Preservation Research at the Library of Congress – the science of cultural materials

Fenella G. France, PhD MBA FAIC

Preservation Research and Testing Division

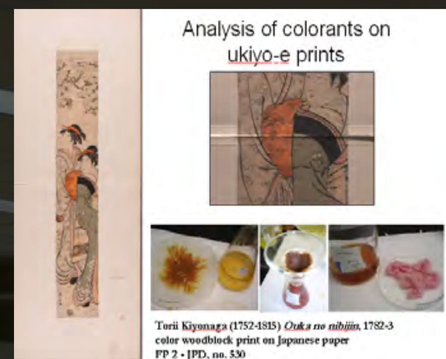
Library of Congress

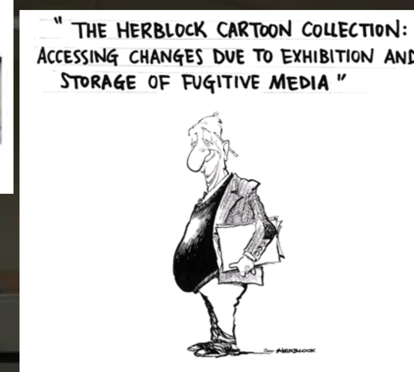

Library of Congress
Preservation Research and Testing Division
Mission:
"To assure long-term uninterrupted access to the intellectual content of the Library's collections, either in original or reformatted form"
Library of Congress collections number over 162 million items

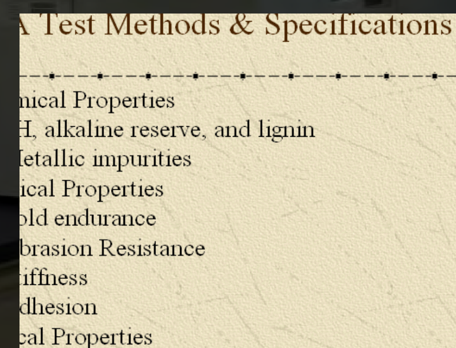
Preservation Research and Testing Division

Programmatic Areas:

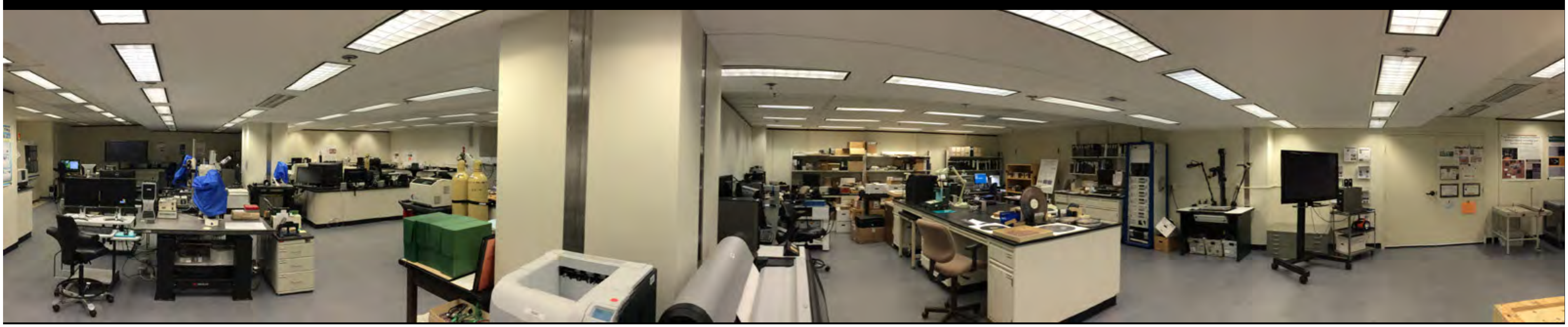
- Analytical Requests
  - *Short term, 1-2 instrument analyses*
- Research Projects
  - *Long term, large scale projects that investigate issues of material degradation that impact large parts of Library collections, or aim to improve methods for analysis and preservation of the collections*
- Quality Assurance
  - *Testing of materials used for housing, storage, building (incl. VOCs), conservation treatments,* ***and*** *evaluation, development and dissemination of material specifications*







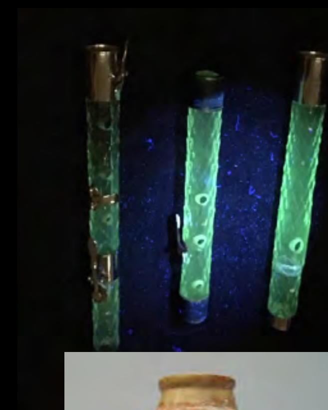
Physical, Chemical and Optical Properties Labs



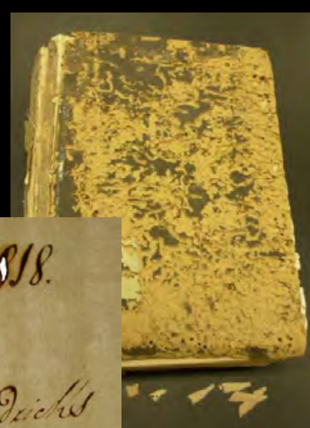
Preservation & Analysis of Large Collections

Impact of:

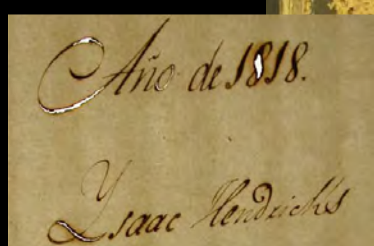
- Use (handling)
- Environment
- Material







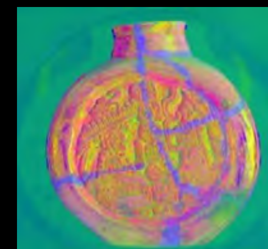


Heritage Science Research

- Diverse range of materials in cultural heritage (CH) with little documentation about history of objects
- Focus on non-invasive techniques to recreate history of use
- Linking reference sample material data with cultural heritage objects
- Modelling damage functions from destructive testing on reference materials
- Baseline imaging – mapping spectral response of a material across an entire object
- Range of complementary analytical techniques *(organic/ inorganic components)*
- Predicting degradation/change from treatments and impact of environmental parameters *(humidity, temperature, light, pollutants)*

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)

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

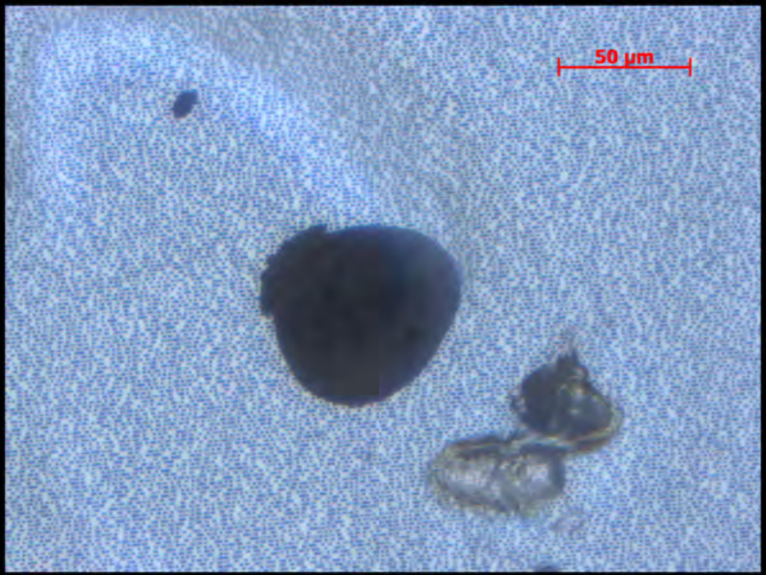
The "go-team"

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

"active learning"

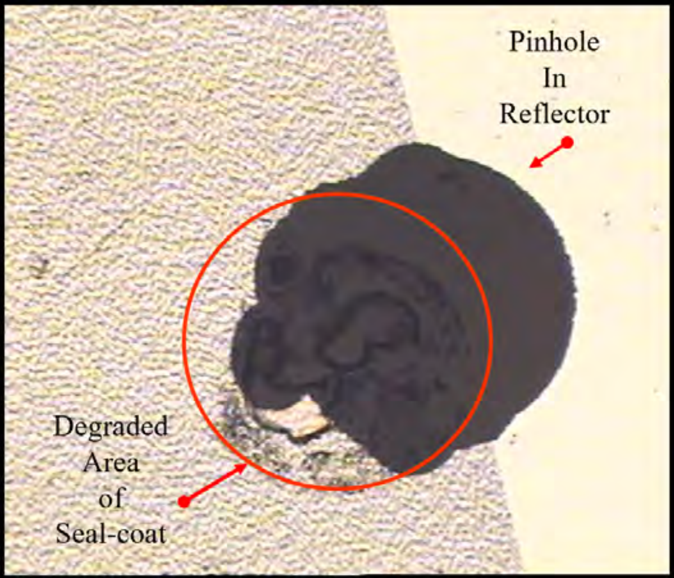
Challenges of Historic (and Modern) Storage Media

CD Degradation – Reflective Layer
'CD-Rot' – Type 1



CD – Read Side

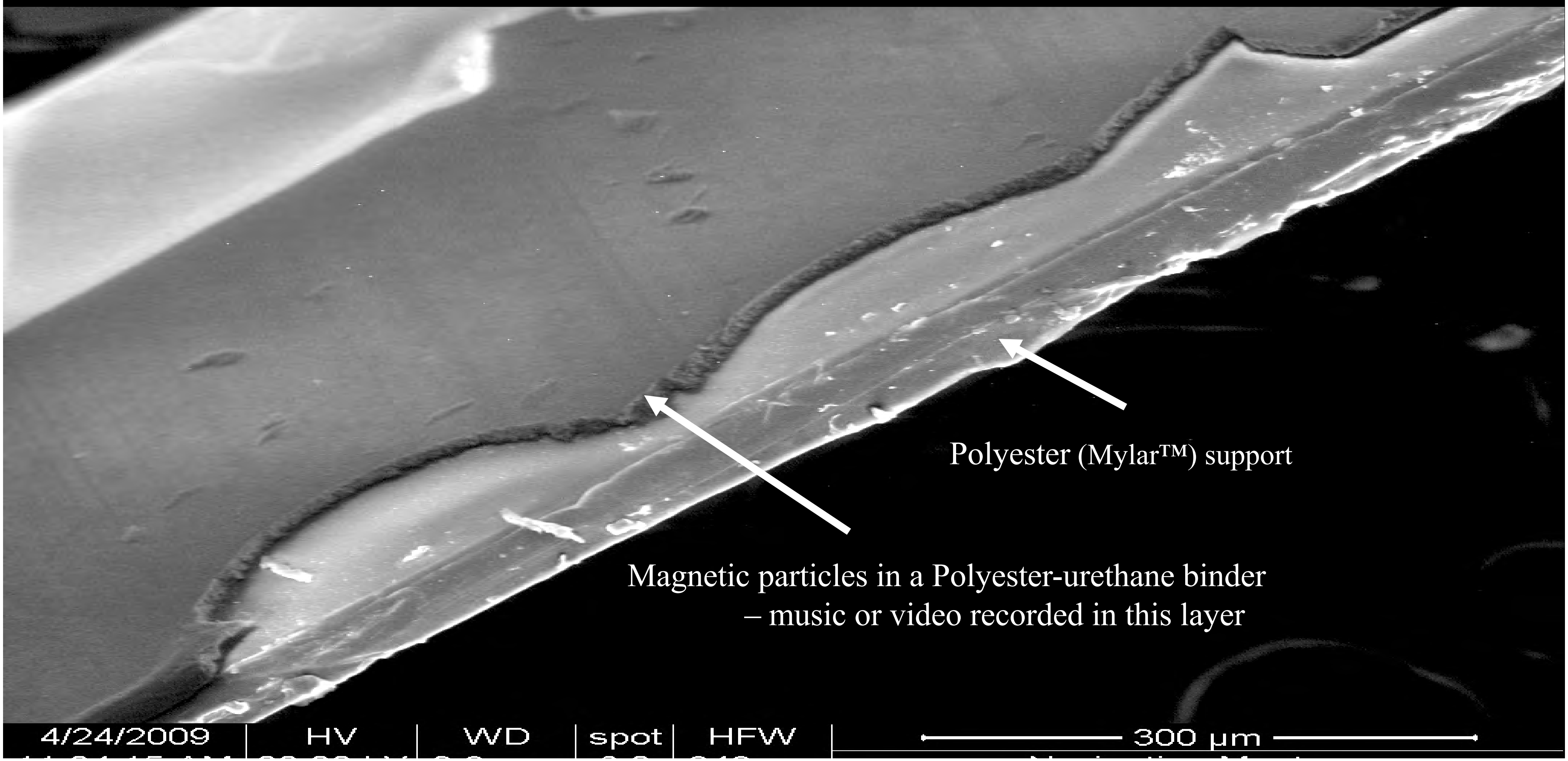
Pin-hole defects in the metal reflector caused by oxidation of aluminum layer, which becomes transparent to the read laser



CD – Label Side

Degradation of the seal-coat apparent above the hole in the reflector

Degradation of *(Early)* Magnetic Tape Formats



The Packard Campus of the National Audio-Visual Conservation Center (NAVCC)

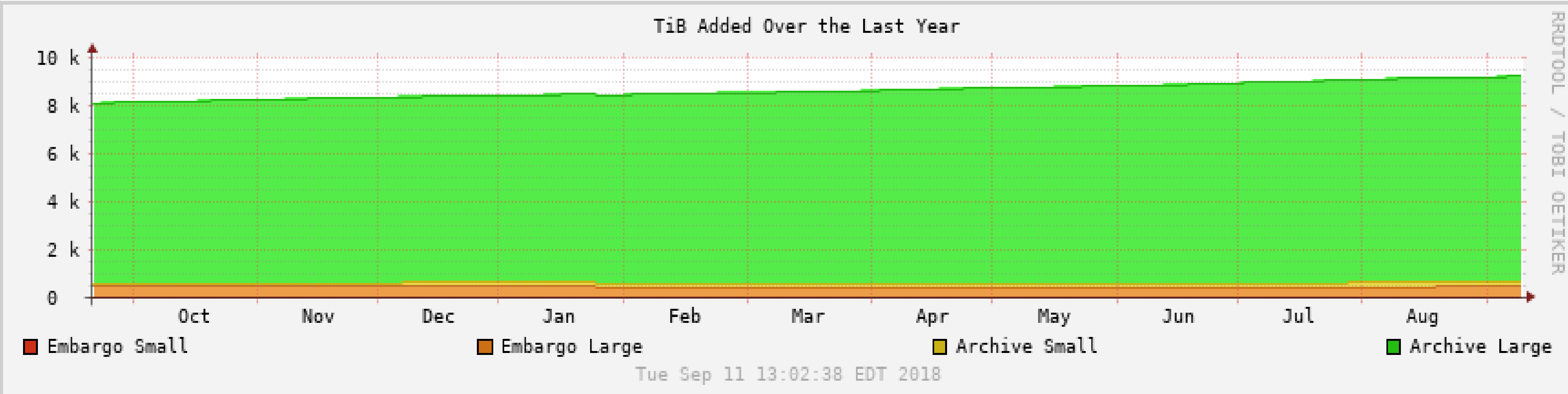
- A center for acquisitions, preservation, access, and partnerships
- 415,000 square feet, more than 90 miles of shelving for collections storage
- 35 climate controlled vaults for sound recording, film, and videotape
- 124 individual vaults for more flammable nitrate film

