if assembled on a cleaning machine or playback device could be used as a playback deterioration symptoms assessment method. A micro-friction tester already exists and is used for research purpose on linear data tape players [13]; this device could be easily adapted to any tape player.</p>
<p>Further developments</p> <p>The laboratory friction tester has to be improved.</p> <p>Friction has to be regarded as a key parameter of the symptoms measurement and is probably the key issue for understanding the relation between physio-chemical properties and playback deterioration symptoms of magnetic tapes.</p>

A.6 SUMMARY

Due to various chemical formulations and deterioration mechanisms, the best markers for condition assessment are related to tape tribological behaviour. These markers are almost exclusively independent from complex chemical considerations. Unfortunately, if they can be used for condition assessment, they do not allow risk evaluation on a long term basis. These markers are:

- surface properties (surface tension, surface acidity)
- mechanical properties (friction, durability)

The parameters that may be useful as conventional “chemical deterioration marker” (if advanced fundamental research efforts are made) are:

- chemical analysis of deterioration products (volatile compounds)
- solvent extractables

Last, the “polymer deterioration size distribution” cannot probably result in a suitable “deterioration marker” due to equipment and skills requirements. Nevertheless, this measurement is mandatory as a laboratory investigation method to support any development of any other “chemical deterioration marker” of polymer condition assessment.

A.7 Part A CONCLUSION

As a general rule, the need for deterioration markers is a common request of many professionals in charge of the preservation of cultural heritage. Because of the lack of knowledge about the composition of ancient, rare or even mass produced objects as tapes, the first task in conservation science is to investigate the object materials itself. Once the constituents are identified, deteriorations of museum objects (esthetical changes, mechanical changes etc) can be investigated and deterioration markers can be proposed. This general approach however, is not as simple for magnetic tapes. This kind of audiovisual carrier requires specific equipment for access. Deterioration assessment depends not only on the intrinsic material stability but also on the player specifications and tolerance to media deterioration as well as tape handling. In addition, the complexity of mass-manufactured tapes with numerous formulations and manufacturing practices result in a variety of deterioration mechanisms.

As a consequence, finding a unique deterioration marker is highly challenging and would probably much more consequent research efforts.

The study carried out during the first 18 months of the project considered all potential deterioration and some analytical tools have been or are still under development. These tools allow the CRDCG to be at the state-of-the-art for further fundamental deterioration studies on magnetic tape materials and further investigations of the relationships between material decay, ageing conditions and symptoms experienced while playing archival magnetic tapes.

Because deterioration processes are highly dependant on the manufacturing practices, a better knowledge of the magnetic tape collections has to be gained. Part B of this report proposes a way to collect, organize, retrieve and use all this information for collection assessment and further magnetic tape ageing studies.

B KNOWLEDGE DATABASE

B.1 KNOWLEDGE DATABASE FOR CONDITION ASSESSMENT: PRINCIPLES

Concepts and considerations that are necessary to build a materials oriented database and related application methods for magnetic tape condition assessment are defined below.

B.1.1 TECHNOLOGICAL FAMILIES AND DETERIORATION MECHANISMS

Magnetic tape technological developments have been driven by a fast growing market in a highly competitive environment. Major improvements have been in terms of mechanical performance and capacity.

Within audiovisual collections, this leads to a “coexistence” of magnetic carriers with a large diversity in materials and chemical formulations. The consequence is large diversity in deterioration behaviour when exposed to the environmental conditions of long-term storage.

The initial phase of all research projects is necessary to gain better knowledge of the materials diversity and to categorize them into more general “technological families” (i.e. with similar chemical composition and structure)

In order to understand the impact of each technological option taken during tape manufacturing on the tape stability, the relation between “technological families” and the nature of the deterioration mechanisms and their severity have to be investigated.

B.1.2 INTRODUCTION TO KNOWLEDGE DATABASE

Contained within the “knowledge database on magnetic tapes” is all the information provided by manufacturers, materials and chemistry laboratory investigations, community feedback and preservation project feedbacks. The database is organized on the fundamental definition of “technological families”.

All these organised information may be useful at a purely technical level (magnetic tape specifications, specificities, known issues etc) but some information can be turned into a real “statistical condition assessment method” for collection of magnetic tapes as shown in this part B. of the report.

B.1.3 CORE CONCEPTS

Collection of Model Samples and Laboratory Investigations

Manufacturers are reluctant to share or simply do not have the information required, so laboratory investigations and reverse engineering approaches are mandatory to feed the fields oriented to the materials and materials stability in the knowledge database.

Unfortunately, laboratory work often implies destructive testing that is rarely possible on a large number of archival samples. The concept of “technological families” allows us to focus laboratory work on some samples considered representative (model) for important batch series affected by similar ageing mechanisms, and therefore to reduce the number of required samples and experiments.

All samples available for destructive testing and representative of “technological families” are gathered in a “collection of model samples”.

In addition to serve as raw materials for laboratory investigations, condition assessment of these samples defines the reference level for the whole technological family. Correction for storage condition and storage period can be made with the help of fundamental stability studies.

“Playability” statistics

We define the probability to encounter playback problems as “playability statistics” for each of the technological families. These statistics represent directly usable information when using the database for condition assessment of a collection. These statistics can be provided by present and future preservation projects dealing with large number of tapes. In order to feed database, reference to technological families has to be made.

User’s feedback

The archive community is an important knowledge resource for the actual deterioration state of magnetic tapes.

The feedback is composed of diverse experiences. Unfortunately this information is often not comprehensive enough, especially in the tape identification method. And it can be quite difficult to re-use this information.

The development of normalized tape identification processes would help to share feedback on tape deteriorations through the knowledge database.

B.1.4 KNOWLEDGE DATABASE AND MANAGEMENT SYSTEM

The database is only an organized collection of data, so a management system has to be developed in parallel, in order to query and retrieve any information. The applications of the management system can be either just a technical response or a statistical condition assessment based on a calculation method linked to a decision making process for individual tape, tape batches or collections.

A calculation method based on a point-based evaluation of the database entries as a function of conservation issues is proposed in the section B10 of this report.

This condition assessment is named “statistical” because it is partly based on “playability statistics” acquired from multiple samples and partly based on the analysis of a small size collection made of “model” samples resulting from a larger statistical sampling of real collections.

A database involving permanent updates and a management system is particularly well suited for on-line applications.

B.2 EXISTING KNOWLEDGE DATABASES

Some categorization and identification protocols for cultural heritage materials already exist. However for A/V carriers, only little information is available and often results from personal initiatives that seldom mention considerations on material preservation. Examples of databases are presented in following table and in appendix 1 to 5.

Appendix	Media	Brand	Information
Appendix 1	Motion picture film	KODAK, DUPONT, FUJI	Reading guide for serial number code for manufacturing site and date.
Appendix 2	Magnetic Audio tapes	3M	Product list and some specifications
Appendix 3	Magnetic tapes	AMPEX	Product list and comments
Appendix 4	Magnetic tapes	Various	Tape categorization by manufacturers / manufacturing period and quick quality assessment.
Appendix 5	Magnetic Audio Tape	Various	Product list and some specifications (pictures included)

B.3 APPLICATIONS

B.3.1 PRESERVATION PROJECTS PLANNING

The collection condition assessment is a mandatory step in planning a preservation project. In some cases, collections which are made of a large number of problematic tapes may lead to an under evaluation of the costs, time and skills required to conduct a proper preservation project.

Evaluation of a collection by comparing it to knowledge database entries is particularly well suited for this specific step. Indeed, often at this early stage, no financial resources are available and it is difficult to undertake instrumental testing of tapes. Comparisons of tapes from the collection to a knowledge database, through a technological family identification, make that condition assessment cost-effective and easier.

B.3.2 MANAGEMENT OF PRESERVATION PROJECTS

Optimisation of the object workflow management implies the ability to prioritise media as function of their condition assessment and to address problems as soon as possible for a specific restoration method. A deficient prioritisation may lead to frequent breaks in the preservation process and frequent human intervention..

The knowledge database approach associates for each technological family a probability to encounter preservation problems and allows prioritisation of the media submitted to a preservation workflow.

B.3.3 TECHNICAL KNOWLEDGE OF THE HOLDINGS

Independently of preservation projects, it is of great concern to the archive community to characterize the materials used in their holdings.

This need for general technical information can be measured by frequent queries from archivists on the *Association for Recorded Sound Collections*¹ (ARSC) or the *Association of Moving Image Archivists*² (AMIA) mailing lists.

These queries are often motivated by conservation issues for tapes with recurrent problems. Some people are also looking for help from the community to identify the origin and the specifications of their carriers.

B.3.4 SUPPORT TO CONSERVATION SCIENTISTS AND MANUFACTURERS

A comprehensive database including the identification of “technological families”, materials characterization and playability statistics represent an important source of information for conservation scientists. For instance, further research could be undertaken on the existing link between fields of the database and the deterioration patterns of each technological family as identified in the database.

This could result in storage and manufacturing recommendations, if specific technological families or tape constituents appear to have poor archival performance.

¹ <http://palimpsest.stanford.edu/byform/mailling-lists/arsclist/>

² <http://lsv.uky.edu/archives/amia-l.html>

B.4 CONSISTENCY WITH PRESTOSPACE WORKING GROUPS

B.4.1 PRESERVATION WORK AREA & WORK PACKAGE 6

The building of a knowledge database and of a related management system fulfil the objectives of the PRE WPO6 work package even if the solution proposed is not based on a chemical marker assessment.

B.4.2 OTHER PRESTOSPACE GROUPS

Strong relationships exist between the proposed approach and specific needs of some tasks of PRE WA WP5 and WA SAM.

B.5 OTHER PROJECT: THE SOUND DIRECTIONS PROJECT

The Sound Directions project [63] funded by the ENH (USA) intends to identify and test best-practices for A/V documents preservation and storage, to initiate preservation programs in universities involved in the project and to preserve critically endangered, highly valuable recording.

Within the framework of the project, the Archive of Traditional Music (Indiana University) is developing a similar approach for collection assessment to what is described in part B of this report. A brief description of the collection assessment tool called: "Field Audio Collection Evaluation Tool" (FACET) follows and this should be released to the archive community in spring 2006:

"The Field Audio Collection Evaluation Tool (FACET) is a points-based tool for ranking field collections for the level of deterioration they exhibit and the amount of risk they carry. This tool helps collection managers construct a prioritized list of collections by the level of risk they represent, enabling informed selection for preservation. [...]. FACET was developed by Mike Casey at the Archives of Traditional Music, Indiana University."

Very little information has been disclosed on FACET up to now, but it appears to have strong similarities with the objectives of the FACET tool and some workgroups of the PRESTOSPACE project including the WP06 Workgroup.

Similarities with collection assessment methods proposed in this part B are:

- condition collection assessment
- point-based approach
- carriers quick identification procedure
- user feedback database

The FACET includes different physical formats but does not have materials chemistry and analytical chemistry inputs.

The existence of such a project reflects the great interest of the community for comprehensive tools assisting collection condition assessment.

B.6 KNOWLEDGE DATABASE STRUCTURE

B.6.1 PRELIMINARY PRESENTATION: THE COLLECTION OF MODEL SAMPLES

“Model samples” as defined in B1.3, allow laboratory investigations to provide the database with materials characterisation and stability information for each technological family. These tapes have to be selected following a well defined sampling procedure

The sampling procedure has to:

- take into account all manufacturers and manufacturing periods.
- include different collections as sample sources.
- two or more samples per technological family.

The size of the “collection of model samples” has to be as large as possible to represent all tapes. However, limitation due to time and cost consuming laboratory work, have to be considered.

If stored in controlled environmental conditions, the periodic assessment of the collected samples would allow keeping the database entries up-to-date. Additional information about modern magnetic tape would also allow the anticipation of future deterioration problems.

Holding a samples collection in various storage conditions, is a commonly used quality control policy of industrial groups.

B.6.2 DATABASE FIELDS RELATED TO TAPE IDENTIFICATION

We define “tape identification” as the identification of the manufacturing operations as precisely as possible only by simple examinations of the tape or of the cassette. Sets of specific identifiers have to be defined.

The link between these identifiers and the technological families enables the “on-shelf” identification of the technological families by simple examination.

The fields of the knowledge database related to the identification of the tape are presented here. The examples given in each table are independent from each other and do not refer to a unique tape format.

DATABASE FIELD: Tape Identification

It is the unique set of identifiers defining unique manufacturing operations. It can be (by order of relevance to manufacturing):

- 1- Unique for each tape (serial number code for each tape)
- 2- Unique for a batch of tapes (serial number code for the batch)
- 3- Unique for all batches from the same commercial product (name, number of the product etc)

Entry Types	Serial number code on the backcoating of the tape Or <u>Serial number code on the cassette</u> / reel /cartridge Or Serial number on the tape/cassette/cartridge case Or Name, type number and manufacturing date
Acquisition method	Simple examination
Skills	Tape handling
Samples	Tapes not already included in the base
R&D efforts	/
Entry Example	N3196857012166

DATABASE FIELD: Tape structure


Tape structure is used as extra-identifier for tape identification. It requires more advanced tape handling.

This information may also be directly useful to define quickly play-back specifications.

Entry Types	<ol style="list-style-type: none"> 1. <u>Tape colour</u> 2. <u>width, thickness, length</u> 3. <u>backcoated or not</u> 4. <u>tape leader aspect</u>
Acquisition method	Simple examination, easy measurements
Skills	Tape handling
Samples	Tapes not already included in the base
R&D efforts	/
Entry Example	<ol style="list-style-type: none"> 1. Black 2. ¾ inch, 30µm , 60min, 3. Backcoated, Translucent leader

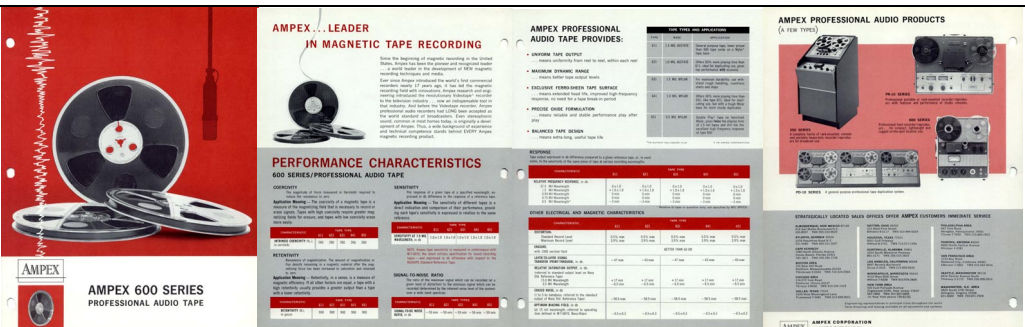
DATABASE FIELD: Tape packaging

This information allows quick on-shelf identification without tape handling. And may serve as extra identifiers for tape identification. Repackaging of the tape may lead to disagreement between database and observation.

Entry Types	<ol style="list-style-type: none"> Pictures of the tape packaging (cassette, reel, cartridge) Pictures of the tape case Any information printed by manufacturer
Acquisition method	Simple examination, pictures
Skills	Tape handling
Samples	Tapes not already included in the base
R&D efforts	/
Entry Example	

DATABASE FIELD: Technical Documentation

This field presents the bibliographic reference of the specsheets and all other documents issued by the manufacturers. If this exists, specific study or scientific publication can be added to that group of documents.

Entry Types	<ol style="list-style-type: none"> Commercial documents Spec sheets Scientific reports/ Studies
Acquisition method	Bibliographic review, call to the community.
Skills	Good knowledge of magnetic tape history
Samples	/
R&D efforts	/
Entry Example	

B.6.3 MATERIALS RELATED FIELDS OF THE DATABASE

These fields belong to the materials and chemistry oriented information gathered by laboratory analysis on the collection of model samples. The most part of the analytical or measurements protocols have been already developed and operational at the CRCDG. However some of them still require R&D. The examples given in each table are independent from each other and do not refer to a unique tape format.

DATABASE FIELD: Tape Chemical Composition

Chemical formulation for a given tape has a direct impact on its stability upon storage. The overall tape stability is dependent on every compound of the formulation. Their chemical behaviour over time when considered independently from each other is a valuable data for assessing the overall formulation's stability.

Some specific research can also be carried out to study the stability of some relevant combination of compounds.

In addition, knowing exactly the formulation of a given technological family can be used as "chemical fingerprint" for tape identification or "technological families" limits definition.

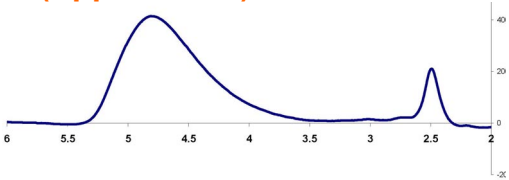
Entry Types	<ol style="list-style-type: none"> 1. Magnetic particles 2. Base Film 3. Additives including lubricants 4. Binder system 5. Backcoating formulation 6. Abrasives
Acquisition method	Laboratory analyses, bibliographic search
Skills	Analytical chemistry laboratory
Samples	Collection of Model samples
R&D Efforts	Some analytical protocols have still to be developed
Entry Example	<ol style="list-style-type: none"> 1. Chromium Dioxide (CrO₂) 2. PET 3. BHT, Palmitic acid butyl ester 4. PUR-ES 5. PUR-ES 6. Aluminium Oxide (Al₂O₃)

DATABASE FIELD: Current deterioration state

“Model samples” being archival tapes sampled through known collections, the conditions of these tapes could be regarded as the reference conditions that may be suspected for all tapes from the same technological family.

Some parameters such as polymeric breakdown, additives loss or conversion, deterioration products could be evaluated by laboratory means and added in the “current deterioration state” fields.

Precautions have to be taken due to differences in storage conditions, tape handling and wear (inducted by multiple playback) between the “model sample” and any tape from the technological family.

Entry Types	Results of analytical laboratory analyses
Feeding method	Laboratory analyses
Skills	Analytical chemistry laboratory
Samples	Collection of model sample
R&D Efforts	Some analytical protocols still have to be developed
Entry Example	<p>Binder state by SEC (Appendix 12)</p> 

DATABASE FIELD: Current tribological behaviour

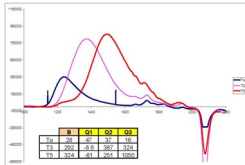
As “current deterioration state”, tribological measurements on the “collection of model samples” could be used for the “current tribological behaviour” assessment of all tapes from each technological family.

