Preservation Research and Testing – the science of cultural materials

Physical, Chemical and Optical Properties Labs

Dr. Fenella G. France, *Preservation Research and Testing Division, Library of Congress*

Focus on Non-Invasive Analytical Techniques

- Prioritizing risk to collections
  - Traditional (e.g. corrosive media)
  - Modern (e.g. sound format, fugitive media)
  - New at-risk areas (e.g. fugitive media, 21st century materials, sound recordings, unstable glass)
- Characterizing materials
  - Degradation mechanisms
  - Tracking change due to environment / treatments
- Scientific reference sample collection
- Scientific data infrastructure
  - Data fusion, data mining, storage, access

Hyperspectral Imaging

Fourier Transform Infrared Spectroscopy (FTIR)

Fiber Optic Reflectance Spectroscopy (FORS))

X-ray fluorescence (XRF)

The "go-team"

Prioritizing and creating a structured approach to resources, time demands and complementary data to answer research questions

"active learning"

Scientific Reference Sample Collection

Materials Types include Barrow Book Collection, magnetic tapes, parchment, papyrus, damaged books, ISR reference papers, ASTM 100-year Paper Aging Study papers, pigments, CDs, DVDs, fabrics, glass, fibers etc.

Materials Characterization Scientific Reference Samples: Development of spectral and spectroscopy databases of reference materials

Center for Library Analytical Scientific Samples (CLASS)

Enhance non-destructive characterization

Expansion of database to include deteriorated substrates / media

Changes from aging, treatments, environment

Audio Tape Degradation – Sticking, Squealing, Shedding

Most common remediation:
thermal baking
54°C for 8-36 hours

	How do you decide when to bake a tape?	What do you do after baking?
User #1	Bake everything	Play it warm
User #2	Bake everything of known vintage	
User #3	No bake until proven sticky	Let it cool
User #4	No bake, ever	

Photos: Christine Folivi, ACS SEED 2018

Thermal Analysis – Differential Scanning Calorimetry

Glass transition point for polymers

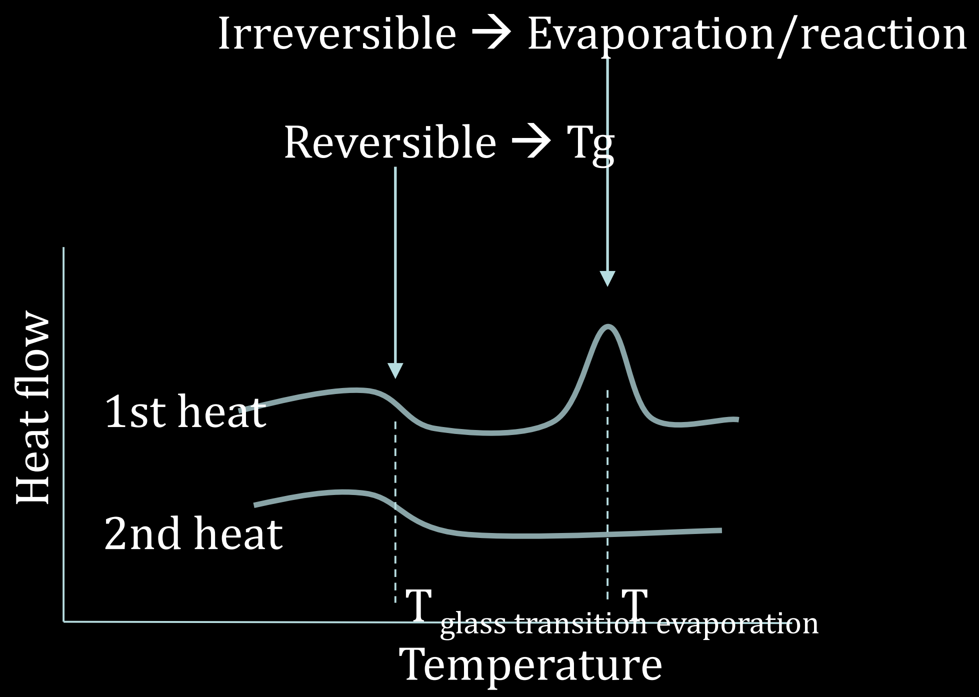
5-10 mg sample is heated at fixed rate (°C/min)

