

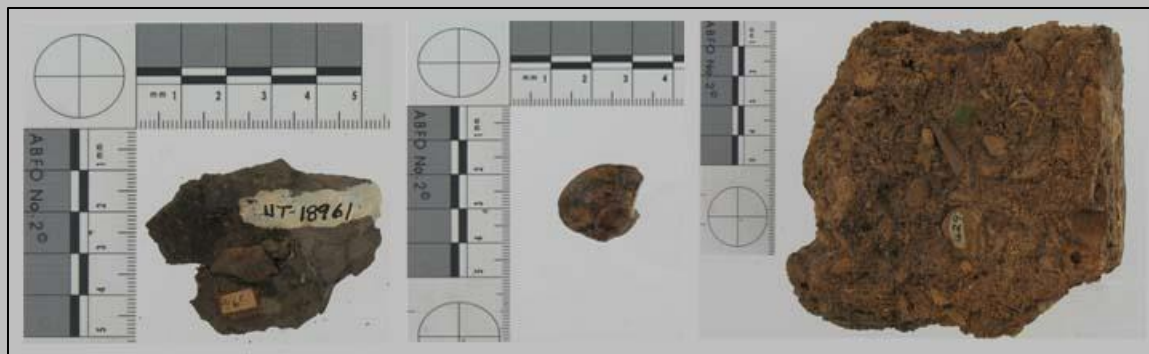
Testing a New Procedure for Removing Aged Consolidants From Historic Collections

Supplementary data and details to accompany the
presentation by Angella Thompson and Chase Shelburne
6th Annual Fossil Preparation and Collections Symposium,
April 20-22, 2013
Royal Tyrrell Museum, Alberta , Canada

The following specimens were sent to Julian Carter, of the Museum of Wales, for consolidant testing. We selected 9 specimens for testing, 3 from each of the large areas of historic collections:

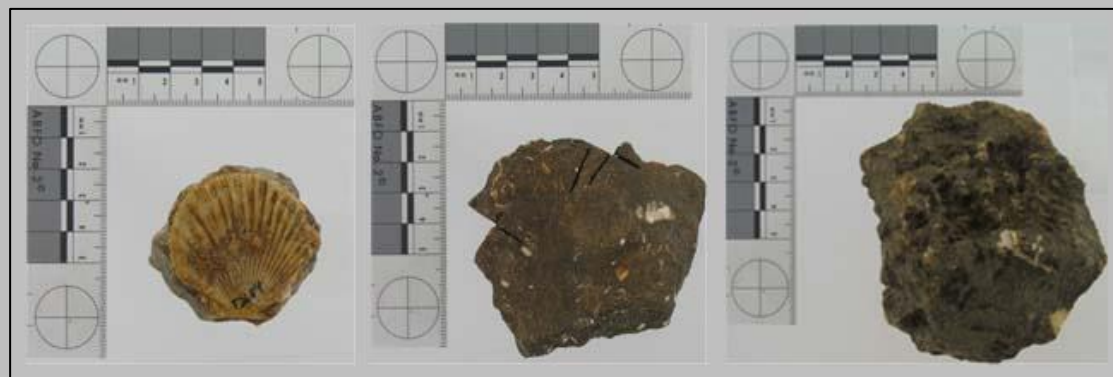
Dumble Survey

- UT18961
- R17587
- White 1582



Rio Bravo

- TX24
- NPL11259
- KX21



Plummer

- NPL6442
- R3289
- P3609



Findings-

Dumble survey

White 1582: Cellulose acetate and animal glue

UT 18961: Shellac

R17587: Cellulose acetate

Plummer Collection

R3289: range of very similar resins came out - pine resin, sandarac and copals all came out as very similar to this sample.

P3609: Cellulose nitrate

NPL 6442: Cellulose acetate

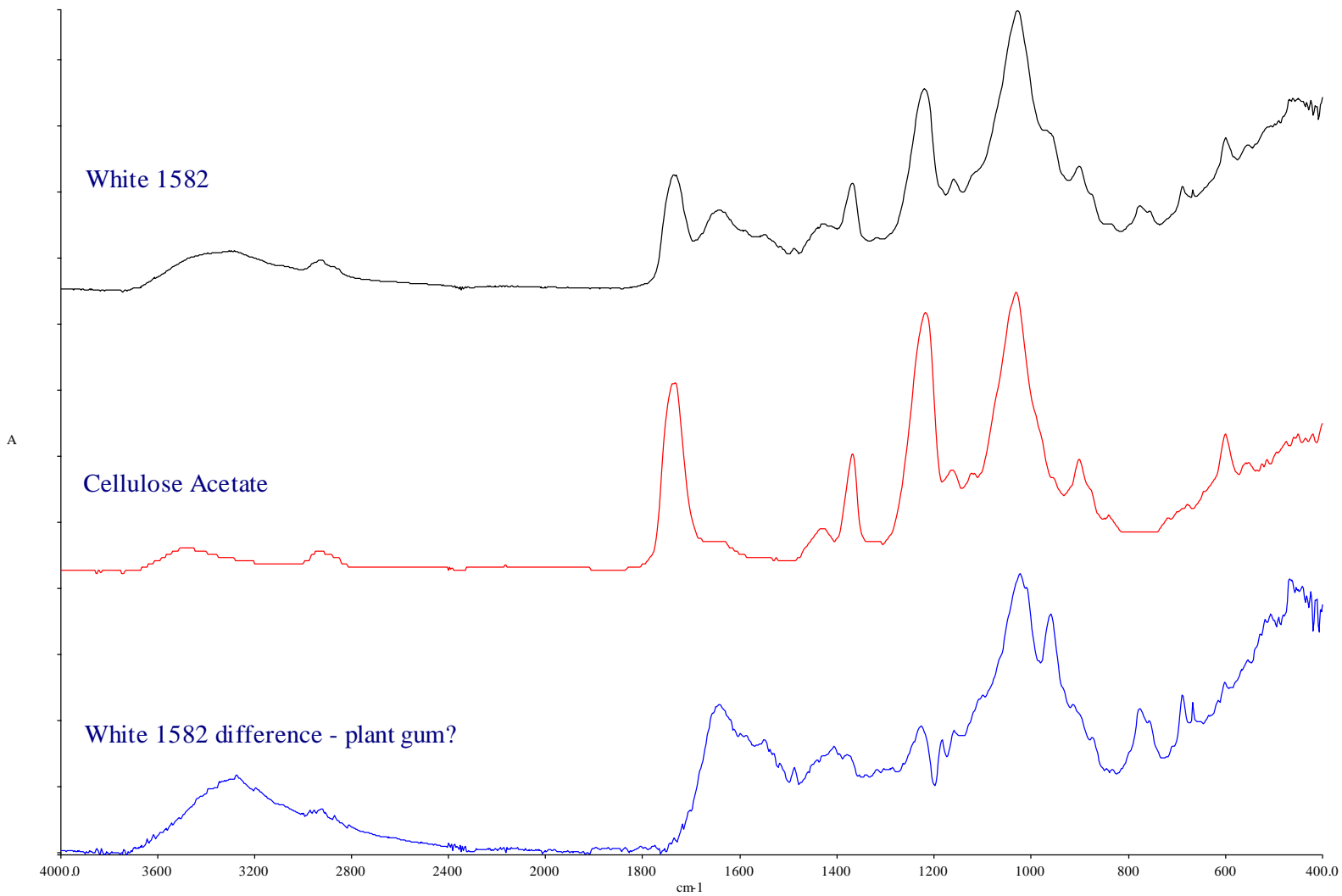
Rio Bravo

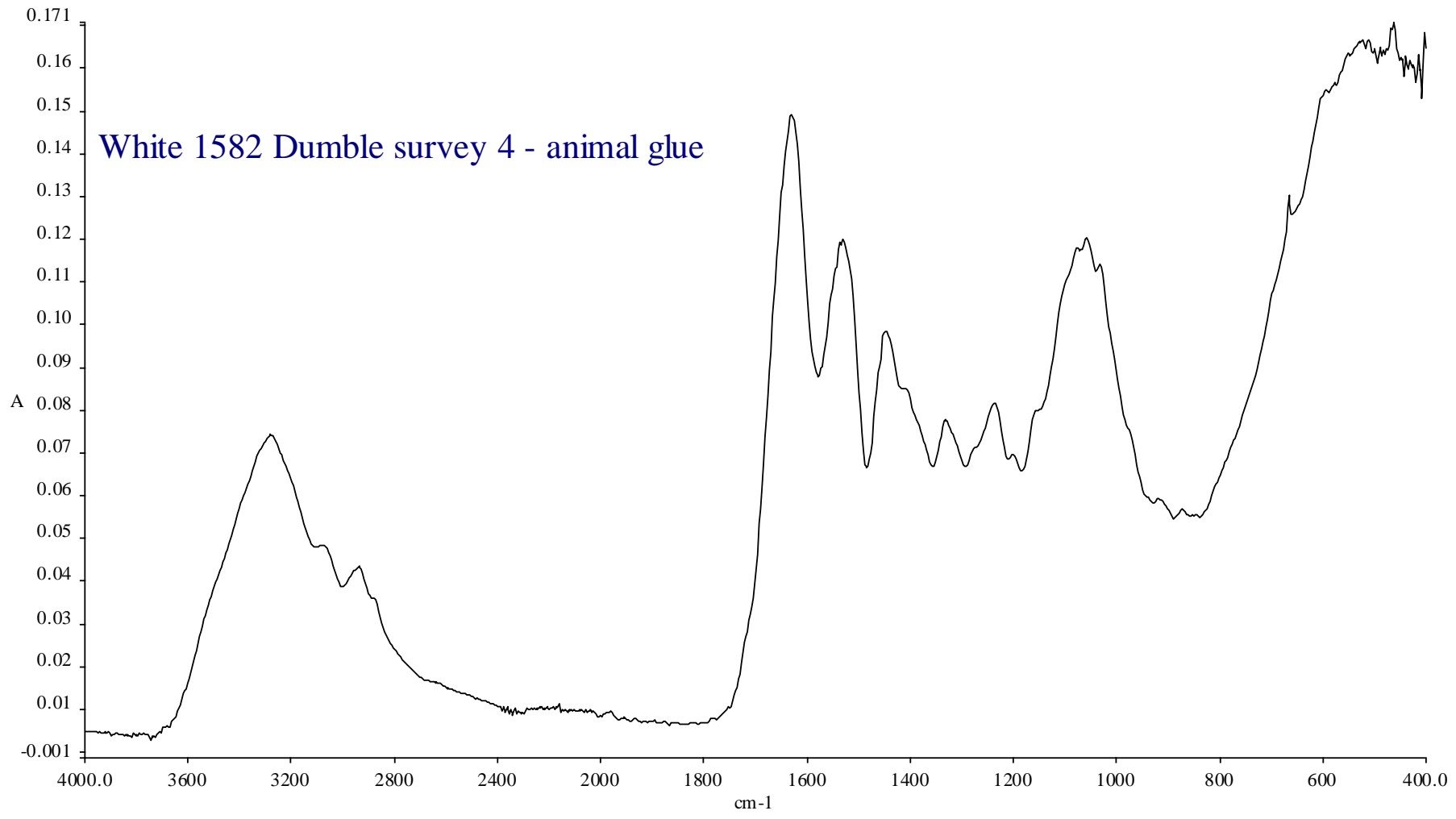
NPL 11259: aged resin along lines of shellac etc - possible resin mix