Entry Types	Tribological behaviour (friction, wear, head clogging)
Acquisition methods	Tribological testing
Skills	Materials Research laboratory
Samples	Collection of model samples
R&D Efforts	Some testing methods have still to be developed
Entry Example	/

DATABASE FIELD: Deterioration susceptibility

Artificial ageing tests in environmental chambers allow us to evaluate the stability of various compounds or objects when exposed to various conditions. In relation with the composition of each technological family as identified in the previous field, this field presents the laboratory evaluation of the stability of each chemical (lubricants, antioxidants...)

In addition, stability of the overall tape after artificial ageing is also evaluated in terms of playback and tribological deterioration symptoms (friction, wear and head clogging).

Entry Types	<ol style="list-style-type: none"> 1. Effect of artificial ageing on chemicals 2. Tribological behaviour after artificial ageing 3. Bibliographic search on stability studies 												
Acquisition method	Artificial ageing, chemical analyses, tribological testing, bibliographic search												
Skills	Analytical chemistry laboratory and materials research laboratory												
Samples	Collection of model samples												
R&D Efforts	Some analytical and measurement protocols still have to be developed												
Entry Example	<p style="text-align: center;">1. Binder stability by SEC (Appendix 12)</p> <p style="text-align: center;">B APX5 kppac930717.1</p>  <table border="1" style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th>Peak</th> <th>Retention Time (min)</th> <th>Intensity</th> </tr> </thead> <tbody> <tr> <td>1</td> <td>20.1</td> <td>1000</td> </tr> <tr> <td>2</td> <td>21.5</td> <td>1500</td> </tr> <tr> <td>3</td> <td>23.2</td> <td>2000</td> </tr> </tbody> </table>	Peak	Retention Time (min)	Intensity	1	20.1	1000	2	21.5	1500	3	23.2	2000
Peak	Retention Time (min)	Intensity											
1	20.1	1000											
2	21.5	1500											
3	23.2	2000											

B.6.4 PRESERVATION FEEDBACK RELATED FIELDS

These two fields are based on an unlimited number of tapes. They are dependant on the community commitment to implement the database or on collaborating organizations in charge of preservation projects at a large scale.

DATABASE FIELD: Playability statistics

The playability statistics represent probably the most important field of the database because of its direct contribution to the final condition assessment. These statistics are obtained without measuring intermediates (chemical analysis, mechanical testing etc) but directly on a playback device.

Reliability of these statistics depends on the sampling procedure and on the definition of standards for playback quality reporting.

Entry Types	<ol style="list-style-type: none"> 1. <u>Digitisation failure rate</u> 2. Advanced quality reporting on playback quality
Acquisition methods	Present preservation projects and advanced playback device
Skills	Migration specialist and service providers.
Samples	As much tapes as possible
R&D Efforts	Quality reporting definition and normalization
Entry Example	<u>Appendix 6</u>

DATABASE FIELD: User Feedback

The community of the archivists dealing with large number of magnetic tapes is an important source of knowledge on technological family stabilities. Large scale survey or online permanent experience sharing and stability update is a direct way to implement “playability statistics”. Relativity of the reliability of the gathered information has to be take into account.

Entry Types	<ol style="list-style-type: none"> 1. Failure rate 2. Survey results
Acquisition method	Survey, online experience sharing
Skills	Archivists, preservation specialists
Samples	As much tapes as possible
R&D Efforts	Questionnaire developement, quality reporting normalisation
Entry Example	/

B.7 DATABASE MANAGEMENT SYSTEM

A management system for the knowledge database is proposed in this section of the report. This system includes: a procedure for tape “technological family” identification, an unformatted data retrieving function, and a calculation method for condition assessment.

Technological family identification process

The objective of this process is to allow a user to identify the “technological family” of the tape (or group of tapes). This identification is mandatory step in order to use entries of the database that are organised following “technological families”.

Database raw information retrieval

This first level of information that a user can retrieve corresponds to all available data, unformatted, on the technological family previously identified. The interpretation and use of the data are up to the user and is dependant upon the objectives and skills (playback specifications, brand name, year or production, chemical formulation etc).

Calculation method for condition assessment

An example of calculation method is provided with the demonstration software and explained in the section B10 of this report.

We define the “archival performance rate” as a number of points attributed to a given database entry as function of conservation issues. The calculation method is based on a mathematical treatment of the “archival performance rate” of each database entry.

The calculation aims to mathematically combine all rates into a unique overall “condition rate” that has to be regarded as a “probability” to experience playback problems. “Archival performance rates” being the result of a statistical approach, an estimation of the reliability of the overall “condition rate” has to be made in parallel and provided by the management system.

Some entries of the database are dependant on the tape samples (model tapes) used for database implementation and on their specific storage conditions.

As a consequence, the calculation result may be under (or over) estimated if storage conditions of the tape (or group of tapes) that a user wants to evaluate have been worse (or better) than the condition the “model tapes” used for database implementation.

A correction for effective storage conditions is possible through the deterioration susceptibility related fields of the database.

If the storage conditions are not known, the “model tapes” storage conditions can be considered as the best available match for condition assessment of the tape (or group of tapes).

The general schema of a management system is presented in the following diagram. The diagram does not include the statistical sampling for technological families mapping of the collections. The method presented here, is focused on the result calculation method for a given technological family stored under known conditions. A method for technological family identification is presented latter in this report (B9).

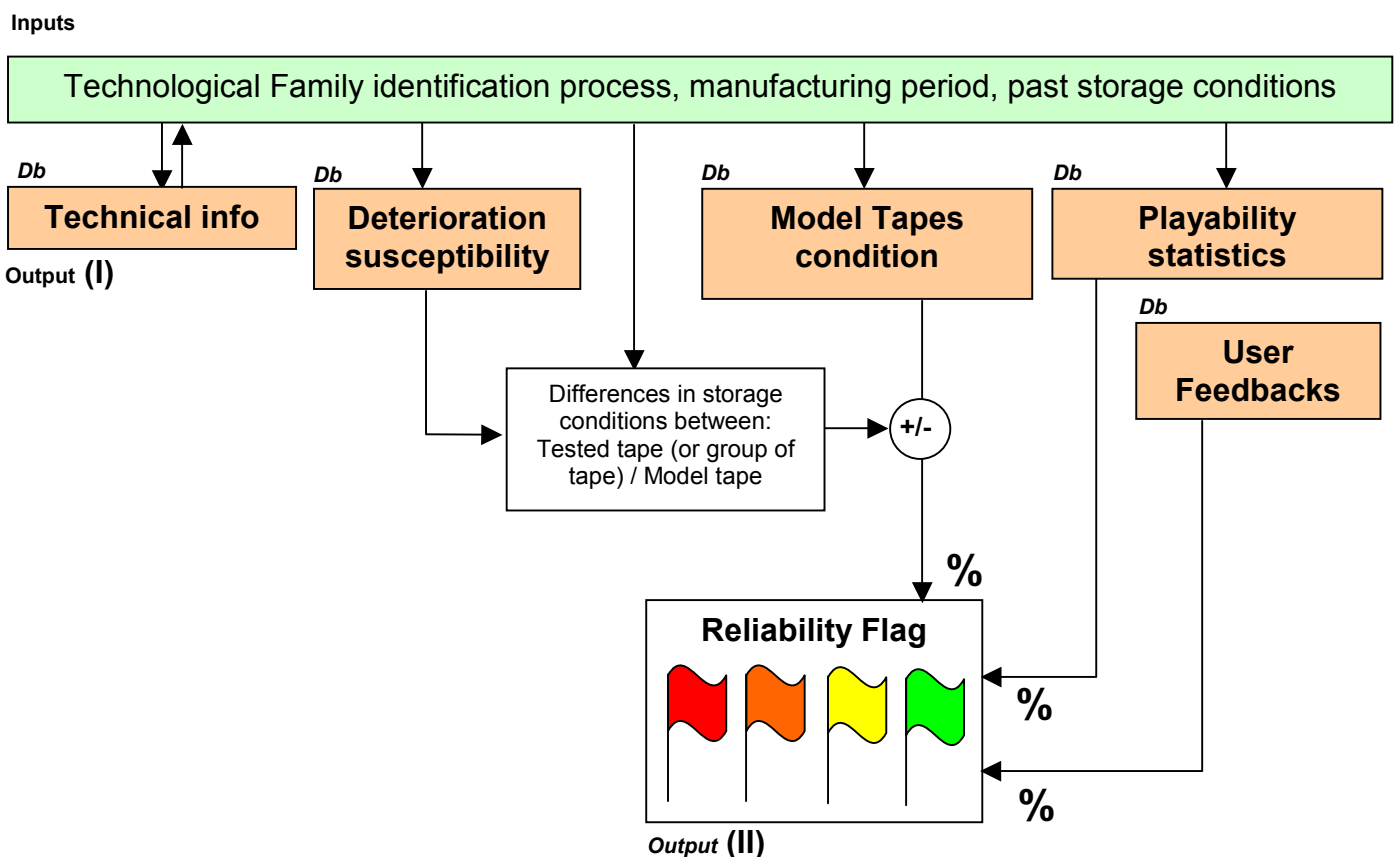


Figure 5 Knowledge management system layout

Inputs

The inputs of the calculation method include the technological family, the past storage condition (if known) and the estimated manufacturing date (if known). The identification process (not shown here) uses the database fields related to tape identification to assist the user. (double sided arrow)

Database (Db)

This diagram presents different blocks called “Db” related to groups of fields from the database.

Technical information - Fields: *tape identification, tape structure, tape packaging, technical documentation, tape chemical composition*

Deterioration susceptibility - Fields: *tape chemical composition and deterioration susceptibility*

Model tape condition - Fields: *present deterioration state, present tribological behaviour*

Playability statistics - Field: *playability statistics*

User feedback - Field: *user feedback*

Output (I): Technical information

This query result corresponds to all information related to the technological family.

Output (II): Statistical condition assessment result (presentation in an easily understandable format)

Tape condition assessment is the result of a calculation based on a combination of all the “archival performance rates” of all database entries relevant to the technological family.

The relative significance towards condition assessment of the different fields is obtained by a “fields weighting”.

Different weights are determined according to the reliability and relevancy of the data towards conservation, as well as the number of model tapes. This weighting is symbolized on the diagram by the ‘%’ sign.

For instance, the fields related to “playability” statistics are the more important parameters compared to others fields. The impact of these entries on the condition

assessment has to be predominant. As a consequence the weight of these fields has to be higher than others.

The different thresholds for flag colour results and the decision making process have to be calibrated.

Optional Output (III) (not shown on the diagram): Risk assessment for long-term storage.

With data on technological family compositions and stabilities being stored in the database, further deterioration risks can be estimated.

This output if possible, could greatly assist the decision making process and optimise the planning actions.

However, some R&D efforts have still to be done.

As told earlier (B3.4), the implementation of the database, and especially the fields related to the technological family identification and the “playability statistics” would greatly improve and guide research efforts.

B.8 LIMITATIONS

Limitations of the database approach

The knowledge approach is based on the categorization of magnetic tape resulting in technological families. Technological families are related to manufacturers and manufacturing practices. Some tapes cannot be included, i.e. those that don't have any method for an "on-shelf" identification of the technological families (distinct patterns, serial numbers code etc).

Most of the video formats present distinct patterns and almost all the cassette formats have serial numbers. However, the manufacturing period/batch/manufacturer of most of audio tapes cannot be identified without chemical analysis.

Considering all the manufacturers and all the tapes ever produced is not possible. Even find a statistically significant number of suitable "model tapes" for destructive testing may be problematic. As a consequence the approach is limited to popular tape manufacturers or popular products.

Limitations of the management system for condition assessment.

The calculation method results only in recommendations and does not guarantee the condition assessment for every single tape. The result has to be regarded as a statistical evaluation.

Common limitations of all condition assessment methods.

Even the best testing method would not be able to take into account manufacturing practices variability or unknown past storage conditions. All condition assessment methods have interpretation difficulties and sometimes poor collection representativeness.

B.9 MANAGEMENT SYSTEM SOFTWARE

Operation of the management system and database update can be made by computer software. This interface would help the user with tape identification and resulting interpretations. In this section, the main functions of the suitable software are described briefly.

B.9.1 INPUTS

Technological family identification:

The first step of the procedure is the technological family identification by simple and quick tape handling or “on-shelf” tape examination.

The following methods can be used:

- Identification by serial number code recognition
- Comparison to reference pictures

It is important for the identification process to be as simple and quick as possible. If serial number codes are used, a full reading is not required. The categorization in the knowledge database is not more accurate than the “technological family” level. In addition, each family may either include many tapes with unique serial numbers or may include different batches (many serial number series).

Once a technological family is proposed by the software, some tape identifiers (called extra-identifiers in B6.2) can be used further verification of the verification of the initial guess.

Output content

The output of the management system has to fit the objectives and skills of the user. Options have to be available to set the level of the output.

Other information

The calculation method includes a correction method for storage conditions. If known or suspected, these conditions have to be provided by the user.

Rough estimation of the manufacturing date and of environmental conditions only are required (levels from “very good” to “very poor” can be defined, explained to the user)

If the storage conditions are unknown, the calculation method cannot correct the condition assessment, and the result will be dependant on the condition of model-tapes that are used for database implementation.

B.9.2 OUTPUTS

As it was told previously, the two main outputs are: unformatted technical information about the technological family and the calculated and corrected information for storage condition assessment of technological families.

The calculation results have to be presented with a statistical “reliability” evaluation calculated on the basis of the number of model samples examined, the reliability of the data sources.

B.9.3 DATABASE UPDATE

Finally, the efficiency of a knowledge database depends on the size of the database and on the number of model-tapes.

As a consequence, a permanent update of the data from the archivists and preservation specialists has to be possible to insure database reliability.

In order to integrate with efficiency the data provided by the archive community, evaluation protocols and update formats have to be defined. Due to the fact that the database is organized in “technological families”, identification of technological families procedure has to be widely disseminated also.

B9.4. Web-based Software

The success of the database knowledge approach depends on the commitment of the archive community and preservation specialists. A web-based application is probably the best way to insure a certain popularity and reliability of the proposed approach.

B.10 DEMONSTRATION SOFTWARE

The first part of the WP06 gave us the opportunity to study magnetic tapes deterioration mechanisms and to develop specific investigation methods.

U-matic tapes have been used as a model format for the laboratory developments. In addition to be particularly representative of the overall magnetic tape media, they used to be very popular for almost 3 decades. Lots of preservation projects on a large scale have been successfully completed providing important conservation feedback on U-matic format. .

The large volume of information and numerous data collected on U-matic tapes by the CRCDG enables to propose a structure for the database.

This knowledge is used here to demonstrate the knowledge database approach and to define the key issues of its implementation.

The choices we made in order to build this demonstration are arbitrary decisions and have to be discussed and supported by additional experimental work. We tried as much as possible, to illustrate in the demonstration software, all the considerations made in this report. For some technical reasons, some concepts described in the report are missing.

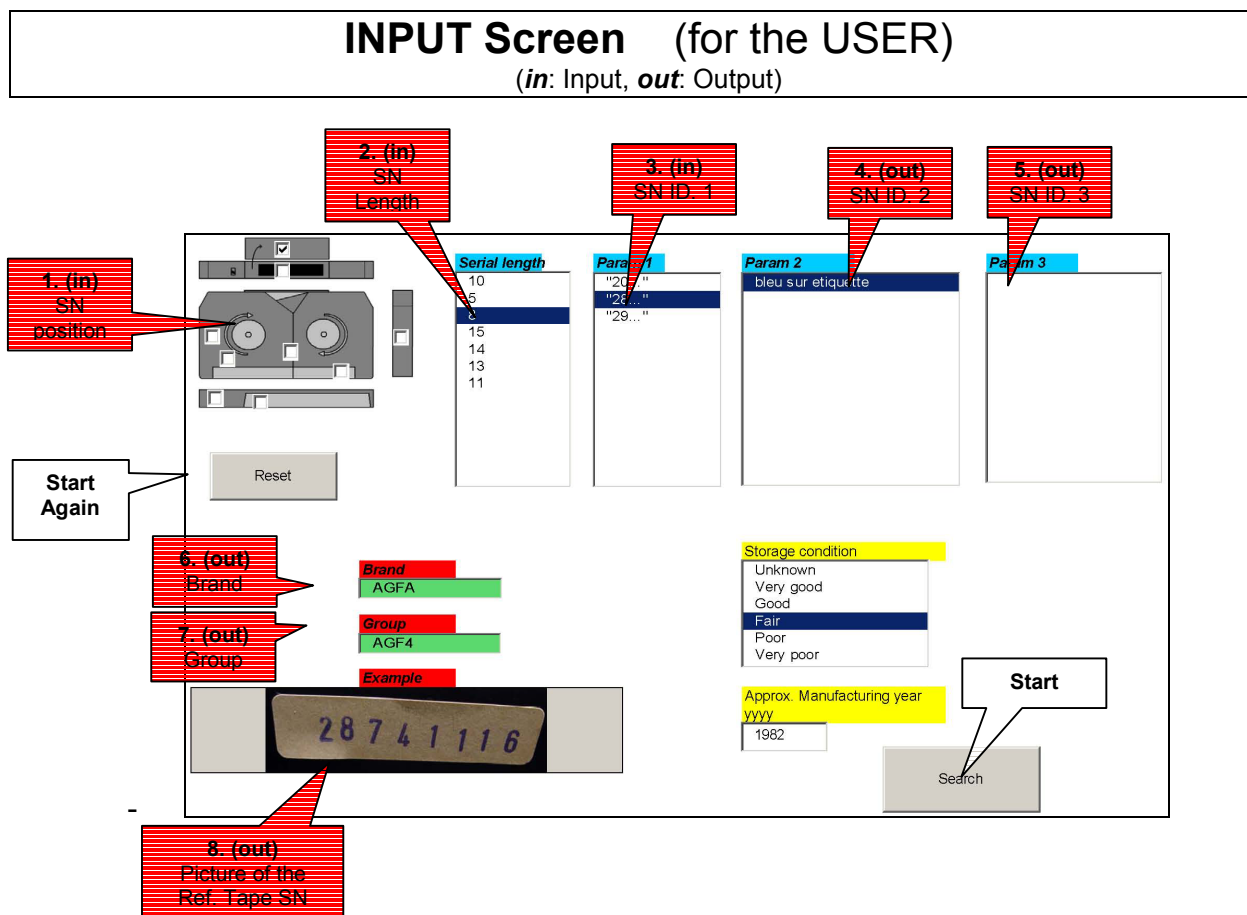
This demonstration software uses Visual Basic and MS Excel programming.