Heat flow (energy) necessary to maintain that rate is recorded

Dips, peaks, step-changes in heat flow

⇩

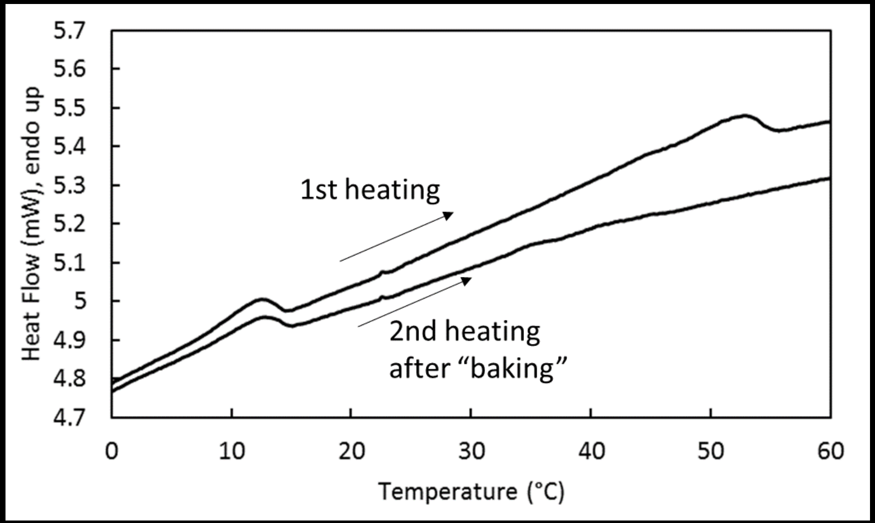
Thermally induced change in state

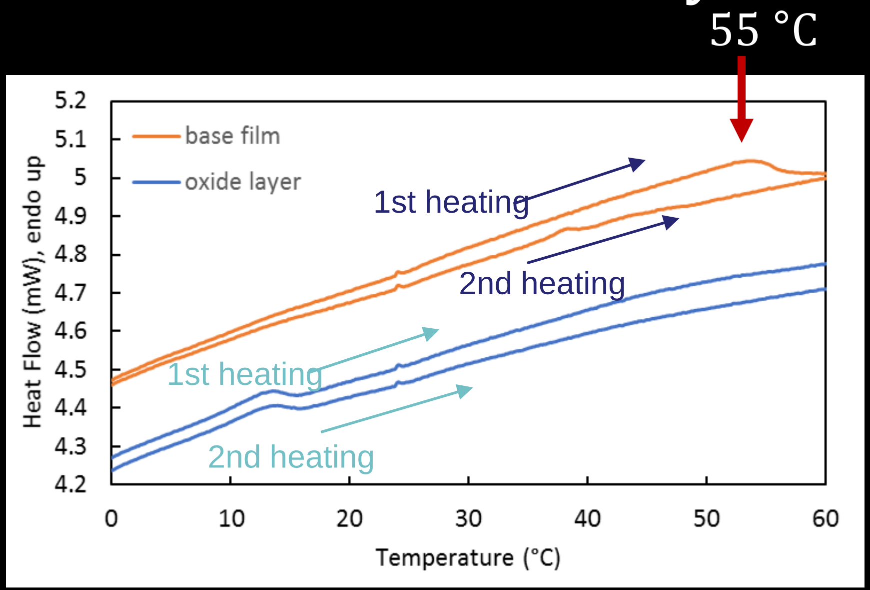


Thermal Analysis – Differential Scanning Calorimetry

Evidence of Thermal Transitions in “Sticky” Tape

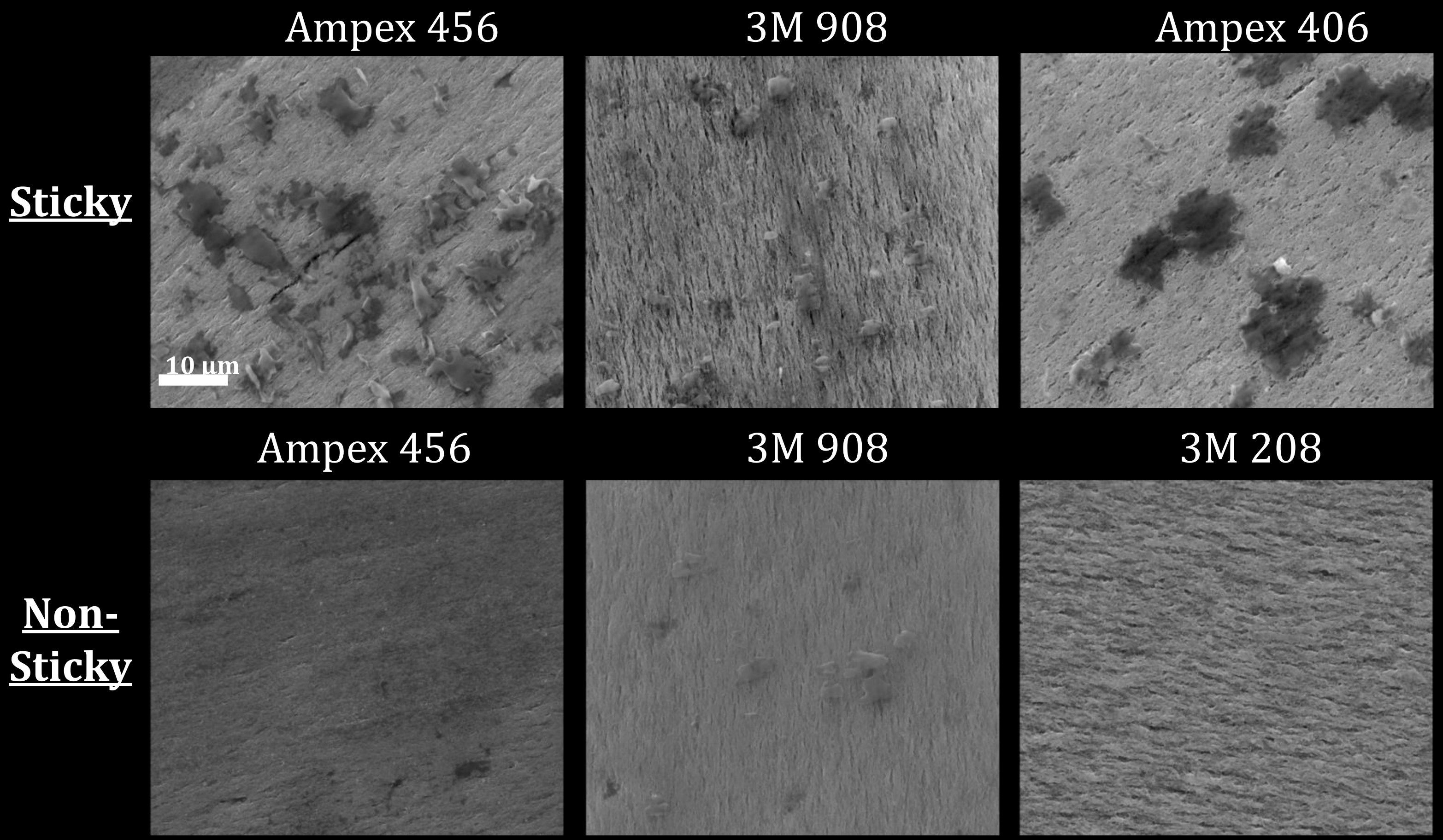
Layer Differences Observable in Thermal Analysis





Using material from separated layers:
Low temperature Tg (15°C) in **oxide layer**
“Bake” temperature transition in **base film**

Electron Microscopy – Tape "twins"