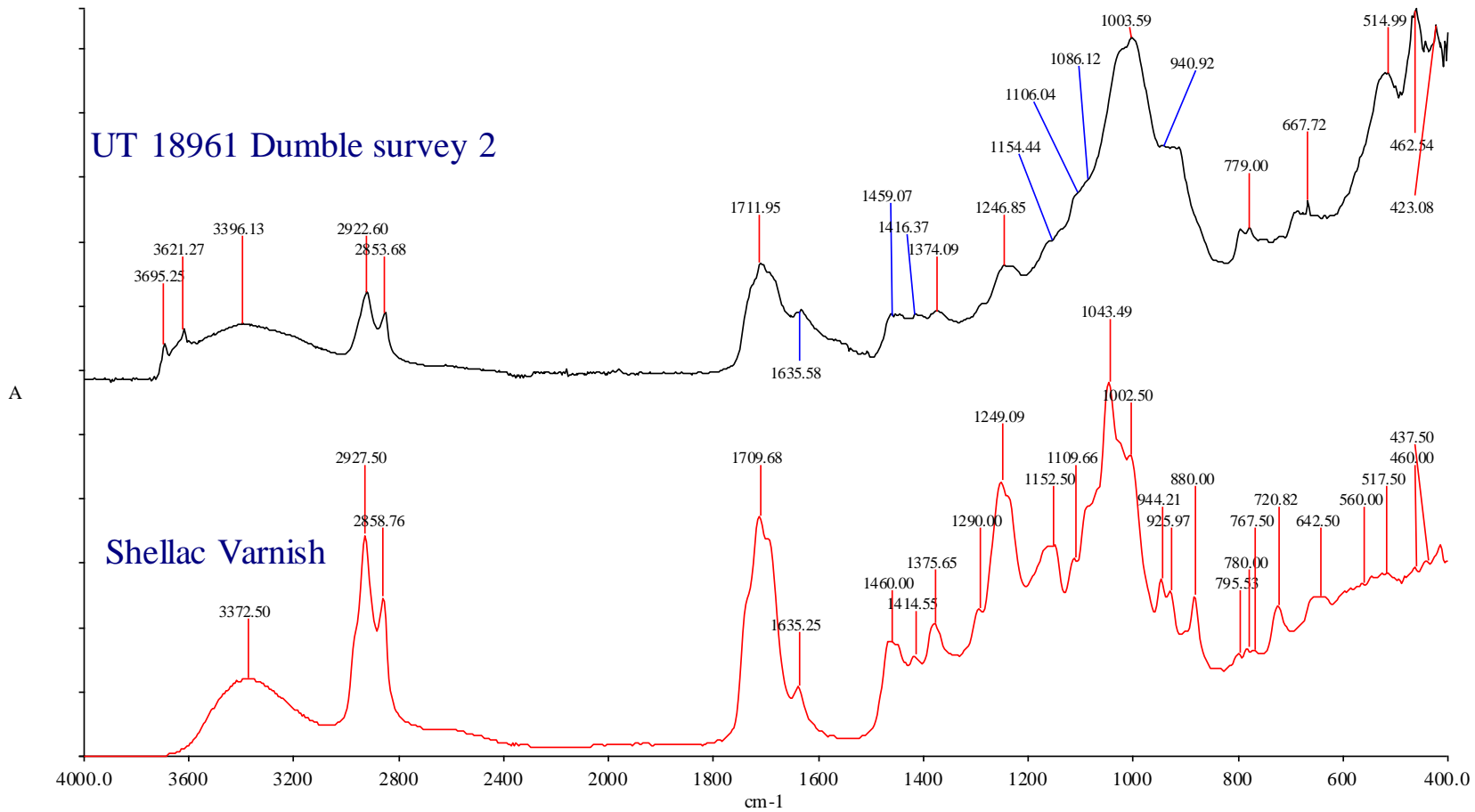
TX24: similar story to above

TX21: again similar to previous two

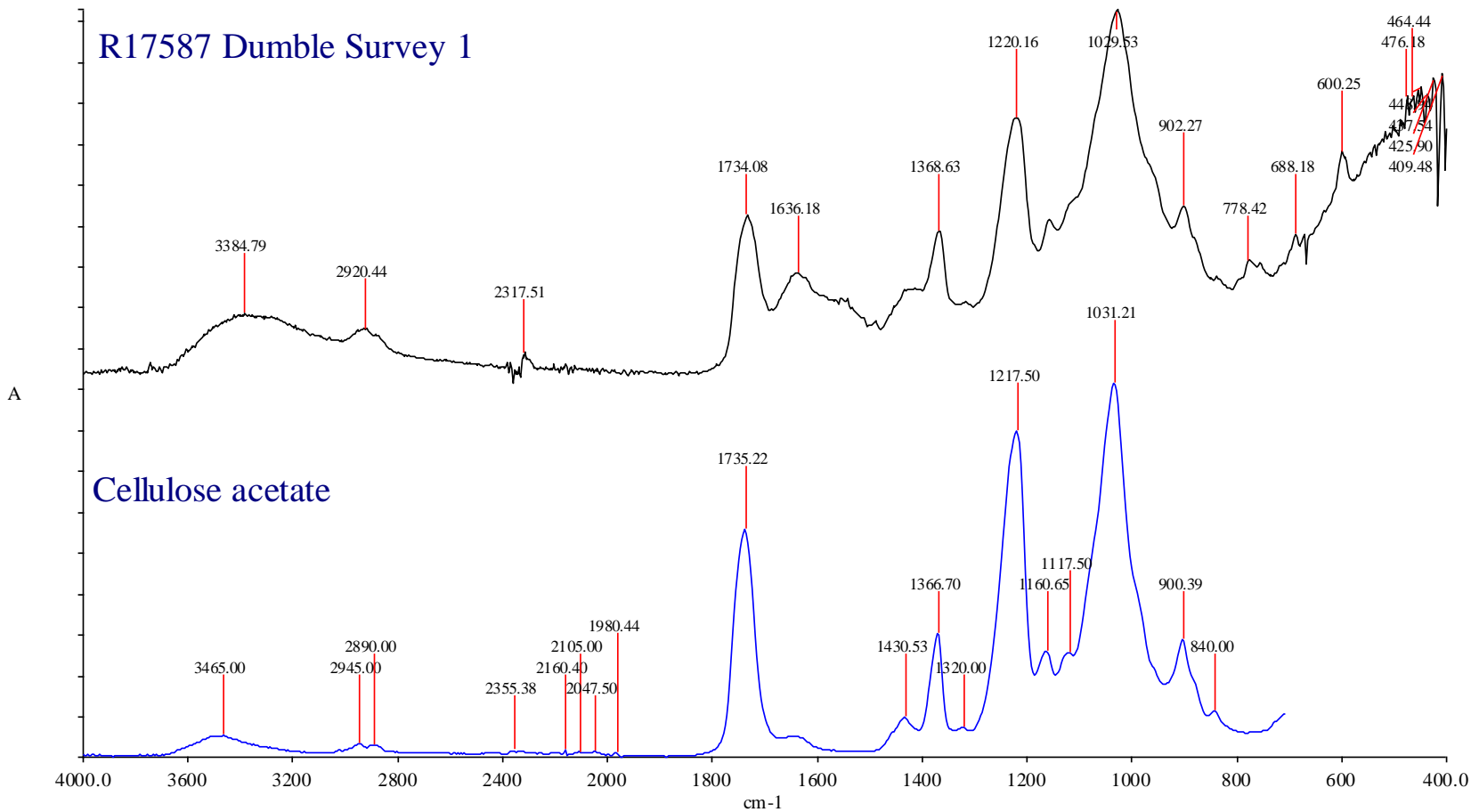
Pinning down the exact components was difficult. The FT IR spectra do show that the chemistry of all the consolidant types is pretty similar. This similarity is more pronounced in the aged and degraded samples.