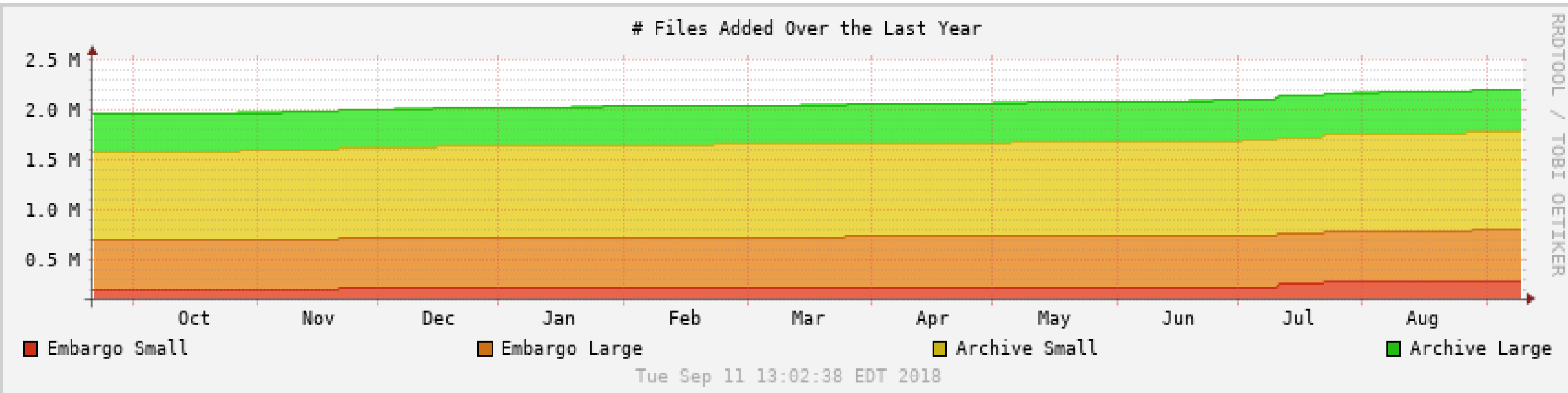
Packard Campus – Many formats

Nitrate Vaults

NAVCC Current State

- Current: 9.6 PB and 2.2 Million files replicated in 2 locations *(2 different media , two different systems)*
  - Compared to 5.1 PB and 410 Million files for Newspapers, internet archive, prints and photographs, etc. Long Term Storage (LCBP)
- 53 Points of Digitization (PODs):
  - 4K preservation for film is new this year
  - Programmatic Orderless ingests for Senate, NFL, and Saturday Night Live
  - 34 Solos (16 in robotic cabinets), 9 Pyramix, 10 Linux(OpenCube, etc) 1 Quadriga, 2 DVD Rippers, 1 CD Ripper, Oxberry, Arrilaser, Spirit, Vario, Clipster
  - Daily each POD can generate: 2GB-150GB for audio and 50GB-1,200GB for video
- Additional workflows coming in the future include Born Digital Copyright Audio submissions (Podcasts), Live capture-264 DVRs, PBS and others


TiB Added Over the Last Year
10 k
8 k
6 k
4 k
2 k
0
Oct
Nov
Dec
Jan
Feb
Mar
Apr
May
Jun
Jul
Aug
Embargo Small
Embargo Large
Archive Small
Archive Large
Tue Sep 11 13:02:38 EDT 2018
RRDTOOL / TOBI OETIKER


Files Added Over the Last Year
2.5 M
2.0 M
1.5 M
1.0 M
0.5 M
Oct
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Embargo Small
Embargo Large
Archive Small
Archive Large
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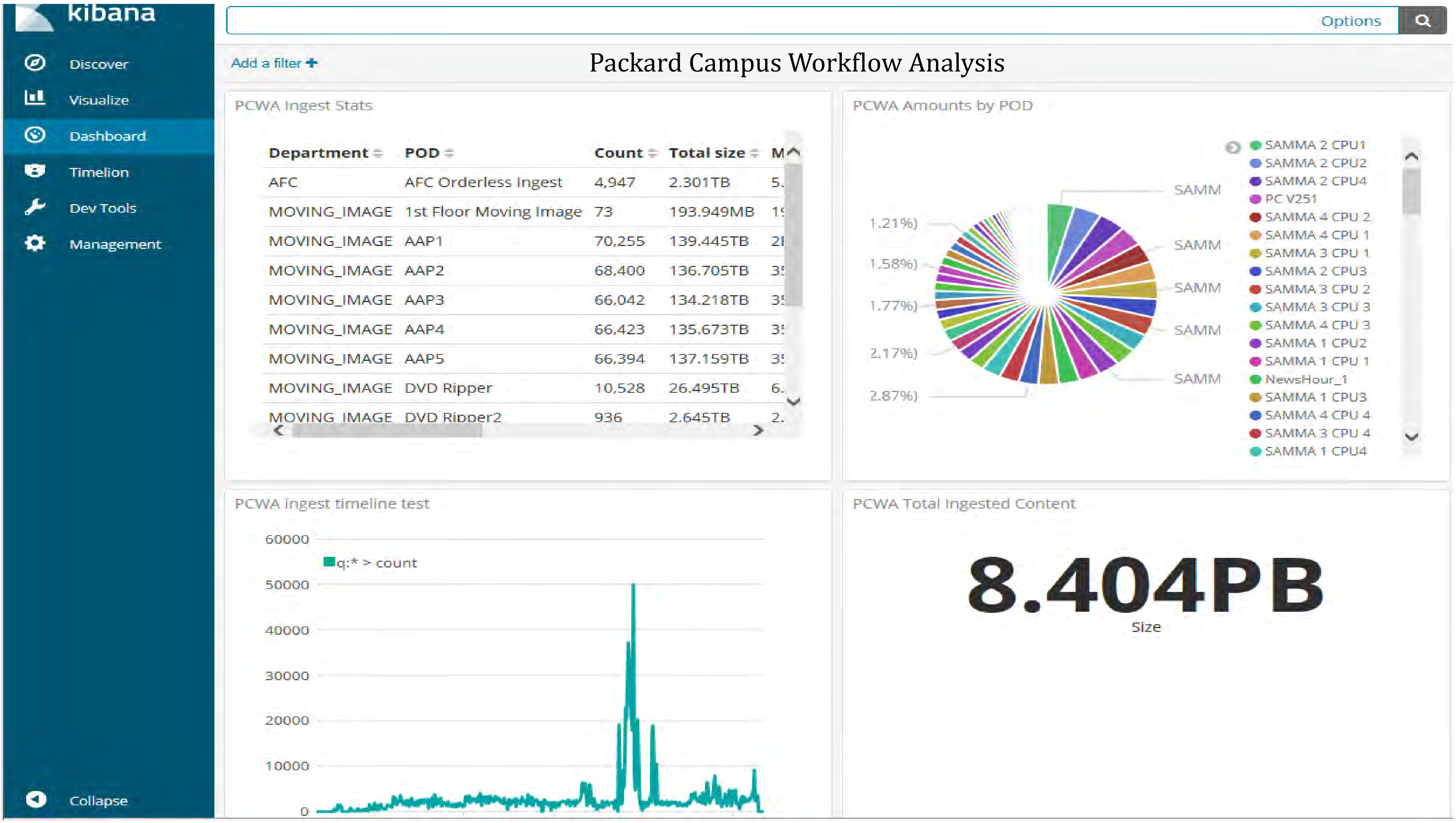
Packard Campus Workflow Analysis

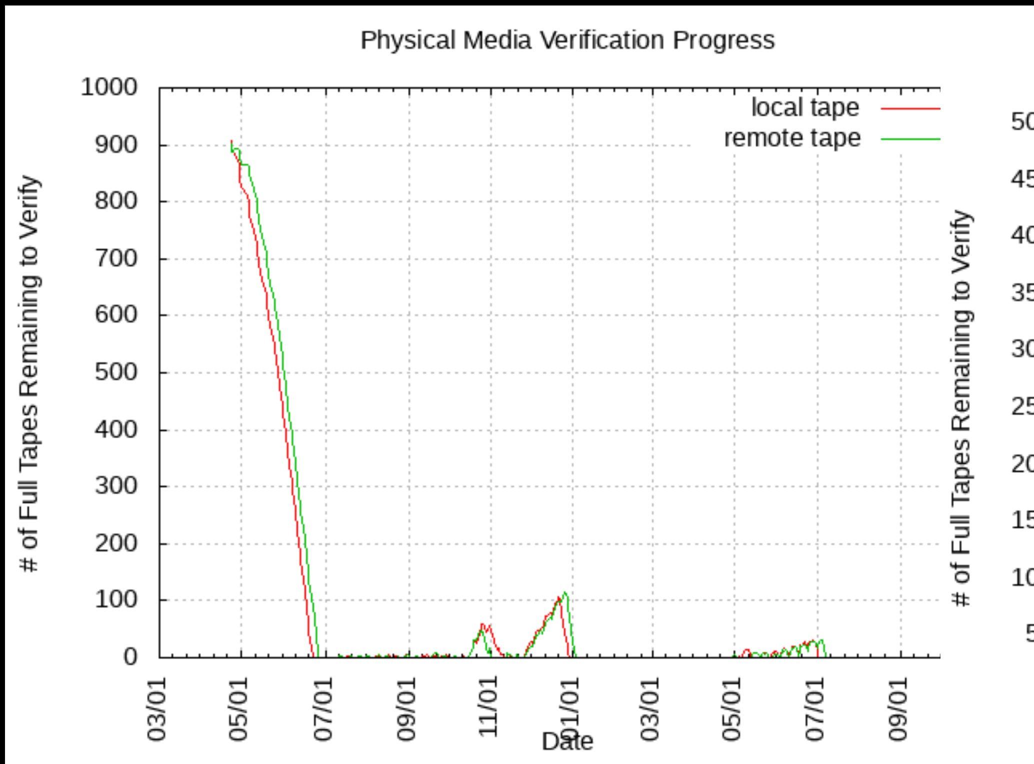


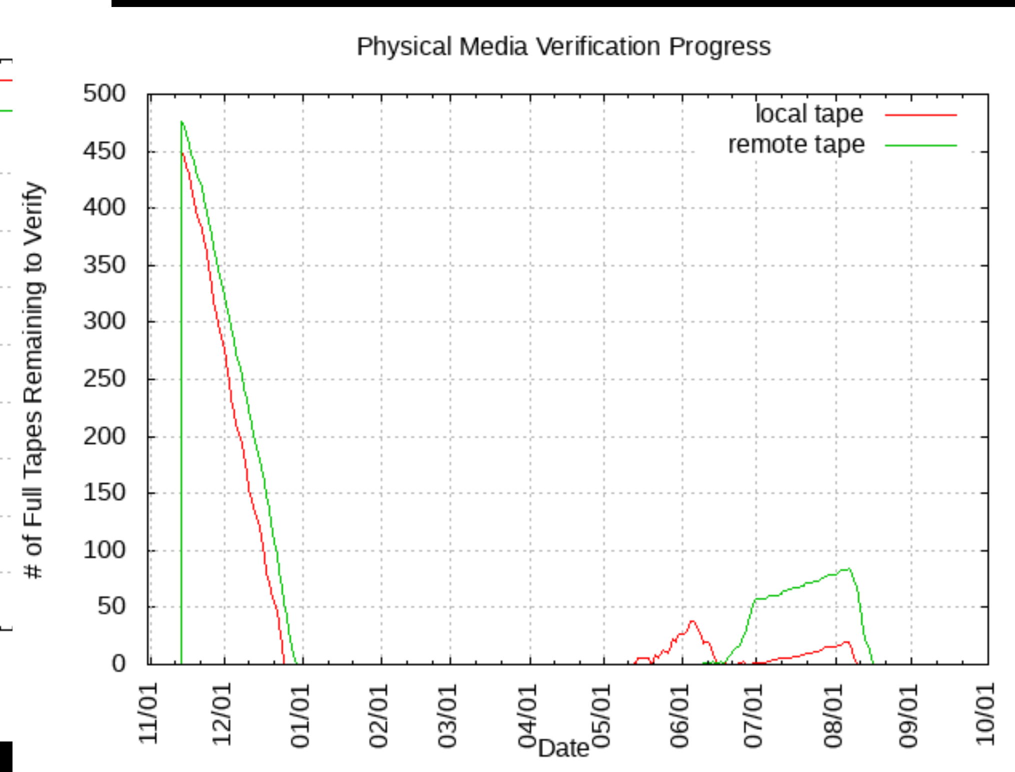
LOC Verify and Correct at the Marginal

Content is different than data

- Reduce the likelihood of content loss while recognizing that data loss is inevitable
- Catch and correct all marginal errors and failures as soon as possible
- Verify all the content at a regular interval
- Some of the regular verification processes that are run:
  - Samfsbackup (meta data backup) 5X/day
  - Verify samfsbackup size and frequency. Send an email if missing
  - Fix damaged files. Occasionally a file will be marked damaged because it cannot be retrieved from tape. Usually because a tape was stuck in a drive/robot/pass thru port. Find these everyday and attempt to stage. If we can't, then send an email. Send an email when we find damaged files so we know issues are occurring and being corrected
  - Stats: Watch the # and size of files waiting to archive. Warn when the # of files or size of files exceeds thresholds. Usually an indication of some marginal error condition. Fix before file system fills up or we fail to deliver a file for customers
  - Samfsck: Run this daily with filesystem mounted. Warns when there are marginal conditions with file system before they are catastrophic
  - # of tapes/TB available: Know when we are running low so we can correct before a failure
  - Tpverify: Verify all tapes with data every 6 months. Verifying header and all blocks of data on tape with CRC.

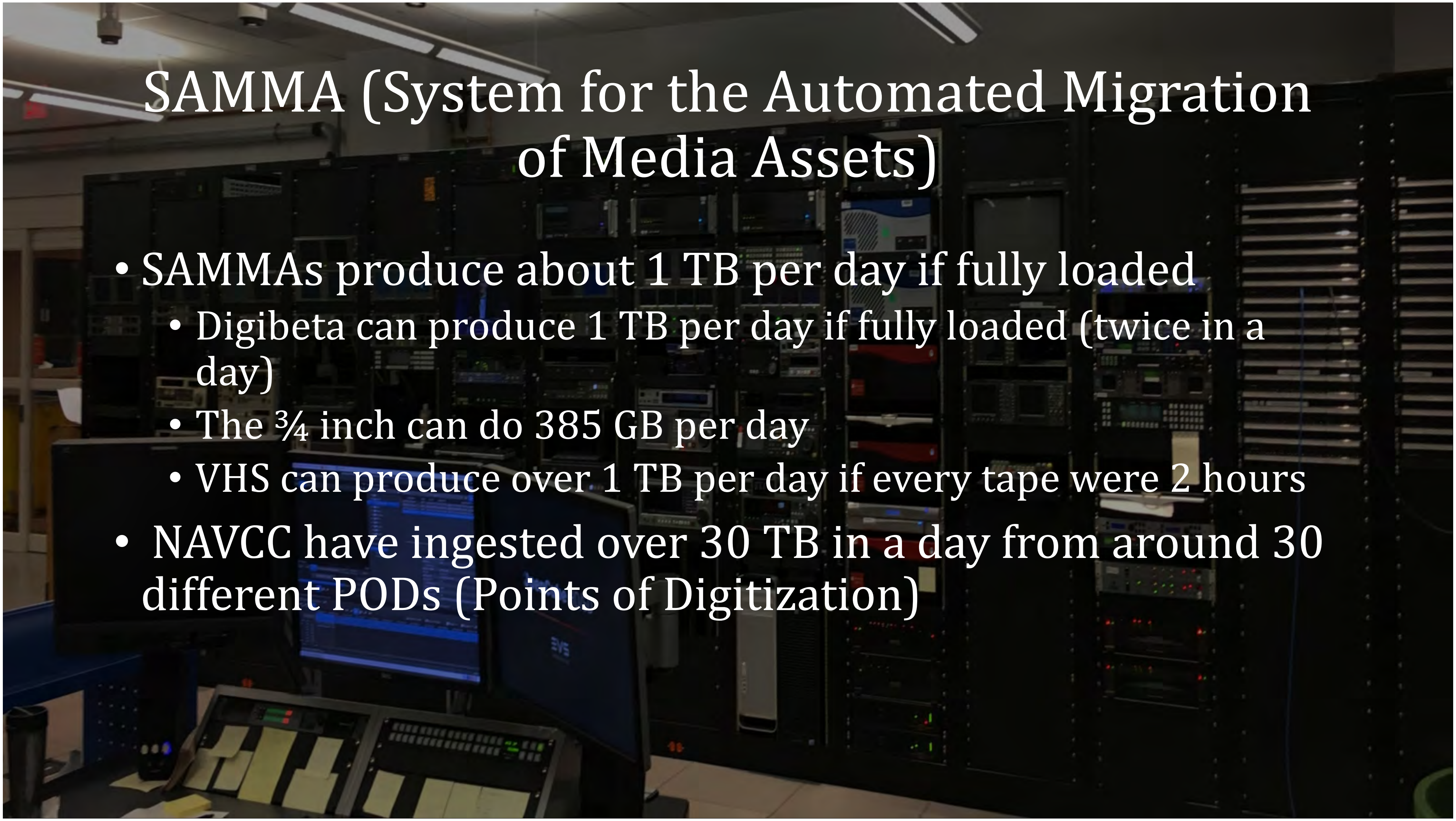
LOC Media Verification (NAVCC and LCBP)