B.10.1 TECHNOLOGICAL FAMILY IDENTIFICATION PROCESS

The identification process is proposed in the INPUT spreadsheet which is in relation with the “Serial Number DB” spreadsheet.

The user is invited to examine a tape and to record the result in dedicated textboxes (*windows in*)

As soon as a sufficient number of elements are provided, a technological family is proposed along with extra-identifiers. (*windows out*). The technological family is identified by a code given by the CRCDG.



Serial Number (SN) associated Database

Group id.	SN Loc.	SN Length	Param. 1	Param. 2	Param. 3	Brand	SN Reading alg.	First	Last	Ref. Tape SN	Ref. Tape Num.	Picture
APX1	12	14	"N..."	other		AMPEX	?	1981	?	N3196857012166	kpcaa8206222	apx1.jpg
APX2	12	14	"N..."	"...000000..."		AMPEX	?	1984	?	N042400000006	kpcab8411292	apx2.jpg
APX3	12	14	"N13..."			AMPEX	?	1985	?	N1321870922063	kppac8509131	apx3.jpg
AGF1	6	10	"H..."	noir sur etiquette		AGFA	?	1987	1993	H71.0421.26	kppac8711032	agf1.jpg
AGF2	6	5	"20..."	bleu sur etiquette	symboles facultatifs	AGFA	?	1980	1983	20474	kppac8304221	agf2.jpg
AGF3	6	8	"20..."	bleu sur etiquette		AGFA	?	1980	1983	20148132	kppac8109281	agf3.jpg
AGF4	6	8	"28..."	bleu sur etiquette		AGFA	?	1982	1983	28741116	kpcaa820616.2	agf4.jpg
S2	9	7	"51..."	"....A8M"	White	SONY	51XXA8B : 0B/198A ; P=dec, M = nov, S = oct (code sony)	1987	?	5181787	KPCAC880901.1	apx2.jpg
S3	9	7	"51..."	"....A4M"	White	SONY/BASF	51XXA4B : 0B/198A ; P=dec, M = nov, S = oct	1987	1987	5111746	kpcab880901.1	apx2.jpg

B.10.2 OUTPUTS FORMATTING

The query is sent to the spreadsheet « Technical DB » with the “Search” button.

The user’s inputs about storage conditions and tape manufacturing year (INPUT screen) are also sent for condition assessment correction.

The condition assessment is displayed on the « OUTPUT » Spreadsheet.

The data retrieved by the software correspond to the “statistics on playability” and the entries related to investigations on the collection of model samples. The number of the sample used for the technological family study is given.

Details on the calculation method arbitrary chosen for this demonstration software are given later in this report (B11).

OUTPUT screen
(blank cell are for unknown data)

Final Grade and flag (see 16.3 for calculation details)

6/20

Tape identification

Brand :	AGFA
Ref. Tape SN :	28741116
Group :	AGF4
First :	1982
Last :	1983
Made in :	leverkusen-germany

Technical documentation and additional

SN reading	?
additional Info :	VIDEOCHROM, leather like tape case
Technical documents :	agf4.pdf

Technology related information

Backcoating	yes
Thickness	?
Oxide Type	Cr
Other particles	?

Chemistry related information

Binder	
Lubricants	palmitic acid butyl ester
Additives	antiox,bht
Volatile compounds	stearic butyl ester, myristic butyl ester, 1,6-Dioxacyclododecan e-7,12-dione
Acidic odouring compounds	yes
Solvent extractable	93%
Extractable mass distribution evaluation	Q1= 3%; Q2= 21%; Q3= 76%

Deterioration symptoms evaluation

Friction	
Binder Hydrolysis	Q1= 3%; Q2= 21%; Q3= 76%
Specific problems experienced	
Reading Failure rate (lab testing)	
Reading Failure rate (user feedback)	

Technical associated Database

Group	Additional information					Technology										
	Tape Series	Made in	full text	Technical Archive	Grade /20	Backcoating	Grade /20	thickness	Grade /20	Oxide	Grade /20	Particles	Grade /20	analytical fingerprint	Lubricant(s)	Grade /20
APX1				apx1.pdf		yes	0			Fe/Co	20	Al		I		
APX2	187 KCA			apx2.pdf		yes	0					?				
APX3	197 BCA			apx3.pdf		yes	0					?				
S2	BRS	made in japan	blue hub	s2.pdf		yes	10					?		F		
S3	BASF		two SN BASF and Sony	s3.pdf		no	20					?		F		
AGF1	Professional	leverkusen-germany		agf1.pdf		yes	0			Cr	0	?		A	palmitic acid butyl ester	
AGF2	VIDEOCHROM	leverkusen-germany	botliers simili cuirs	agf2.pdf		no	20			Cr	0	?		A		
AGF3	VIDEOCHROM	leverkusen-germany	botliers simili cuirs	agf3.pdf		no	20			Cr	0	?		A		
AGF4	VIDEOCHROM	leverkusen-germany	botliers simili cuirs	agf4.pdf		yes	0			Cr	0	?		A	palmitic acid butyl ester	

Chemistry															
Binder	Grade /20	additives	Grade /20	VOC	Grade /20	Acids / lactones	Grade /20	Solvent extractables (%)	Grade /20	Q1(%) >30000 g/mol	Grade /20	Q2(%) 30000 g/mol >M> 4000 g/mol	Grade /20	Q3 < 4000 g/mol	Grade /20
								65	7	62	7.6	27	14.6	11	17.8
		antiox		aldehydes, propyl glutaric acid, cyclohexanone, toluene		No	20	41	11.8	32	13.6	45	11	21	15.8
		bq, antiox		stearic butyl ester, myristic butyl ester, hexanoic dibutyl ester, cyclohexandiolic butanol		Yes	0	61	7.8	7	18.6	34	13.2	59	8.2
		antiox.bht		stearic butyl ester, myristic butyl ester, 1,8-Dioxacyclododecane-7,12-dione		Yes	0	93	1.4	3	19.4	21	15.8	76	4.8

Playability											
Hydrolysis	Grade /20	Friction	Grade /20	Head-clogging	Grade /20	Other problems	Grade /20	Failure rate (%) (lab)	Grade /20	Failure rate (%) (users)	Grade /20
	141										
	138										
	107										
	3:1										

B.10.3 CALCULATION METHOD FOR CONDITION ASSESSMENT

A calculation method used in the demonstration software is presented here.

The result of the calculation is displayed as a coloured cell ranging from green to red respectively for tapes in good condition and for tapes with a high probability of playback problems.

The colour is a function of an overall rate resulting of the calculation method. This rate is calculated by a linear combination of all “archival performance rates” given to the entries of all fields of the database having a responsibility in playback problems.

To take into account different level of responsibility for different fields, proper database fields weighting is applied. (determination of the linear combination coefficients)

Coefficients related to the environmental stability properties (for example: hydrolytic stability etc) are calculated values depending on the storage conditions provided by the user. These coefficients are responsible for the “correction” of the condition assessment.

The general expression for the calculation of the overall rating for a given tape belonging to a given technological family OR_{TF} is:

$$OR_{TF} = N \times \left[\sum_i a_i \times v_i + \sum_j b_j \times v_j \right]$$

where N is a normalization factor, a are the constant coefficient for all fields (i) related to properties or measurements of model-tapes (including playability statistics) and user’s feedback. v are the “archival performance rates”, b are coefficients function of the storage conditions differences between the model tapes and the tested tapes. b are applied to the fields (j) related to the deterioration susceptibility of the technological family.

For some technical reasons, the corrective term is not included in the software demonstration and all weights are set to “1”.

B10.3.1. “Archival Performance Rates” Attribution Methods

The “archival performance rates” are attributed following rules (rating systems) dependant on the nature of the evaluated fields.

Each particular rating system is defined to allow ranking of all entries as function of their responsibility in playback problems, or as function of their role in a given deterioration mechanisms.

All rates are between 0 and 20 (0: poor archival performance, 20: best archival performance).

In the demonstration software arbitrary choices have been made, further discussions and experimental work would lead to more accurate rating systems.

It can be defined two systems whether the entries are “properties” (characterization data) or “values” (experimental measurement or result).

Properties are: chemical nature, technology, tape structure. For each field, a finite number of possibilities exist. In this case, rating system can be simplified by ranking all the entries in each field as function of their archival performance.

Values are: tribological measurement, deterioration susceptibility measurement... They are continuous over a range of measured values. Rating system is a normalization of the measurement over the score range. Here are two examples of rating systems for “properties” and “experimental results”.

1) A rating system of a Property:

Example: Chemical nature of magnetic particles

The magnetic particles of U-Matic tapes are made of chromium oxide or of cobalt-doped ferrous oxide.

It is known (and experimental studies show it) that chromium oxide based particles have a negative effect on the binder system stability. Ranking of these properties give: 1. cobalt-doped ferric oxide, 2. chromium dioxide).

The “archival performance rate” of the entry “chromium dioxide” in the field “chemical nature of magnetic particles” is the lowest: “0”. Whereas the “archival score” of ferrous oxide based magnetic particles is the highest: “20”.

If more than two entries are possible for a given field, the rates will reflect the rank of each possibility towards their archival performance.

Only the catalysis effect on binder system deterioration is considered here. Magnetization stability over time may be considered as well.

2) Rating system for an experimental measurement result:

The example given here illustrates the rating system related to measurement and experimental results. This particular case is complex but allow to describe the different considerations and assumptions required to turn experimental result into significant values. Additional information on this specific example can be found in Part A, section A5.1.1 and appendix 7 and 12.

Example: Binder study by size exclusion chromatography. (SEC):

First step:

from curves to significant quantities

B APX5 kppac930717.1

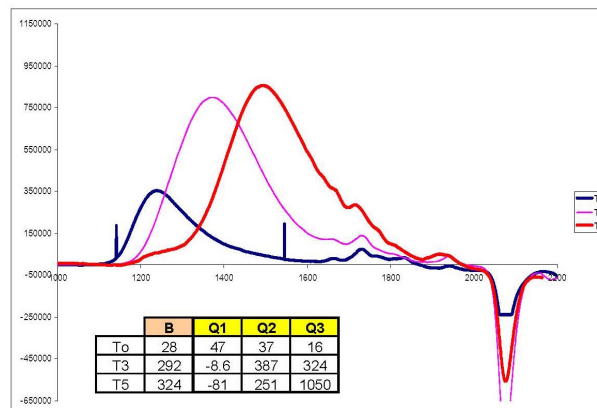


Figure 6 SEC analysis of magnetic tape binder molecular weight distribution before (blue curve) and upon artificial ageing (pink and red curves). Fragment molecular weigh decrease from left to right, intensity is proportional to quantity. The table summarizes the measured significant quantities.

The Figure 5 shows the results of the SEC investigation for an AMPEX model-tape that belongs to the technological family of the APX5 group (CRCDG group reference).

The following assumptions are made to define the significant characteristics (or measurable quantities) to be taken into consideration for archival performance rating. Polymer condition can be represented by 4 significant and measurable quantities (Q1, Q2, Q3 and Q) extracted from the experimental SEC results. Polymer hydrolytic stability can be represented by the percentage of evolution of these 4 quantities upon artificial ageing. All these quantities are summarized in the table presented in Figure 5. More details about the table are given latter.

Q3 Value:

Only smaller polymer fragments can migrate within the polymer matrix up to the polymer surface. It can be supposed that they are responsible for head clogging and surface properties deterioration of the magnetic layer surface (mainly increased adhesivity).

Q3 is the amount of polymer fragments having a molecular size lower than 4000 g/mol eq. PS. Q3 is equal to the area of chromatograms eluted after 1500 seconds on the SEC chromatogram.

Q2 Value:

Fragments with a molecular weight ranging from 4000 to 30000 g/mol eq. PS are likely not as mobile as Q3 fragments, but are responsible (as Q3) for an increased acidity of the tapes surface (due to binder breakdown by hydrolysis). This acidity favours the water uptake and catalytic effect on the hydrolytic breakdown. Q2 is equal to the chromatogram area eluted between 1300 s and 1500 s on the SEC chromatogram.

Q1 Value:

Fragments with a molecular weight higher than 30 000 g/mol eq. PS. have little consequences on playback problems even if they are probably the precursors of Q2 and Q3 fragments upon hydrolytic breakdown.

Q1 is equal to the area of chromatograms eluted before 1500 seconds on the SEC chromatogram.

Q Value

Q quantity is the sum of all extractable (Q1, Q2, Q3) fragments for a given tape. This quantity is directly related to the total amount of solvent-extractables.

Hydrolytic stability

The hydrolytic stability is investigated upon artificial ageing and by a size exclusion chromatography study. The evolution of the Q1, Q2, Q3 and Q quantities upon artificial ageing can be considered as an evaluation of the hydrolytic stability.

Thanks to significant ageing times and conditions (T0 = 0 days; T3=16 days; T5 = 36 days), the evolution (in percentage) of the quantities Q1, Q2, Q3 and Q can be regarded as quantities that measure the hydrolytic stability.

Second step:

from significant quantities to “archival performance rates”

All the quantities defined previously can be easily turned into rates ranging from 0 to 20. The following table (Figure 5) calculated for each SEC analysis allows rapid extraction of the significant quantities that are added to the database.

	B	Q1	Q2	Q3
To	A	B	C	D
T3	A	E	F	G
T5	A	E	F	G

A: Solvent extractable quantity (Q) (percent of a reference value).

B: Q1 (percent of Q)

C: Q2 (percent of Q)

D: Q3 (percent of Q)

E: Q1 variation (ageing time T3 or T5) (percent of evolution)

F: Q2 variation (ageing time T3 or T5) (percent of evolution)

G: Q3 variation (ageing time T3 or T5) (percent of evolution)

B10.3.2 Database Fields Weighting (linear combination coefficients calculation)

No weights optimisation has been completed for the demonstration software giving the same importance to all fields.

The weights optimisation can be the result of arbitrary choices supported by deterioration mechanisms and kinetic knowledge.

Statistical approach can also be used for weights optimisation based on feedback of present preservation project.

B.11 PART B: CONCLUSION

The knowledge database approach as described in the part B of this report, if accompanied by the implementation of a suitable management system, can be regarded as a condition assessment method for anticipation of playback problems.

The key issues of such approach are:

- the identification of all the major technological families
- the evaluation of their deterioration susceptibility
- the statistical evaluation of their playability

Technological family identification (including chemical characterization) and related “playability statistics” have to be added in priority in order to implement and deliver a management system for condition assessment.

The deterioration susceptibility and the laboratory measurement on each technological family (i.e. polymer breakdown, friction etc) are important to allow correction for storage condition of the evaluated collection, precise risk evaluation on a long term storage, and further research on magnetic tape materials.

Beyond the applications of the database presented in the part B, all the knowledge gathered are of great importance for conservation scientists involved in magnetic tape research. This database would allow a better understanding of the relations between materials, storage condition and degradation symptoms.

Keeping in a controlled environment, updating with new tapes, and frequent re evaluation of the collection of “model sample” would enable condition assessment to be more accurate and reliable. Anticipation of future preservation issues (including for tapes that so far, do not exhibit playback problems) could be greatly facilitate by such approach.

C APPENDICES

APPENDIX 1

Serial Number Code Reading For Motion Picture Film

(Source: <http://www.sabucats.com/?pg=datecode>)

EASTMAN KODAK DATE CODE CHARTS				DUPONT DATE CODE CHART	
CANADA		ROCHESTER USA			
1925 1936 1947 ● L	1916 1936 1956 1976 ●	1982 ●■X	1956 KL		
1926 1937 1948 ●-	1917 1937 1957 1977 ■	1983 X▲X	1957 KN		
1927 1938 1949 ●◐	1918 1938 1958 1978 ▲	1984 ▲■▲	1958 KS		
1928 1939 1950 ◐●	1919 1939 1959 1979 ●●	1985 ■●▲	1959 LN		
1929 1940 -●	1920 1940 1960 1980 ■■	1986 ▲●▲	1960 LS		
1930 1941 ◐●	1921 1941 1961 1981 ▲▲	1987 ■▲▲	1961 NS		
1931 1942 ◆	1922 1942 1962 ●■	1988 ++▲	1962 K		
1932 1943 ◆●	1923 1943 1963 ●▲	1989 X+▲	1963 L		
1933 1944 ◆-	1924 1944 1964 ▲■	1990 ▲+▲	1964 N		
1934 1945 ◆L	1925 1945 1965 ■●	1991 X+X	1965 S		
1935 1946 ◆◐	1926 1946 1966 ▲●	1992 ■+▲	1966 KLT		
	1927 1947 1967 ■▲	1993 +▲▲	1967 KNT		
	1928 1948 (1968 IS ++)	1994 +●▲	1968 KST		
	1929 1949 1969 +	1995 +■▲	1969 LNT		
	1930 1950 1970 ▲+	1996 X●▲	1970 LST		
	1931 1951 1971 ●+	1997 X■▲	1971 NST		
	1932 1952 1972 ■+	1998 X▲▲	1972 KT		
	1933 1953 1973 +▲	1999 ●X▲	1973 LT		
	1934 1954 1974 +●	2000 ■■▲	1974 NT		
	1935 1955 1975 +■	2001 ▲▲●			
UK		PART OF YEAR*		FACTORY ID CODE	
1917 1936 ◐	1927-?(US), 1936-?(UK/CAN)	1/16"	S [●] A F E T Y - R O C H E S T E R		
1918 1937 L	first half of year		S A [●] F E T Y - C A N A D A		
1919 1938 -	1927-?(US), 1936-?(UK/CAN)	3/16"	S A F E [●] T Y - E N G L A N D		
1920 1939 ◐◐	second half of year		S A F E T [●] Y - F R A N C E		
1921 1940 LL	(later date)	1/16"	S A F E T Y [●] - A U S T R A L I A		
1922 1941 --	first quarter of year				
1923 1942 ◐L	(later date)	1/8"			
1924 1943 -L	second quarter of year				
1925 1944 ◐-	(later date)	3/16"			
1926 1945 -◐	third quarter of year				
1927 1946 L-	(later date)	1/4"			
1928 1947 L◐	last quarter of year				
1929 1948 +					
1930 1949 +◐					
1931 1950 ◐L					
1932 +-					
1933 ◐+					
1934 L+					
1935 --+					

THESE ARE THE DATE CODE CHARTS FOR EASTMAN KODAK MOTION PICTURE FILM AND DUPONT FILM. READ THE SYMBOLS ON THE EDGE MARKINGS OF THE FILM FROM LEFT TO RIGHT, AND COMPARE TO THE CHART. THIS WILL TELL YOU WHEN THE FILM WAS MANUFACTURED AND WHERE. WHITE PRINTING TELLS THE INFORMATION ABOUT THE NEGATIVE, AND BLACK PRINTING TELLS THE INFORMATION ABOUT THE POSITIVE PRINT. EXAMPLE: THE BLACK PRINTING READING LEFT TO RIGHT ON A PRINT OF "THE PRODUCERS" IS " + + ". THE CHART TELLS US THAT THIS IS A PRINT STRUCK IN 1968, WHICH INDEED IS WHEN THE FILM WAS RELEASED. FOR FUJI FILM, THERE IS A 4 DIGIT CODE ON THE PERFORATIONS. THE FIRST 2 NUMBERS ARE THE YEAR THE FILM WAS MANUFACTURED. FOR EXAMPLE: "8 3 J M" IS A PRINT MANUFACTURED IN 1983.