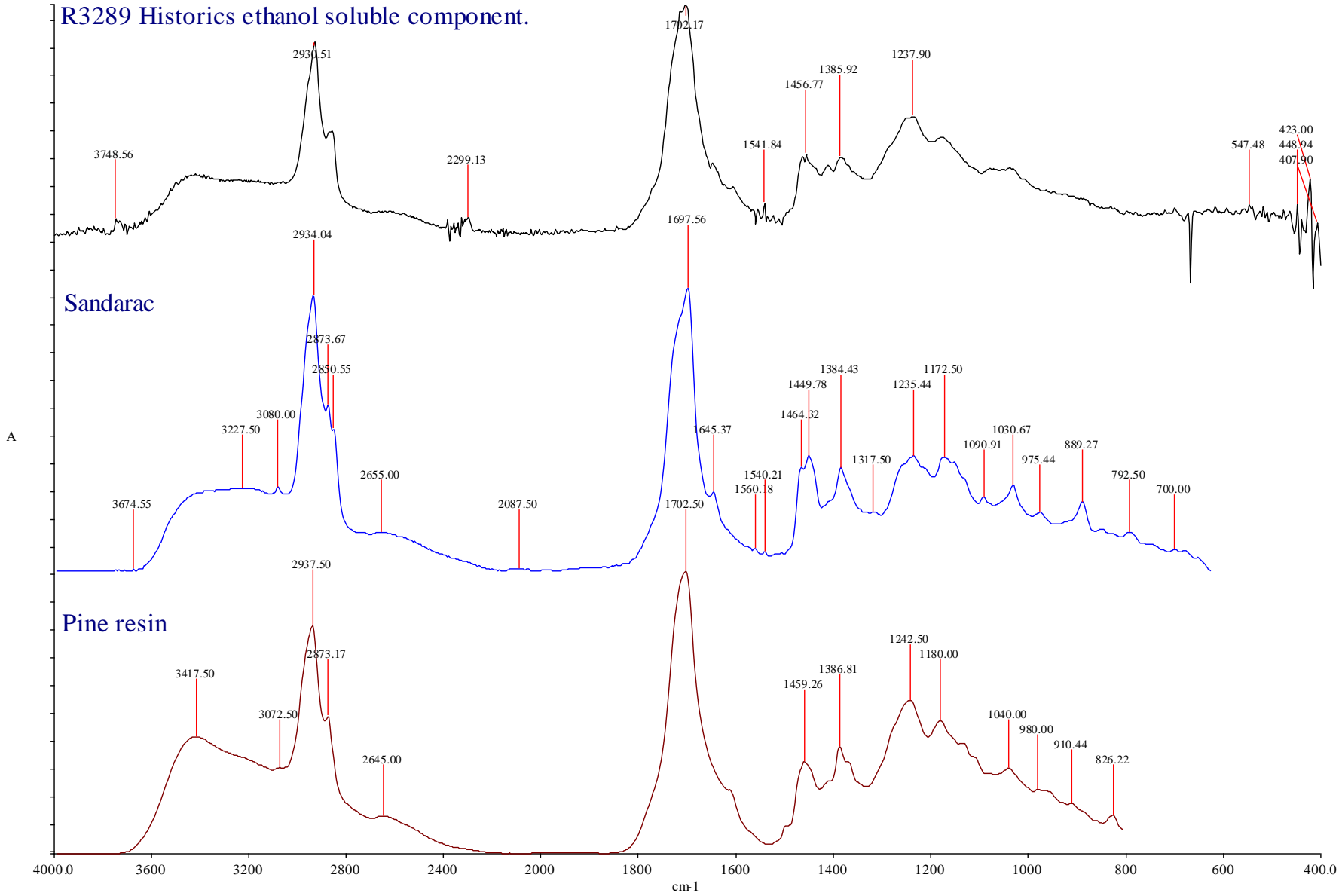




R17587 Dumble Survey 1

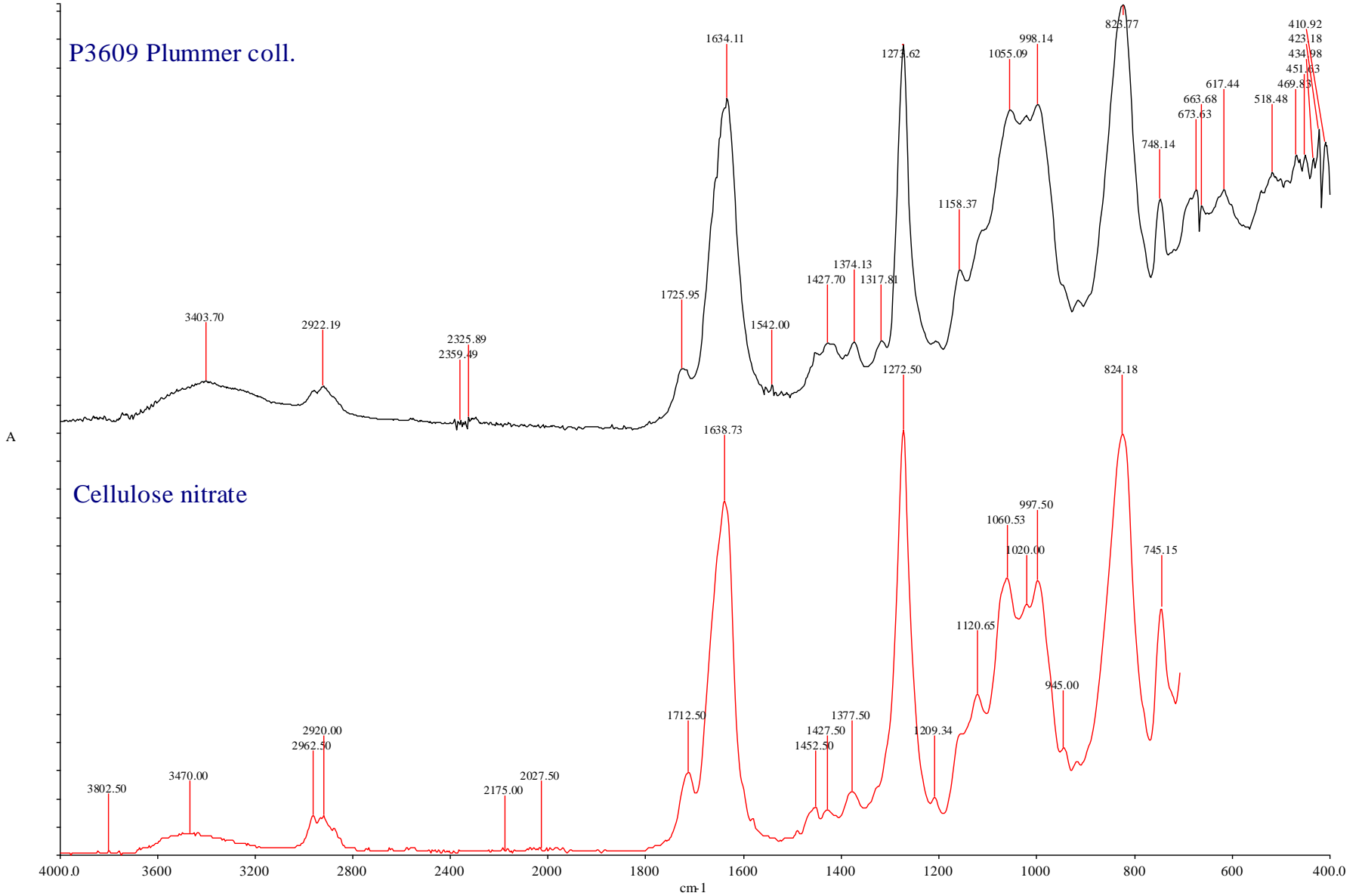


R3289 Historics ethanol soluble component.

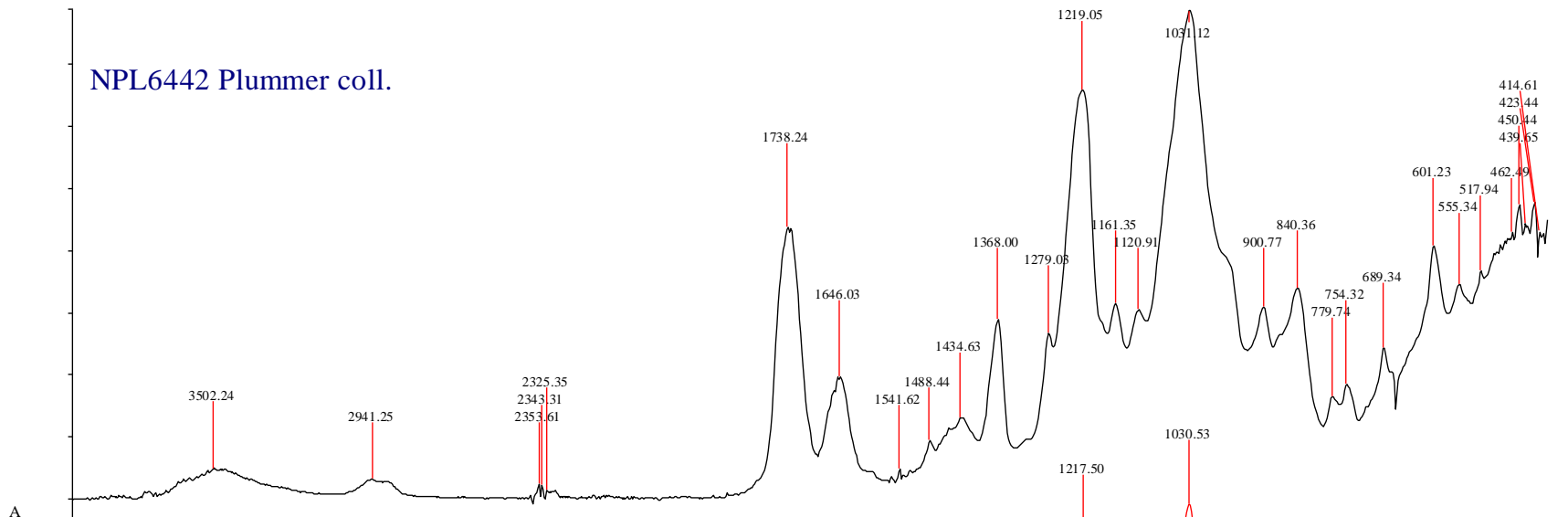


P3609 Plummer coll.