- Every tape is loaded to a tape drive every 6 months and the CRC codes for each block are checked


YEA 46 NAY 53
S.J. Res. 1, the Mitchell/Nunn
Persian Gulf Resolution
172.22.6.234



SAMMA (System for the Automated Migration of Media Assets)

- SAMMAs produce about 1 TB per day if fully loaded
  - Digibeta can produce 1 TB per day if fully loaded (twice in a day)
  - The ¾ inch can do 385 GB per day
  - VHS can produce over 1 TB per day if every tape were 2 hours
- NAVCC have ingested over 30 TB in a day from around 30 different PODs (Points of Digitization)

NAVCC Observations

- Migrating from T10K-C to T10K-D was cost-effective
- Doubled the capacity and reduced floor space requirements for future growth
  - Plan on reducing tape cabinet needs and clearing floor space
  - Last migration completed without issues
  - Verification after write to tape necessary, even if only sampling
- Next migration will be LTO, if tape is still the best TCO
  - LC is taking advantage of Cloud where effective
- SSD offers power and cooling that fits NAVCC limitations:1 PB in 1 RU?
- Customers require TB of short term storage for projects
  - Force them to request for short periods. Program deletions from shared NAS

Storage at LC (Media, Data, Content)

- For LC current storage greater than 100 PB
  - Data 54 PB
  - Content 20 PB
  - NAVCC 9.6 PB
- Four data centers – 4 locations
- At NAVCC, about 150 TB of disk storage for daily content capture
  - Disk storage transferred to 2 copies of tape, 1 at the NAVCC location
  - The file contents are typically deleted within a week and a pointer is left on the system
  - A 22 GB file can be restored in 5 min

Audio Tape Degradation – Sticking, Squealing, Shedding

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

Photos: Christine Folivi, ACS SEED 2018

	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	

Thermal Analysis – Differential Scanning Calorimetry

Glass transition point for polymers

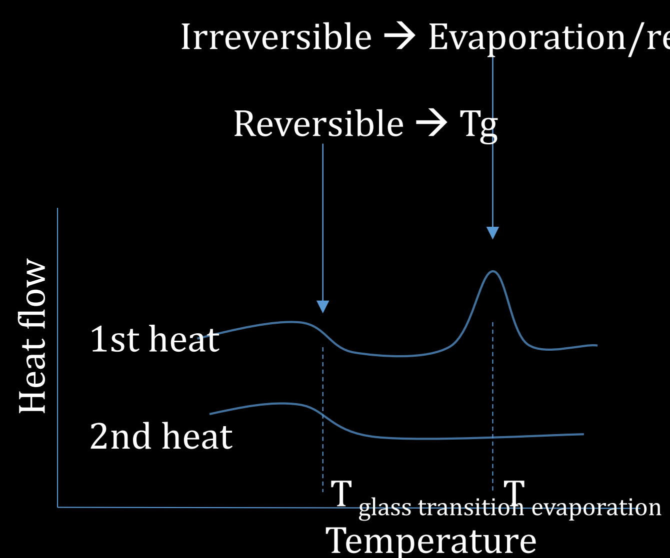
5-10 mg sample is heated at fixed rate (°C/min)

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

Dips, peaks, step-changes in heat flow

⬇

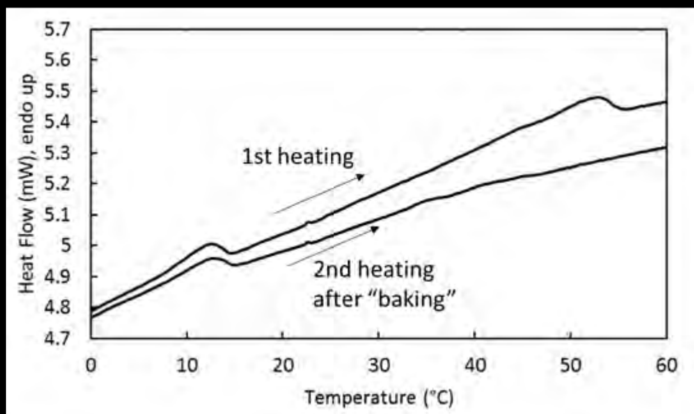
Thermally induced change in state

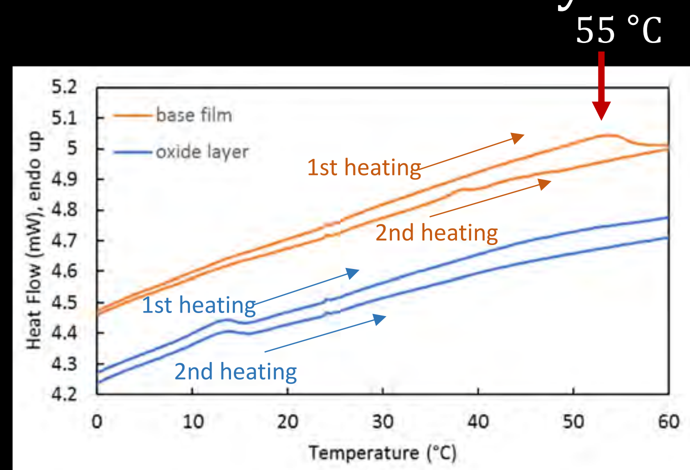


Thermal Analysis – Differential Scanning Calorimetry

Evidence of Thermal Transitions in “Sticky” Tape

Layer Differences Observable in Thermal Analysis



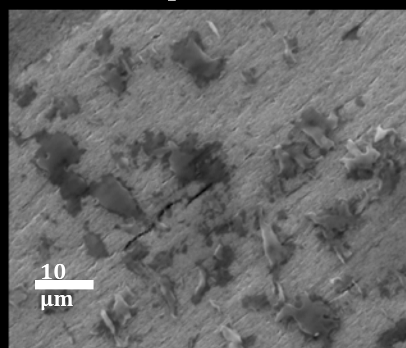


Using material from separated layers:
Low temperature Tg (15°C) in **oxide layer**
“Bake” temperature transition in **base film**

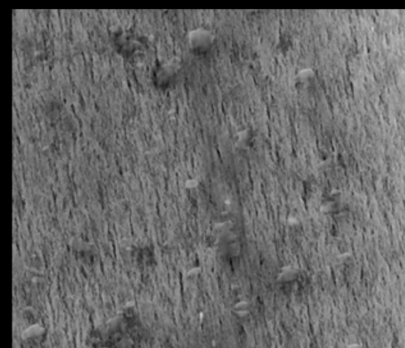
Electron Microscopy – Tape “twins”

Sticky

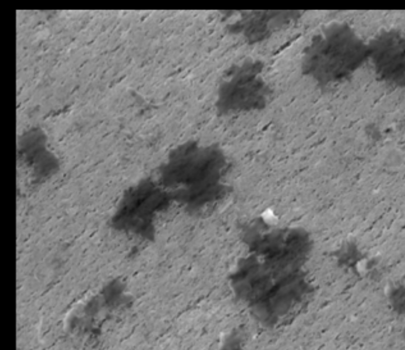
Ampex 456



3M 908



Ampex 406

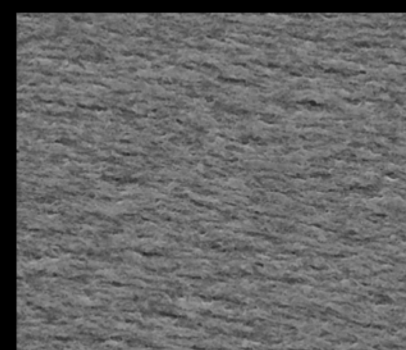


Non-Sticky

Ampex 456

3M 908

3M 208

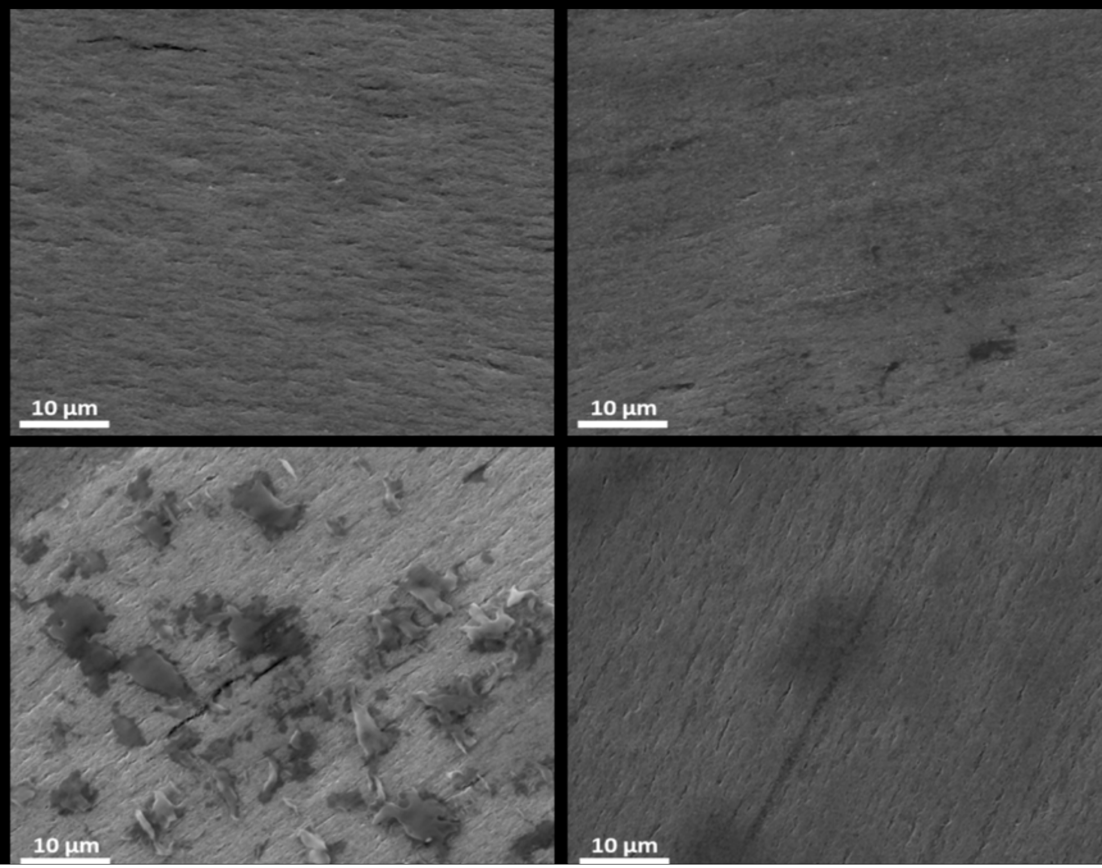


Electron Microscopy of Baked Tapes

Unbaked | **Baked**

Ampex 456 non-sticky

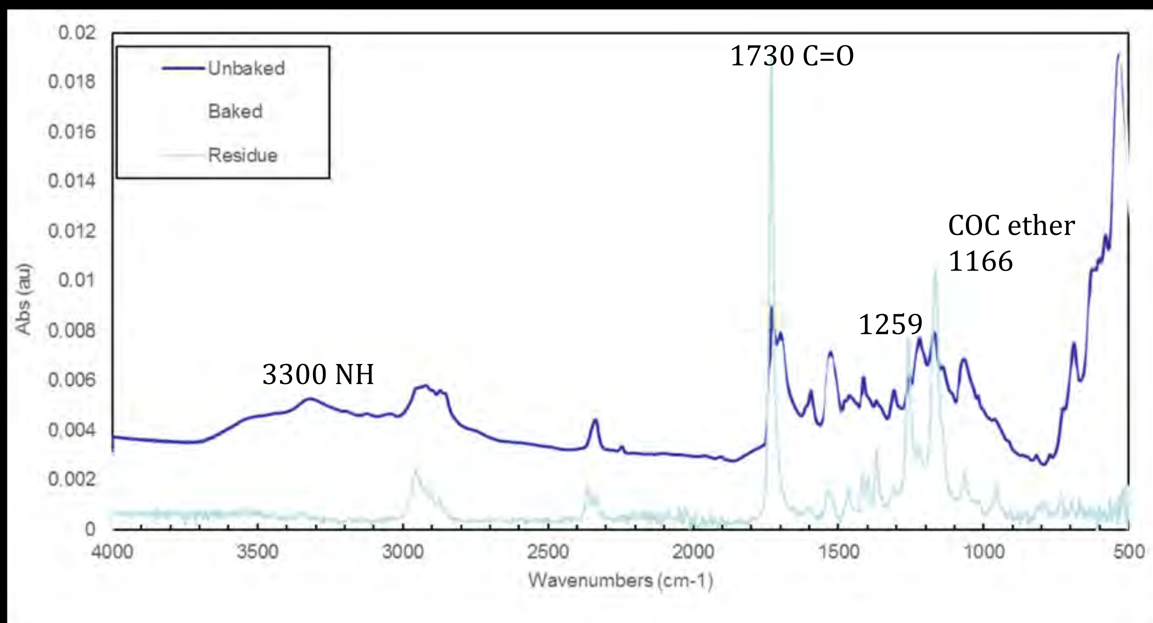
Ampex 456 sticky



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

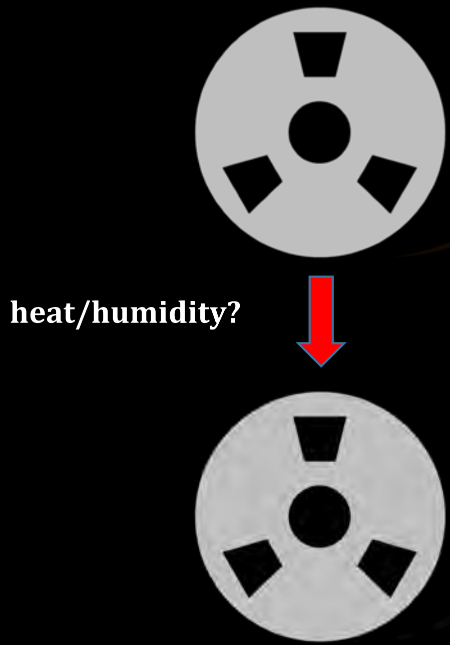
Results suggestive of lubricant/plasticizers, NOT degradation from PU

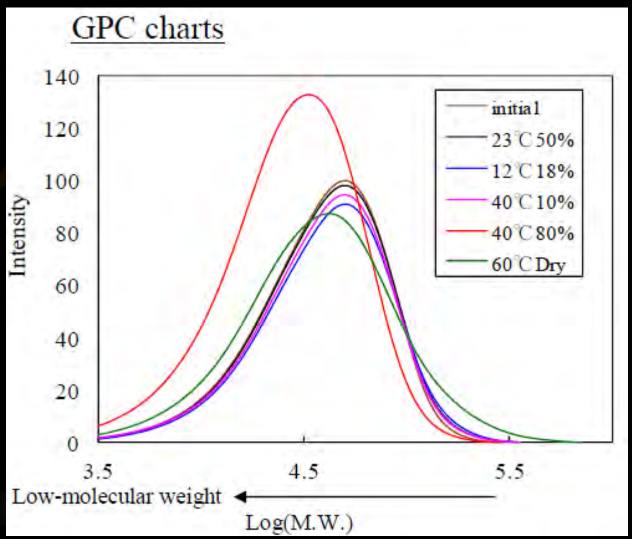
Strongest peaks (1730, 1259, 1166 cm^{-1}) correlate to peaks found to decrease after baking (both here, and other studies)



Fourier Transform Infrared Spectroscopy (FTIR)

Can we make a sticky tape?





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

80°C/80%
40°C/80%
40°C/10%
60°C/0%
...