*A SMALL VERTICAL BAR IS SPACED BETWEEN THE WORDS "EASTMAN" OR "KODAK", AND "NITRATE" OR "SAFETY". THE YEAR SYMBOL USUALLY FOLLOWS THIS BAR. THE DISTANCE BETWEEN THE BAR AND YEAR SYMBOLS INDICATES THE FIRST OR SECOND HALF OF THE YEAR.

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 www.sabucats.com



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 Phone: (661) 538-9395
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 info@sabucats.com

APPENDIX 2

List of 3M Audio Open Reel Tapes

<http://www.aes.org/aeshc/docs/3mtape/aorintr1.html>

Introduction to 3M Audio Open Reel Tape List

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APPENDIX 3

List of Ampex Magnetic Tapes

(Source: <http://recordist.com/ampex/docs/apxtape.txt>)

Ampex tape list -- Compiled by Scott Dorsey, NASA Langley Research Center

This list started out as a response to the huge number of people asking questions about surplus instrumentation tape, developed into a short description on identifying Ampex tapes, and then developed into an attempt at categorizing all Ampex tapes made. This list is far from complete but is a good beginning:

All tapes backcoated unless otherwise indicated (NB= Non-Backcoated)

```

-----
021-- 1 mil rejected NB acetate tape (sold as Shamrock) (early 60s)
031-- 1.5 mil rejected mastering tape (maybe 406, 456, etc. depending on batch)
      (Was available in 2500' pancakes as well as 7" reels in 1983)
041-- 1 mil version of 031 in 7" only
051-- 0.5 mil rejected NB acetate tape (sold as Shamrock) (early 60s)

101-- Betamax half-inch videocassette (intro before 1978)
102-- VHS videocassette (late seventies)
142-- Helical 2" format video tape for VR-660/1500 (1962-?)
144-- Transverse scan video tape (1964-?) for quad-format machines
145-- Helical 2" formulated for VR-660/1500 video recorders (1966-?) backcoated
147-- A-format tape formulated for VR-7000 video recorders (1965-?)
161-- 1" videotape (1968-?)
163-- 1/2" helical scan industrial videotape (1968-?)
164-- 1/2" helical scan videotape for color use
167-- U-Matic videocassette (replaced by 187/197) (late seventies, sold in 83)
      (Sometimes this is labelled KCA-xx where xx indicates running time)
174-- 1/2" helical scan industrial videotape (1970s?)
175-- 2" Quad tape (backcoated) replacing 144 (sold until 1990)
187-- U-Matic videocassette
      (Sometimes this is labelled KCA-xx where xx indicates running time)
188-- Older Beta videocassette
189-- VHS videocassette, NB (discontinued 199x?)
190-- 1" IVC/Sony format videotape (1983)
192-- 1" videotape (1970s) (replaced by 196)
194-- 1/2" helical scan industrial videotape (1970s through 1983)
196-- 1" B-format helical videotape (comes in two versions, earlier chrome
      later cobalt oxide), wound oxide out.
197-- U-Matic 3/4" videocassette (coexists with 187, better quality tape)
198-- Beta videocassette (oxide) (replaced by 208) (1987-??)
199-- VHS videocassette, NB (? to some time between 1990 and 1995)

208-- Betacam 1/2" broadcast videocassette (oxide, not metal) (changed to BC in
1998)
211-- 1.5 mil NB Irish ??? (1963 sample)
219-- D-1 digital videocassette (replaced by 229) (1990)
220-- Degaussing and head cleaning cartridge for cassettes (not real tape)
228-- Degaussing and head cleaning cartridge for 8-track tape (not real tape)
229-- D-1 digital videocassette (renamed DIV in 1998)
288-- Hi-8mm videocassette
289-- S-VHS videocassette
292-- 1.0 mil polyester 1/4" audio backcoated on 7" reel, black oxide.
296-- 1" C-format helical videotape
297-- U-Matic SP videocassette
298-- Betacam SP 1/2" videocassette (metal) (replaced by 398) (1990)

301-- General purpose audio NB tape available on reel or cassette (1968-?)
304-- Tape optimized for low speed logging use (1968-?)
311-- Irish version of 511 (1962)
319-- D-2 digital videocassette (replaced by 329) (1988)
321-- Irish version of 521 (1962)

```

329-- D-2 digital videocassette
 331-- Irish version of 531 (1962)
 341-- Irish version of 541 (1962)
 342-- 1.5 mil "Plus Series" NB consumer 1/4" tape (185 nWb/m) (1970s-1980s)
 344-- 1 mil PE, low noise, non-backcoated 1/4" tape. (available in 1973)
 345-- 1.5 mil red oxide tape (recommended for B&K 7004 recorder) (av in 1976)
 351-- Irish version of 551 (1962)
 352-- LO-NOISE compact cassette (lowest grade) (1978)
 361-- Compact cassette tape (1969-?)
 364-- 20/20 Plus cassette tape (mid grade) (pre-1978 to 1983)
 365-- Grand Master type I cassette tape (pre-1978 to 1983)
 366-- Grand Master type II cassette tape (pre-1978 to 1983)
 370-- HOLN cassette tape lowest-grade (discontinued 1983)
 371-- Plus Series cassette tape (lower grade) (1978)
 372-- Ampex 406 on 7" reels for consumer use (mid/late 70s). "20-20 series"
 373-- 1 mil version of 372
 381-- Low grade 8-track cartridge (1976)
 382-- Plus Series 8-track cartridge (1978)
 388-- 20/20 Plus Series 8-track cartridge (1978)
 389-- Grand Master 8-track cartridge (1978)
 398-- Betacam SP videocassette (metal) (renamed BSP in 1998)

401-- 1.5 mil mastering tape.... probably pre-1965
 404-- low-noise NB mastering tape (1965-?)
 406-- 1.5 mil mastering tape (260 nWb/m)
 407-- 1 mil version of 406
 408-- 406 wound slightly differently for Nagra recorders. (Intro 1997)
 411-- 1.5 mil NB (sample dated 1964)
 434-- 1.5 mil non-backcoated high-output brown-oxide consumer tape (1960s)
 444-- 1 mil version of 434
 456-- 1.5 mil mastering tape (370 nWb/m)
 457-- 1 mil version of 456 (available only in 7" 1/4" reel)
 460-- Digital audio tape (backcoated) for 3M machines (deleted 1982)
 467-- 4mm DAT tape backcoated, now called DAT (1998)
 467-- U-Matic oxide tape for digital audio, now called DAU (1998)
 467-- oxide 1/4" NAGRA-D, 1" for Pro-Digit, 1/2" for DASH tape for digital audio
 472-- Stereo audio cassettes (CrO2, NB) (250 nW/m)
 478-- 1.5 mil low-print mastering tape (250 nWb/m)
 480-- 1.5 mil Quantegy equiv. of 3M 908, modified 478 coat w/ smoother B (1997) for Nagra
 488-- 8mm tape designed for DTRS (Tascam DA-88) use, backcoated. (now DA8 in 98)
 489-- S-VHS tape designed for Alesis ADAT use, backcoated. (now ADAT in 98)
 499-- 1.5 mil mastering tape (520 nWb/m)

511-- 1.5 mil acetate version of 531 (1962)
 521-- 1 mil acetate version of 531 (1962)
 531-- "premium tape for semi-professional and home use" 1.5 mil NB Mylar 5", 7" (1962). Red oxide
 541-- 1.0 mil mylar base version of 531 (1962) Looks like later 641 tape.
 551-- 0.5 mil mylar base version of 531 (1962)

601-- .66 mil Cassette duplicating pancakes (NB) (1978)
 602-- .44 mil version of 601
 603-- .66 mil High performance cassette pancake (NB) (1979)
 604-- .44 mil version of 603
 607-- Thick standard bias cassette loads (NB) (1983)
 608-- Thin standard bias cassette loads (NB) (1983)
 609-- Thick chrome cassette loads (NB) (1983)
 610-- Thin chrome cassette loads (NB) (1983)
 611-- 1.5 mil brown oxide NB acetate (7" samples dated to 1963-1971)
 617-- 0.5 mil FeO2 cassette bulk-load pancake

618-- 0.3 mil version of 617
619-- .62 mil cassette duplication pancake (pre-1988-1993)
620-- .46 mil version of 619 (pre-1988-1993)
621-- 1 mil version of 611
631-- 1.5 mil non-backcoated "red oxide" voice-grade audio tape (185 nW/m)
632-- 1.5 mil non-backcoated "brown oxide" LNHO duplicating tape (185 nW/m)
638-- Empty 7" 1/4" accessory reel (not tape)
641-- 1 mil version of 631 (1960s-present)
642-- 1 mil equivalent of 632
651-- 0.5 mil version of 641
660-- HOLN black shell bulk cassettes (discontinued 1983)
661-- 0.5 mil version of 641 with thinner oxide (1968-present) (cassette or 1/4)
671-- 0.75 mil LNER non-backcoated extended-length tape (cassette or 1/4)
672-- FeO2 audio cassette (discontinued 1993)
674-- Cassette, low noise for high speed duplication. (existed in 1978, not now)
675-- 8-track cartridge pancakes, back-lubricated
678-- Cassette (discontinued)

700-- 1 mil NB low speed logging with very thick coating (1978)
701-- 1 mil NB low speed logging tape (replaced by 704) (1978)
702-- 1 mil backcoated tape with same coating as 701 (1978)
703-- 0.5 mil version of 702 (1978)
704-- 1 mil non-backcoated low speed logging tape
705-- 1 mil backcoated low speed logging tape (same coating as 704)
706-- 0.5 mil backcoated low speed logging tape (same coating as 704)
721-- 1 mil Digital PCM instrumentation tape (cobalt)
722-- 0.8 mil Double Density digital PCM instrumentation tape (cobalt)
731-- Data cartridge for Ampex Digital Cassette Recorder (lower error rate)
733-- Data cartridge for Ampex Digital Cassette Recorder
738-- ?? NB instrumentation-
741-- ?? NB instrumentation- (circa 1962)
756-- 1 mil Intermediate band NB instrumentation- replaced by 766 in 1984
760-- Wideband instrumentation tape, NB (1966)
761-- 1 mil standard coat 1" or 1/2" NB low band FM instrumentation (1965)
762-- 1 mil thin coating 1" or 1/2" NB low band FM instrumentation (1965)
763-- 1.5 mil standard coat 1" or 1/2" NB low band FM instrumentation (1965)
764-- 1.5 mil thin coating 1" or 1/2" NB low band FM instrumentation (1965)
766-- 1 mil Intermediate band NB instrumentation- replaced by 767 in 1986
767-- 1 mil Intermediate band instrumentation tape
770-- Wideband instrumentation tape, NB (1966)
771-- Tape for FR-1800 instrumentation recorder (1965)
775-- 2" transverse scan instrumentation tape (probably selected 175)
782-- 1 mil wideband NB instrument tape (1972 or so) (replaced by 787)
786-- 1 mil NB High Resolution (1978)
787-- 1 mil backcoated High Resolution (1978) (replaced by 797)
795-- High Resolution (intermediate band with low drop-outs) instrumentation
79A-- Variant of 797 made for government use only
797-- 1 mil wideband instrumentation tape
799-- 1 mil High bit density digital PCM tape

831-- computer tape (1966)
832-- 1/2" computer tape (1965-?)
834-- computer tape (1966)
836-- 1/2" 1600 bpi computer tape (1965-late 70s?)
837-- computer tape (1966)
838-- 1/2" computer tape (1965-?)
836-- computer tape (1966)
870-- 1/2" IBM-compatible computer tape (CATT) with new binder (1968)

8206-- 1 mil backcoated low speed logging tape (a 3M design), similar to 705.

ADAT-- Formerly part 489 (1998)
BC -- Formerly part 208 (1998)
BSP-- Formerly part 398 (1998)
CDR-- Compact Disc Recordable (1994)
CDR1-- Data tape cartridge (1968)
DA8-- Formerly part 488 (1998)
DAU-- Formerly part 467 in U-Matic (1998)
DAT-- Formerly part 467 in DAT (1998)
DBC-- Digital Betacam cartridge, metal particle tape (1998)
DDS-- 4mm DAT tape tested to DDS standards (1995)
DD1-- 19mm oxide tape for D-1 video format
DD8-- 8mm data cartridge (Exabyte style) (1995)
D1V-- Formerly part 229 (1998)
DV -- Tape for Ampex DCT recorder, 19mm metal D2-type cartridge (1998)
GP9-- 1/2", and 2" backcoated 320 nW/m high output mastering tape (1998)
HG -- A VHS videocassette offered until 1997.
ID2-- 19mm metal D-2 videotape.
MDR-- Recordable Minidisc (1998)
MOD-- Rewritable magneto-optical disk (1995)

APPENDIX 4

Evaluation of Some Magnetic Tapes

(Source: <http://www.tonbandwelt.de/texte/baender.htm>)**Inoffizielle Bändertauglichkeitsliste**

Ich habe einmal zusammenzustellen versucht, was im Forum zur Bänderfrage gepostet wurde. Dabei ist auffällig, dass es die Artenvielfalt bei BASF wohl eine Tabelle für sich benötigen würde. Somit dient diese eher grobe Liste als Leitfaden für den (Gebraucht-)Bänderankauf. Verbindlich sind die Aussagen jedoch nicht. Wer eigene Erfahrungen einbringen möchte, sollte dies bitte im Forum unter 'Diskussionen/Bänderfrage' tun, jedoch bitte mehr als eine handvoll Bänder besitzen - Ausreisser wird es wohl immer geben.

<i>Band</i>	<i>Beschreibung</i>	<i>Bewertung</i>
Maxell	XL II EE	Unbedenklich, neigt aber zum Schmieren (lagerproblematisch?)
Maxell	Alle Sorten	Unbedenklich
TDK	Alle Sorten	Unbedenklich
Agfa	alt ohne Rückseitenbeschichtung	Unbedenklich
Agfa	neuer mit Rückseitenbeschichtung	Bedenklich
BASF	alle Typen, vor ~ 1978	Unbedenklich
BASF	alle Typen, ab ~ 1978 bis ~ 1985	Bedenklich
BASF	alle Typen, nach ~ 1985	Unbedenklich
Emtec	alle Typen	Unbedenklich
Revox	601	Unbedenklich, jedoch höherer Abrieb, nicht gut für Neuaufnahmen
Revox	641	Unbedenklich
Revox	621 und 631	Bedenklich
Shamrock	mit Rückseitenbeschichtung	Bedenklich
Shamrock	schwarzes Bandmaterial	Bedenklich
Shamrock	dunkelbraunes Bandmaterial	Unbedenklich, aber schlechte Qualität
Shamrock	hellbraunes Bandmaterial	Unbedenklich und zumeist gute Qualität
Sony	alle Typen	Bedenklich
Scotch	207	Unbedenklich
Scotch	212 LP	Bedenklich
Scotch	3M / Scotch 806 PR7	Bedenklich
Scotch	Classic	Bedenklich
Scotch	alle anderen Typen	Bedenklich (vermutlich)
Ampex	alle Typen	Bedenklich
Braun	1022 (?)	Unbedenklich, jedoch gelber Abrieb
Braun	1025 (?)	Unbedenklich
Philips	Alle Typen	Unbedenklich, die Großspuler sind qualitativ hervorragend
Orwo	Alle Typen	Unbedenklich
Quantegy	Alle Typen	Unbedenklich, jedoch wenig Feedback

Natürlich kann man die schönste Liste knicken, wenn man sie mit Bändern vergleicht, die aus dem Baggersee gezogen wurden. Die Bänder sollten schon ordentlich gelagert worden sein. Aber selbst bei schlechter Lagerung (Keller, Dachboden) halten sich manche Bänder wacker!

APPENDIX 5

Magnetic Tape Technical Directory

(Source: <http://mb.abovenet.de/magnetofon/openreelgallery/>)

Alle Marken sind eingetragene Warenzeichen Ihrer jeweiligen Eigentümer.