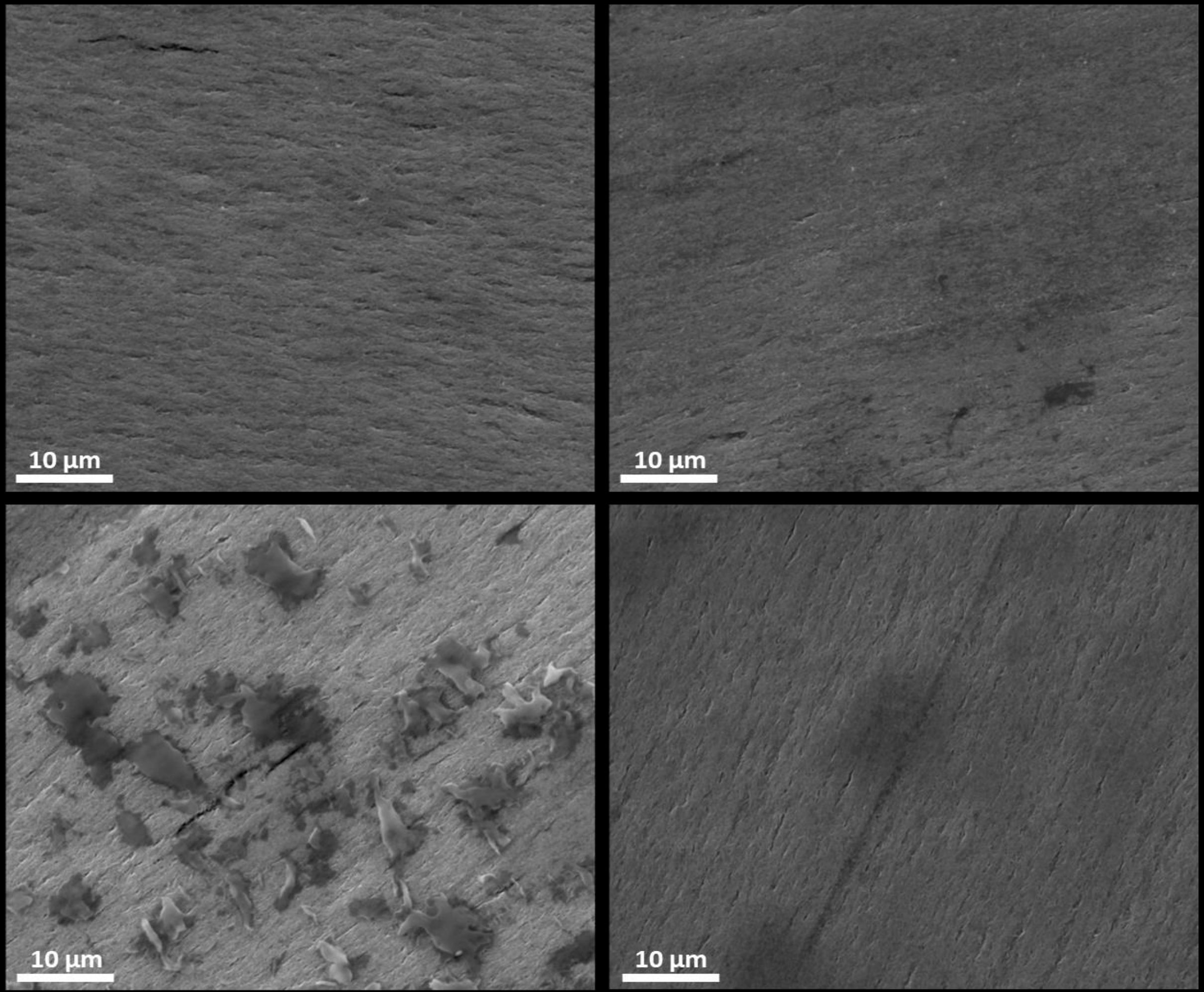


Electron Microscopy of Baked Tapes

Unbaked **Baked**

Ampex 456 non-sticky

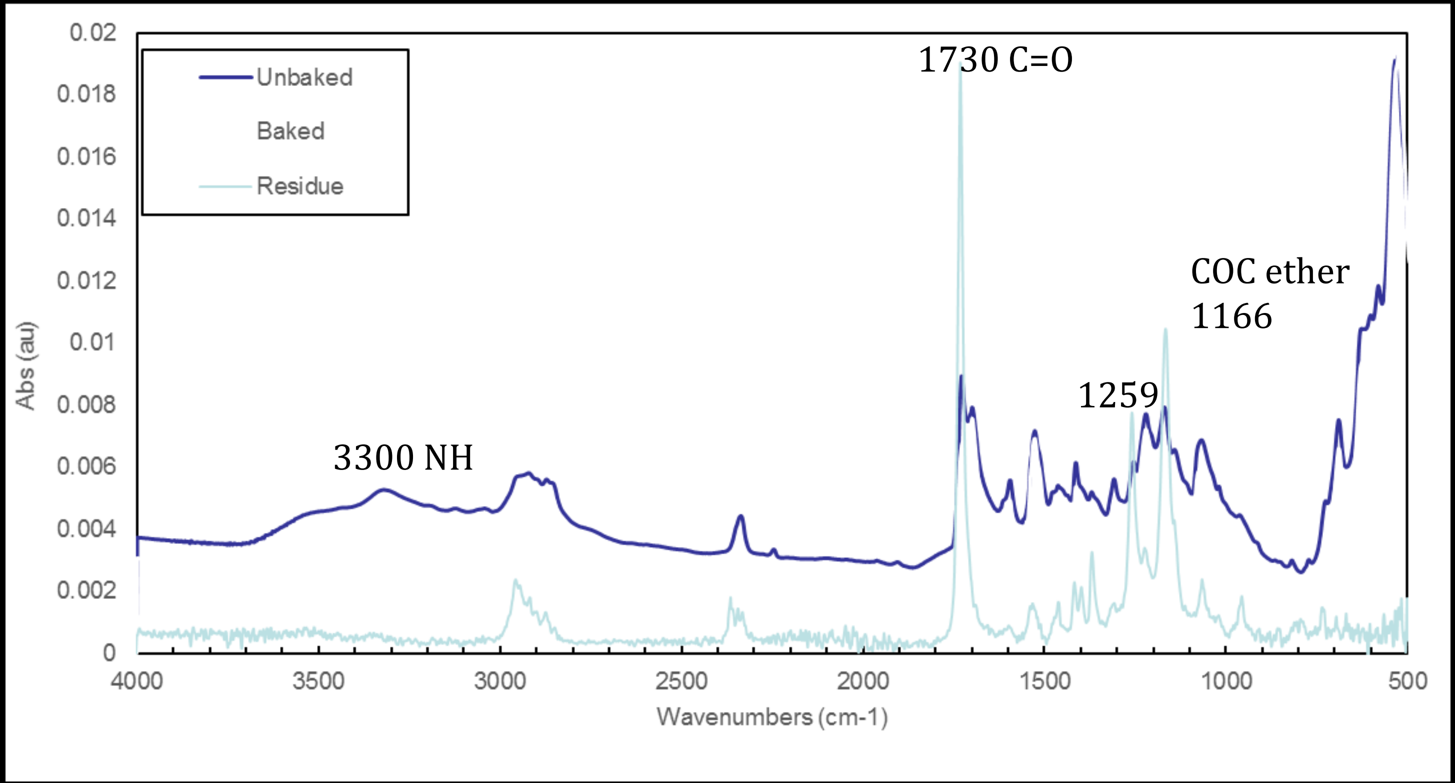
Ampex 456 sticky



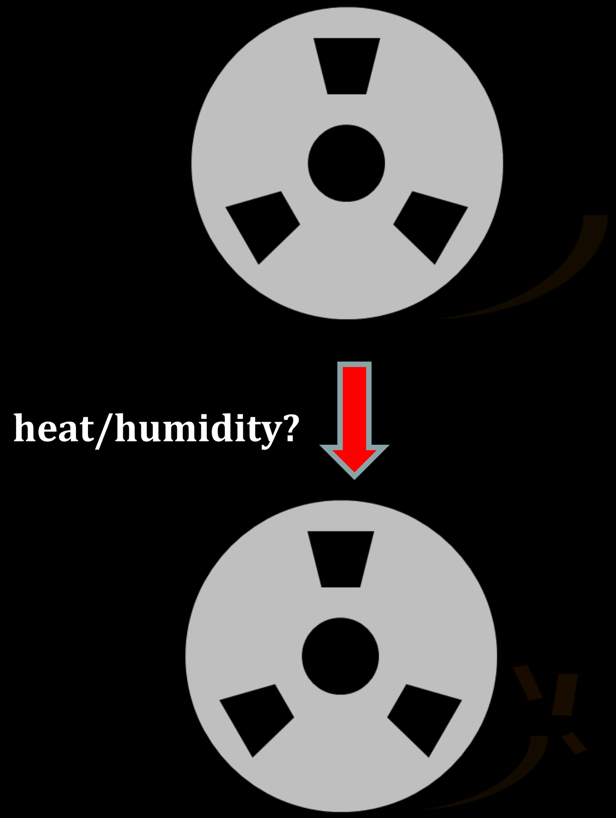
Removed surface residues with gentle swab, analyzed by FTIR and compared to baked and unbaked oxide layer of same tape

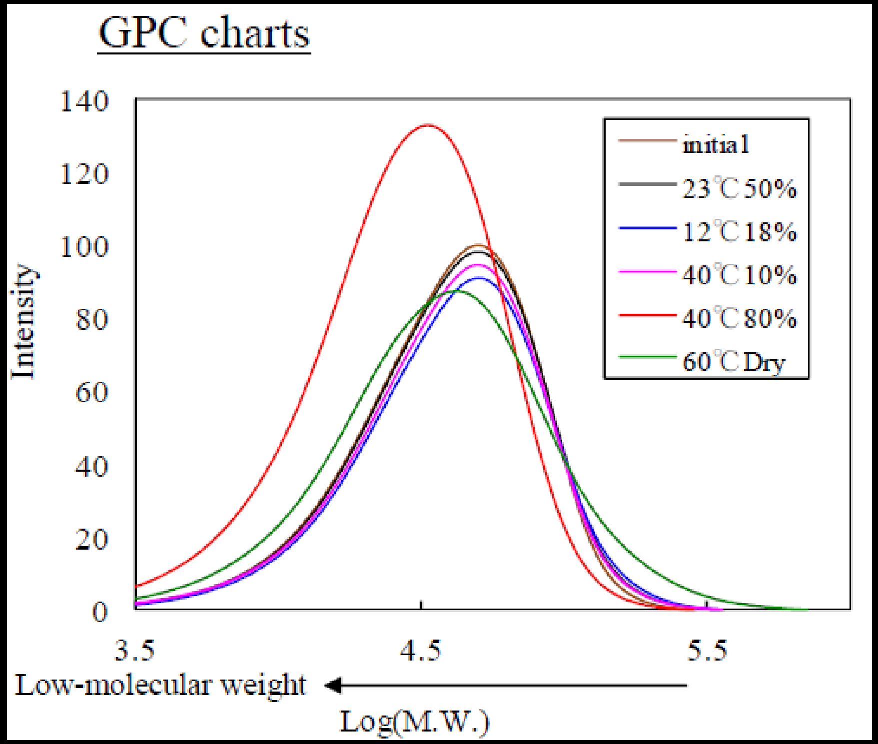
Results suggestive of lubricant/plasticizers, NOT degradation from PU

Strongest peaks (1730, 1259, 1166 cm^{-1}) correlate to peaks found to decrease after baking (both here, and other studies)



Can we make a sticky tape?