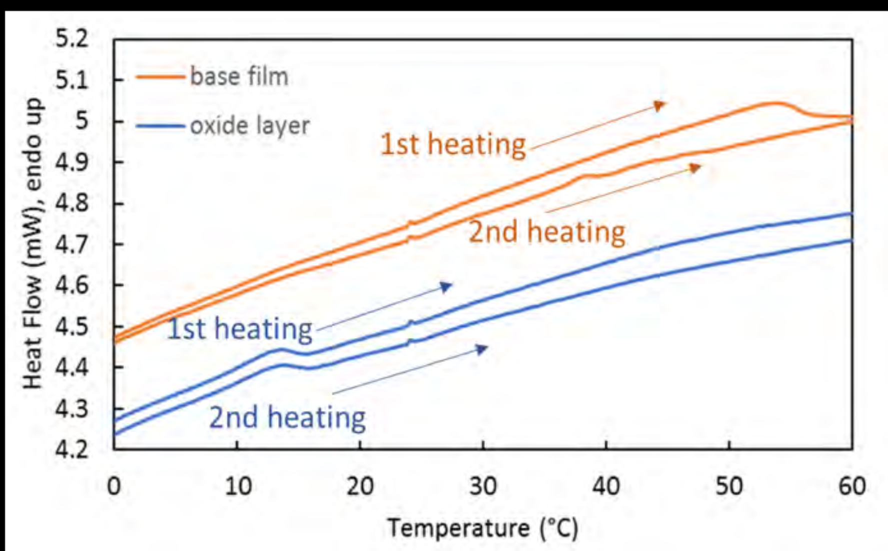
Can break down a tape, but difficult to reliably mimic a "sticky" tape

In collaboration with FujiFilm Japan

Analyses of stickiness and baking

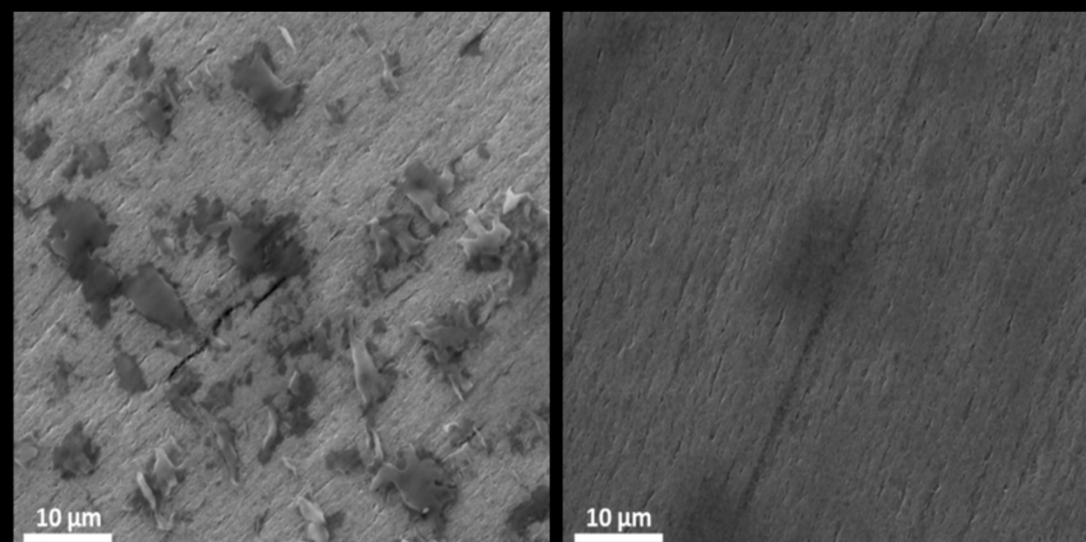
DSC thermal data

base film contributes to baking process



Microscopy data

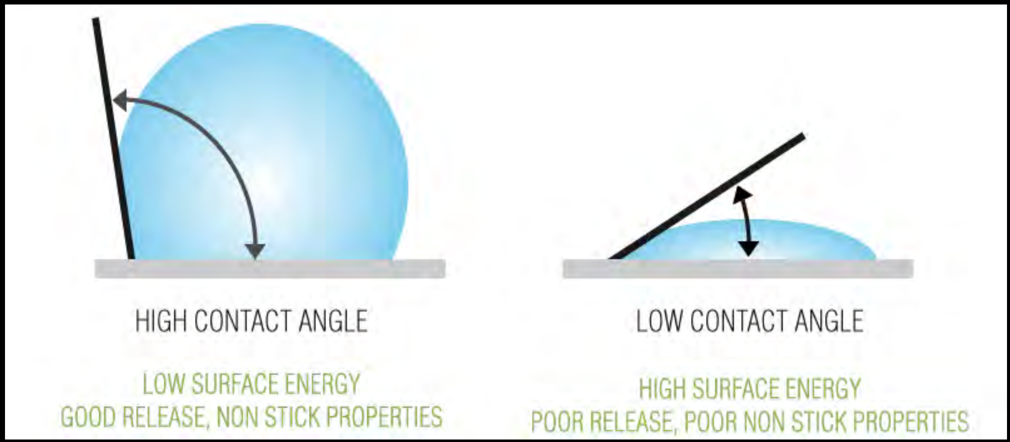
oxide layer shows visible restorative changes during baking



Water Contact Angle of Magnetic Media

Non-sticky

Sticky



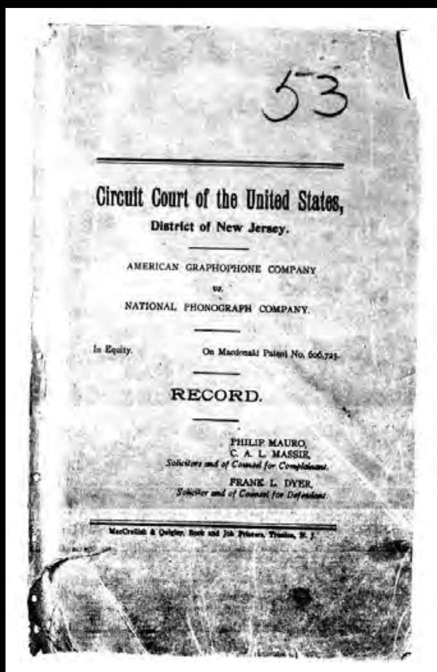
ptfecoatings.com

<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.

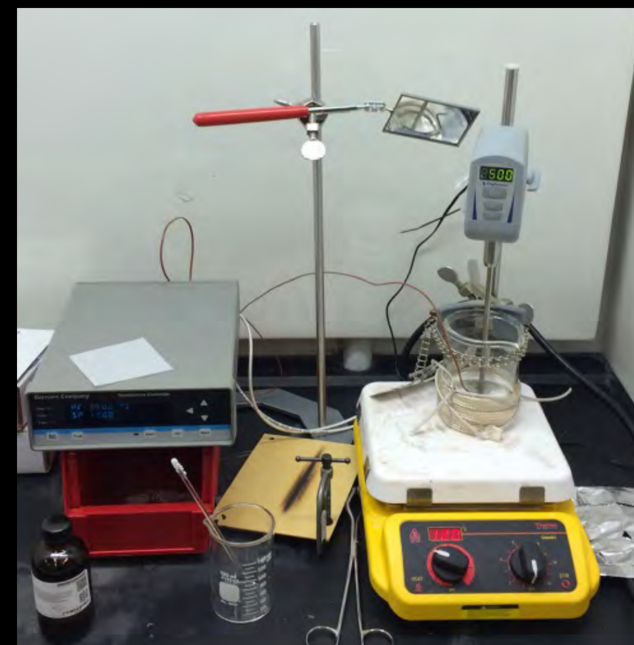
PHILIP MAURO,
C. A. L. MASSIE,
Solicitors and of Counsel for Complainant.

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

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

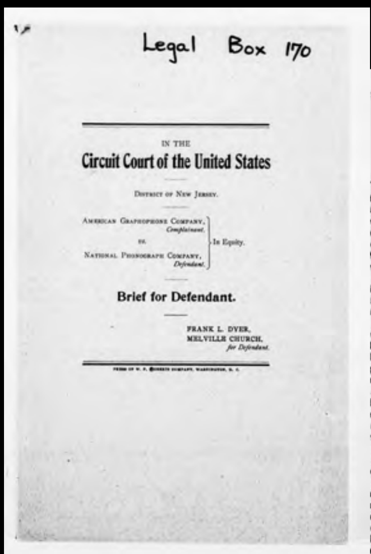
Historical Records

Chemical and Physical Testing



Laboratory Synthesis

Edison Papers Project @ Rutgers
Digitized lab notebooks



Legal Box 170

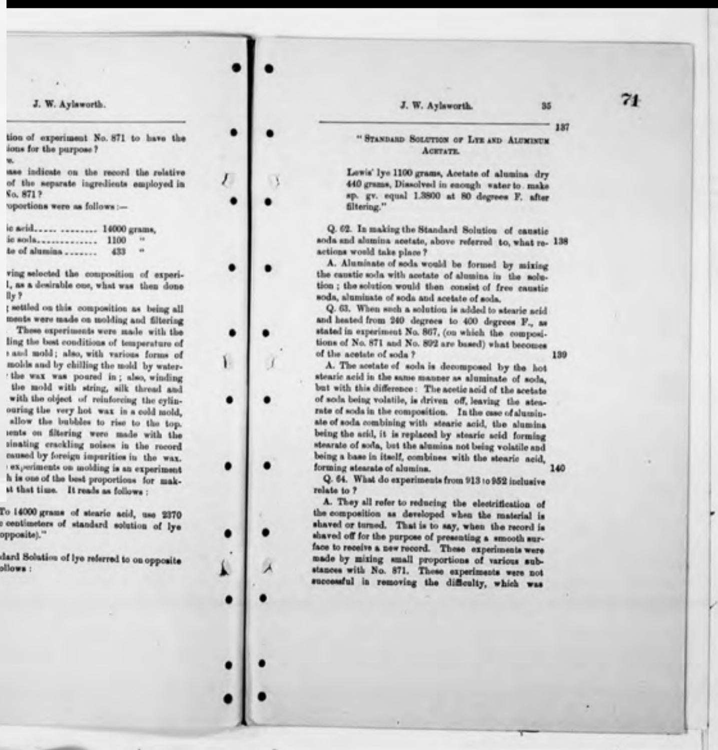
IN THE
Circuit Court of the United States

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.



J. W. Aylsworth.

"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 reactions would take place? 138

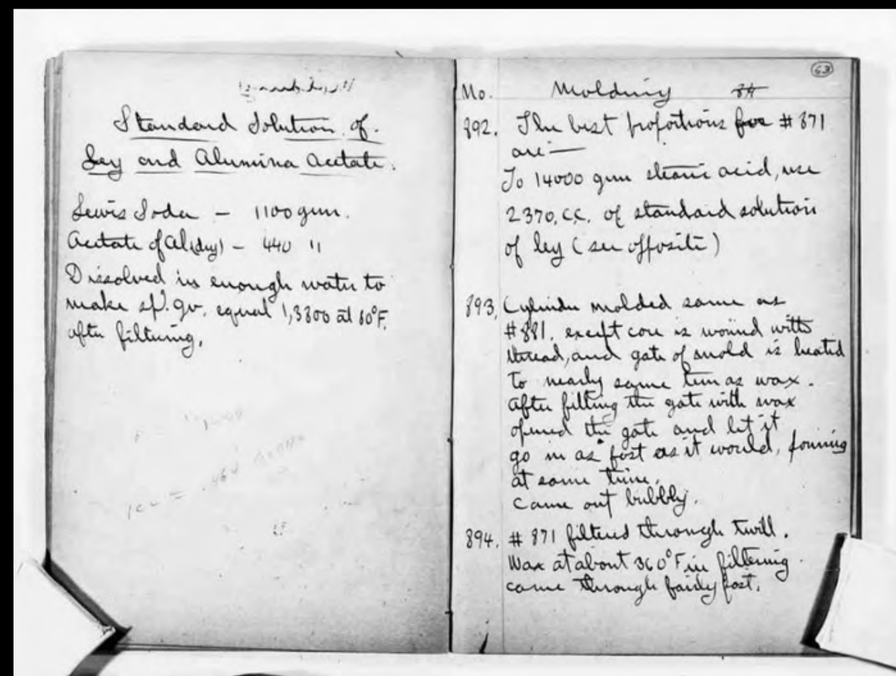
A. Aluminate of soda would be formed by mixing the caustic soda with acetate of alumina in the solution; the solution would then consist of free caustic soda, aluminate of soda and acetate of soda.

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



Standard Solution of
Lye 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.

63

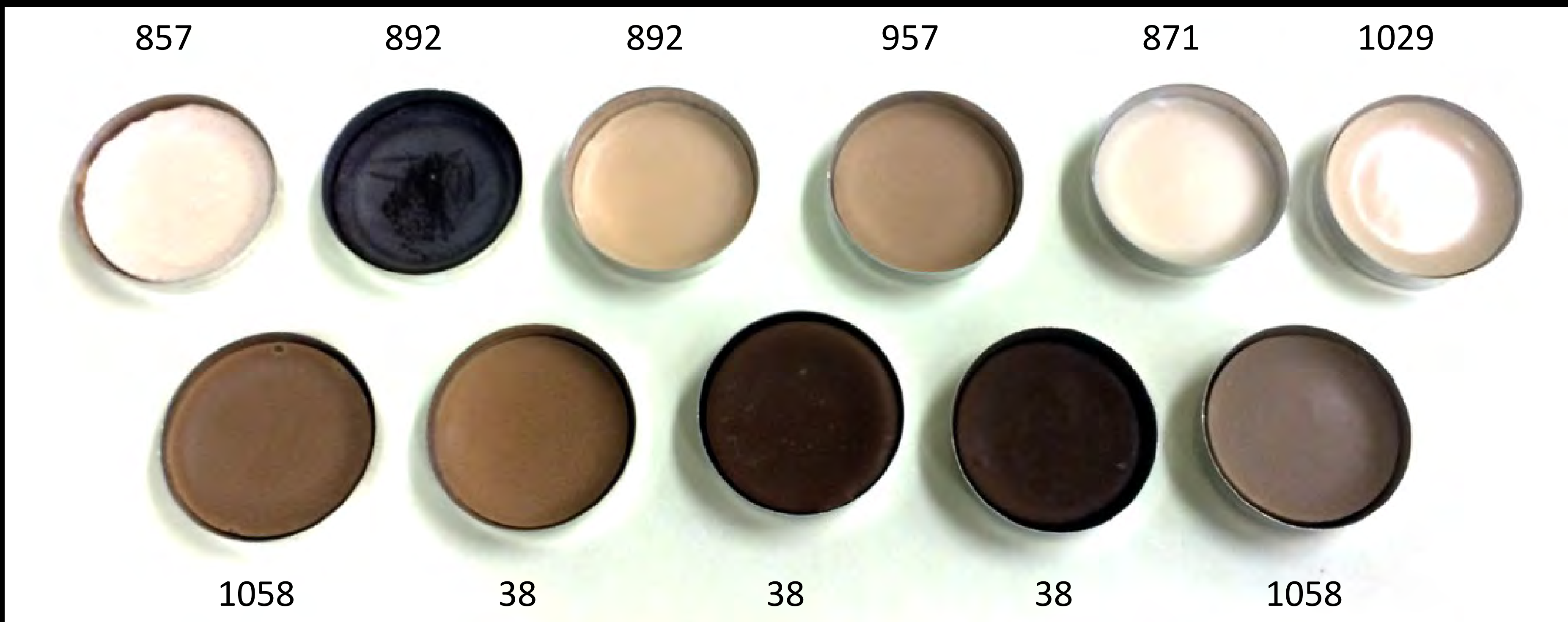
No. Molding

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

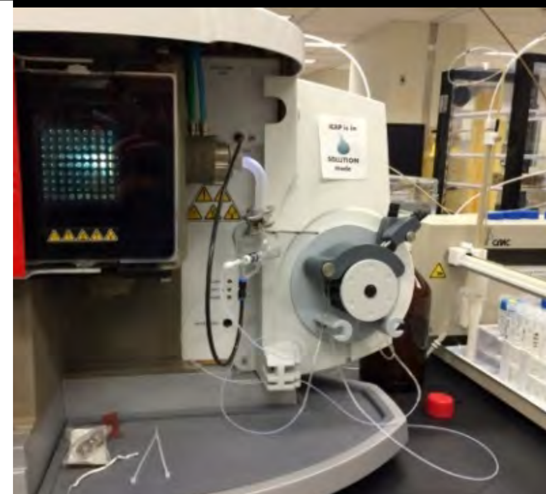
893. Cylinder molded same as #881, except core is wound with thread, and gate of mold is heated to nearly same temp as wax. After filling the gate with wax opened the gate and let it go in as fast as it would, forming at same time. Came out bubbly.