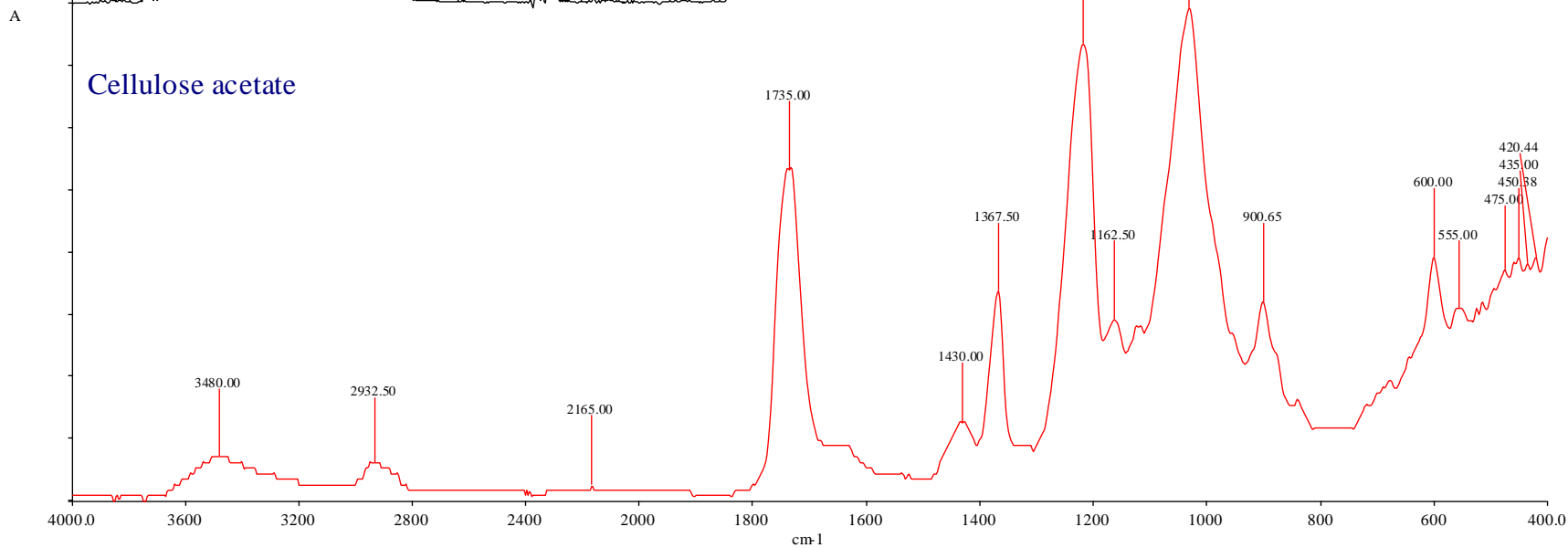
Cellulose nitrate



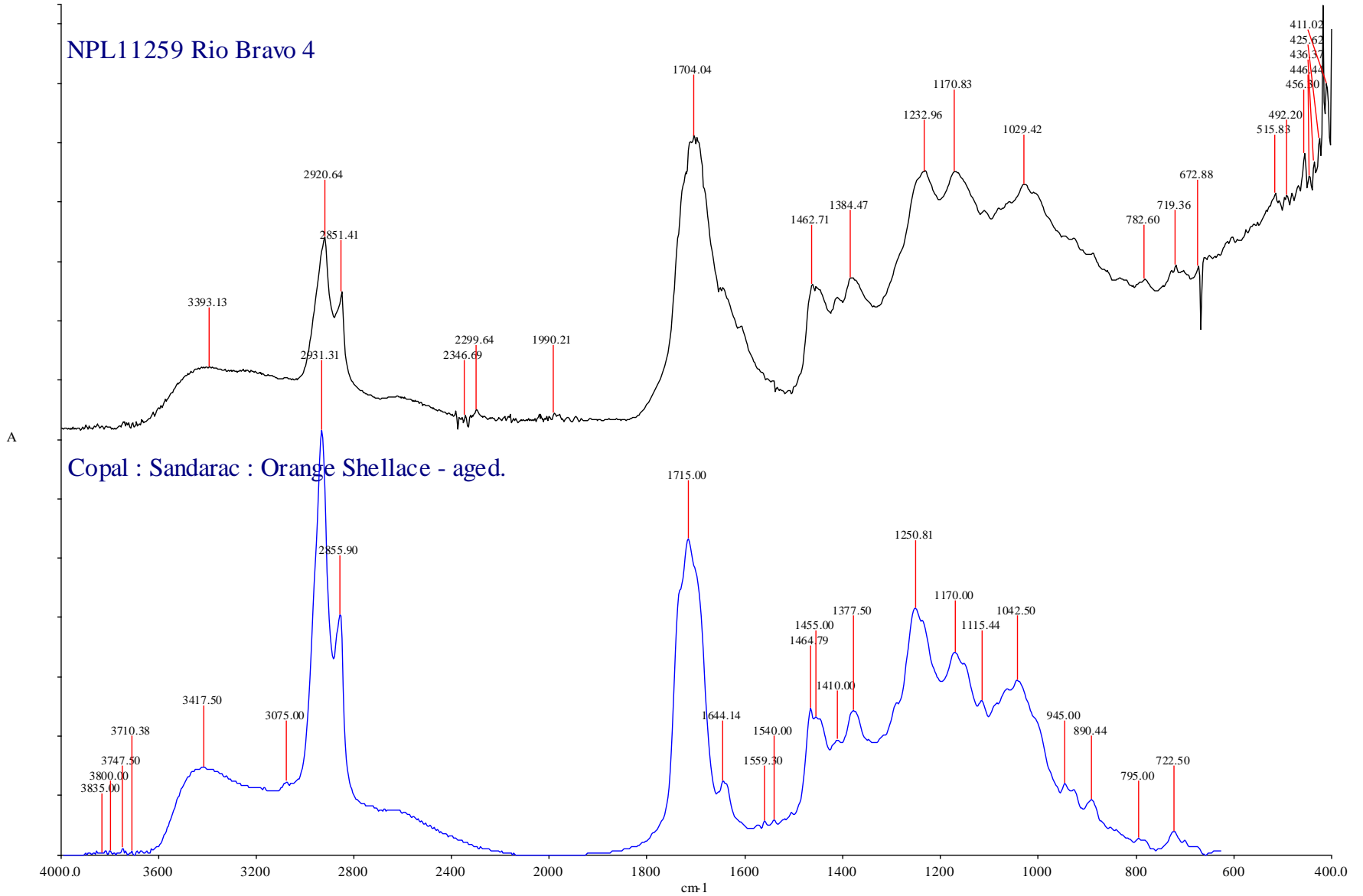
NPL6442 Plummer coll.



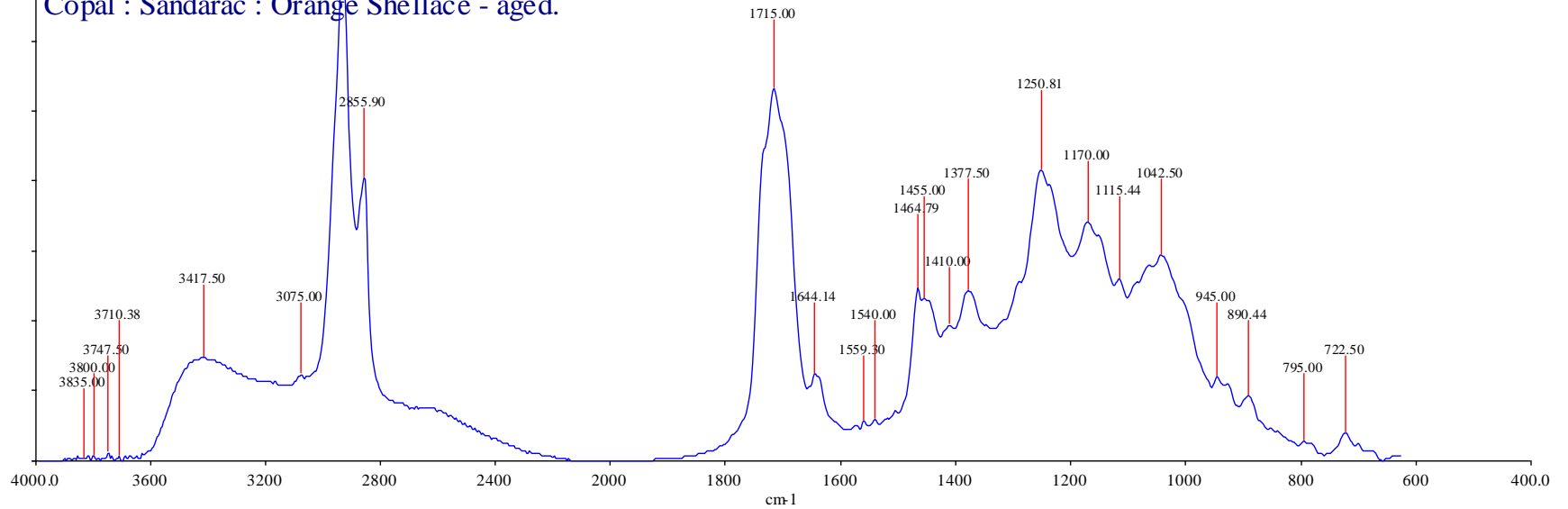
Cellulose acetate



NPL11259 Rio Bravo 4



Copal : Sandarac : Orange Shellace - aged.