All brands and trademarks are property of their respective owners

- [Agfa-Gevaert \(10\)](#)
- [Agfa Wollen \(3\)](#)
- [Akai \(1\)](#)
- [Ampex \(3\)](#)
- [Aristona \(2\)](#)
- [BASF \(14\)](#)
- [Bel-Cleer \(1\)](#)
- [Capitol2 \(1\)](#)
- [Combo \(1\)](#)
- [Concord \(1\)](#)
- [E-Leven \(1\)](#)
- [EMI \(3\)](#)
- [Emtec \(1\)](#)
- [Exclusiv \(2\)](#)
- [Gevaert \(1\)](#)
- [Grundig \(3\)](#)
- [Karstadt \(1\)](#)
- [Kodavox \(1\)](#)
- [Magna \(1\)](#)
- [Maxell \(14\)](#)
- [Memorex \(1\)](#)
- [No Name \(1\)](#)
- [Orwo \(6\)](#)
- [Permaton \(2\)](#)
- [Philips \(7\)](#)
- [Quantegy \(4\)](#)
- [Revox \(4\)](#)
- [Revue \(1\)](#)
- [Scotch \(6\)](#)
- [Shamrock \(2\)](#)
- [Sony \(5\)](#)
- [Tandy \(1\)](#)
- [TDK \(3\)](#)
- [Telefunken \(1\)](#)
- [Uher \(1\)](#)
- [Universum \(1\)](#)
- [Vendomatic \(1\)](#)
- [Veritone \(1\)](#)
- [Yukami \(1\)](#)

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Sony

Sony PR-150 (18 cm)

- [PR-150 \(18 cm/ 7"\)](#)
- [PR-200 \(18 cm/ 7"\)](#)
- [SLH \(18 cm/ 7"\)](#)
- [Ferrichrome \(18 cm/ 7"\)](#)
- [Ferrichrome \(26 cm/ 10 1/2"\)](#)



Marke Brand	Typ Type	Herkunftsland Origin	Spulengröße Reel Size	Spulentyp Reel Type	Verpackungstyp Package Type
Sony	PR-150 (18 cm)	Japan	18 cm/ 7"	Dreizack-Aufnahme, Kunststoff Three-winged hub, plastic	Pappschachtel/ Cardboard Box
Bandlänge Tape Length	Banddicke Tape thickness	Rückseitenbeschichtung Back coating	Angeboten von/ bis Distributed from/ until	Hinzugefügt Added on	Supplied by Zur Verfügung gestellt von
540 m/ 1,800 ft	35 Mikron (Langspiel) 35 microns (long play)	Nein/ No	-?-	08.05.05/ May 08, 2005	Franz Munsch
Kommentar Comment					
-					

APPENDIX 6

Migration Project Statistics on U-Matic Playability

(Source: confidential)

Bilan de performance des différentes bandes U-matic numérisées.							
<i>Fabricants</i>	<i>Quantité totale</i>	<i>Nombre d'échecs</i>	<i>Pourcentage d'échec (%)</i>	<i>Moyenne niveau de RF</i>	<i>Moyenne nombre de prélecture</i>	<i>Rapport bande passante: Normale/Réduite</i>	<i>Qualité visuelle (%)</i>
Agfa	287	68	23.693	4.244	2.040	0.187	96,8% B 1,36% M 1,81% P
Scotch	336	13	3.869	3.700	2.191	0.177	97% B 3% P
PYRAL	36	1	2.778	2.613	2.895	0.156	97,22% B 2,78% P
SONY	3600	40	1.111	3.388	2.211	0.293	97,09% B 0,67% M 2,24% P
BASF	1267	4	0.316	2.632	1.851	1.053	98,57% B 0,48% M 0,95% P
FUJI	232	0	0.000	3.609	2.021	1.648	99,14% B 0,86% P
3M	108	0	0.000	3.829	2.054	0.009	100% B
AMPEX	23	0	0.000	4.361	2.042	0.263	100% B
KODAK	4	0	0.000	4.100	2.000	0.333	100% B

APPENDIX 7

Tape Binder Mass Distribution Analysis by Size Exclusion Chromatography (SEC)

Objectives

The chain length (number of repeating monomers) of a polymer is strongly related to its mechanical properties. Quantitative analysis of the chain length distribution (equivalent to molecular mass distribution) and its evolution upon ageing allows us to understand properties-structures relationships of the tape binder.

Experimental procedure overview

Approx. 0.2 g of tape are extracted with approx. 2mL of HPLC grade Tetrahydrofuran (THF) at room temperature for 2 hours in screw capped glass vials set on an automatic shaker.

The solvent is then filtered through a 0.2 μm porosity PTFE syringe filtering unit with a glass syringe. 10 μL of the sample is eluted on a series of 3 Phenogel SEC columns (porosity ranging from 10⁶ to 50A^o porosity) at a flow rate of 1mL/min HPLC Grade Tetrahydrofuran. The detector used is a Differential Refractometer. The system is calibrated with PolyStyren (PS) SEC standards between 400 000 g/mol and 1100 g/mol. 3 linear fatty acid methyl esters are used as mass standards at 354 g/mol, 270 g/mol, 186 g/mol.

The chromatograms are normalized to 0.2g / 2mL THF and integrated following the procedure presented in section B11.3.1.

Artificial ageing procedure is given in appendix 12.

Results & Discussion overview

More than 200 analyses have been carried out on $\frac{3}{4}$ Umatic tapes from various brands and ages. The results show three typical chromatographic profiles (*fig. 1*). *Shape A*: most of the macromolecules have a weight in the 50 000 g/mol eq. PS ; *Shape C*: most of the macromolecules have weights in the 1000 g/mol eq. PS Domain ; *shape B*: macromolecules weights are in the intermediate domain.

These large discrepancies in mass distribution indicates large differences in binder chemical properties and consequently in overall mechanical properties

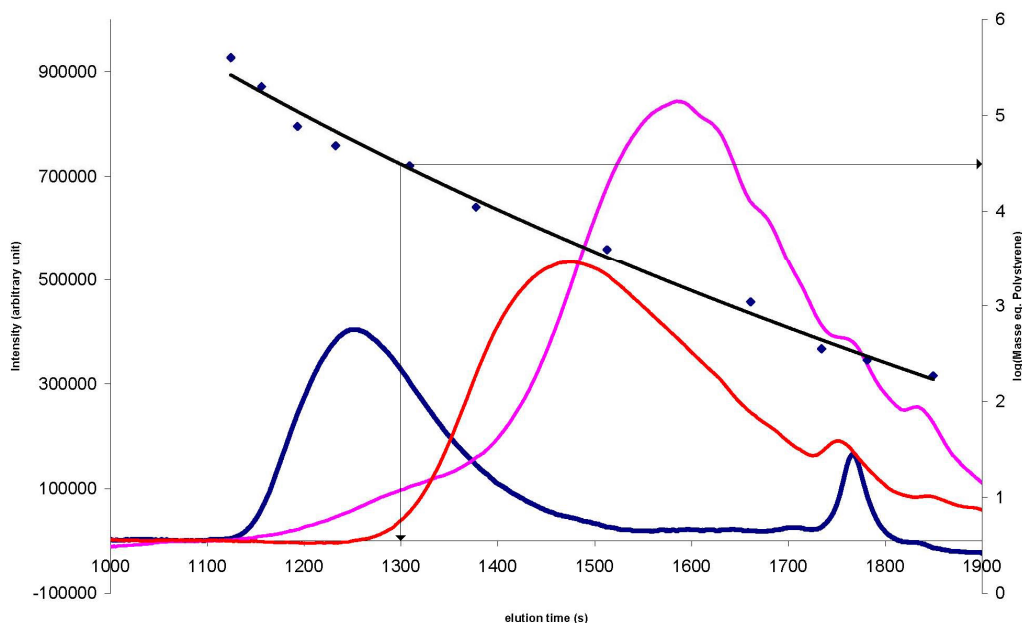


Fig. 1 Typical GPC Chromatograms: Blue: shape A; Red: shape B; Pink: shape C; Black: Calibration curve

Artificial ageing in high humidity level (100% RH) and hot temperature (75°C) were applied on tapes having all three chromatographic shapes (All tapes tend to show the same shape C upon ageing when submitted to conditions that promote hydrolysis. It can be supposed that the mass distribution goes from chromatographic shape A to C upon ageing at various speeds. Consequently, the time to reach a highly hydrolysed binder state (shape C) in a given artificial condition allows hydrolytic stability comparisons between PUR-ES technologies. Tapes with chromatographic shape C before artificial ageing might already have been highly hydrolysed during their storage period. It can be noticed that for longer ageing time, peaks corresponding to small fragments appears. The formation of product with specific masses demonstrates that end-product are produced (products that are not affected anymore by hydrolysis). These products may correspond to the monomers used for PUR-ES manufacturing. Appendix 18 shows the formation of white crystals of adipic acid (one of the widely used basic molecule) that are exuded from the tape surface upon artificial ageing.

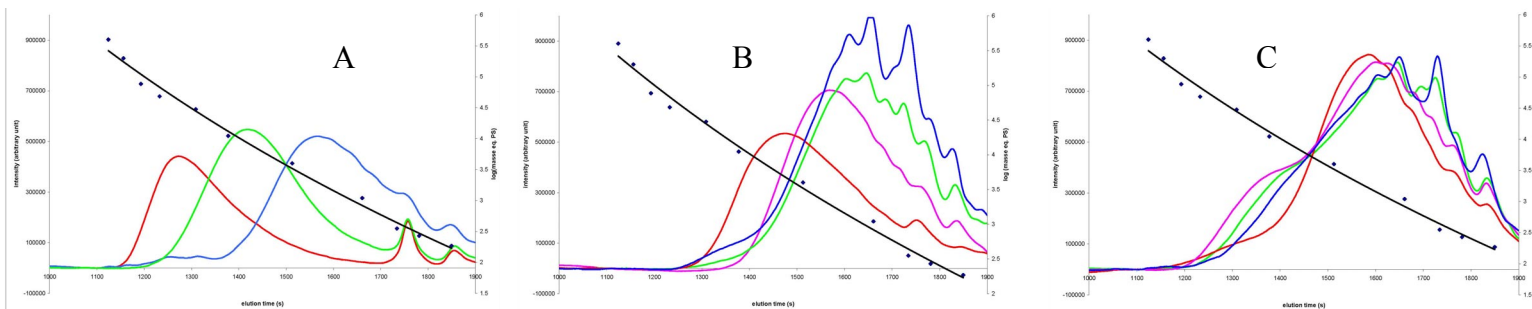


Fig. 2 GPC Chromatogram evolution; ageing at 100%RH, 75°C ; shape A, shape B, shape C after 0 day (red), 14 days (pink), 25 days (green), 49 days (blue)

Conclusion overview

Further studies on the relation between polymer structure and playability of the tape, would make SEC characterisation an efficient method to evaluate the deterioration state of the binder. Stability study with SEC and artificial ageing (Appendix 12) would allow evaluation of deterioration susceptibility of the binder. Short term risks endured by tapes stored in a non-controlled environment could as well be studied. Storage or manufacturers recommendations concerning tapes having hydrolytic stability could be determined.

APPENDIX 8

Identification of Volatile Organic Compounds Emitted by Naturally and Artificially Aged Polyester-Urethane Magnetic Tape Binders

Objectives

Some naturally aged tapes are giving off strong odours when handled after a long storage period. Odours are caused by volatile molecules present at given concentrations in the air surrounding the tape. Characterization of all VOC and their sources is required to identify the odour causing VOC. Because odour formation seems to be related to tape deterioration, chemical marker for deterioration assessment may be found among them. Hydrolytic artificial ageing will be carried out in order to accelerate and emphasize the release of volatile degradation products.

Experimental procedure overview

VOC are extracted and concentrated by Headspace Solid Phase Micro Extraction (SPME) then injected into a Gas Chromatography coupled with Mass Spectrometry (GC-MS) device for separation and characterization. SPME fibres coated with a polymeric blend of Divinylbenzene / Carboxen / Polydimethylsiloxane were used. 30 cm of tape is introduced in 20 mL glass vial hermetically closed with a PTFE septum. After a 3 min nitrogen purge applied through the septum with two syringe needles the vial was heated to 60°C for 5 minutes prior to introduction of a preconditioned SPME fibre mounted on a special fibre retractable holder. After 1 hour of extraction at 60°C, fibre is quickly transferred into the injection port of the GC-MS. Analytical conditions are: Splitless Injection port temperature: 230°C, Column CPSil 8, Constant Helium flow of 1mL/min Oven temperature program: 10 min at 35°C, 5°C/min ramp to 270 °C, 20 min isothermal hold at 270 °C. Transfer line temperature: 280 °C, Quadrupole Mass spectrometer in electron ionisation mode (EI) at 70eV scanning from m/z 39 to m/z 600 in 500ms.

Results and discussion overview

Table 1 presents the VOC identified for U-matic tapes (approx. 100 tapes from various brands and ages). The suspected origins are proposed on the basis of the chemical characterization and the common use of the characterized products.

These products are emitted exclusively by the magnetic layer and/or the backcoating.

The aliphatic series of acids and lactones (Figure 2) are emitted in large quantities by some tapes. These compounds are the major compounds extracted at room temperature and comparison to the pure products commercially available, show that they are responsible for the main part of the odour.

A strong smelling tape is submitted to advanced testing to investigate the origin of the series of acids and lactones.

The magnetic layer of the tape has been removed in acetone and washed several times. This procedure left the magnetic coating clean and chromatographic analysis shows no more VOC after cleaning. This clean magnetic coating then undergoes artificial ageing. Samples are introduced in hermitically sealed glass vial at inner conditions of 100%RH 75°C and 10%RH, 75°C. The glass vials are periodically submitted to SPME/GC/MS analysis.

VOC identified from U-Matic tapes	
Pentanoic acid	Deterioration products of some tape binders
Hexanoic acid	
Heptanoic acid	
Octanoic acid	
Nonanoic acid	
Decanoic acid	
4-Ethyl-butylolactone	
4-Pentyl-butylolactone	
4-Hexyl-butylolactone	
4-Heptyl-butylolactone	
4-Octyl-butylolactone	Probable deterioration products of some binders
4-Nonyl-butylolactone	
4-Propyl-butylolactone	
Heptanal	
Octanal	Lubricants
Nonanal	
Decanal	
Benzaldehyde	
Palmitic acid butyl ester	Antioxidants or product of deterioration of antioxidants
Myristic acid butyl ester	
Stearic acid butyl ester	
Lauric acid butyl ester	Unreacted precursors of binders or by-products
2,6-Di-tert-butylbenzoquinone	
Benzoquinone	Solvents introduced during the tape manufacturing
Butylated Hydroxytoluene (BHT)	
1,6 dioxacyclododecanone	
3 propylglutaric acid	
Cyclohexanone	
Butanone	
Toluene	
Styrene	

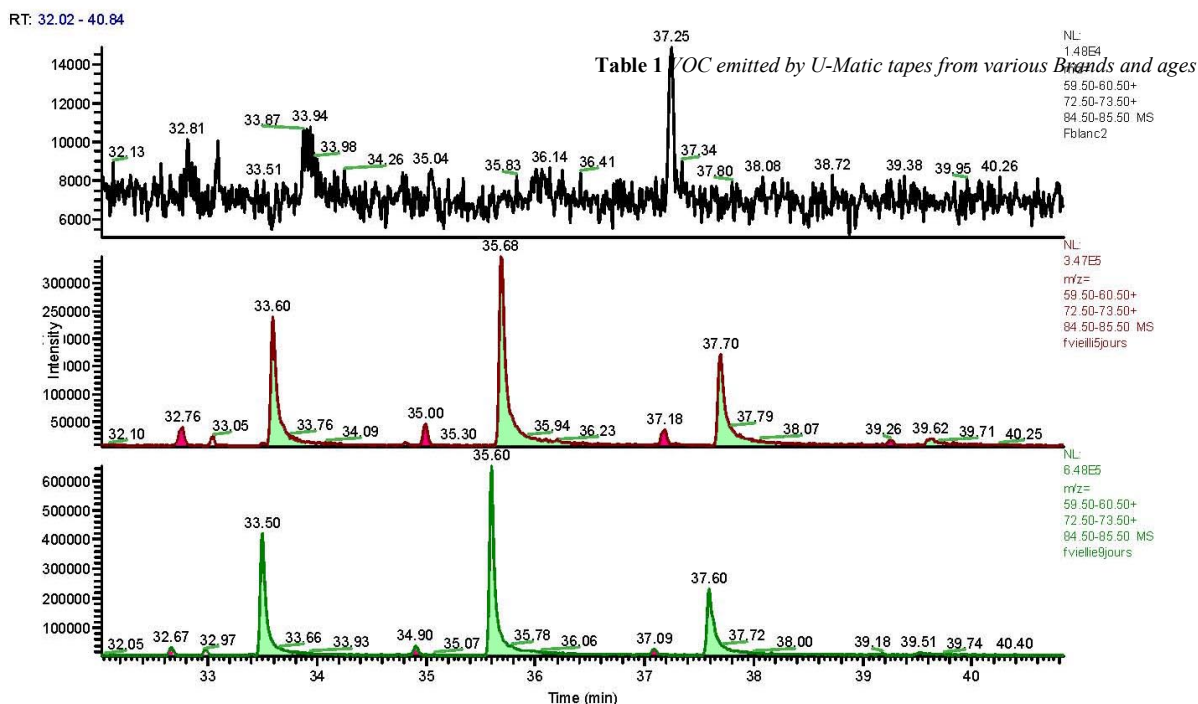


Figure 1. SPME/GC/MS chromatograms of ions with m/z 73; 60 and 85 extracted from the TIC for clean magnetic coating exposed at 100% RH, 75°C during 0 (up), 5 and 9 days (bottom). [carboxylic acids (green), lactones (red)]

Figure 1, shows the chromatograms at different ageing times (100% RH, 75°C) of the ions m/z 60 and 73 which are characteristic of the carboxylic acids and of the ion m/z 85 which is specific to the series of identified lactones.

An increase in peak areas for the lactone series and acids series occurs upon ageing.

Figure 3 shows the SMPE/GC/MS TIC of the clean magnetic coating after 15 days of artificial ageing in humid conditions (100% RH, 75°C) and low RH conditions (10%RH, 75°C). Some VOC were produced in both conditions, but the intensity of the TIC for dry conditions is 5 times less.

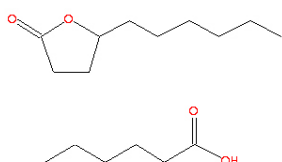


Figure 2 Structure of 4-Hexyl-Butyrolactone(upper), hexanoic acid (lower)

Conclusion overview

Further studies are required to understand the mechanisms of aliphatic acids and lactones production. As these products are easily detectable, if their impact on tape playability is shown, they could be an efficient chemical marker to assess the tape condition.