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

Initial reproductions of Edison cylinder formulations

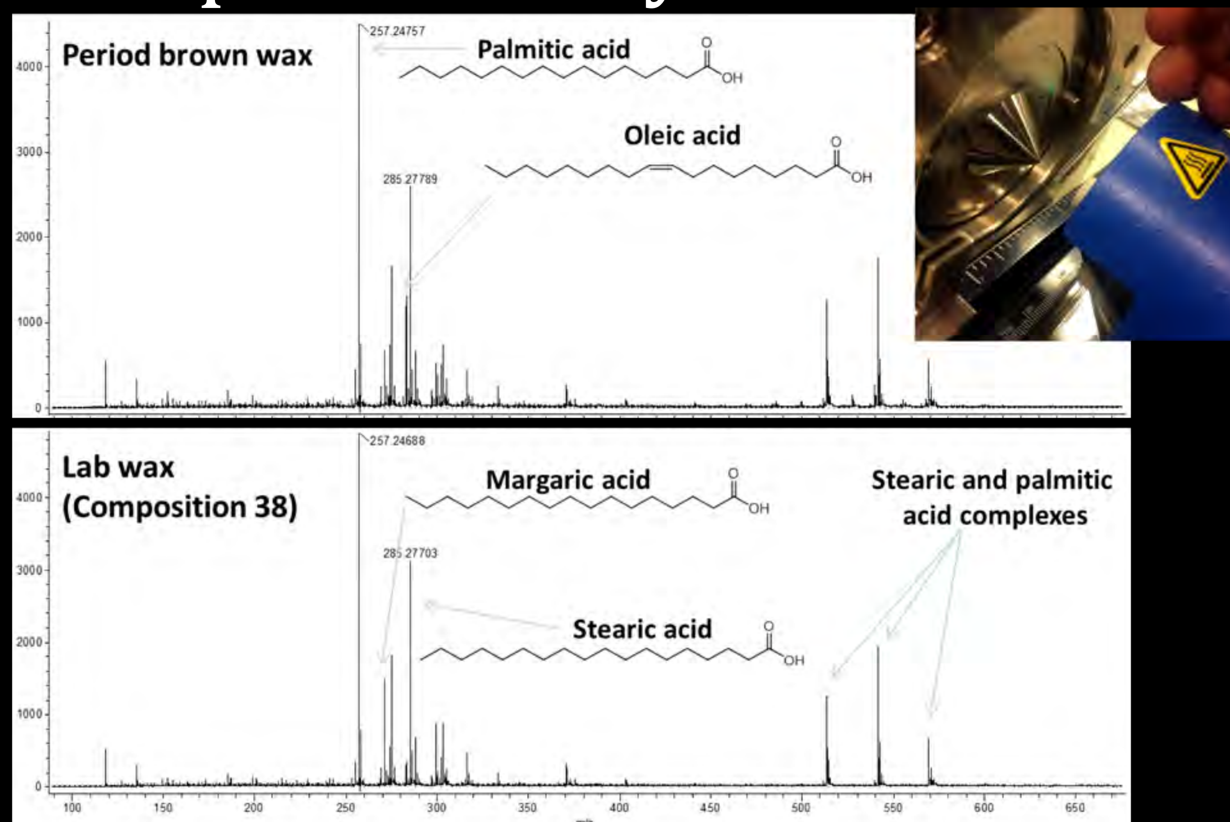


Examining metals content by ICP



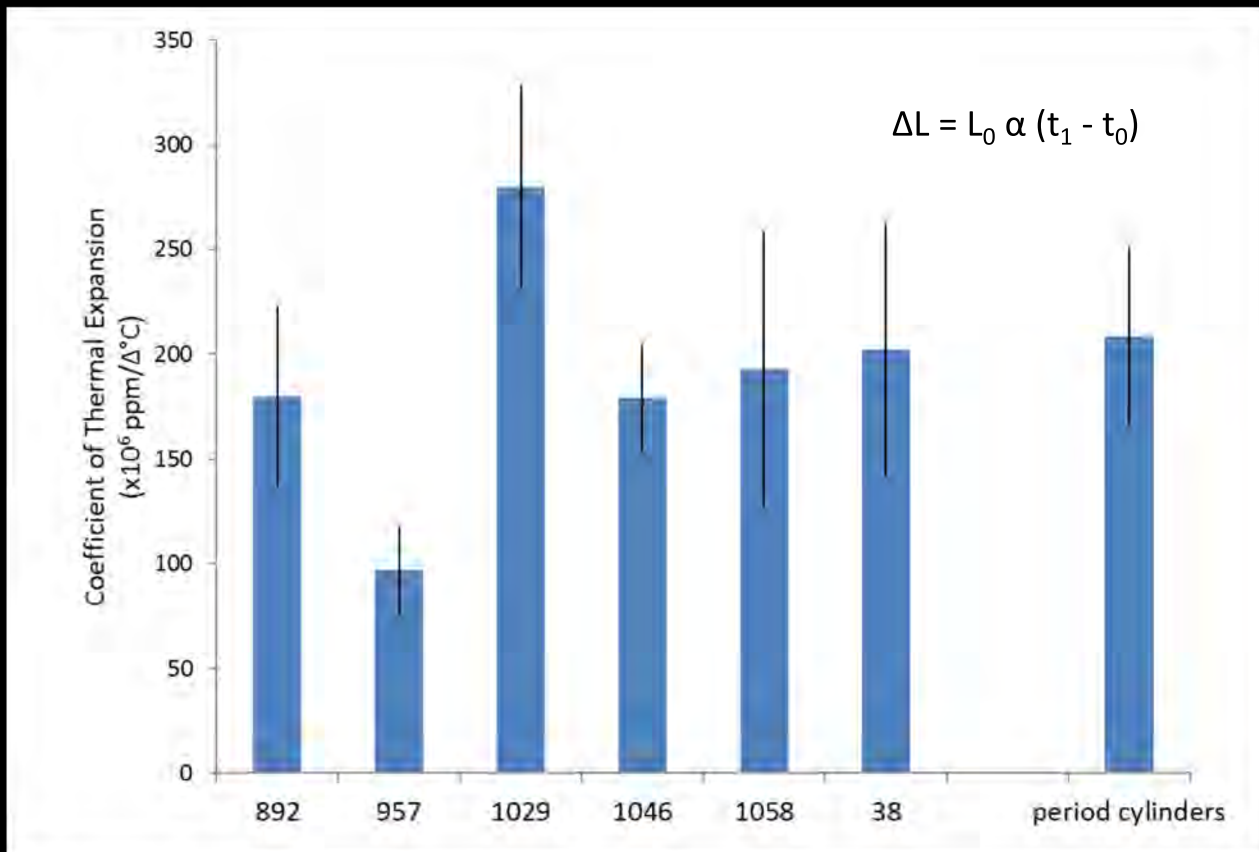
All values in
parts-per-million (ppm)

Organic compound analyses



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

Coefficient of thermal expansion

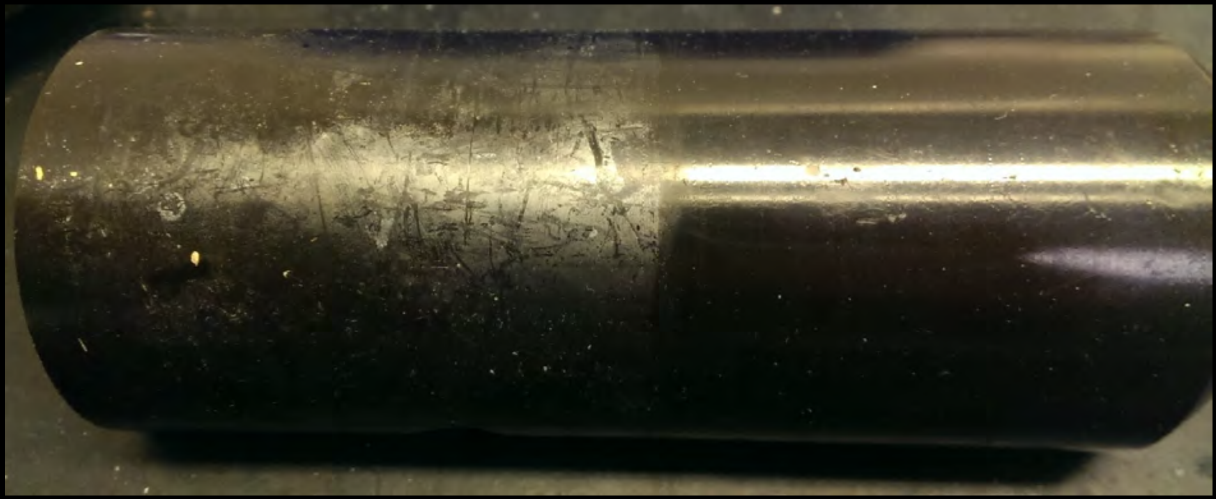


Creation and destruction of “pseudocylinders”

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…

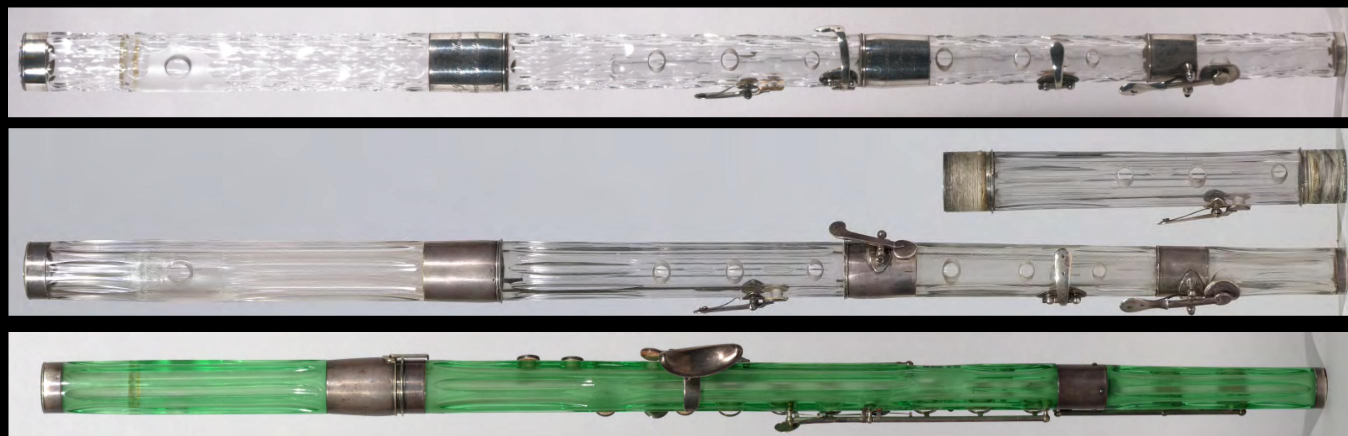
- Prototype solutions contained high acetonitrile for optimum cleaning, particularly during rinse
- Evaporative cooling could lead to rapid thermal change at surface – leading to breakage
- Not comfortable with the inherent risk

Technical Study of Claude Laurent’s Glass Flutes

DCM 378
(1813)

DCM 475
(1814)

DCM 11
(1844)



- Assessment of Dayton C. Miller collection at LC - some 19th century glass flutes made by Claude Laurent were showing “fogginess”

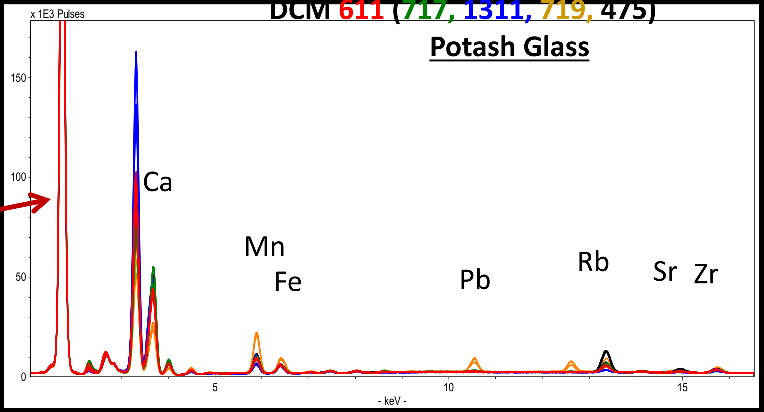
Project goals:

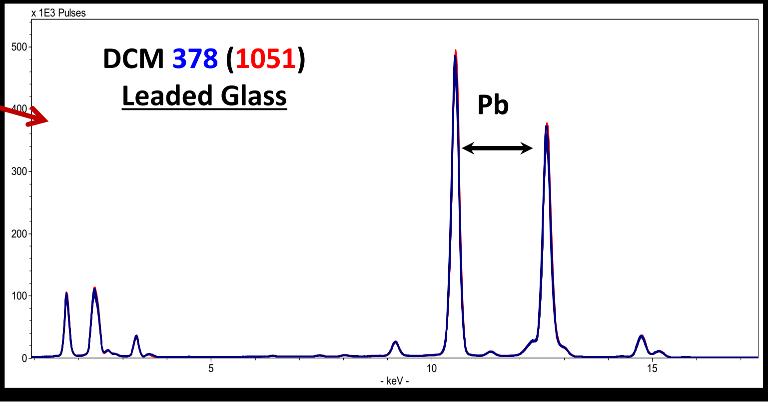
- Understand relationship between flute composition and chemical stability
- Develop ‘simple toolkit’ to analyze glass and in preservation

Determining Glass Composition

UV light examination (365 nm)

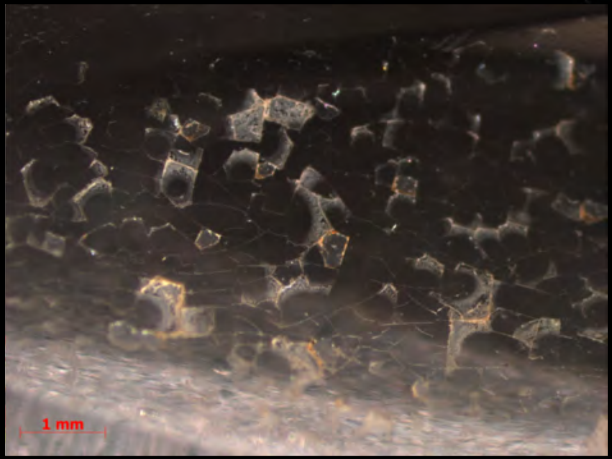
X-ray fluorescence (XRF)





Understanding and Quantifying Deterioration

DCM 717, upper body joint



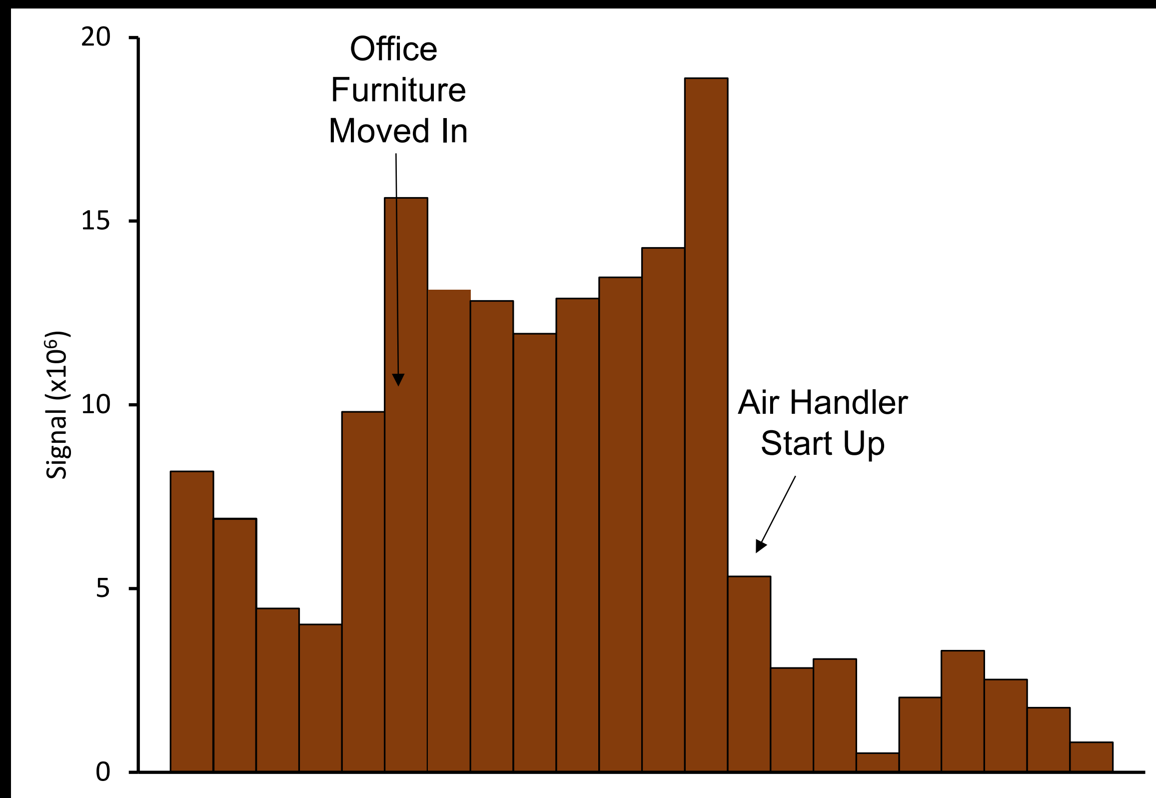
Light microscopy shows severe microcracking on flute surface