In collaboration with FujiFilm Japan

Have tried artificial aging at various combinations of temperature and humidity:

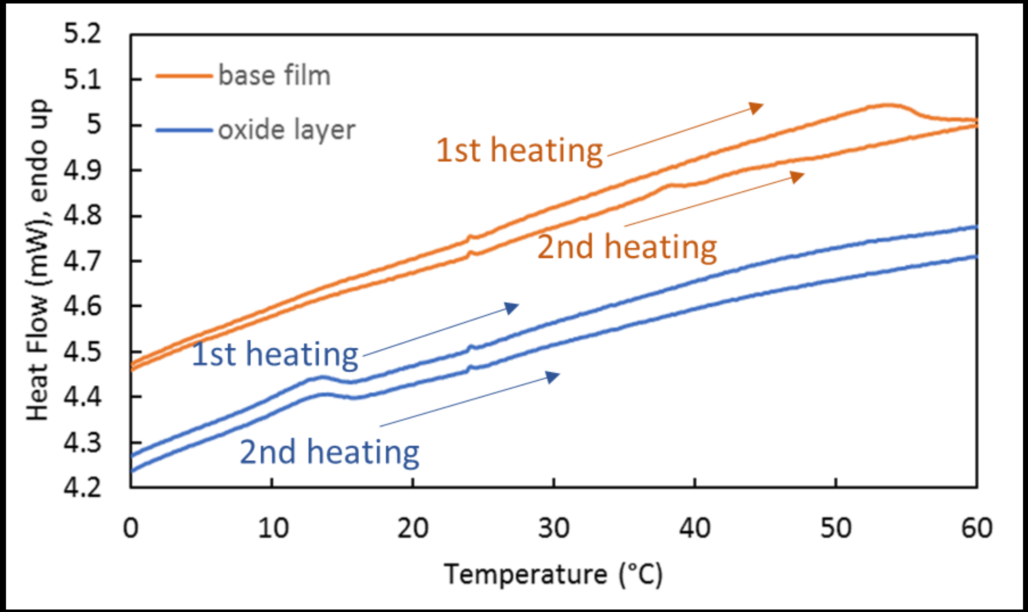
80°C/80%
40°C/80%
40°C/10%
60°C/0%
…

Can break down a tape, but cannot reliably mimic a "sticky" tape

Analyses of stickiness and baking

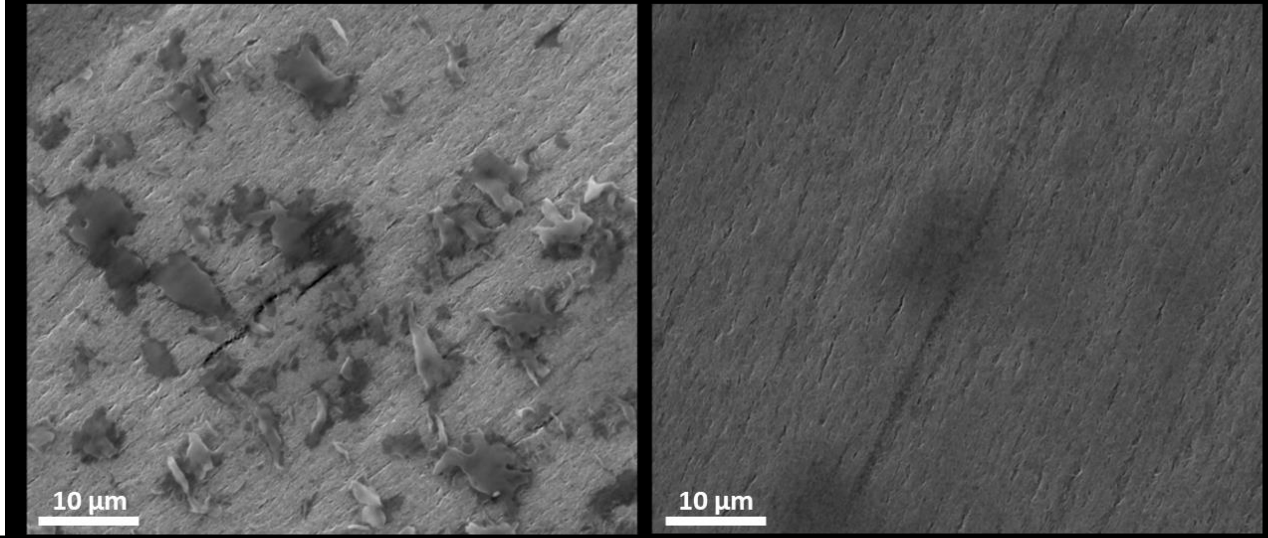
DSC thermal data

base film contributes to baking process

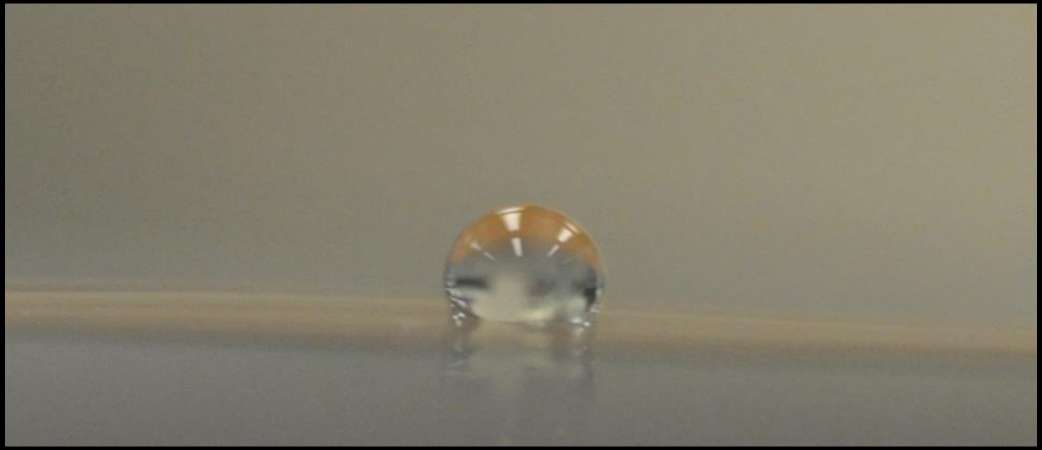


Microscopy data

oxide layer shows visible restorative changes during baking

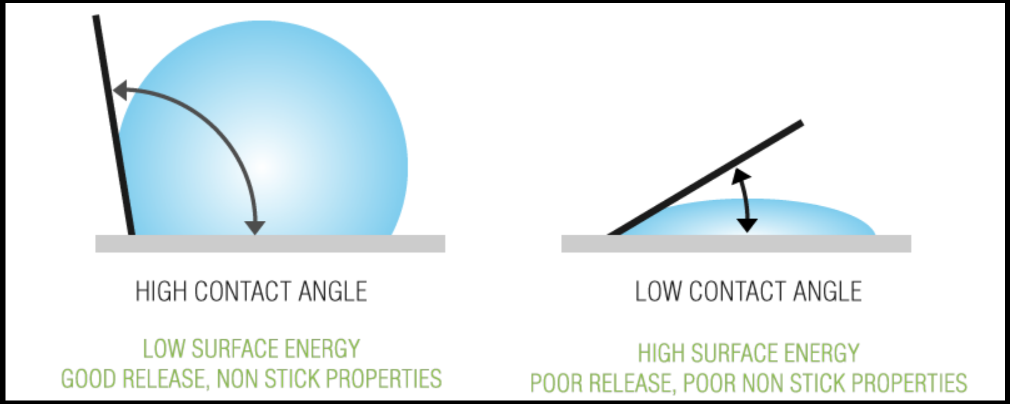


Water Contact Angle of Magnetic Media



Non-sticky

Sticky



ptfecoatings.com