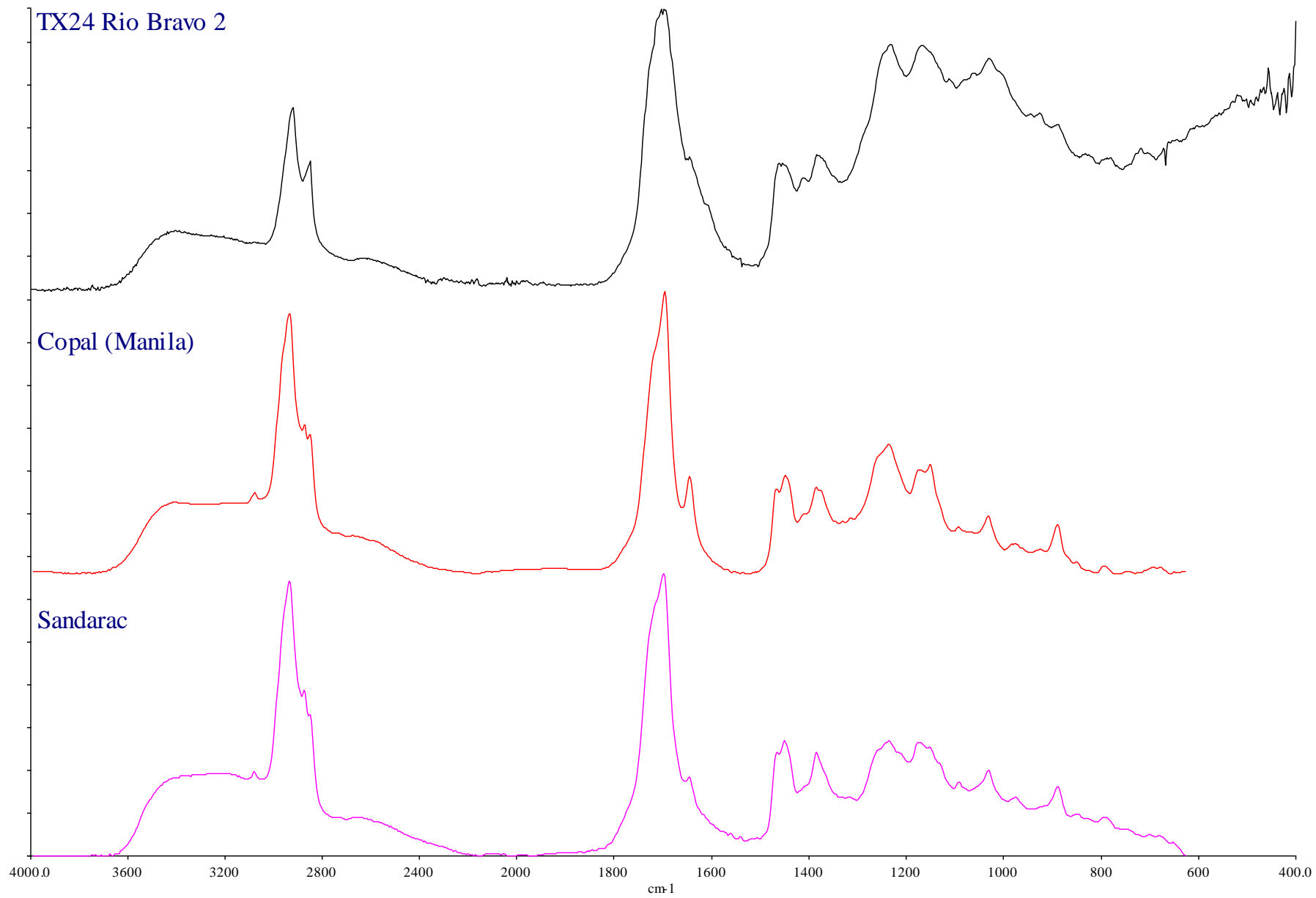


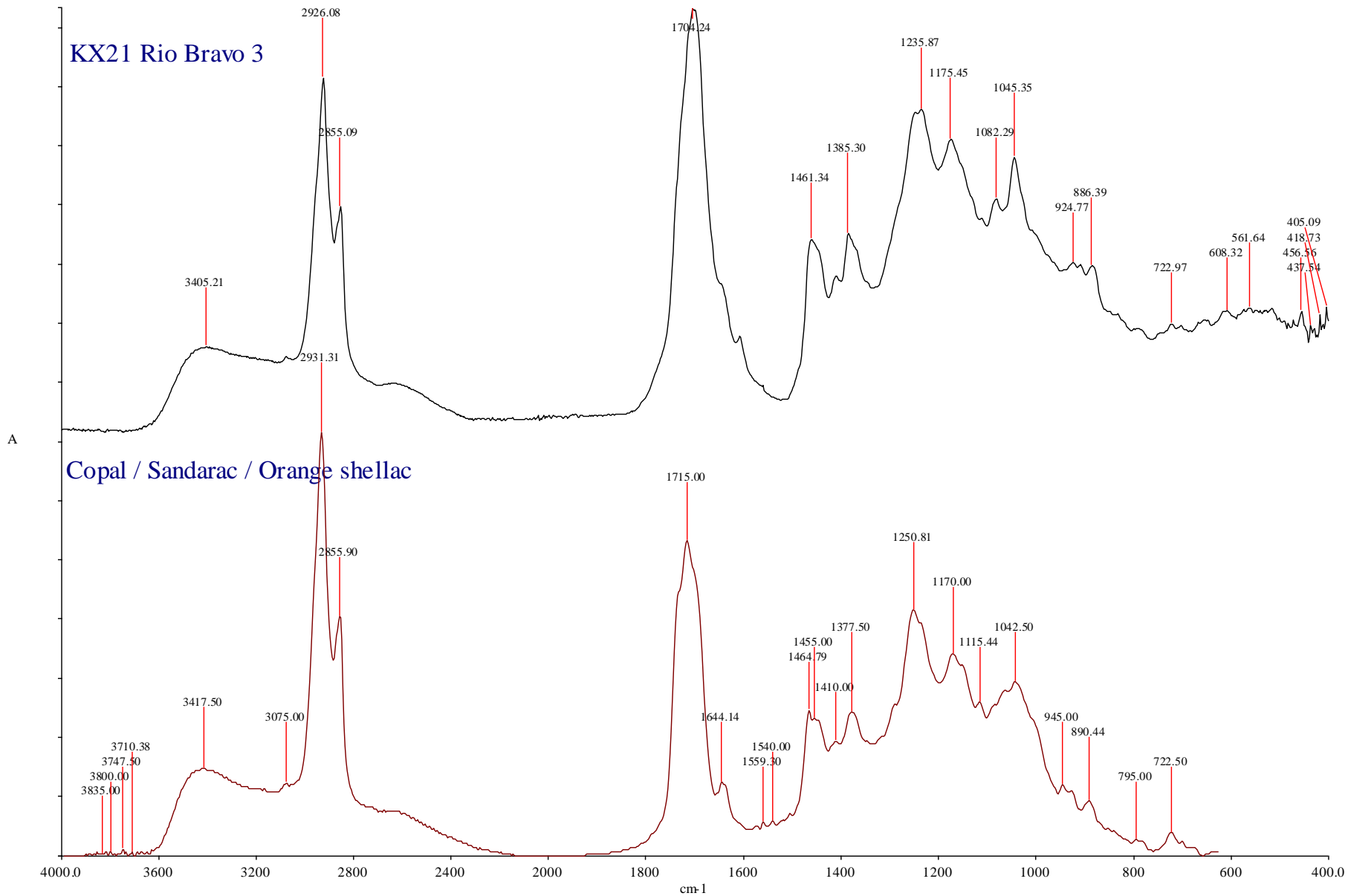
TX24 Rio Bravo 2

Copal (Manila)

Sandarac

A





Outline for Slide 1

- At the Non-vert paleo lab at UT Austin, our repository houses specimens that represent over a century worth of collecting.
- Many of these specimens are from localities that are no longer accessible, or have eroded away.
- Our historic collections have some tricky conservation problems that make their care challenging.
- This quote you see behind me illustrates really nicely ground 0 for some of our problems.
- With collections from this era one of the challenges we face is reconstructing these historical methods.
- Inconsistent record keeping
- When records are present, they are not always precise

[Howe asks]” ...but they’re [the bones] are so busted up; how do you ever expect to get them out, now they’re in so many pieces?”

“Don’t worry; watch this,” Carl said, reaching for the shellac can.

...He dribbled shellac from his flooded brush into the cracks and fractures of the badly broken surface. The dry bone bits drank in the yellow fluid thirstily. “When that sets,” Carl went on, “the bone won’t be in any danger of falling apart before we’re ready to take it from the ground.”

Excerpt from:

“Bones for Barnum:
Adventures of a Dinosaur
Hunter”

-by R.T. Bird

Conversation between Carl Sorensen, an AMNH perpetrator
and Rancher Howe

Slide 2

- true shellac
- tree resins
- animal Glue

- synthetic nitrocellulose, acrylic lacquer
-

We need to know the consolidants used because often they are involved in modern conservation problems.

A shellac by any other name...