Lower % K than bulk glass

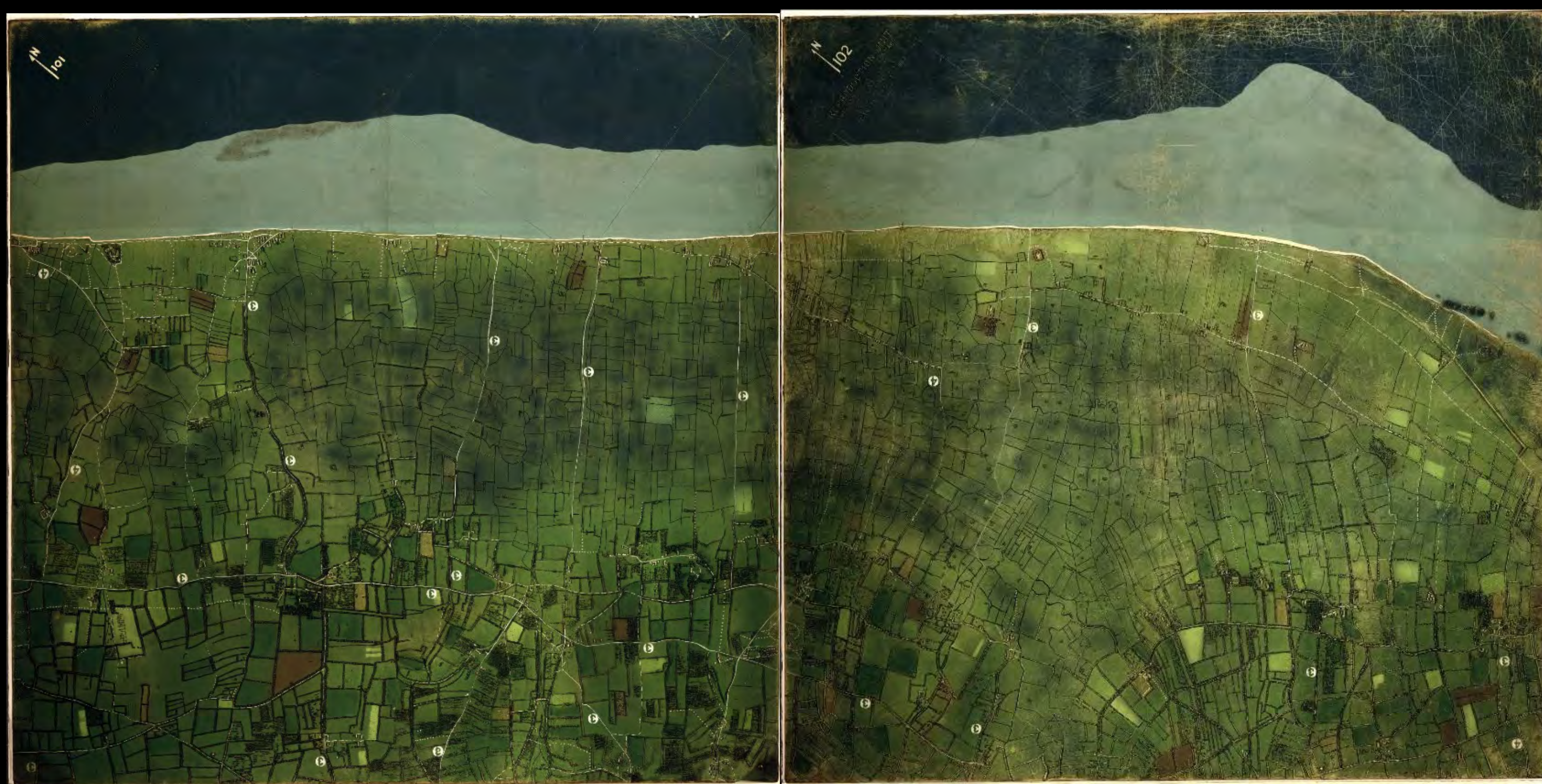
Electron microscopy of a glass chip cross section shows surface alteration layer

Renovation of collection storage spaces
Testing for Volatile Organics

Sampling storage area during renovations



Application of field sampling – WWII Map

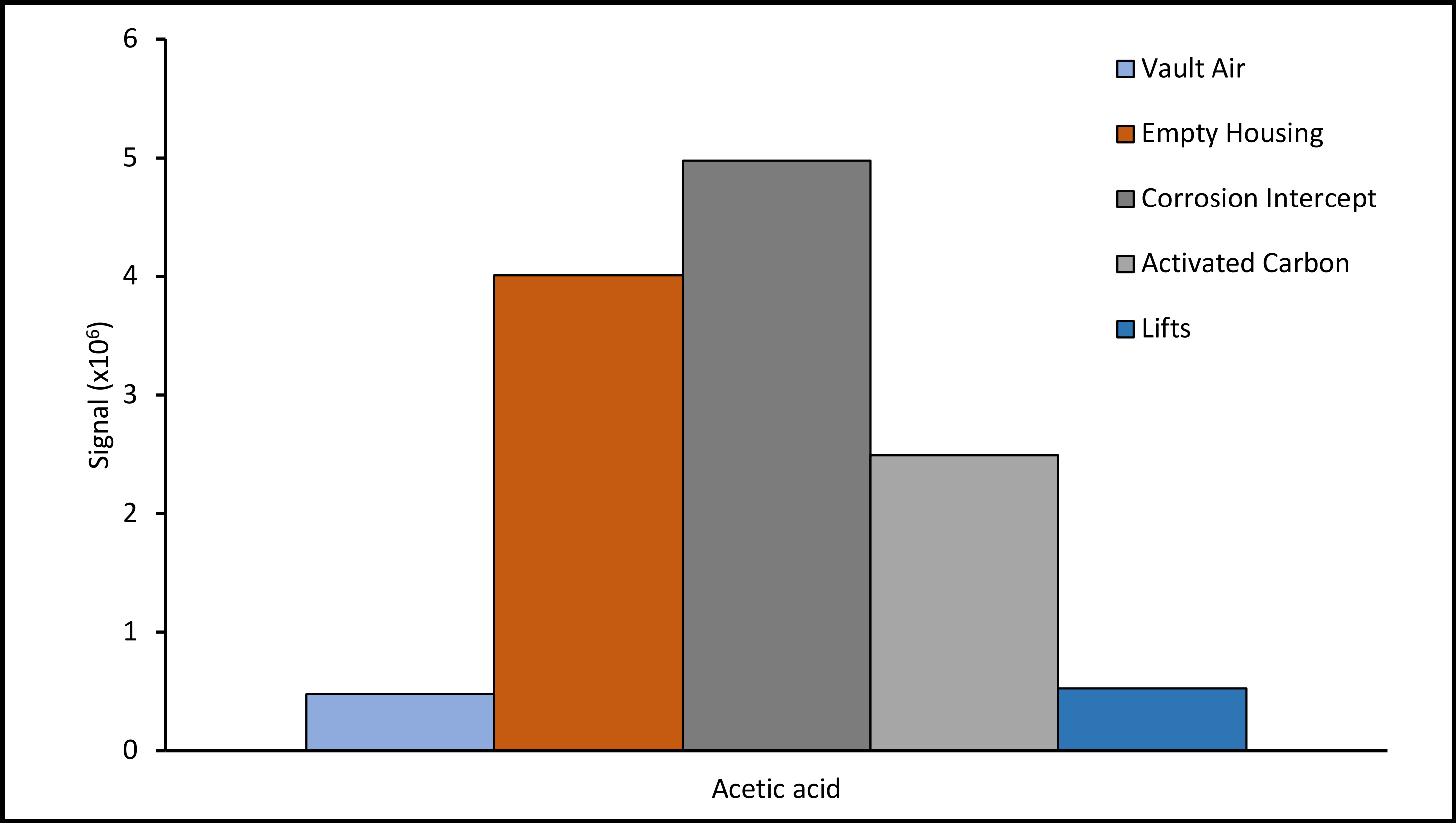


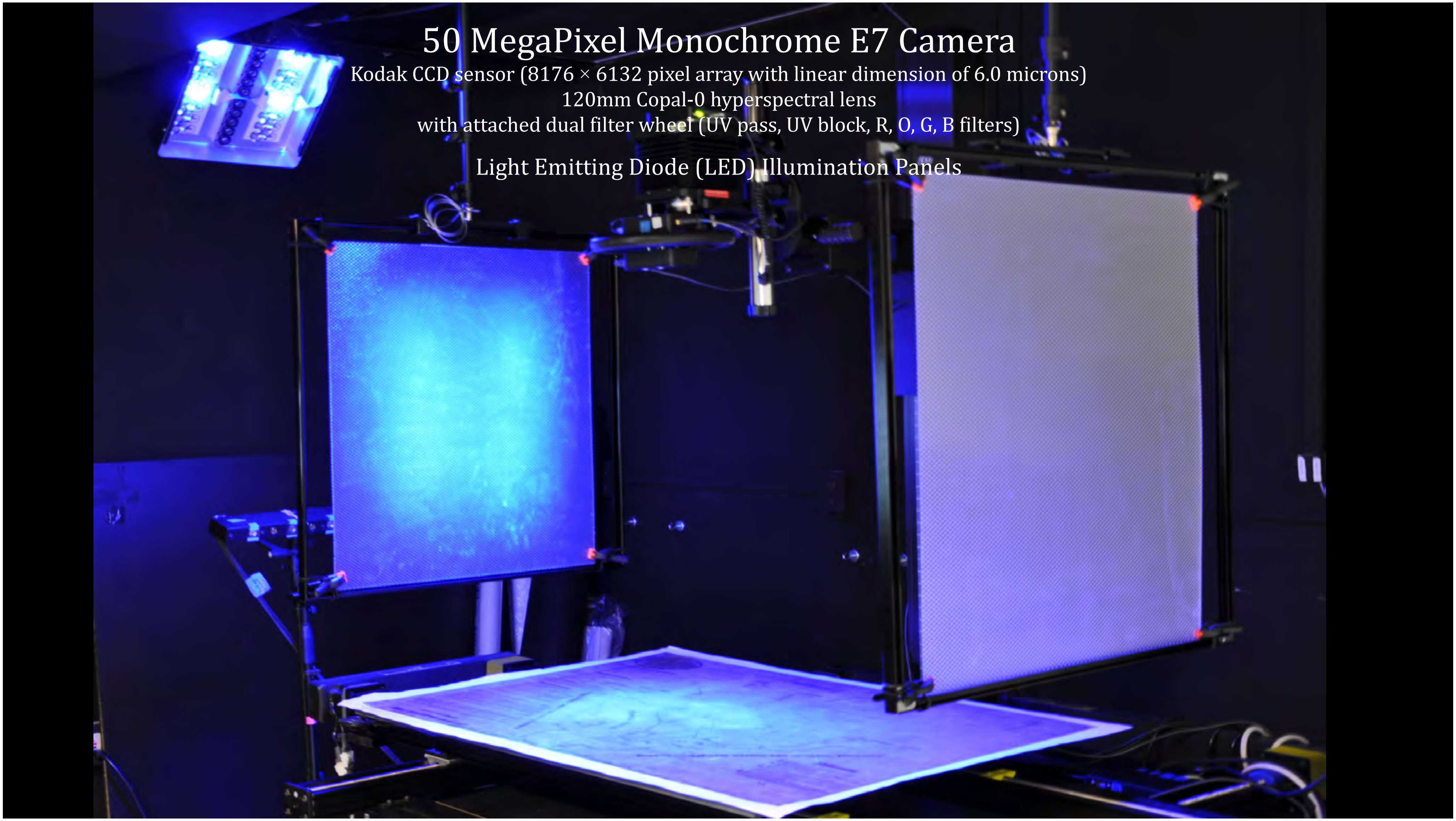
Application of field sampling – WWII Map

Thermal desorption of “self-sampled” material

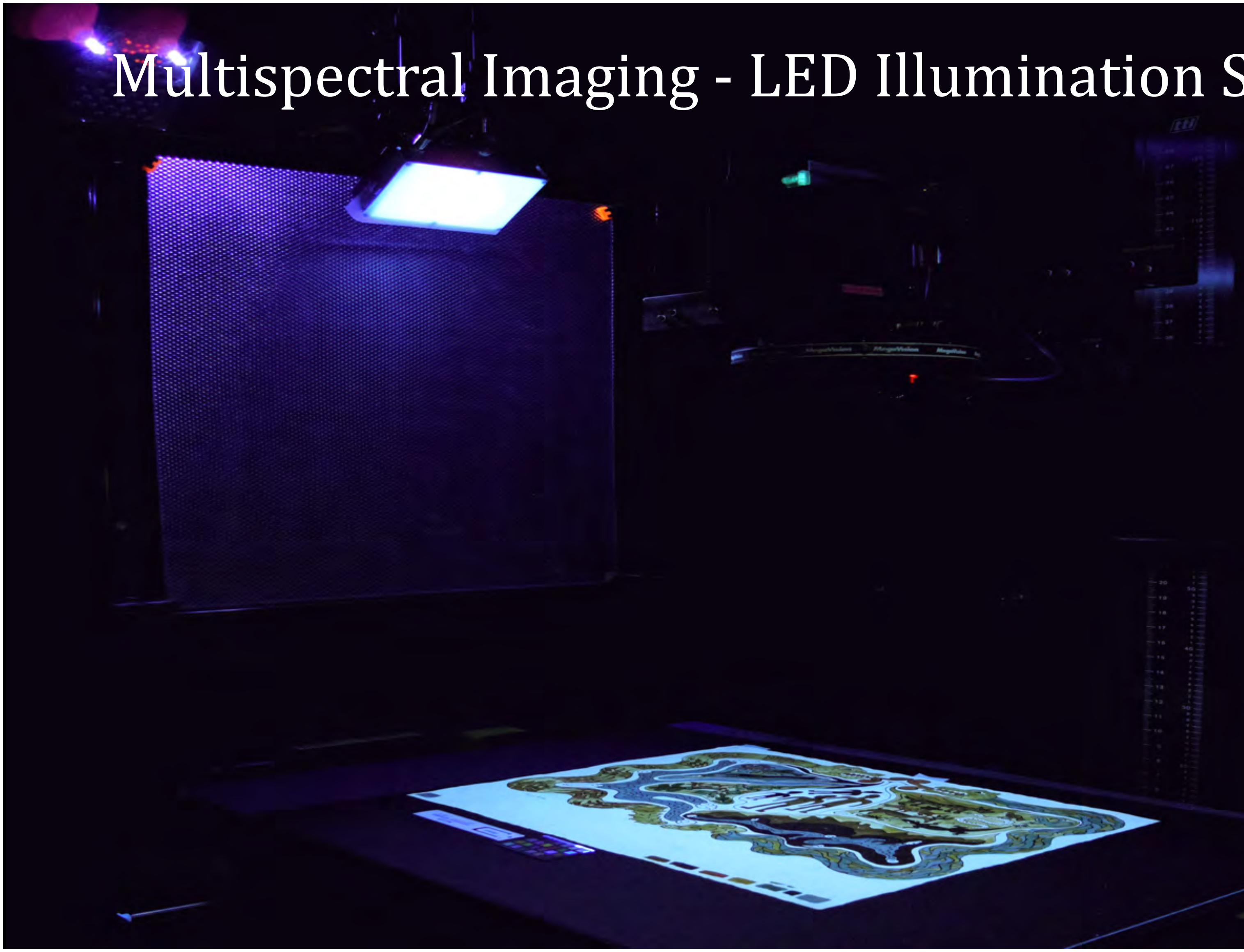
Retention time	Peak Area	Peak ID	Concentration (ppm)
1.836	4895585	acetone	15.5
2.275	14451487	butanone	45.8
2.546	5420474	formic acid	17.2
2.976	50362512	acetic acid	159.6
3.081	770128	pentanone	2.4
3.613	1262767	propanoic acid	4.0
5.319	830776	?	2.6
7.148	8290134	2-butanone	26.3
9.639	1490986	2,5-Hexanedione	4.7
10.581	2283669	2(3H)-Furanone, dihydro-5-methyl-	7.2
12.286	679150	1-Hexanol, 2-(hydroxymethyl)- ??	2.2
14.156	1709434	2-Acetyl-5-methylfuran	5.4
14.305	1746681	2(3H)-Furanone, 5-ethenyldihydro-5-methyl-	5.5
14.911	3068449	2-Heptanone, 6-methyl-	9.7
15.076	842069	2-Cyclohexen-1-one, 3-methyl-	2.7
16.264	11340497	Pentanoic acid, 4-oxo-	35.9
19.093	711014	? Ketone/ester-like	2.3
19.496	1134623	? Ketone/ester-like	3.6