<u>Factors affecting contact angle:</u>

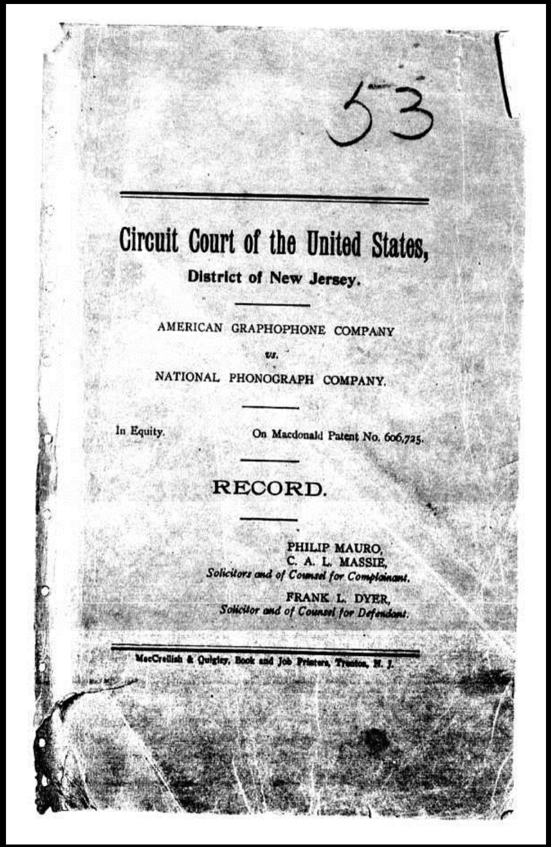
Surface roughness

Surface chemistry

KEY: **SURFACE**

Challenges with Wax Cylinders

Taking a multipronged approach


53

Circuit Court of the United States,

District of New Jersey.

AMERICAN GRAPHOPHONE COMPANY

vs.

NATIONAL PHONOGRAPH COMPANY.

In Equity. On Macdonald Patent No. 606,725.

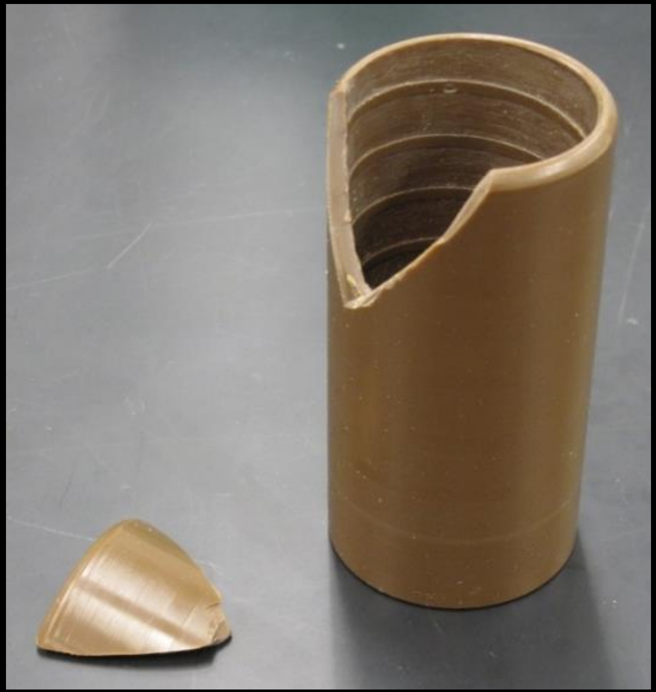
RECORD.

PHILIP MAURO,
C. A. L. MASSIE,
Solicitors and of Counsel for Complainant.

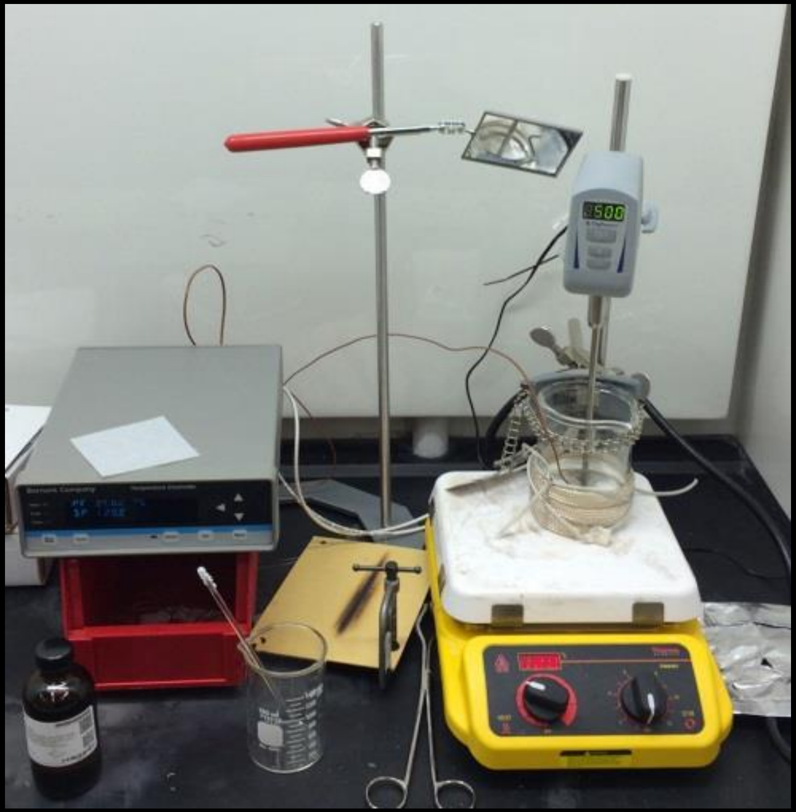
FRANK L. DYER,
Solicitor and of Counsel for Defendant.

MacCrellish & Quigley, Book and Job Printers, Trenton, N. J.