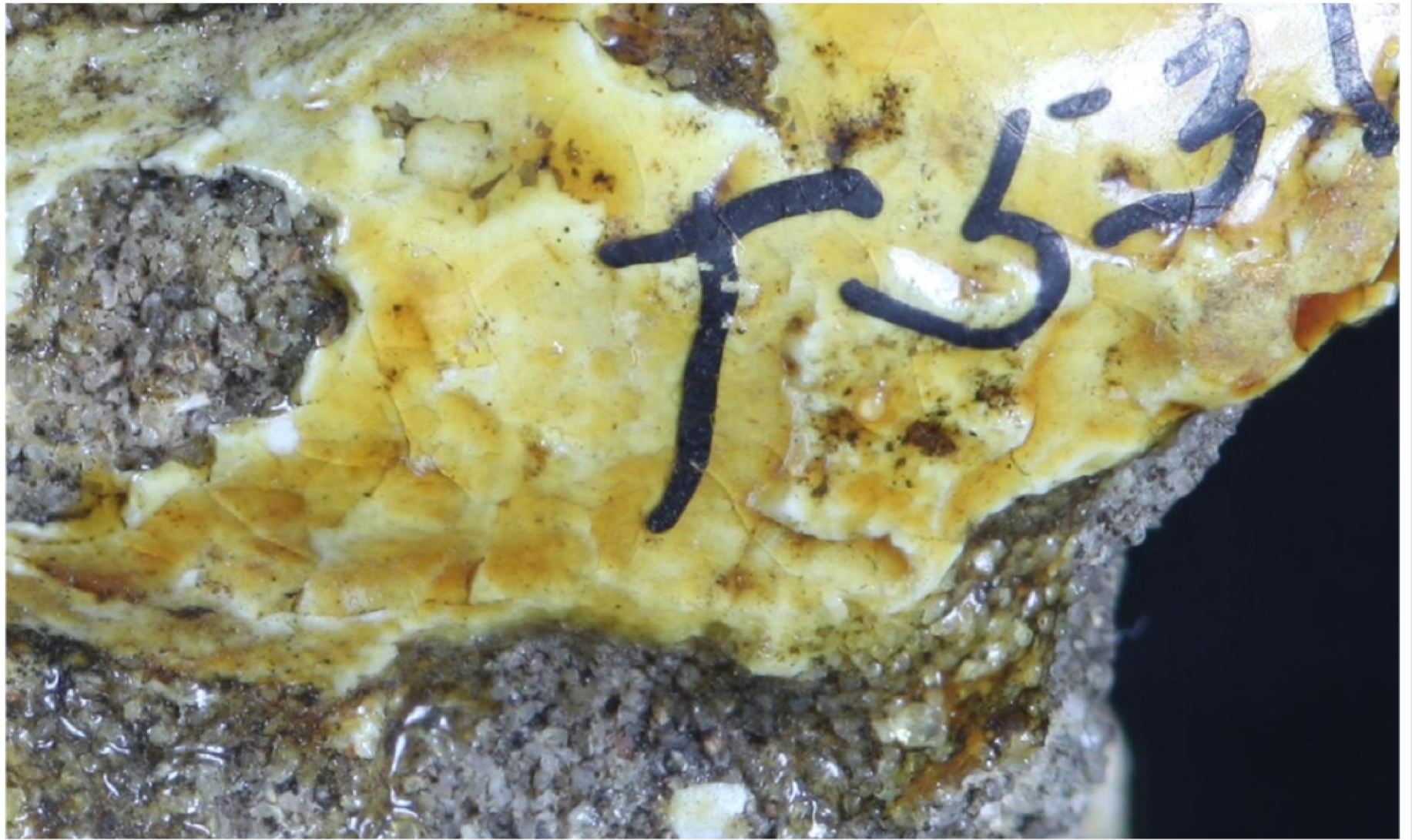
- true shellac
- tree resin
- animal glue



And a variety of synthetic resins

Slide 3-5

- Cracking
- sometimes just of the consolidant, sometimes the crack invades the fossil
- darkening
- obscures margin between fossil and matrix
- can't make out colors
- texture can't be made out
- Peeling and flaking
- Cross-linking
- softening over time, becoming viscous dust magnets



Cracking



Darkening

Peeling and Flaking

The diagram consists of three horizontal rectangular frames stacked vertically. Each frame contains a blue rounded rectangle with a gradient and a drop shadow. The top frame contains the text 'Peeling and Flaking', the middle frame contains 'Softening', and the bottom frame contains 'Cross linking'. The frames are connected by thin blue lines at the top and bottom edges.

Softening

Cross linking

Slide 6

While our main collection area is climate controlled, we have a warehouse building that is not.

- warehouse is where we find dramatic examples of aged consolidants
- dramatic temp and RH swings
- these swings worsen cracked peeling and flaking consolidants

At NPL wanted to identify our most at-risk collections and move them into more modern housing.

- We have a giant collection, but limited climate controlled space to put them in.
- Goal to Identify most at-risk collections that we are hoping rehouse in our main climate controlled area.
- Some specimens are at higher risk from shellac damage than others
- Particularly old specimens, and fragile or friable specimens are more likely to be damaged by the shellac as it expands and contracts



Heat



Deterioration
of
Consolidants

Humidity

Slide 7

- Dumble Survey
- Plummer Collection
- Rio Bravo
- We want aged consolidant removal to be part of the conservation effort for these specimens.

At Risk Collections

Dumble Survey

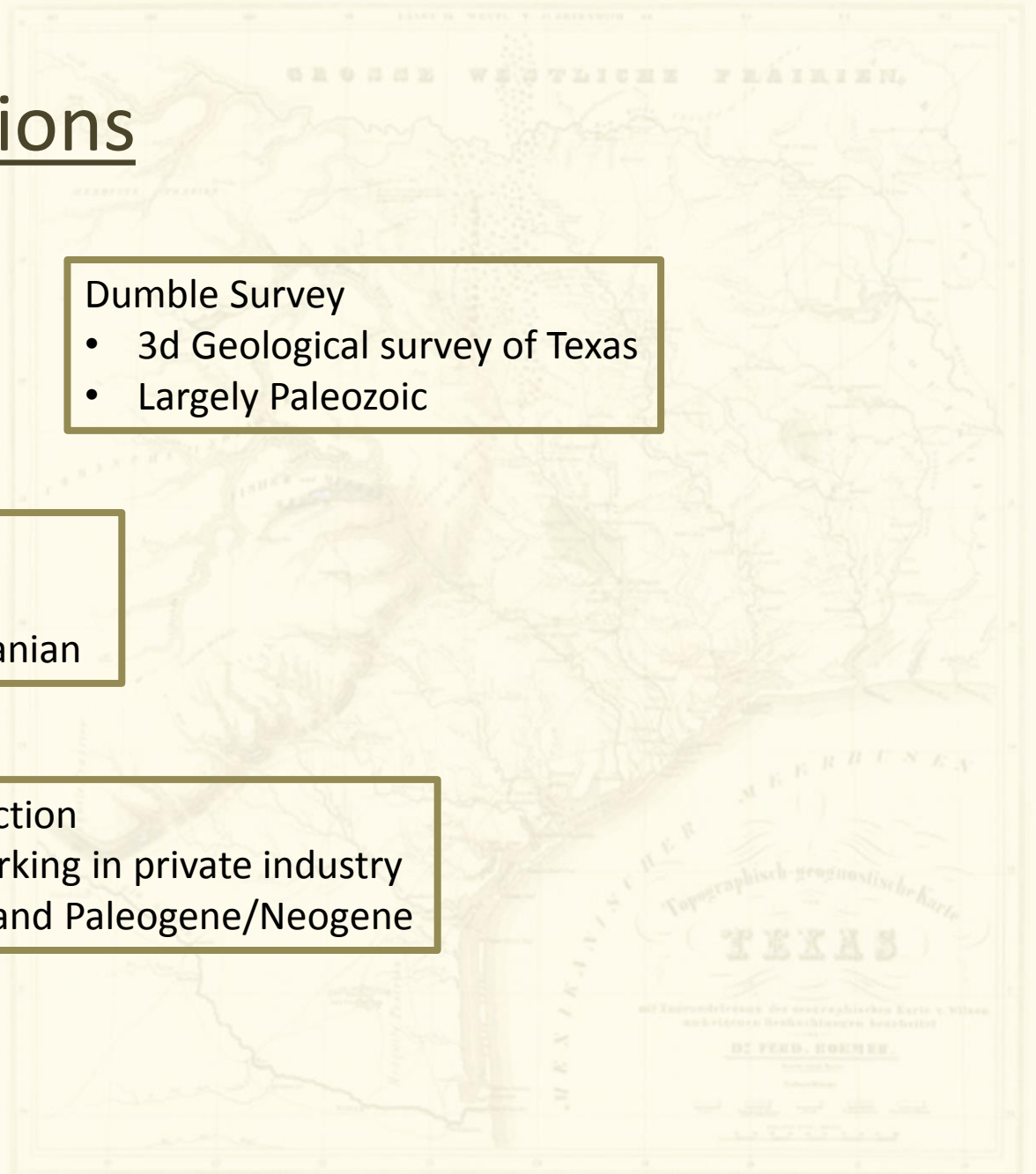
- 3d Geological survey of Texas
- Largely Paleozoic

Plummer Collection

- Texas Geology
- Cenozoic and Pennsylvanian

Rio Bravo Collection

- Dumble, working in private industry
- Cretaceous and Paleogene/Neogene



Slide 8

- Usual methods of removing consolidant
- Chemical solvent
- Applied carefully
- Scraped off with pin vice.