Tests to reduce acetic acid (and other VOCs) in housing




50 MegaPixel Monochrome E7 Camera
Kodak CCD sensor (8176 × 6132 pixel array with linear dimension of 6.0 microns)
120mm Copal-0 hyperspectral lens
with attached dual filter wheel (UV pass, UV block, R, O, G, B filters)
Light Emitting Diode (LED) Illumination Panels

Multispectral Imaging - LED Illumination Sequence



Ultra-Violet

365 nm

NON-Visible

---

400 nm
445 nm
450 nm
470 nm
505 nm
530 nm
560 nm
590 nm
615 nm
630 nm
655 nm

Visible

---

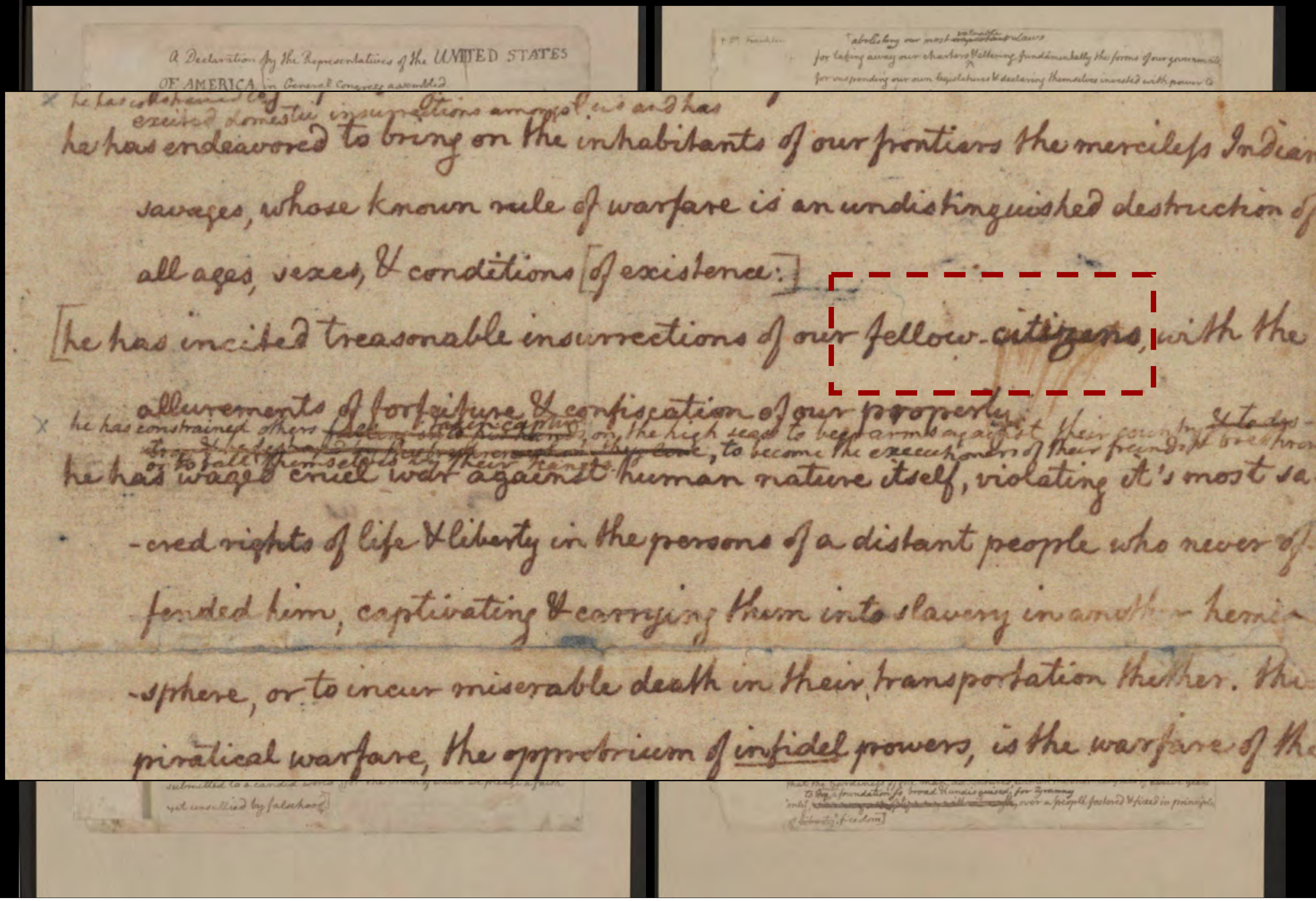
700 nm
735 nm
780 nm
870 nm
940 nm

Infrared

NON- Visible

- 445 nm raking
- 910 nm raking

Jefferson's Handwritten Draft of the Declaration of Independence

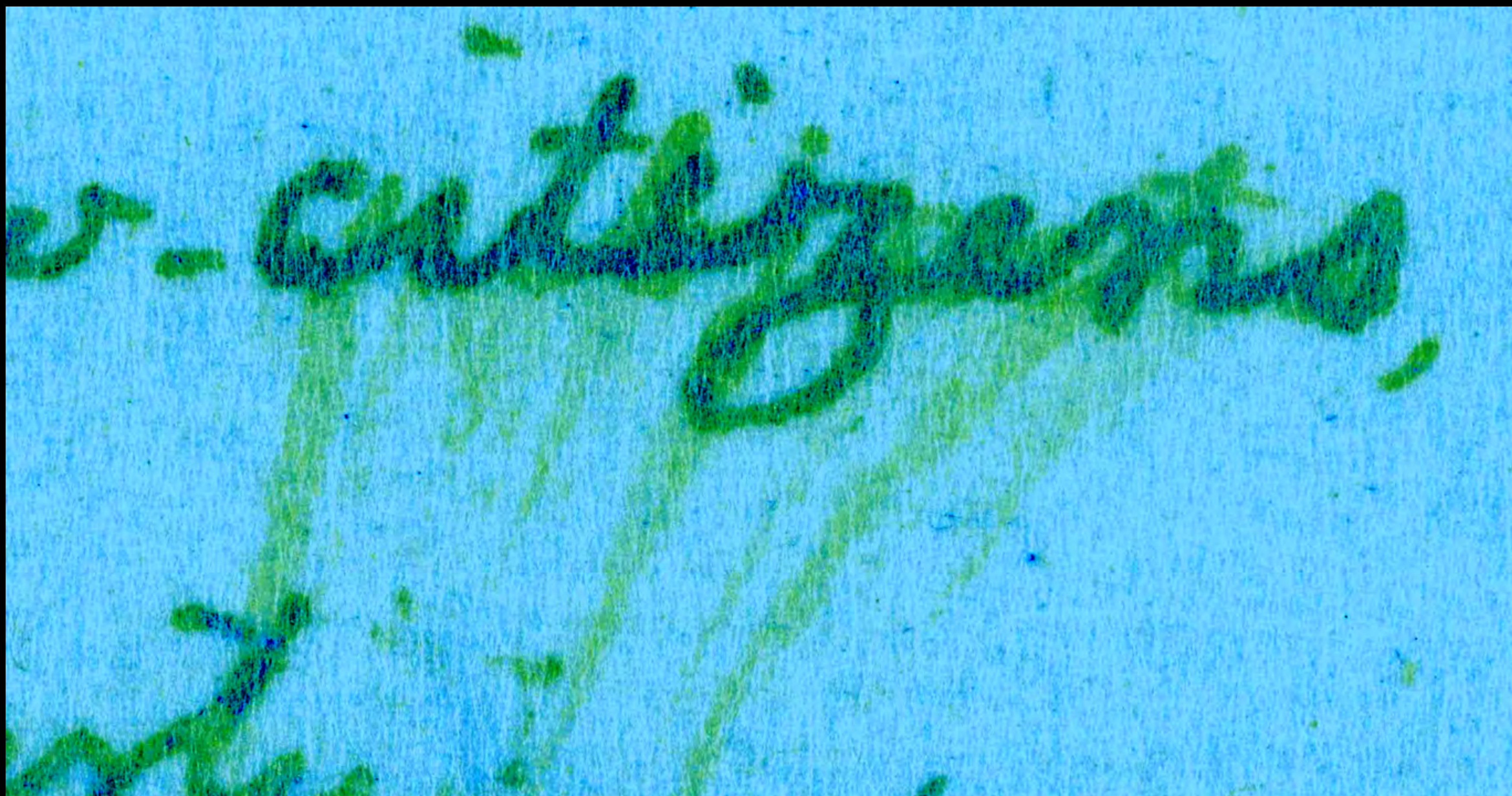


excited domestic insurrections amongst us and has
he has endeavored to bring on the inhabitants of our frontiers the merciless Indian savages, whose known rule of warfare is an undistinguished destruction of all ages, sexes, & conditions [of existence.]

[he has incited treasonable insurrections of our fellow-citizens, with the allurements of forfeiture & confiscation of our property.

he has constrained others on the high seas to bear arms against their country, to become the executioners of their friends & brethren, or to fall themselves by their hands.

he has waged cruel war against human nature itself, violating it's most sacred rights of life & liberty in the persons of a distant people who never offended him, captivating & carrying them into slavery in another hemisphere, or to incur miserable death in their transportation thither. this piratical warfare, the opprobrium of infidel powers, is the warfare of the



Subtraction of bands 10-5 (NIR – green visible) image inverted, with overlay

a. Difference between 2 bands (IR and visible) difference imaging and ratios between wavebands

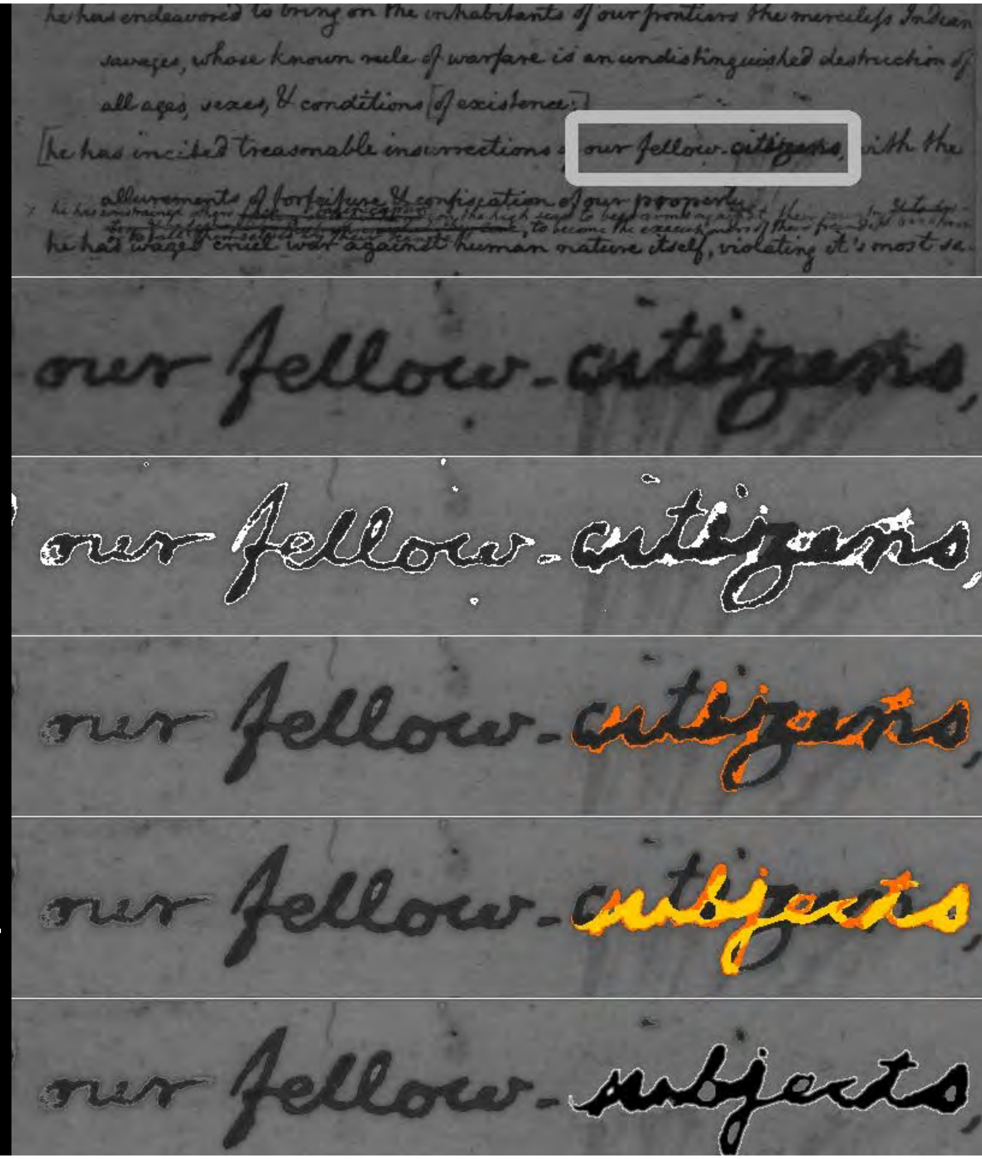
b. Cropped version of (a)

c. “Otsu” multi threshold technique to enhance different intensity ranges

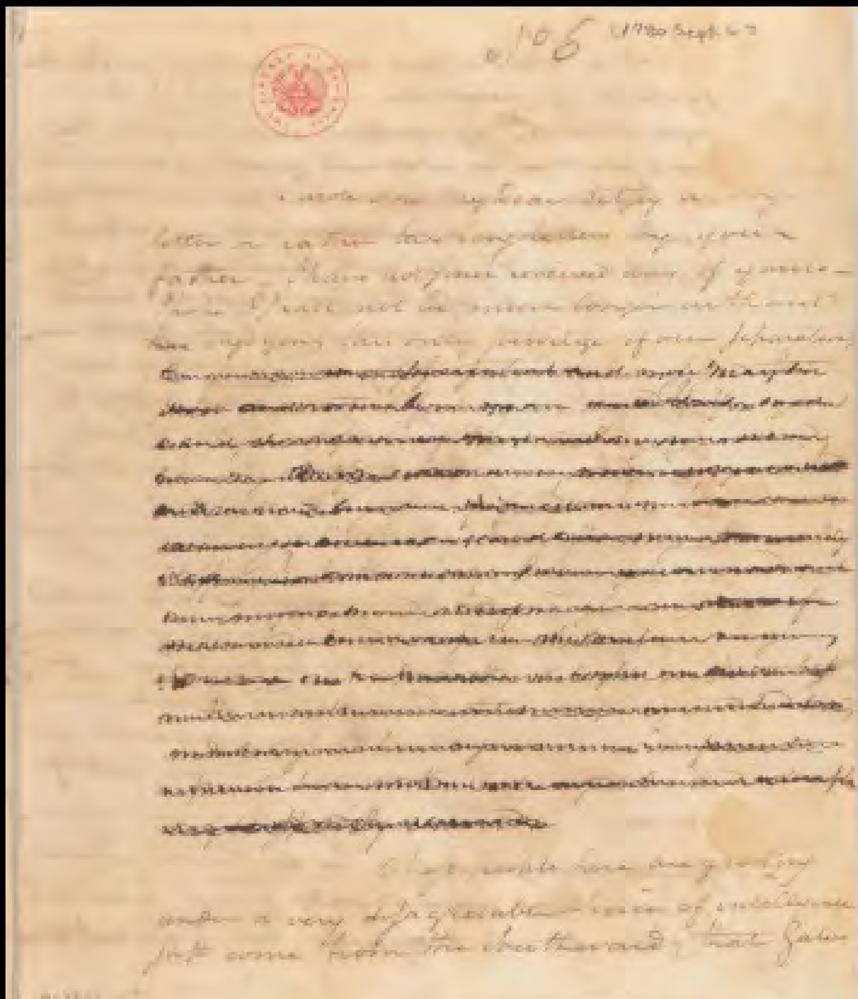
d. Assigning pseudo color (remove transition edges from (c)