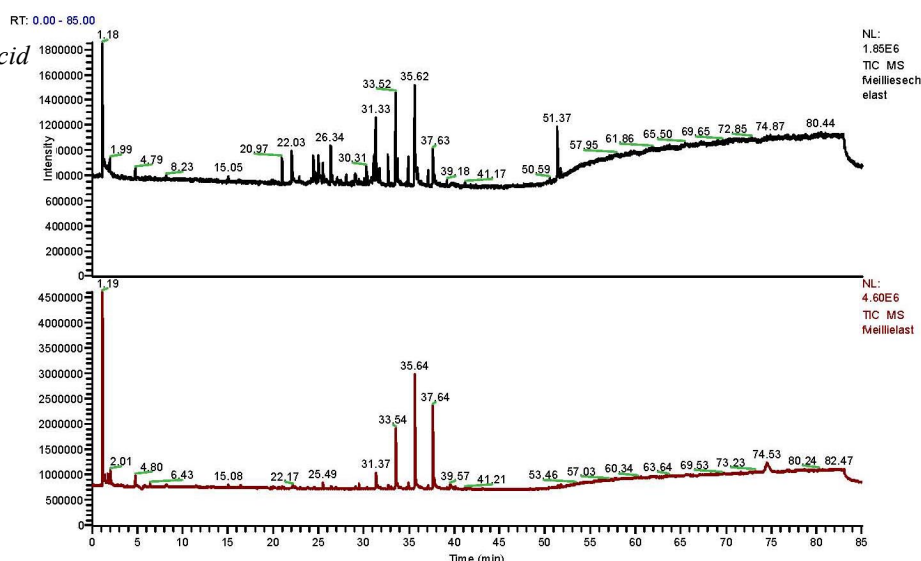


Figure 3 SPME/GC/MS TIC chromatograms of clean magnetic coating after 15 days of artificial ageing at 10% RH, 75°C (up), 100% RH, 75°C (bottom).

APPENDIX 9

Surface Acidity Evaluation for Magnetic Coating by Contact Angle Titration

Objective

The environmental hydrolytic deterioration of polyester-urethane PUR-ES leads to ester bonds cleavage by water, increasing the number of acidic and alcoholic functions on cross-linked polymers chains. In addition, ester-type lubricants present in magnetic coating for most U-Matic tapes are also prone to degrade by hydrolysis leaving the related fatty acids in the binder matrix. These acidic functions are not readily extracted in water, consequently usual pH measurement by glass electrode are not suited to evaluate magnetic coating acidity. An original approach by contact angle titration may overcome water extraction problems for surface acidity measurement. The method is based on acid-base interactions of the tape surface with acid aqueous droplets deposited on it. These interactions lead to drop-shape variations evaluated by the contact angle measurement.

Experimental procedure overview

A stock solution of 20 mL NaCl (0.01M) is decarbonated by nitrogen bubbling for sufficient time to get a neutral pH controlled by glass electrode. Acidification (alkalinisation) of the solution is obtained by addition of calculated volumes of HCl 0.1M (NaOH 0.1M) under constant nitrogen bubbling and pH glass electrode monitoring.

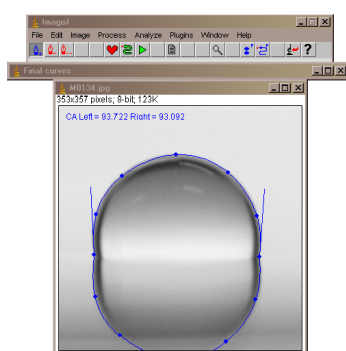


Figure 2 Contact angle measurement software

After each addition and pH stabilisation, a 10 μ L glass syringe is rinsed and filled with the solution. The syringe is quickly installed on the measurement system (fig. 1). A first drop is suspended to the syringe needle to allow camera alignment and focus optimisation, and then the drop is discarded. A 1 μ L drop is suspended manually to the syringe needle, the sample is placed under the needle tip not allowing the drop to touch the tape surface. The syringe is then lowered slowly to transfer the suspended drop onto tape surface. The picture of the drop formed on the surface is quickly taken. One syringe allows 10 measurements. The tape sample was conditioned at 50%RH and 23 °C for one night before measurement. Each measurement has been made 4 times.



Figure 1 Contact angle measurement system

The pictures for all pH values are processed afterward with measurement software able to measure contact angles. (fig. 2) The repetition error on the angle value was less than 0.5%.

Results and discussion overview

A coherent titration curve can be obtained (fig. 3) with a contact angle drop for a droplet pH of 10.6 which correspond to an equivalent acidity of $4 \cdot 10^{-4}$ mol per "droplet contact area". For accurate measurement, accurate contact area measurements have to be done. Pictures from the deposited droplet taken in parallel plan above the drop area can be used. Evaluation of the area by mathematical approximations on the basis of the contact angles measurement can also be made.

Results after artificial ageing (not shown here) show a significant modification of the curve shape and a shift of the equivalent point, but no accurate measurement of the shift have been possible due to poor data quality.

Conclusion overview

This technique is satisfactory but is still highly experimental and has to be improved to be fully operational for large number of samples.

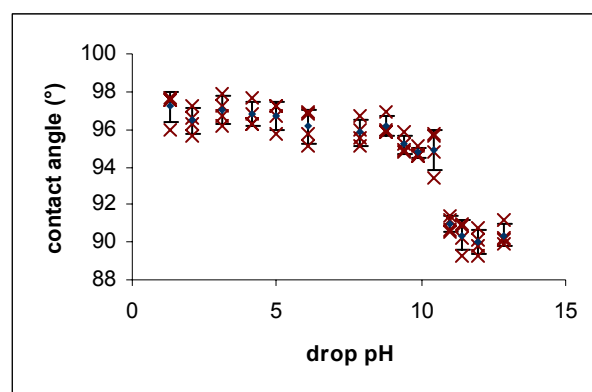


Figure 3 Contact angle titration curve for a U-matic magnetic coating.

APPENDIX 10

Head-Clogging Study by High Speed Imaging

Objective

Head-clogging is the most common problem for archival magnetic tape playback and consequently trouble with access to the recording. This deterioration symptom does not affect the recorded information itself, but the access to the recorded document. Tape restoration specialists can often deal with light head clogging by advanced tape cleaning or multi pass digitisation. However, migration project at a large scale, cannot afford any stop in the digitisation process for human intervention. In addition, severe head-clogging can not be fixed. A device built by the CRCDG allows to take pictures of the moving reading-heads of a helical tape player with a stop-motion method while playing archival tapes. At a rate of 5 frames per second, a stop-motion video can be recorded. Clogging materials build-up can be observed and related to document play back quality.

Experimental procedure overview

A conventional U-Matic Tape player has been modified to allow the synchronization of a strobe light to the drum revolution frequency. The conversion of the drum motor supply voltage into a TTL signal is performed thanks to a especially designed electronic controller. The controller features an adjustable phase delay to allow observation of different parts of the drums. To avoid overheating of the light bulb, one flash over 5 revolutions is applied. A CCD Camera with a 55mm macro lens aiming at the drum is used to observe the head. The CCD video camera is set at an exposure time of $\frac{1}{4}$ s and is operated in a dark room to avoid blinking video.

Analysis of the deposited materials were performed by microsampling and Infrared Spectroscopy.

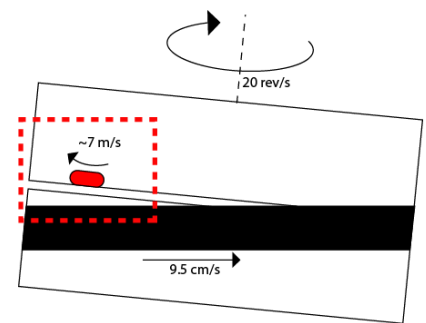


Figure 1 U-Matic player rotary (upper) part and stationary (bottom) part of a drum. In black: magnetic tape wrapped on the drum around the drum. In red: reading head, Red dot-lined Square: area observed by the CCD.

Results and discussion overview

A video sample showing a reading head being clogged while playing an AGFA tape from the '80s is available upon request. Suspected lubricants were identified by GC and FTIR spectroscopy.

Testing performed for different magnetic tapes tend to demonstrate that in most cases lubricants build up is the main cause of head clogging. The device allows to observe and detect early and very light head clogging before the play back quality is affected.

It can be noticed that when the deposited is transferred at the rear of the head back to the tape surface when its size is too large to fit the gap between the head and the tape. Consequently it can be supposed that these large particles remain between the tape wraps when the cassette is stored. Head-clogging can be regarded as a cause of depletion and overloading of lubricants observed at different spots on the tape surface.

Manufacturers used to produce tapes with well dissolved lubricants in the magnetic layer. But upon storage, it seems that lubricants migrate to the tape surface as their properties may change or as a consequence of polymer matrix ageing to water uptake increased.

Conclusion

The device developed at the CRCDG allows observation of head-clogging formation while playing back an archival tape. Comparative studies can be performed to quantify the head-clogging ability for all tapes. In addition, effect of restoration practices (baking, dry cleaning, and wet cleaning) or the effect of player settings (tape tension) on head-clogging can be evaluated.

APPENDIX 11

Magnetic tape Friction Tester

Objective

When a tape is played back onto a given tape-path, friction is required to allow tape movement and alignment, but also have to be reduced for head to tape contact and drum sliding. Most of the magnetic tape deterioration symptoms can be related to friction problems through incorrect lubrication, local overheating and wear. In addition, concerning the environmental ageing of materials, surfaces are the most affected. The friction of two sliding surfaces is controlled by many different and complex parameters related to the mechanical properties and the chemical properties of the surfaces. Thus determination of friction coefficient is essential to monitor when studying deterioration mechanisms of archival magnetic tapes.

Experimental procedure overview

Pictures and schematic representation of the friction tester built by the CRCDCG together with the INA A/V maintenance department can be found on section A2.5.1 of this report. A loop of approx. 1.55 m of magnetic tape is set with the magnetic layer side in contact with a friction cylinder. A U-Matic tape player rotary drum serves as friction cylinder. A moving tape guide mounted on a lever with an adjustable weight that allows tape tension regulation. A capstan and a motor are used to run the tape at various speeds. The tension is recorded before and after the friction cylinder along the tape loop. The tension sensors are identical as those used for tension control in some U-Matic tape players. Friction is calculated from the difference between the outputs of the two tension sensors and tests showed a linear response for a tension weight ranging from 0 to 100g.

Results and discussion overview

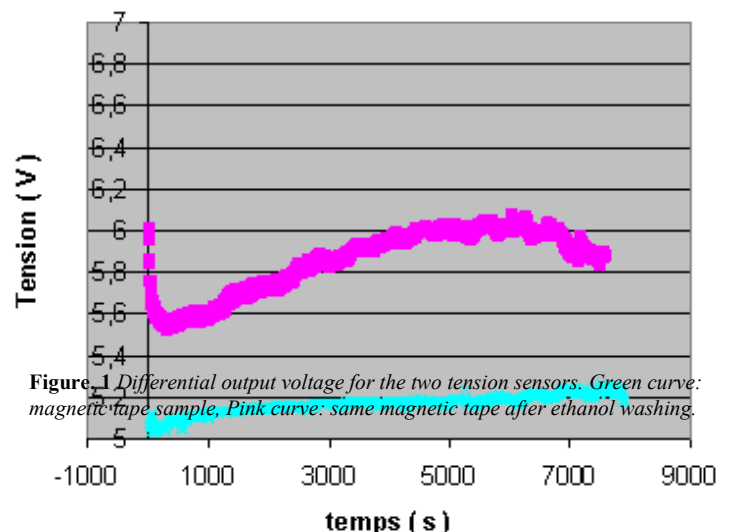
The overall system have been tested for sensor noise, sensor sensitivity and tension regulation efficiency and some improvements have to be made mainly on the tape tension measurement. A poor repeatability was obtained. This result may be related to the measurement system or variation of the friction for the same cassette but for different lengths of tape. Each measurement requires a new tape sample.

However, coherent results were obtained for comparative evaluation of different friction values.

Figure 1. The differential output voltage recorded for a given magnetic tape containing lubricants (green) and after removal of lubricants (pink) by washing the tape with ethanol. Note that the differential voltage output is higher for tape without lubricants meaning a higher measured friction.

Conclusion

More developments have to be done to use the friction tester in a magnetic tape deterioration study. Sensor replacement by commercially available force sensors is an option.



APPENDIX 12

Hydrolytic Stability Study of Magnetic Tape Binder

Objective

For most of magnetic tapes, the magnetic layer is made of magnetic particles dispersed in a polymeric binder and coated onto a base film. The binder is generally a polyurethane-ester. This type of polymer includes a wide range of materials having different structures (foam, flexible, rigid), different mechanical and chemical properties. This versatility originates from the numerous chemical compounds available for polyurethane-ester manufacturing. Consequently, this explains the different stability of tape binders when they are exposed to humidity. The stability to artificial ageing of tape binder allows the evaluation of the hydrolytic stability of the magnetic layer.

As explained in appendix 7, the relevant parameter to measure in a study of the polymer hydrolytic breakdown is the macromolecules weight distribution.

Experimental procedure overview

The analysis of the macromolecular weight distribution is described in detail in appendix 7. Ageing testing was achieved for 100% RH and 75°C in hermetically closed glass vessels. Humidity was kept constant thanks to a small glass vial filled with distilled water, uncapped and inserted within the ageing vessel. 0.2 g of tape was inserted and the deterioration vial was sealed with a Silicon/ Teflon septum. An oven with precise temperature regulation was used to maintain the deterioration vessel at 75°C. No loss of water was observed. After a period of time, the vial was unsealed and the tape was allowed to dry up before analysis by Size Exclusion Chromatography (SEC). This was repeated for different periods of time (up to 78 days)

At the same time, some investigations tend to demonstrate that the extractable fragments are extracted from the polyurethane-ester binder and not from the polyester base film.

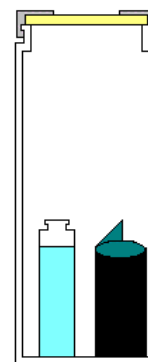


Figure. 1 Ageing vessel

Results and discussion

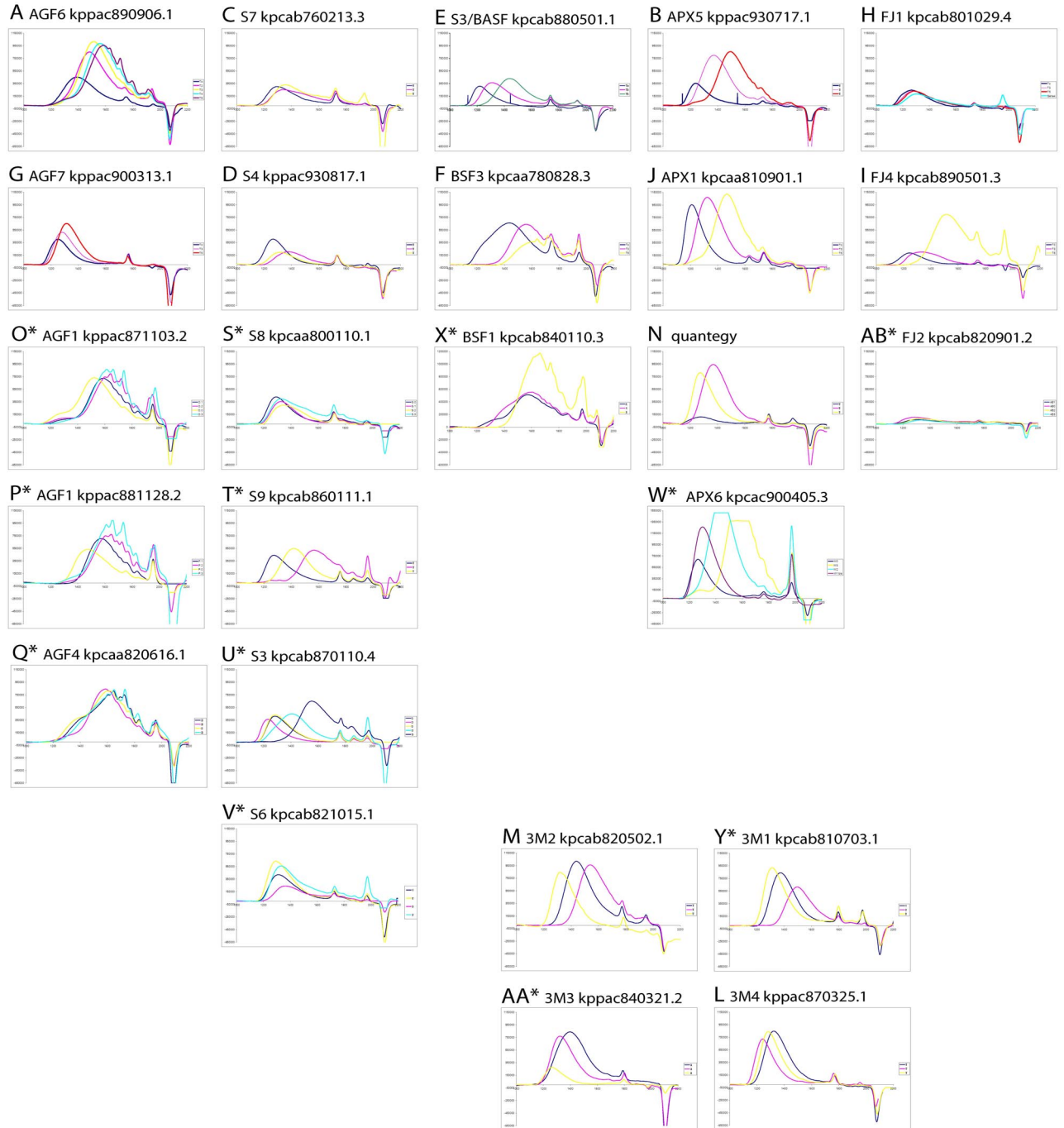
The results of the ageing experiments are presented in the next page. The information above each series of chromatograms corresponds to the cassette identifier in the lab (Letter), the group identifier according to serial number code (Appendix 14), the INA reference of the recording.

As suspected the different binder systems have different hydrolytic stabilities. Some binders are almost not affected by hydrolysis breakdown (H, AB, G...), others are far less stable (A, U, B...).

Conclusion

The comparison of the testing results for each group of tapes allows the evaluation of the hydrolytic stability of the different binder systems used by manufacturers to produce tapes from a same group. A quantification method for hydrolytic stability evaluation and comparison is proposed in section B11.3.1.

SEC ANALYSIS RESULTS



X : 100%RH, hermetique, 75°C ; chrom. norm. 0,2g/2mL ; T1 : 4j, T2 : 9j, T3 : 16j, T4 : 23j, T5 : 36j, T6 : 49j, T7 : 66j, T8 : 78j ;
 X* : 100%RH, hermetique, 75°C ; T1 : 14j, T2 : 25j, T3 : 49j

APPENDIX 13

Infrared Spectroscopy for Polyurethane Characterization

Objective

For most magnetic tapes, the magnetic layer is made of magnetic particles dispersed in a polymeric binder and coated onto a base film. The binder is generally a polyurethane-ester. This materials family is large and covers a wide range of polymers having different structure (foam, flexible, rigid), different mechanical and chemical properties. The versatility comes from the numerous available starting chemical compounds for polyurethane-ester manufacturing. As a consequence, polyurethane-esters have different stabilities when exposed to humidity. The identification of the exact nature of the polyurethane-ester used by manufacturers for different tape products would allow us to build groups of tapes with similar binder composition and so similar hydrolytic stability.