Chemical Solvents

- Acetone
- Ethanol
- Xylene *



Applied Carefully

- Poultice
- Brush & Blot



Mechanical Removal

- Pin Vice

Slide 9

- Why the usual methods won't work for us
- Shell composition
 - Calcium Carbonate and Aragonite
 - Soft and porous
 - Acid test
- Had to find another way

Fossil Shells

Calcium Carbonate

Aragonite

- Soluble in weak acid
- Hardness: 3.5-4
- Tenacity: Brittle

Calcite

- Soluble in weak acid
- Hardness: ~3
- Tenacity: Brittle

Neither are particularly hardy, invertebrate fossils are notoriously fragile and susceptible to their environment.

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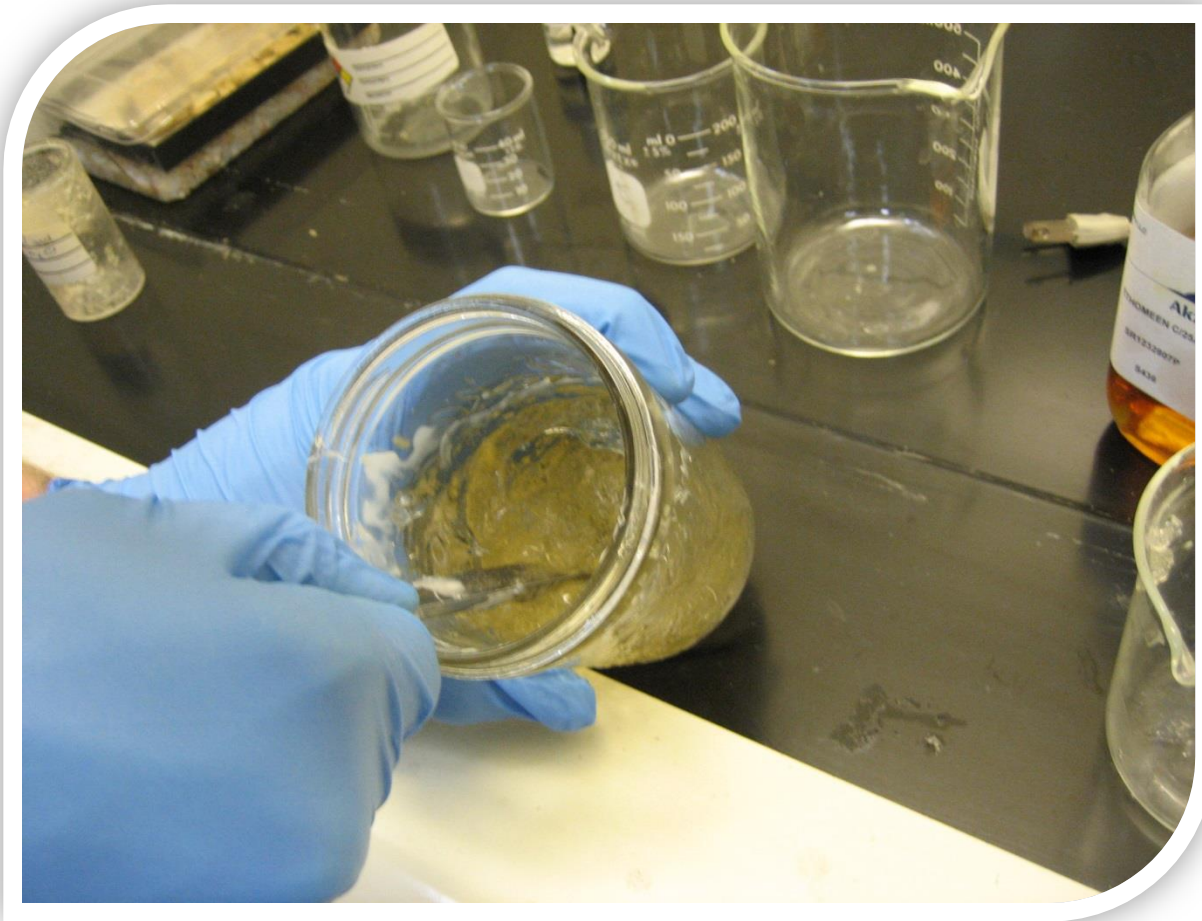
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<http://www.jstor.org/stable/27784654>

Slide 10

- We experimented with a gel formulation intended to quickly, and easily remove aged consolidant
- Originally used in the field of art conservation to restore paintings without damaging them
- Adapted by Williams and Doyle to remove aged consolidant from vertebrate teeth
- We've adapted their formula to be used to remove aged shellac from less resistant, Calcite and aragonite shell material

The Gel



Originally used for
art conservation

Adapted to remove
consolidant from
vertebrate teeth

Slide 11

- History seems to have taught us no
- Scientists have had bad luck with slathering goop, (mainly consolidants), in the past, because they have negative aging effects such as discoloration, blistering, or cross-linking of polymers, which results in difficulty during removal.
- This increases the likelihood of damage to the specimen beneath.
- There are several general types of consolidants
 - Reaction Consolidants (Cyanoacrylate, epoxies)
 - Solution Consolidants (Paraloid, Butvar)
 - Shellac and other natural resins
 - What our project focuses on

Is Slathering Goop a Good Idea?

Historically, no. Lack of understanding of consolidant ageing effects has damaged, or has the potential to damage many specimens



Slide 12

- In order to work with either, the composition and nature of the consolidant or consolidant remover needs to be understood
 - How well does/did the chemical penetrate the fossil?
 - Will the chemical react with the surface to damage or discolor the fossil or matrix?
- What are the ageing effects of the chemical?
 - Nature of the underlying fossil must be understood, (smooth, rough, friable, etc.). Some surface textures are easier to work with, (for example, rough surfaces tend to retain more residue).
 - Can the fossil stay in one piece after having the consolidant removed, (after all, the consolidant was put on there for a reason!).
 - Will the fossil chemically react to the consolidant/consolidant remover and become chemically damaged?

Things to consider

```
graph TD; Root[Things to consider] --- L[The Chemicals]; Root --- R[The Specimens]; L --- L1[Consolidant Type]; L --- L2[Ageing Effects]; L --- L3[Potential for Reaction]; L --- L4[Reversibility]; R --- R1[Porosity]; R --- R2[Texture]; R --- R3[Post-treatment Stability]; R --- R4[Matrix Stability];
```

The Chemicals

Consolidant Type

Ageing Effects

Potential for Reaction

Reversibility

The Specimens

Porosity

Texture

Post-treatment Stability

Matrix Stability

Slide 13

- Why is the gel different? Are we repeating the same mistakes? The history of fossil conservation is paved with good intentions, but how can we be sure we're not causing potential, long-term damage to specimens in the same way as the consolidant we're trying to remove?
- The gel dissolves, and draws the consolidant into it, meaning it doesn't harshly contact the fossil itself.
- It's a gel, so it isn't readily absorbed by the fossil
- It leaves very little residue; most of which can be easily removed, (note: the long term effects of this residue are not yet understood)
- Ageing effects require further study (talk about later)
- Be Cautious!

Are we making the same mistakes? How can we be sure we're not causing the same sort of long-term damage?

Leaves little
residue

No harsh
contact

Not readily
absorbed

Proceed with
CAUTION!

Slide 14

- How to decide which specimens to be unconsolidated.

- Some specimens are better candidates for consolidant removal than others. Those in immediate danger of being damaged by their consolidant are a priority, but not all features of a specimen are conducive to consolidant removal.
 - This method is more effective on smooth specimens than rough ones. Gel residue tends to cling more tenaciously to rough specimens.
 - Will the specimen fall apart upon having its consolidant removed? If so, do not remove it.
 - Are the specimens of great importance? If so, the nature of the gel residue should be fully understood before risking type specimens. (No specimens treated at NPL were type specimens).
 - Is the matrix stable, or might it chemically, (or physically), react with the gel or its components?
 - Can the gel be easily applied to the specimen, and can it subsequently be effectively removed from every crack and crevice?

At high risk from consolidant damage?



What's the surface texture?



Can the specimen survive without consolidant?

Is the specimen of great importance?

Is the matrix stable?



Can the gel be easily applied and removed?

Slide 15

-Cost, time, and labor invested

Specimens usually require only one or two coats of gel applied in a relatively thick layer.

Numerous small specimens can be unconsolidated using relatively little gel, composed of even less of the constituent ingredients.

However, when attempting to clean large groups of specimens, or entire collections, the price and time investment will also increase.

One of the main constraints of this procedure is time. Not including prep-time, residue clean-up, and the possibility of multiple coatings of gel, a minimum of 20 minutes of set time is required for the gel to do its work.

For very large collections, this time factor can be mitigated by performing the procedure on numerous specimens at a time.

Each specimen requires the gel to be removed after ~20 minutes, however, so performing this procedure on very large batches may mean some specimens will sit with gel on them for longer than 20 minutes, which may not be good for the fossil.

Having multiple people working, or gelling specimens in smaller batches may alleviate this risk.

The 20 minute set time for the gel gives little time to work on other things, meaning multitasking while performing this procedure can be difficult.

Despite the investment, both financially and time-wise, the results are dramatic enough, and the methods are simple enough to consider this procedure for any available candidate specimens.

Now how do you go about performing this procedure?

The materials and chemicals
don't cost much...