Historical Records

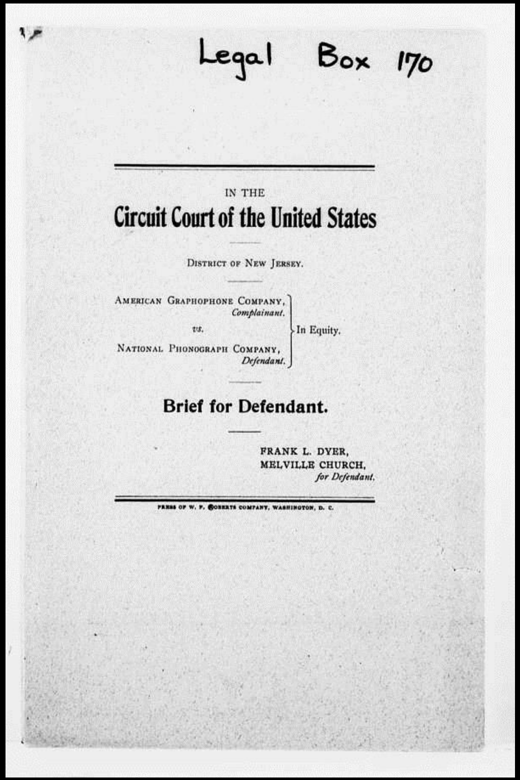


Chemical and Physical Testing



Laboratory Synthesis

Edison Papers Project @ Rutgers: Digitized lab notebooks



Legal Box 170

IN THE

Circuit Court of the United States

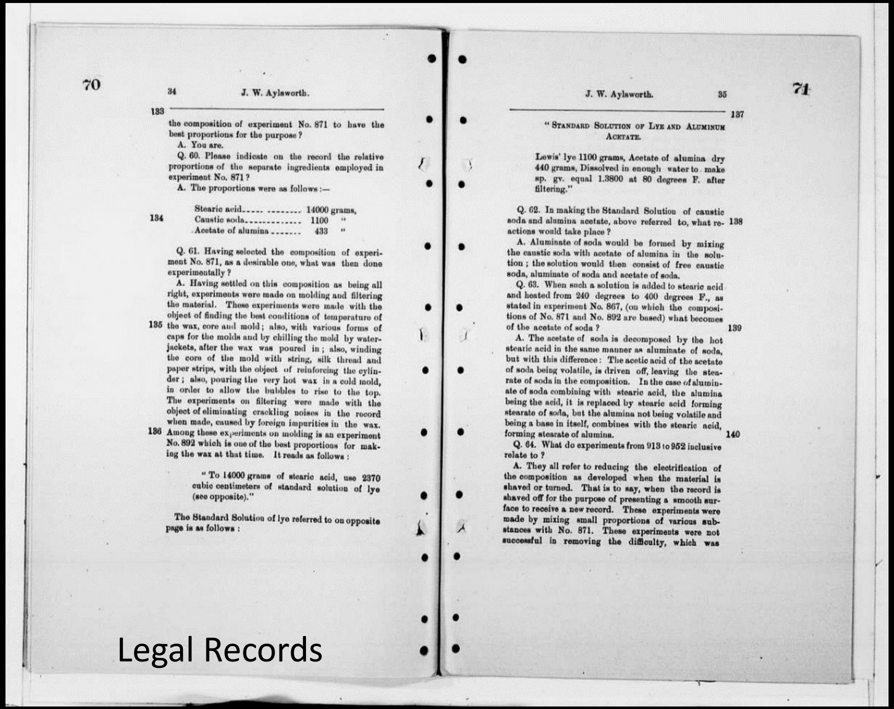
District of New Jersey.

American Graphophone Company, *Complainant.*
vs.
National Phonograph Company, *Defendant.*
} In Equity.

Brief for Defendant.

FRANK L. DYER,
MELVILLE CHURCH,
for Defendant.

PRESS OF W. F. ROBERTS COMPANY, WASHINGTON, D. C.



70

34 J. W. Aylsworth.

133 the composition of experiment No. 871 to have the best proportions for the purpose?

A. You are.

Q. 60. Please indicate on the record the relative proportions of the separate ingredients employed in experiment No. 871?

A. The proportions were as follows:—

Stearic acid............ 14000 grams,
134 Caustic soda............ 1100 "
Acetate of alumina........ 433 "

Q. 61. Having selected the composition of experiment No. 871, as a desirable one, what was then done experimentally?

A. Having settled on this composition as being all right, experiments were made on molding and filtering the material. These experiments were made with the object of finding the best conditions of temperature of
135 the wax, core and mold; also, with various forms of caps for the molds and by chilling the mold by water-jackets, after the wax was poured in; also, winding the core of the mold with string, silk thread and paper strips, with the object of reinforcing the cylinder; also, pouring the very hot wax in a cold mold, in order to allow the bubbles to rise to the top. The experiments on filtering were made with the object of eliminating crackling noises in the record when made, caused by foreign impurities in the wax.
136 Among these experiments on molding is an experiment No. 892 which is one of the best proportions for making the wax at that time. It reads as follows:

"To 14000 grams of stearic acid, use 2370 cubic centimeters of standard solution of lye (see opposite)."

The Standard Solution of lye referred to on opposite page is as follows:

J. W. Aylsworth. 35

71

137

"Standard Solution of Lye and Aluminum Acetate.

Lewis' lye 1100 grams, Acetate of alumina dry 440 grams, Dissolved in enough water to make sp. gv. equal 1.3800 at 80 degrees F. after filtering."

Q. 62. In making the Standard Solution of caustic soda and alumina acetate, above referred to, what re- 138
actions would take place?

A. Aluminate of soda would be formed by mixing the caustic soda with acetate of alumina in the solution; the solution would then consist of free caustic soda, aluminate of soda and acetate of soda.

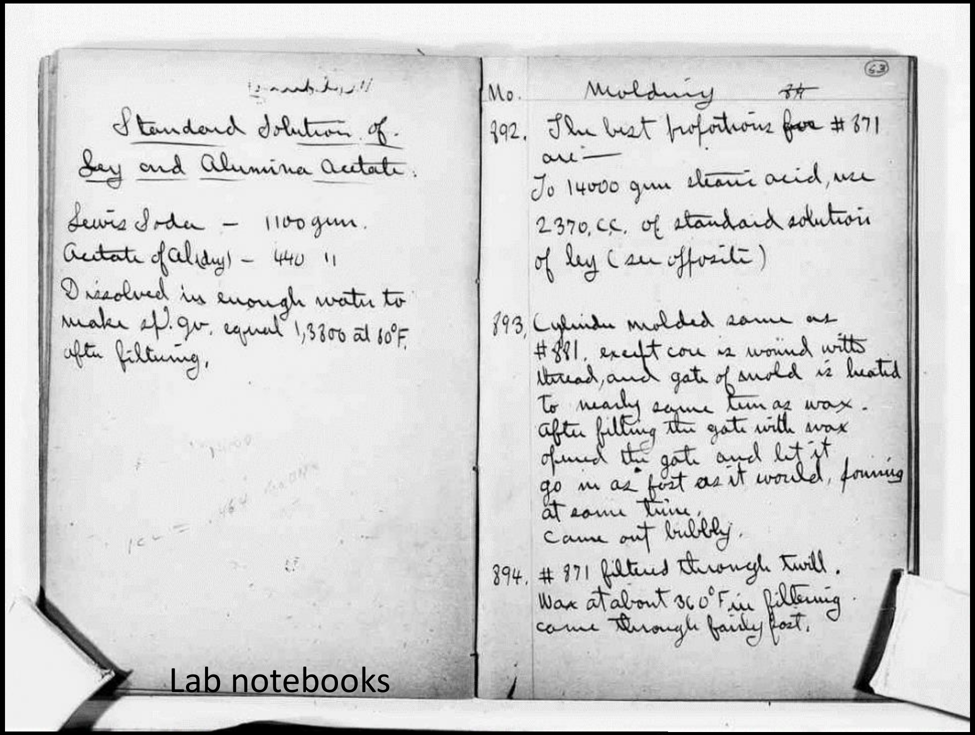
Q. 63. When such a solution is added to stearic acid and heated from 240 degrees to 400 degrees F., as stated in experiment No. 867, (on which the compositions of No. 871 and No. 892 are based) what becomes
of the acetate of soda? 139

A. The acetate of soda is decomposed by the hot stearic acid in the same manner as aluminate of soda, but with this difference: The acetic acid of the acetate of soda being volatile, is driven off, leaving the stearate of soda in the composition. In the case of aluminate of soda combining with stearic acid, the alumina being the acid, it is replaced by stearic acid forming stearate of soda, but the alumina not being volatile and being a base in itself, combines with the stearic acid,
forming stearate of alumina. 140

Q. 64. What do experiments from 913 to 952 inclusive relate to?

A. They all refer to reducing the electrification of the composition as developed when the material is shaved or turned. That is to say, when the record is shaved off for the purpose of presenting a smooth surface to receive a new record. These experiments were made by mixing small proportions of various substances with No. 871. These experiments were not successful in removing the difficulty, which was

Legal Records



Standard Solution of Ley and Alumina Acetate.

Lewis Soda – 1100 gm.
Acetate of Al (dry) – 440 "
Dissolved in enough water to make sp. gr. equal 1,3800 at 80°F after filtering.