Instead of characterizing the binder molecules, this study aims to compare “infrared spectroscopy fingerprints” for each manufacturer in order to categorize tapes with identical binders. The tapes having the same fingerprint are likely to have the same deterioration susceptibility.

In addition, this categorization method supports the definition of the “technological families” that is a fundamental concept used in the section B of this report.

Experimental procedure overview

60 cm of tape is inserted into a 15mL glass vial. Approx 10mL of acetone (technical grade) is added and the vial is placed on an automatic shaker overnight. The solution is filtered on a paper filter and the solid particles and tape remains are rinsed twice with 10 mL of acetone (HPLC grade). The tape fragments are inserted back into the vial and 5 mL of tetrahydrofuran (HPLC Grade kept as dry as possible over molecular sieves) is added. The vial is placed on the automatic shaker for 1 hour. The solvent is then filtered with a PTFE filter syringe unit of 0.45µm of porosity mounted on a glass syringe. The resulting solution is poured into a 15 mL glass vial. The solvent is dry evaporated under nitrogen at constant room temperature.

The dry deposit is redissolved into 1.5 mL of tetrahydrofuran (HPLC Grade). The resulting solution is spread onto 0.5 g of dry KBr crystals and ground in an agate mortar until total evaporation of the solvent. (This operation is performed under a laboratory hood). The powder is then inserted into the KBr pellets die. The die is air vacuumed for 5 minutes before 10T pressure is applied for 10 minutes under constant vacuum. The pressure is released and the pellet is mounted on a pellet sampling card for analysis by transmission infrared spectroscopy by Fourier Transform Infrared Spectroscopy (FTIR). The resultant spectrum is added to the spectrum library for further comparisons.

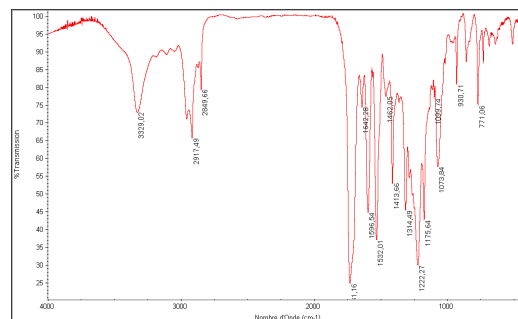


Figure 1 FTIR spectrum of a magnetic tape THF extract (KBr pellet method)

Results and discussion overview

A polyurethane-ester spectrum obtained for a U-Matic tape is given in figure 1. Only small differences exist between two spectra of two slightly different binders.

The automatic comparison method provided by the FTIR software has been used. The correlation index between all spectra from the library has been calculated. These values are shown in Figure 2. A red cell indicates a high correlation index whereas lower correlation values range from red to green. The wide coloured area tends to demonstrate that similar polyurethane-esters have been used. AGFA, AMPEX and BASF tapes are similar with the exception of the “agfa kppac930820.1” and “basf kpcab910124.5”. Indeed, these two tapes form clear lines within the correlation chart.

However, precautions have to be taken due to the correlation index calculation method that takes into account the overall intensity of the Infrared bands. This intensity differs from tape to tape due to different extraction protocol efficiencies. As a consequence, the correlation index calculated does not represent only the fingerprints similarity.

##	78	84	89	53	65	65	85	69	83	82	84	52	38	28	74	31	36	69	69	76	38	agfa kpcaa	820616,2	
78	##	75	76	42	63	56	53	67	69	68	60	61	46	49	20	59	19	32	68	54	53	32	agfa kppac	810928,1
84	75	##	83	65	81	81	81	81	84	87	81	84	50	37	27	73	29	35	86	67	72	38	agfa kppac	910613,1
89	76	83	##	50	71	80	81	79	84	84	71	74	59	49	32	76	37	47	88	67	73	36	agfa kppac	920212,1
53	42	65	50	##	47	44	47	61	51	49	46	49	33	25	25	44	16	22	46	55	45	22	agfa kppac	930820,1
85	63	81	71	47	##	81	69	82	84	83	72	80	40	26	16	54	27	32	68	48	53	19	ampex kpcaa	810901,1
85	56	81	80	44	81	##	98	74	87	83	68	80	33	25	25	63	20	27	77	47	64	15	ampex kpcaa	810901,2
85	53	81	81	47	69	89	##	83	84	84	73	80	41	26	17	63	27	32	78	54	66	20	ampex kppac	870502
89	67	81	79	61	82	74	83	##	81	87	87	92	50	35	21	71	32	36	82	62	73	28	ampex kppac	870902
83	69	86	84	51	84	87	84	91	##	91	88	89	48	33	27	71	30	36	87	63	69	28	ampex kppac	910901,2
82	68	87	84	49	83	83	84	87	91	##	84	87	51	34	28	73	36	39	87	67	74	35	basf kpcaa	780208,3
82	60	81	71	46	72	66	73	87	88	84	##	91	46	28	30	59	34	37	74	61	84	27	basf kpcab	860110,4
84	61	84	74	49	80	80	80	82	89	87	81	##	49	28	24	64	41	40	77	66	74	30	basf kpcab	860111
52	46	50	59	33	40	33	41	50	48	51	46	46	##	55	40	72	52	50	60	63	54	39	basf kpcab	880501
38	49	37	49	25	26	25	26	35	33	34	28	28	55	##	42	53	31	46	42	47	20	32	fujii kpcab	801029
28	20	27	32	25	16	25	17	21	27	28	30	24	40	42	##	56	38	35	42	57	32	59	fujii kpcab	820901,1
74	59	73	76	44	54	63	63	71	71	73	59	64	72	53	56	##	50	51	82	71	69	65	fujii kpcab	861204,3
31	19	29	37	16	27	20	27	32	30	36	34	41	52	31	38	50	##	82	42	65	53	29	fujii kpcab	890501,3
36	32	35	47	22	32	27	32	36	36	39	37	40	50	46	35	51	82	##	44	59	44	26	fujii kpcab	910124,5
81	66	86	88	46	68	77	78	82	87	87	74	77	60	42	42	82	42	44	##	76	78	44	scotch kpcaa	790118,2
69	54	67	55	48	47	54	62	63	67	61	66	63	47	57	71	65	59	76	##	82	55	55	scotch kpcab	761031,1
76	53	72	73	45	53	64	66	73	69	74	64	74	54	20	32	69	53	44	78	82	##	46	scotch kppac	870325,1
38	32	38	36	22	19	15	20	28	28	35	27	30	39	32	59	65	29	26	44	55	46	##	sony kpcab	821015,1
820616,2	agfa kpcaa																							
810928,1	agfa kppac																							
910613,1	agfa kppac																							
920212,1	agfa kppac																							
930820,1	agfa kppac																							
810901,1	ampex kpcaa																							
810901,2	ampex kpcaa																							
870502	ampex kppac																							
870902	ampex kppac																							
910901,2	ampex kppac																							
780208,3	basf kpcaa																							
860110,4	basf kpcab																							
860111	basf kpcab																							
880501	basf kpcab																							
801029	fujii kpcab																							
820901,1	fujii kpcab																							
861204,3	fujii kpcab																							
890501,3	fujii kpcab																							
910124,5	fujii kpcab																							
790118,2	scotch kpcaa																							
761031,1	scotch kpcab																							
870325,1	scotch kppac																							
821015,1	sony kpcab																							

Figure 2 Correlation factor table for different U-Matic tape binder IRTF spectra

Conclusion

Even if this comparison method seems to be promising, extra investigations are required to optimise the solvent extraction protocol, the normalization of the spectrum intensity. A suitable correlation method would probably have to be develop for this specific FTIR application.

APPENDIX 14

Serial Number Code for U-Matic Tapes

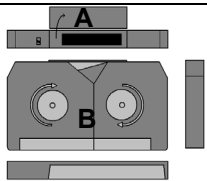
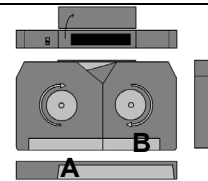
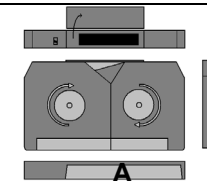
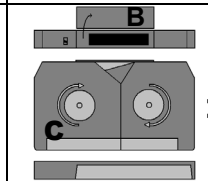
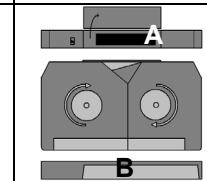
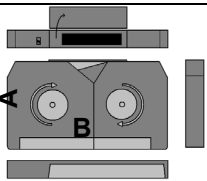
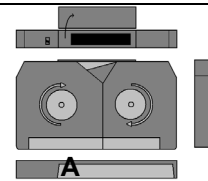
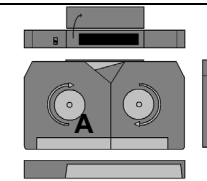
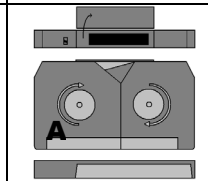
Objective

The only method to identify visually and definitively a tape on-shelf is the serial number code given by manufacturers and printed on the cassette itself. This code express either a unique serial number for the cassette, a manufacturing date or a batch number unique for series of tapes. However, because these codes and the printing patterns (colour, position, fonts...) are related to one production period, one production place it can be used as a marker of production practices, materials formulation.

Some restrictions exist for the use of that serial number for tape full characterization: poor quality control policy, great and frequent variability in the materials and manufacturing practices.

Results

The following table summarize the different locations of the serial number code on U-Matic cassettes according to the manufacturer. Some cases may miss.

AGFA		AMPEX		QUANTEGY		BASF		SCOTCH/3M	
									
A:	Sticker under the cassette door	A:	Blue ink often under the corner of the cassette sticker Or White or yellow ink on grey or black colored cassette	A:	Under the sticker or next to it	A:	Sticker. Sometimes double coded on C position (black ink)	A:	Sticker on the cassette under the cassette door
B:	Yellow ink on white cassette	B:	Sticker			B:	Sticker under the cassette door	B:	Red ink often under the cassette sticker
FUJI		KODAK		SONY		PYRAL			
									
A:	Black ink	A:	Black ink	A:	Black or white ink	A:	White ink		
B:	Black ink								

APPENDIX 15

Comparison of Analytical Fingerprints (magnetic tape acetone extracts)

Objective

In order to quickly identify tapes with the same materials formulation, an ‘analytical fingerprints method’ was developed and more than 200 tapes, considered as representative of all manufacturers and of the whole manufacturing period, were analysed and compared.

Experimental procedure overview

30cm of U-matic tape sample is cut into small pieces and inserted into a 15 mL glass vial. Approx 5 mL of acetone (SPECTRO GRADE) is added. The vial is then capped. The inner part of the cap is protected by an aluminium foil to prevent any contamination from the plastic cap. The vial is mounted on an automatic shaker for 2 hours at room temperature. The solvent is then filtered with a PTFE syringe filter unit (porosity 0.2 µm) mounted on a glass syringe. The filtered solution is transferred into two 3 mL glass vials for analysis. An automatic GC sampler is loaded with the vials.

The GC-FID analysis is performed on a FFAP J&W 30m capillary column under a constant flow rate of 1mL/min. The injector temperature is set at 250°C in 1/20 split mode, the FID detector is set at 270°C. The oven temperature is kept constant at 125°C for 10min and then a heating rate of 10°C/min is applied until 250°C. The temperature is kept constant for 15min.

Results and discussion overview

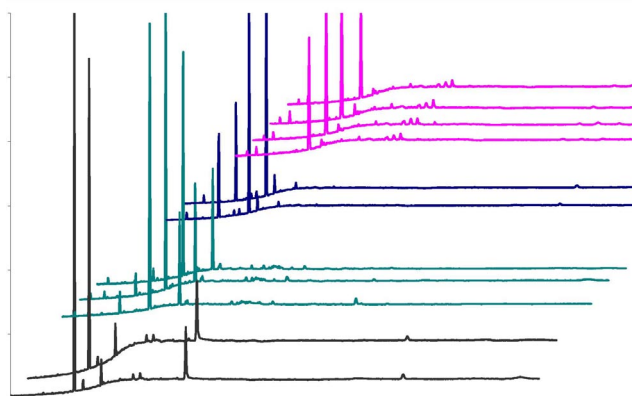


Figure 1. 3-dimensional overlay of the chromatograms for 11 Sony tapes produced between the ‘80s and the ‘90s. Colours correspond to highly similar chromatograms

The comparison of the chromatograms from tape acetone extracts allows identifying tape chemical contents. These chromatograms are dependent of the additives, the residual manufacturing solvents, un-reacted manufacturing products, and impurities. These chromatograms can be considered as “analytical fingerprints” and are representative of a given set of manufacturing practices.

Differences observed are different products (different retention times) or differences in relative concentration. Figure 1 shows comparison of different analytical fingerprints for Sony tapes. 4 different groups can be made according to 4 sets of similar patterns. There is no perfect match between chromatograms due to manufacturing variability and past storage conditions.

Among the extracted products, the analytical fingerprints depend mainly on the additives (the biggest chromatographic peaks). The dependence of the analytical fingerprint towards the binder system which is poorly extracted by acetone is questionable. Some other investigations by mass spectroscopy revealed that some peaks of the analytical fingerprints are un-reacted chemicals related to the binder system manufacturing. This result, if further investigated, would allow us to regard the analytical fingerprints as representative of the entire magnetic layer (additives and binder). Study of the magnetic layer by an analytical method more suitable for polymer characterization would support fingerprinting approach. FTIR fingerprint, may partially solve this issue. (Appendix 13)

Conclusion

This approach allows us to categorize tapes in “technological families” on the basis of the additives. Further FTIR study may help to refine the “technological family” categorization by including the binder nature.

APPENDIX 16

Elementary Analysis of the Magnetic Coating by Energy Dispersive X-Ray (EDX)

Objective

Elementary analysis of the magnetic coating allows identification of magnetic particles and their optional alloying for protective treatment, the abrasives and elements other than carbon, oxygen and nitrogen that enter in the organic composition of the layer. Regarding the conservation issues, the elementary analysis results are of importance concerning tape tribological behaviour (abrasives), deterioration catalytic effect (magnetic particles) or magnetization stability (magnetic particles nature and treatments).

In addition the elementary analysis can be used as a fingerprint characterization of tapes technologies and manufacturing practices.

Experimental overview

30 cm of magnetic tapes are cut into small pieces, weighed and inserted into a small porcelain vessel. The vessel is introduced into a high temperature oven turned off prior to introduction. The temperature is then slightly raised to 500°C over 1 hour. The potential toxicity of the polyurethane thermal-decomposition products requires placing in the oven under a laboratory hood. After 30 minutes the oven is cooled down to room temperature and the porcelain vessel is extracted. The powdery residue corresponding to the inorganic materials is weighed. A double sided adhesive tape suitable for scanning high vacuum chamber is covered of the magnetic tape residue and placed on the sample stand of the SEM. Analysis is performed on non metal-sputtered samples with the EDX detector of a JEOL SEM in low vacuum mode, 20 keV, magnification of 1000.

Results and discussions overview

The concentration of the minor elements (abrasives...) within the magnetic coating is too low in order to obtain a reliable signal directly on the tape surface. The combustion procedure is necessary to gain better signal intensity.

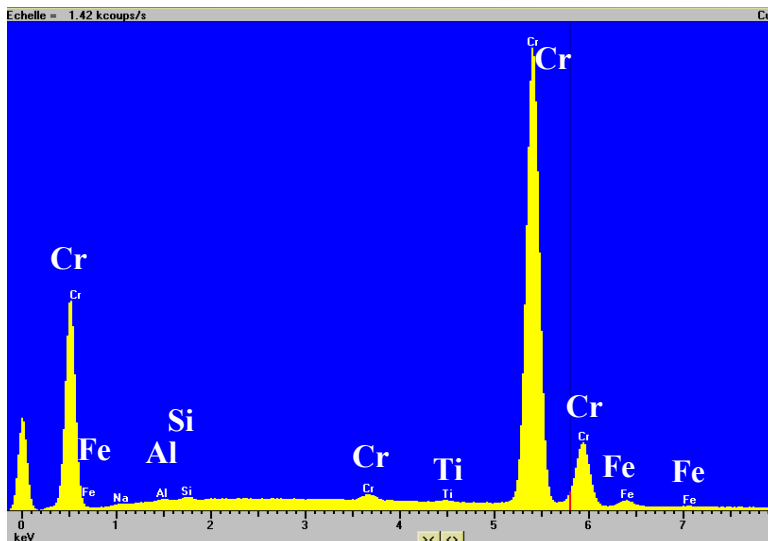


Figure 1 Energy spectrum of the combustion residue of an Agfa tape.

Figure 1 shows the energy spectrum for an **AGFA** tape. The magnetic particles are probably chromium oxide. Aluminium, silicium, titanium and iron rays are weak, this means that only traces of these elements are present or that this tape may have been cross-contaminated by other particles on the tape player.

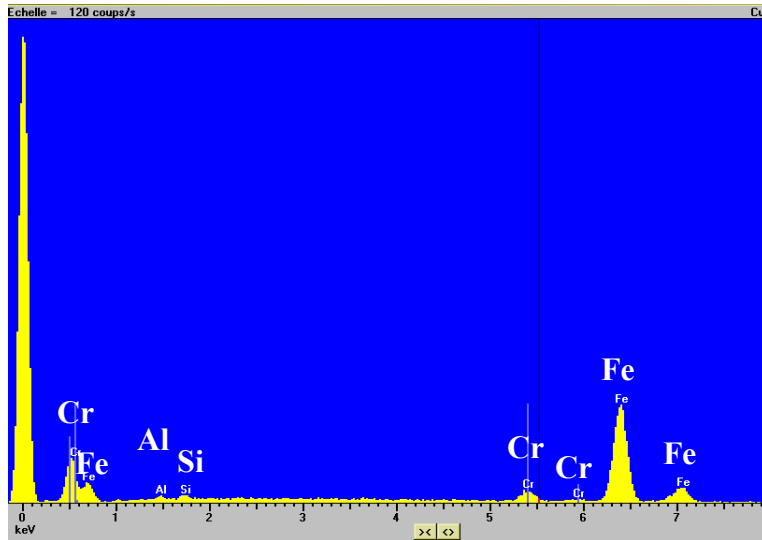


Figure 2 Energy spectrum the magnetic coating of a Sony tape.