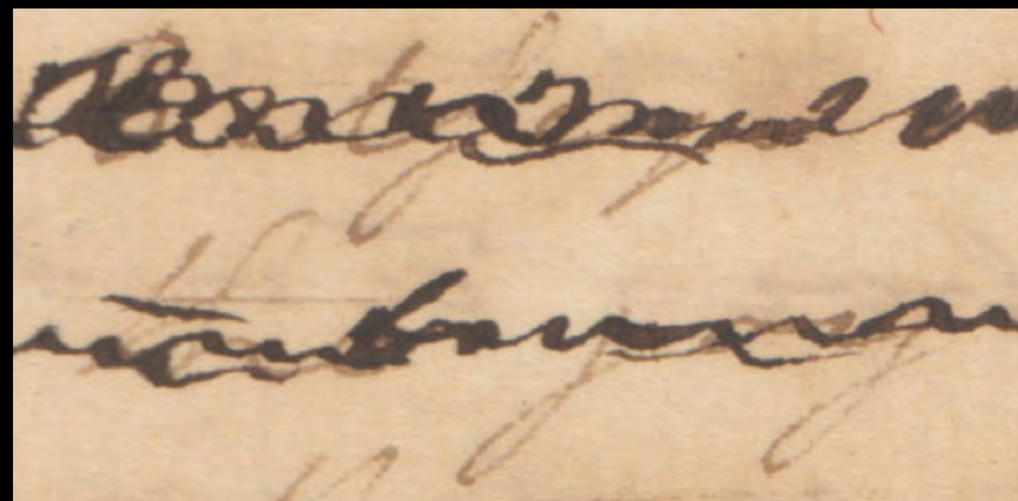
e. Joining the dots - pseudo color (artistic interpretation)

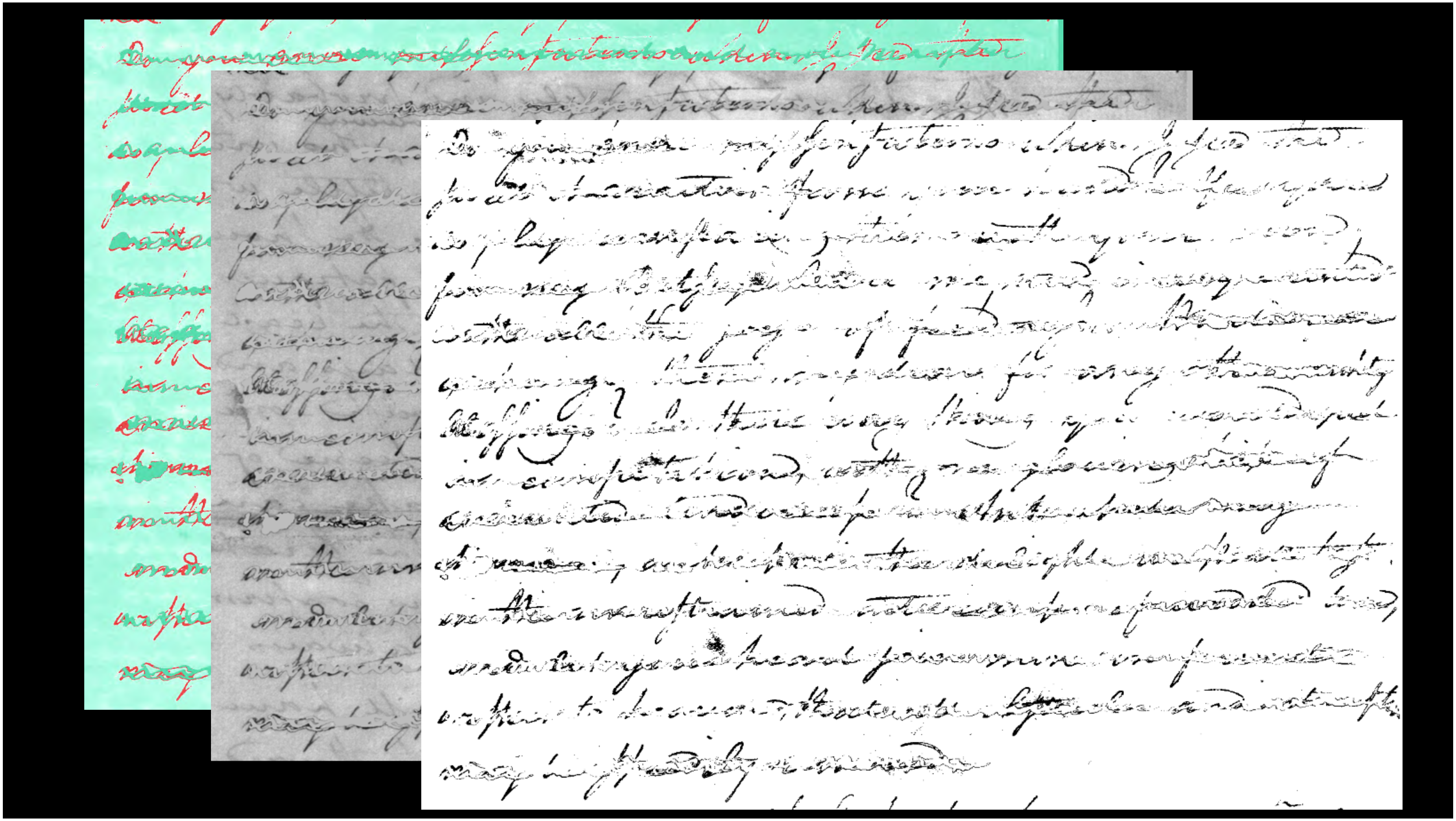
f. Removed citizen for clarity of viewing (make equal to

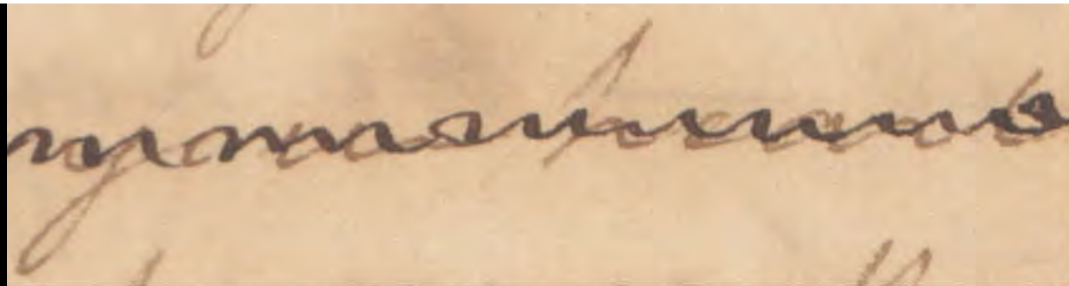


Alexander Hamilton Letter to Elizabeth Schuyler, September 6, 1780



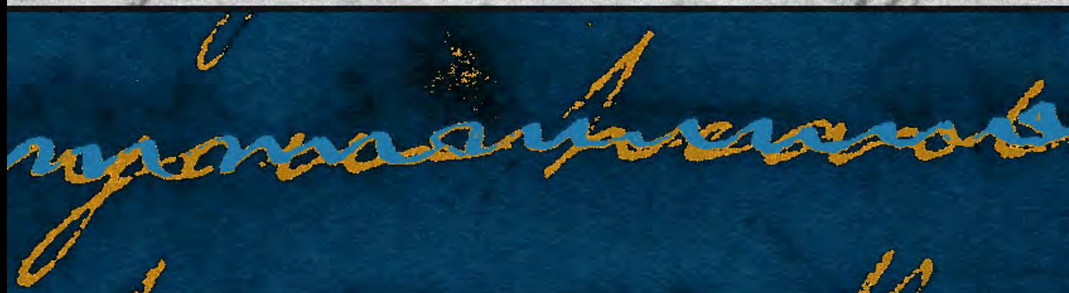






Detail of color image showing orginal text and crossout

Principle component analysis combining two wavelengths to suppress crossout and enhance original text



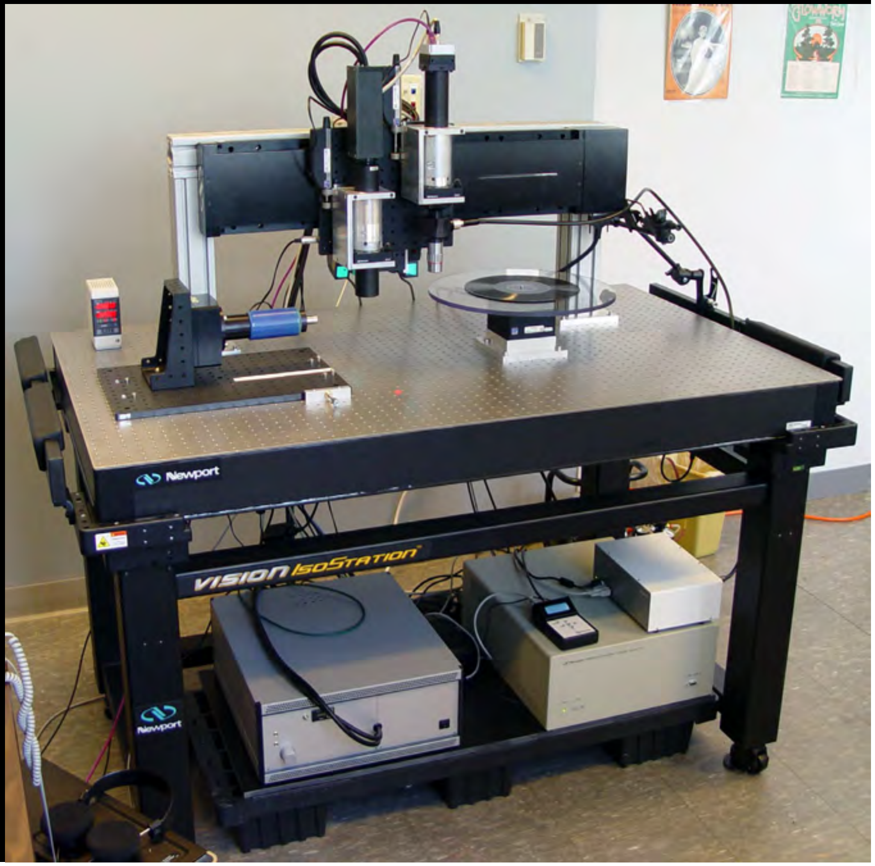
False color applied for additional visual enhancement of undertext

Removal of crossout and connection of original text (artistic interpretation)

Do you know my sensations when I see the sweet characters from your hand? Yes you do, by comparing them with your own, for my Betsey loves me and is acquainted with all the joys of fondness. Would you exchange them my dear for any other worthy blessings? ls there any thing you would put in competition, with one glowing kiss of animated tenderness? Anticipate my [unknown], anticipate the delights we [unknown] [unknown] in the unrestrained intercourses of wedded love, and bet your heart joins mine in fervent wishes to heaven that all obstacles and interruptions may be speedily removed.

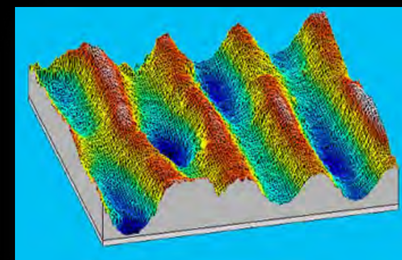
IRENE: Image Reconstruct Erase Noise Etc.

- IRENE is a suite of hardware and software developed to digitally preserve and provide access to mechanical (grooved) sound carriers
- Developed as a collaboration between the Library of Congress and the Lawrence Berkeley National Laboratory
- IRENE uses non-invasive imaging to create high resolution data sets of discs and cylinders and numerical methods to perform restoration and extract audio



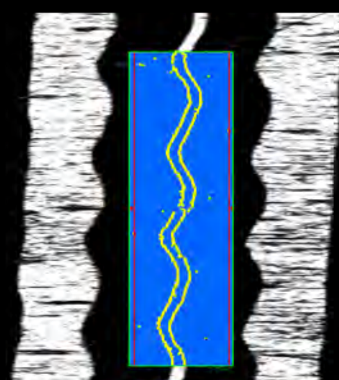
Capture Process

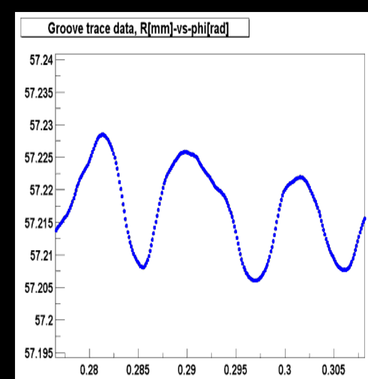
Scan

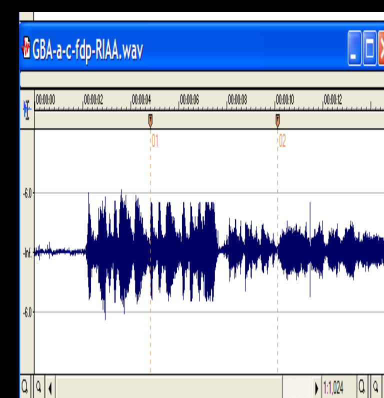


3D Probe

2D Probe







Image

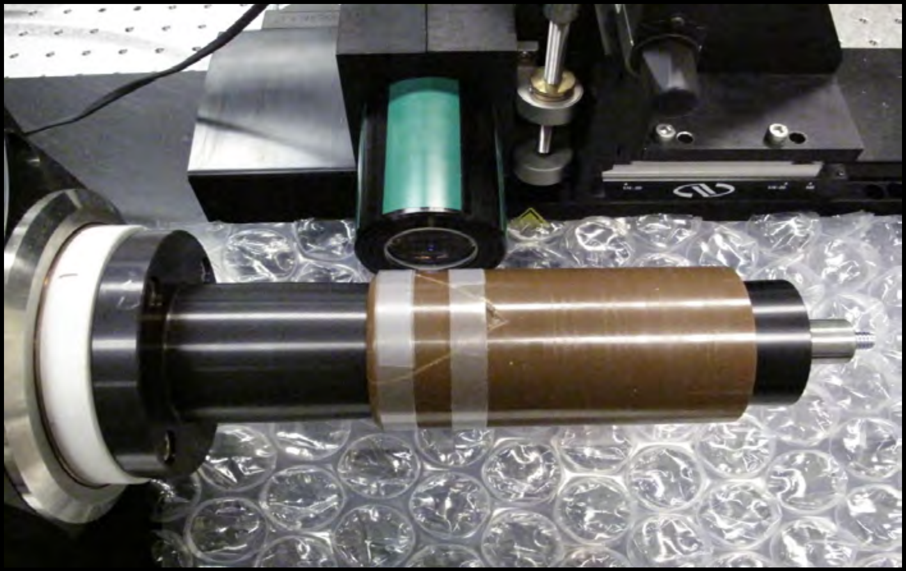
Process (Edge Detection)

Extraction

Audio

Broken Wax Cylinder Record

Song from the ritual of the White Buffalo Hide sung by Wa-kon-mon-thin.
Recorded in September 1895 by Francis LaFlesche
(an Omaha/Ponca tribal member and adopted son of Alice Fletcher).



Folklife Center Collection
Fletcher 25 temporarily held together with conservator's polyethylene straps for imaging

Uniquely identified recording (1885) of Alexander Graham Bell: “hear my voice”

“This record has been made by Alexander Graham Bell, in the presence of Dr. Chichester A. Bell, on the 15th of April, Eighteen hundred and eighty five, at the Volta Laboratory, 1221 Connecticut Ave, Washington, DC, in witness whereof, hear my voice, Alexander Graham Bell”


"Custodians for future generations"
Acknowledgements
Preservation Research and Testing Division Staff, LC
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Fenella France frfr@loc.gov