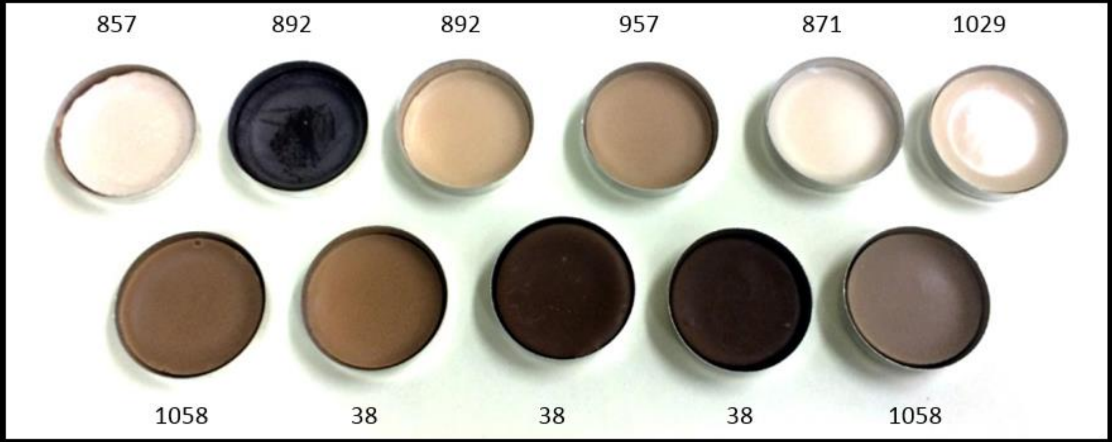
No. Molding

892. The best proportions for #871 are— To 14000 gm stearic acid, use 2370 cc. of standard solution of ley (see opposite)

893. Cylinder molded same as #881, except core is wound with thread, and gate of mold is heated to nearly same temp as wax. After filling the gate with wax opened the gate and let it go in as fast as it would, forming at same time. Came out bubbly.

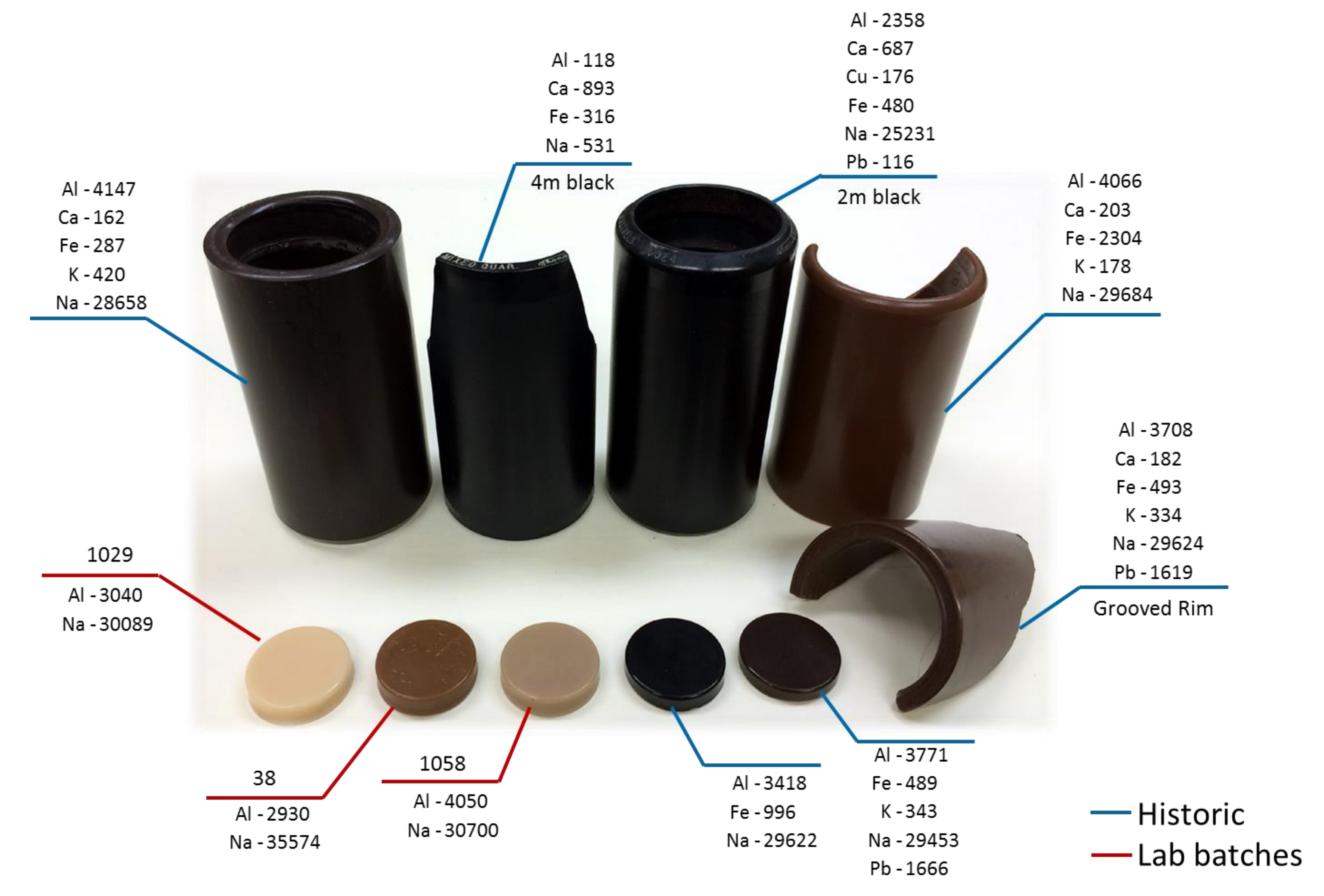
894. #871 filtered through twill. Wax at about 360°F in filtering came through fairly fast.