Figure 2 shows the spectrum of a **SONY** tape surface – without combustion. The ray intensities are 10 times weaker compared to the analysis result when carried out after combustion. This tape is probably made of Fe₂O₃ magnetic particles. The presence of Chromium (Cr) can be attributed to either cross-contamination of the tape surface or the use of a protective treatment of the particles.

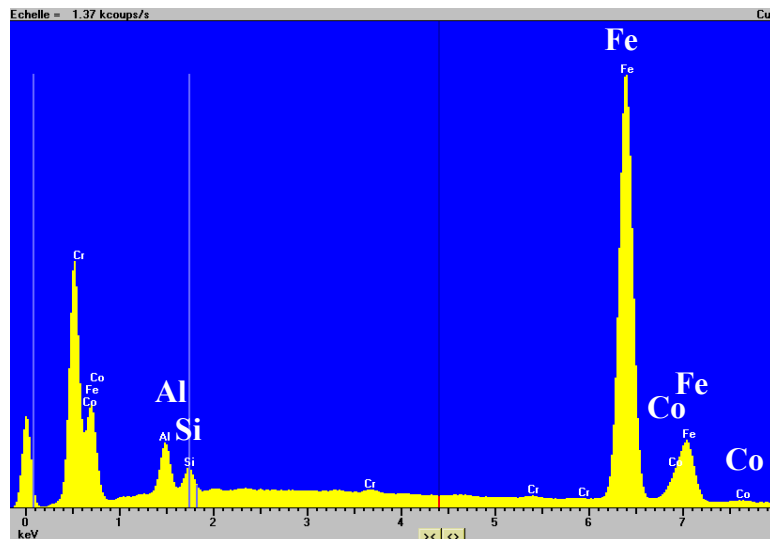


Figure 3 Energy spectrum of the combustion residue of an AMPEX tape.

Figure 3 shows the spectrum of a combustion residue of an **AMPEX** tape. The magnetic particles used are probably cobalt-doped ferric oxide. Presence of aluminium can be noticed. This is probably due to the addition of aluminium oxide as abrasive to the magnetic coating formulation

Conclusion:

The technique presented here allows further characterization of the materials entering in the formulation of the magnetic coating. “Portable” device for X-ray fluorescence analysis is now available at the CRCDG. This new instrument will probably simplify greatly the experimental protocol.

APPENDIX 17

Test Markers Pens for Surface Tension Measurement

		ACCUDYNETEST			
		DIVERSIFIED ENTERPRISES			
Overview	Accu Dyne Test	Contact Angle Measurement	Tensiometry	Searchable Bibliography	Bulletin Board

USING ACCU DYNE TEST™ MARKER PENS TO MEASURE SUBSTRATE SURFACE ENERGY



This report describes how **ACCU DYNE TEST™** marker pens can be used to measure the surface energy of films and other non-absorptive substrates. This method parallels ASTM Std. D2578¹, which covers the testing of polyethylene (PE) and polypropylene (PP) films via the application of formamide/ethyl Cellosolve* solutions. The fluids used in **ACCU DYNE TEST™** marker pens are based on those specified in ASTM D2578; the critical difference between this test and the ASTM technique is the manner in which the fluids are applied to the test sample.

In general, the ability of a substrate to anchor inks, coatings, or adhesives is directly related to its surface energy. If the substrate surface energy does not significantly exceed the surface tension of the fluid which is to cover it, wetting will be impeded and a poor bond will result. Thus, for most solvent based printing, plastics need to be treated to 36 to 40 dynes/cm; water based inks usually require 40 to 44 dynes/cm; some laminating and coating applications require surface energies of 50 dynes/cm or more. Clearly, surface energy must be assessed before printing, coating, or laminating is attempted.

ACCU DYNE TEST™ marker pens perform well on most non-absorptive materials. It is critical that the test fluid does not alter the surface properties of the substrate. For example, if the test fluid permeates a fibre substrate (such as paper) and causes swelling, results will indicate unrealistically easy wetting. A chemical reaction between the test fluid and the substrate invalidates results altogether.

To ensure replicability of this test, material preparation and test technique must be standardized. ASTM Std. D618² documents suggested conditioning methods. Unfortunately, this standard is untenable for treated film testing; conditioning times

Unfortunately, this standard is untenable for treated film testing; conditioning times range from 24 to 96 hours. Such rigorous controls may be of value for R & D, but for normal QC testing, much shorter conditioning times should be used. Standardization of ambient, substrate, and test solution temperature is critical, as is inspection methodology. Have one trainer instruct all testers to minimize variability. Relative humidity should not be excessive; higher RH tends to increase data variability. Finally, the elapsed time between extrusion or coating to test (or from test to printing, etc.) must be controlled.

TEST PROCEDURE

IMPORTANT SAFETY NOTICE:

The fluids contained in **ACCU DYNE TEST™** marker pens are considered hazardous materials. Avoid contact with skin. Use with adequate ventilation. Avoid contact with eyes. Pregnant women should not perform this test. For further information, refer to product MSDS or call Diversified Enterprises at 800-833-4644 or (603)543-0038.

For the results of this test to be meaningful, the following four points are absolutely essential and must be followed:

- 1) Do not touch or in any way contaminate the surface to be tested. Dirty surfaces lose their wettability.
- 2) Do not use contaminated or outdated **ACCU DYNE TEST™** marker pens.
- 3) Never retest the same location on a sample; move along the sample, or pull a new one.
- 4) Store and use **ACCU DYNE TEST™** marker pens at room temperature.

1.0 Materials/Equipment

1.1 ACCU DYNE TEST™ Marker Pens

1.2 Subject Material

1.3 Clean Level Test Area

1.4 Thermometer and Hygrometer

2.0 Method

2.1 Pull test sample. Be sure to pull a good specimen; surface aberrations cause poor results. For extruded film, one entire web cross-section should suffice. Do not touch the surface.

2.2 Place the sample on a clean, level surface. If necessary, anchor the edges to avoid curling or other deformation.

2.3 Record ambient temperature and relative humidity. If sample temperature differs from ambient, allow it to stabilize.

from ambient, allow it to stabilize.

2.4 Test at least three points across the sample; 1/4, 1/2, and 3/4 across the film section. It is good practice to test the outer edges as well. For non-film materials, test locations must be determined in-house.

2.5 Determination of Wetting

2.5.1 Choose an **ACCU DYNE TEST™** marker pen of a dyne level you believe is slightly lower than that of the test sample.

2.5.2 Press applicator tip firmly down on subject material until the tip is saturated with ink.

2.5.3 Use a light touch to draw the pen across the test sample in two or three parallel passes. Disregard the first pass(es); to flush any contamination from the tip, and to ensure that the test fluid layer is thin enough for accurate measurement, evaluate only the last pass.

2.5.4 If the last ink swath remains wetted out on the test sample for three seconds or more, repeat steps 2.5.2 and 2.5.3 with the next higher dyne level marker. If the last ink swath beads up, tears apart, or shrinks into a thin line within one second or less, repeat steps 2.5.2 and 2.5.3 with the next lower dyne level marker. If the ink swath holds for one to three seconds before losing its integrity, the dyne level of the marker closely matches that of the sample.

This is a relatively accurate surface energy measurement technique; used in standard 2 dyne/cm increments, **ACCU DYNE TEST™** marker pens can generally produce results with a precision of +/- 2.0 dynes/cm. Repeated use of **ACCU DYNE TEST™** marker pens should enable testers to estimate surface energy to within +/- 1.0 dyne/cm.

To investigate discrepancies between obtained and expected results, a more precise measurement method should be considered; application of **ACCU DYNE TEST™** surface tension test fluids with cotton swabs (per ASTM D2578-84) or by use of a drawdown rod is recommended. Alternatively, if results are suspect, replicate the test with a set of unused markers. This test has proven itself on a wide variety of substrates; it is, however, theoretically more prone to contamination than some other techniques. For this reason, even in the absence of unexpected results, you should establish a quality assurance plan which calls for regular audits during the phase-in stage of **ACCU DYNE TEST™** marker pen use. The effect of **all** changeovers from one substrate to another should be monitored especially closely. Slip and other additives tend to bloom to the surface of extruded sheets and films; transferring surface-active additives from one material to another can have a profound effect on surface energy measurement. In general, once you demonstrate that a switch from substrate A to substrate B has no effect, it is safe to assume that future changeovers from A to B will act similarly. Thus, the frequency of backup audits decreases over time.

Finally, a few qualifying words. Surface energy is critically important to many converting operations. Unfortunately, it is not the sole determinant of product suitability. Other factors, such as surface topography, coating rheology, and chemical incompatibility, must also be considered. This is why broad-based communications with vendors and customers is so important. But at least by systematically measuring substrate surface energy, you will have a sound starting point from which to resolve

substrate surface energy, you will have a sound starting point from which to resolve other problems which may arise.

¹Annual Book of ASTM Standards, *Wetting Tension of Polyethylene and Polypropylene Films*.

²Annual Book of ASTM Standards, *Conditioning Plastics and Insulating Materials for Testing*.

*ethyl cellosolve is a registered trademark of Union Carbide Corp. for ethylene glycol monoethyl ether (2-ethoxyethanol).

This report is provided by Diversified Enterprises, 101 Mulberry St., Suite 2N, Claremont, NH 03743. It is intended to provide quality control information for converters and others who need to measure surface energy. We believe all information contained herein is accurate. Diversified Enterprises will not, however, under any circumstances be held responsible for any losses or damages incurred by any party using this report.

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Diversified Enterprises
101 Mulberry St., Suite 2N
Claremont, NH 03743 U.S.A.
(603) 543-0038 or 1-800-833-4644; fax (603) 543-1334
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APPENDIX 18

Adipic Acid Formation upon Artificial Ageing

Adipic acid (Figure 1) is a bi-functional carboxylic acid widely used as raw materials for Polyurethane-Ester production. When reacted with a glycol (mainly butanediol) the polyester formed is the polybutyleneadipate that tends to demonstrate a poor hydrolytic stability.

The hydrolysis of this polyester extended to an important degree of breakdown would theoretically lead to adipic acid recovery.

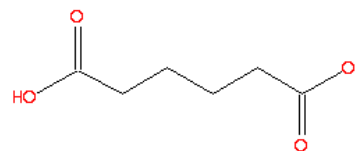


Figure 1 Structure of adipic acid

This phenomenon was observed upon artificial ageing in two cases:

- environmental chamber, 90%RH 50°C for more than 1 year, whole U-Matic AGFA reel form 1980
- hermetically closed vessel, 100%RH, 75 °C for more than 100 days; 30cm of U-Matic AMPEX tape from 1981

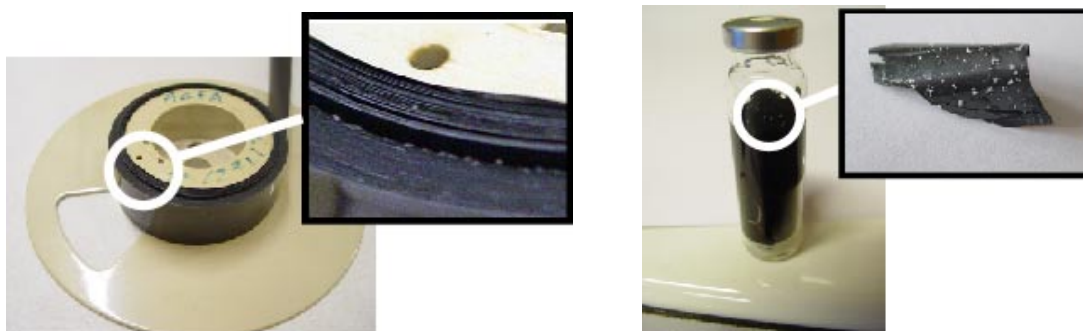


Figure 2 Adipic acid exudation and crystallisation upon artificial ageing in environmental chamber (left), in sealed vial (right)

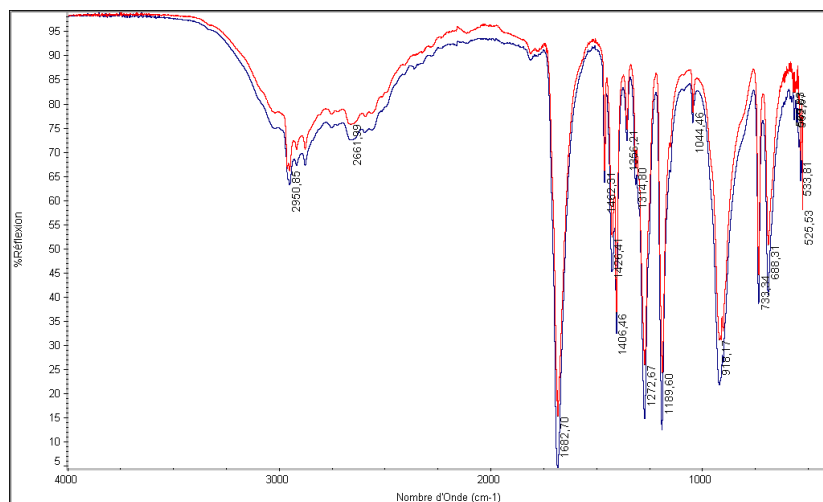


Figure 3 Infrared spectra of white crystals sampled from artificially aged tapes (red) and of pure adipic acid (blue).

Infrared spectra (reflection mode on ATR diamond) of the white crystals deposited on the top of the spool and on the tape surface are identical and can be compared to the spectrum of pure adipic acid.

This observation tends to demonstrate that deterioration upon hydrolysis can continue until total depolymerisation of the PUR-ES.

This phenomenon can be correlated to the hydrolytic production of end-products as observed in Size Exclusion Chromatography (Appendix 7).

All tapes are not concerned

by this phenomenon, this tends to demonstrate that PUR-ES used for magnetic coating have highly different hydrolytic stabilities.

APPENDIX 19

CRCDG U-Matic Tapes Initial Sampling Procedure

Introduction

Due to the variety of formats, brands, magnetic tape types and manufacturing practices, the CRCDG had to restrict the study to a given format. The U-Matic format (fig. 1) has been chosen as a case-study for some reasons presented in section A1.3.

The French *Institut National de l'Audiovisuel* (INA) holds a considerable number of U-Matic tapes and gave the CRCDG access for U-Matic sampling to more than 20 000 tapes already digitised. Tapes were stored in the vaults located at *Bry sur Marne, France* and *Les-Essarts-Le-Roi, France*. These tapes were available for destructive testing.

In addition, the INA gave us access to about 40 tapes that had not been successfully digitised for materials deterioration reasons. 290 tapes have been retrieved from the INA and brought to the laboratory.



Figure 1 U-Matic cassette.

Sampling procedure

The tapes have been chosen according to the following procedure:

For each brand, as identified in the INA holdings database, 2 tapes (duplicates) with close recording date and with same origin have been chosen. The sampling period was three times in one year (in most cases January, may, November) between 1976 and 1997. The 290 tapes are represented on the chart in figure 2.

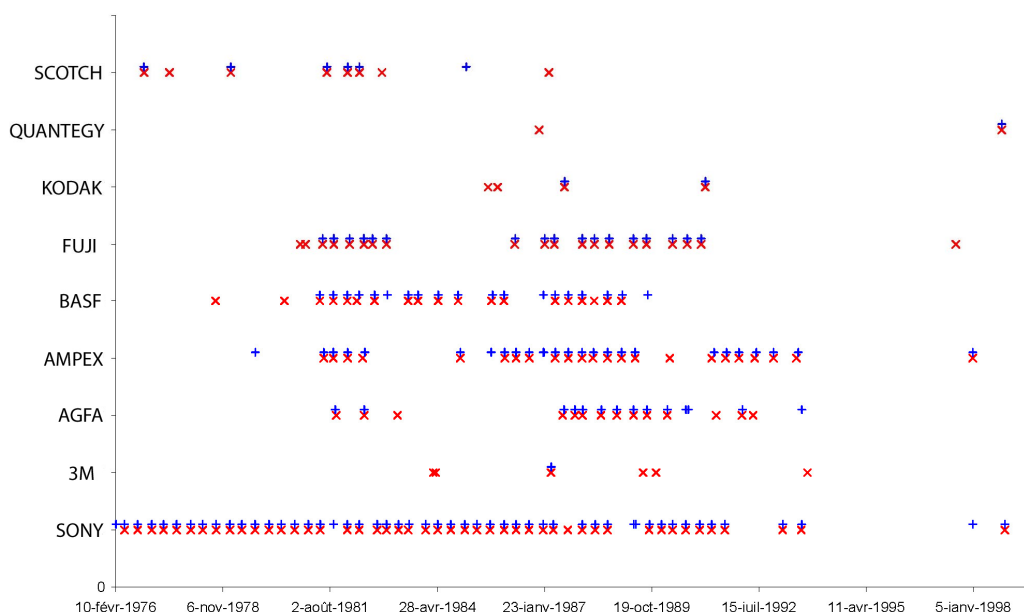


Figure 2 U-matic tapes sampled through INA collection, stored at the laboratory and available for destructive testing. The red and blue crosses correspond respectively to the two tapes with same origin and with same recording date (duplicates).

Discussion

In most cases duplicates are tapes with same batch number or close serial number code. It can be assumed that these tapes have been manufactured in the same conditions, recorded by the same operator and stored in the same conditions.

For tape selection we relied on the holding database of the INA which records the brand as it appears on the cassette case. This information may differ from the real manufacturing date due to lack of tape

labelling or wrong cases. The manufacturing date can only be approximated by using the recording date.

Important errors can be made due to the tape storage period prior to recording; transfer of oldest recording onto newer cassettes and use of old cassettes to record newer recording.

Fortunately, most tapes have been used as soon as received and handled a few times until now.

After examination of the 290 tapes, correction of the sampling chart has been made. In some cases, investigations on the serial number attribution method allowed us to correct the brand and the manufacturing date.

The initial sampling is not perfect (lack of tapes for some brand/manufacturing period), but enables statistical investigations and a review of the different manufacturing practices and technological families.

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