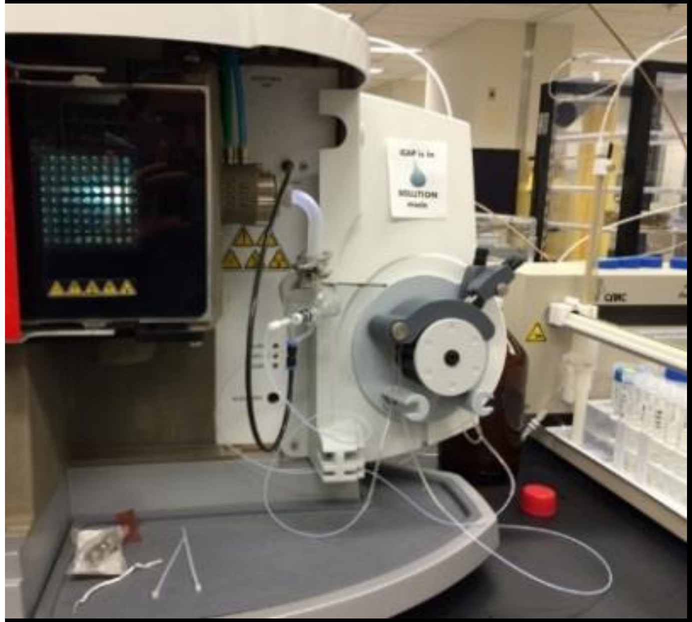
Lab notebooks



Reproductions of Edison cylinder formulations

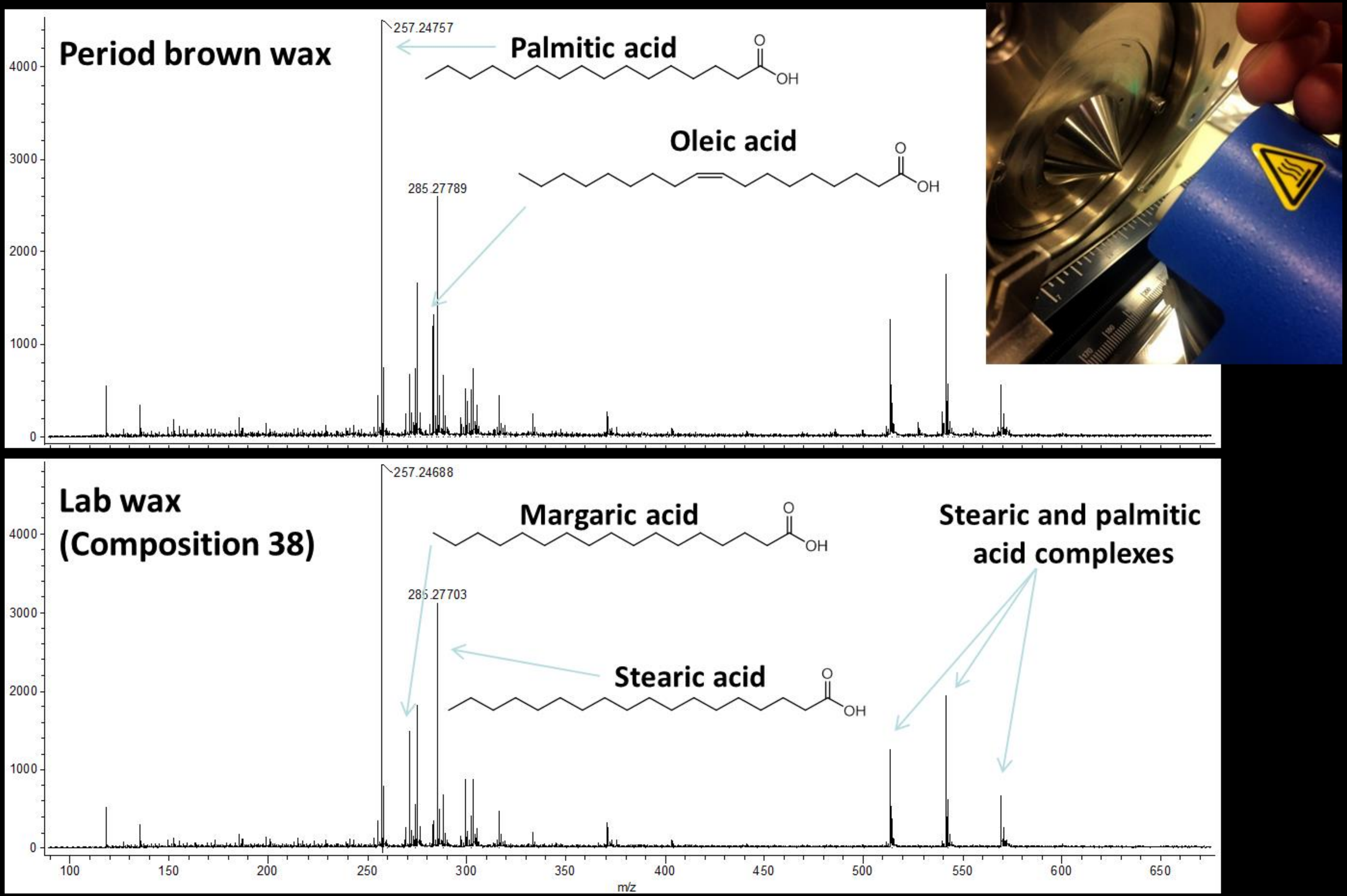
Examining metals content by ICP





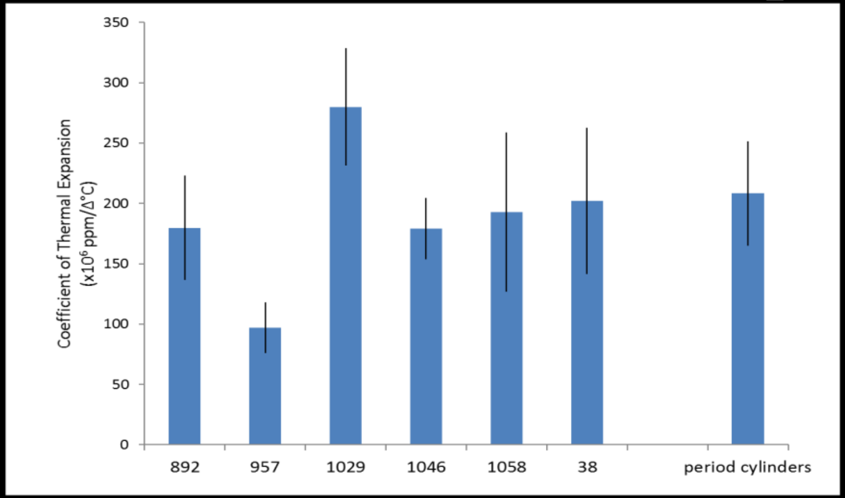
All values in parts-per-million (ppm)

Organic compound analyses



Results showed no chemical change between original swab samples and new lab formulations

Coefficient of thermal expansion



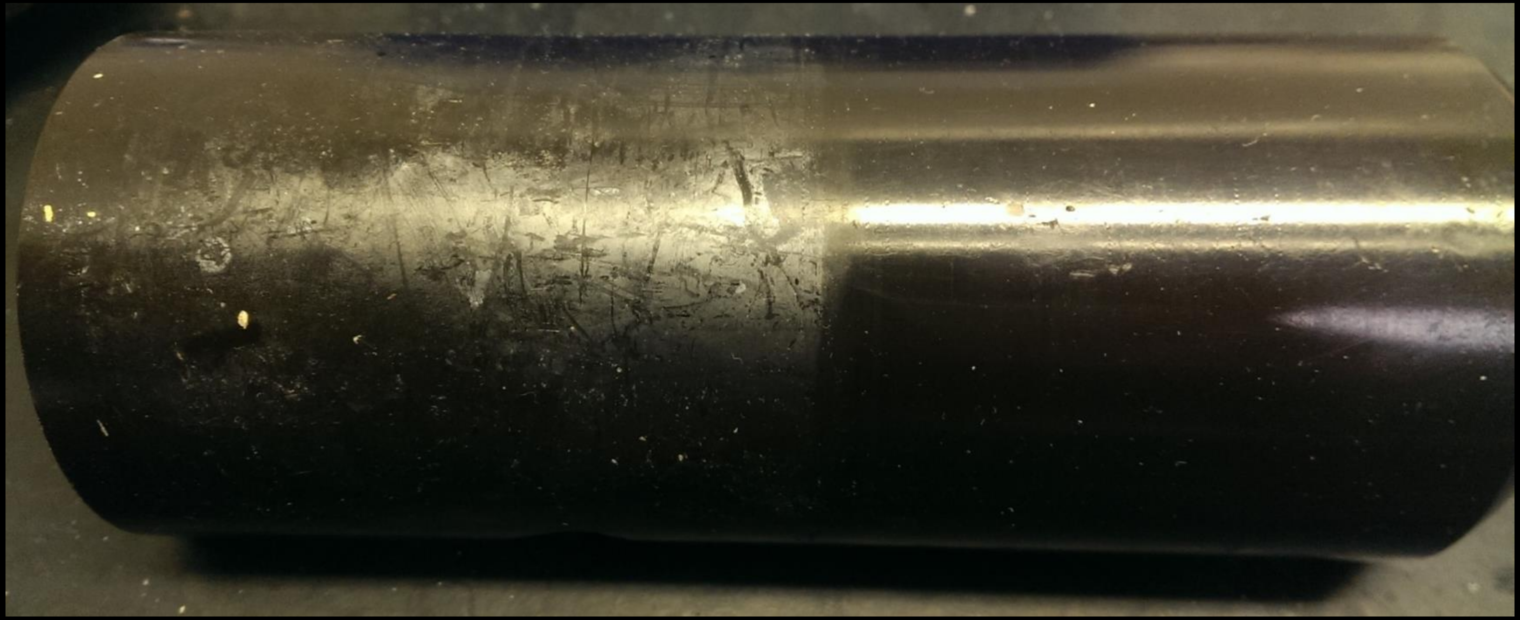
Creation and destruction of "pseudocylinders"

Thermal cycle 100 °F to 0 °F (8-12x)

Lab trials of prototype cleaning solutions

- Acetonitrile and water solutions (1:3, 1:1, 3:1)
- 2.5% Tween 20, Triton X-100, or Tergitol 15-S-7

Lab trials of prototype cleaning solutions



Promising. But…

- Prototype solutions contained high acetonitrile for optimum cleaning, particularly during rinse
- Evaporative cooling could lead to rapid thermal change at surface – leading to breakage
- Not comfortable with the inherent risk

"Custodians for future generations"

Acknowledgements

Preservation Research and Testing Division Staff, LC
Esp[eciallyt Drs. Andrew Davis, Eric Monroe,
and ACS SEED fellow 2018 Christine Folivi
NAVCC Colleagues
FujiFilm Japan

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