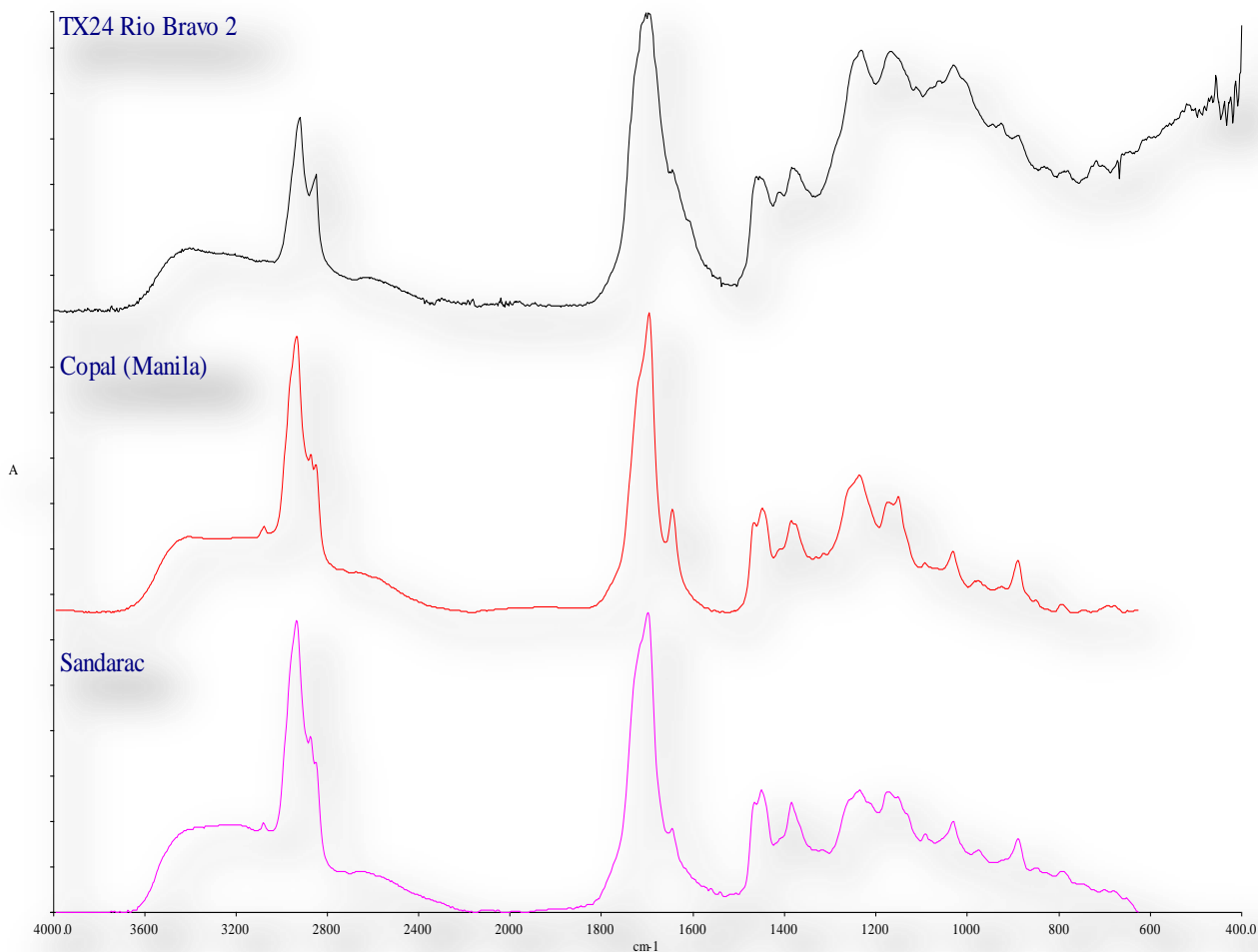


...But the procedure **does**
take time.

Slide 16

- Testing specimen consolidants & IR Spectroscopy
- The composition of the consolidant must be tested before beginning the experiment
- Infrared (IR) Spectroscopy is used to determine chemical composition of the consolidant
 - Based on the absorptive qualities of the chemicals present
 - Peaks indicate which chemicals are present (See: graphs)
- 3 collections tested (Sent to National Museum of Wales)
 - Dumble: 3 specimens sent; 1 animal glue, 1 cellulose acetate, 1 shellac
 - Plummer: 3 specimens sent; 1 unknown, [possibly sandarac, copal, or pine resin], 1 cellulose acetate, 1 cellulose nitrate
 - Rio Bravo: 3 specimens sent; 3 shellac

Infrared (IR) Spectroscopy



Tests the composition of the consolidant

Peaks indicate the chemicals present

Slide 17

- Selecting specimens to unconsolidate
 - Rio Bravo collection selected to test, as all 3 specimens shared the same consolidant
 - We proceeded assuming all specimens in this collection shared this consolidant
 - Identical consolidant allows for fossil texture to be the variable tested in each treatment, and gel pH between trials
 - Specimens in this collection tend to have an unstable matrix, and can be quite friable
 - Rio Bravo collections at high risk, (no climate control; high heat, high humidity)
- A smooth, rough, and friable specimen was selected to be tested in each trial
 - No type specimens were selected
 - Four trials were performed on the specimens, testing 3 different formulations

Rio Bravo Collection

Specimens shared consolidant (shellac)

Collection at high risk

No climate control

High heat/humidity

3 specimens selected
for each trial

Smooth

Rough

Friable

4 trials performed

Slide 18

- 1 step method
- 2 step method

- Acidic formulation
- Basic formulation

Formulation Variants Used

- Ethanol: 200 mL
- Acetone: 200 mL
- Xylene: 50 mL
- Ethomeen: 30 mL
- Carbopol: 6 g
- di Water: 50 mL
- Ethanol: 100 mL
- Acetone: 100 mL
- Xylene: 25 mL
- Ethomeen: 55 mL
- Carbopol: 3 g
- di Water: 25 mL

Specified formulation
acidic (pH ~5)

45 additional mL of
Ethomeen added

10 extra mL of Ethomeen
added to raise pH to 6.0

Basic (pH ~8.5) as specified
by original publication

Slide 19

- gel application (similar for all trials)
 1. Gel applied using a toothbrush in a relatively thick layer
 2. Cheesecloth firmly placed over gelled area
 3. Upon removal, cheesecloth allowed us to easily see if shell material had come off with the gel
 4. Specimen placed in a tight plastic bag, or piece of plastic firmly placed over cheesecloth
 5. Specimen allowed to sit undisturbed for 15-20 minutes

Gel Application

- Gel applied in thick layer (>8mm)
- Cheesecloth placed over specimen
- Specimen tightly bound in plastic bag
- Specimen allowed to sit 15-20 minutes

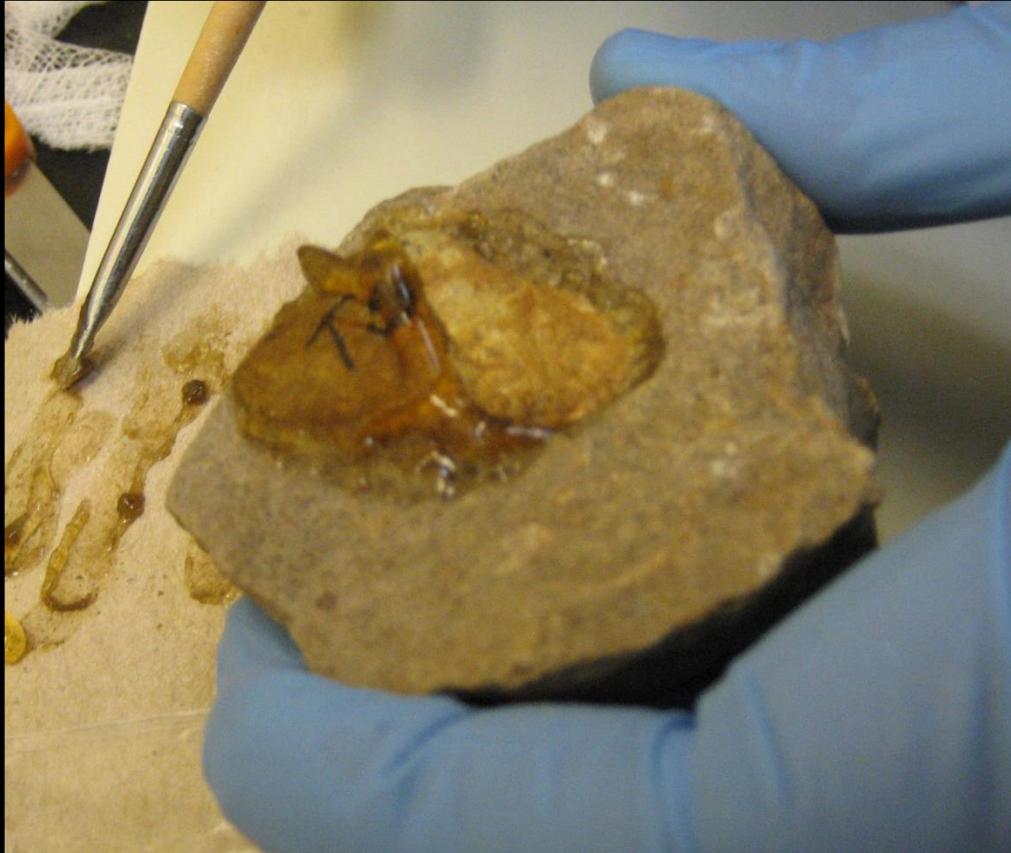


Slide 20

Gel removal

- Plastic and cheesecloth removed carefully, and gel brushed away using a stiff-bristle brush or acid brush
 - A long-bristle acid brush was much more effective at removing gel, and was more gentle on the specimen
 - In specimens that had gel removed using a stiff-bristle brush, clear marring was seen in gel residue under microscope
 - Specimen taken under microscope to inspect for residue or remaining shellac
 - If shellac remains, second treatment performed
 - If only residue present, removed using water and a soft bristle brush

Gel Removal



- Plastic and cheesecloth removed
- Gel removed using (preferably) a long-bristle acid brush
- Inspected under microscope for residue and remaining consolidant
- Possible 2nd treatment (if shellac remains)
- Residue removed using water and a soft-bristle brush

Slide 21

Results

- 1-Step method (Acidic)
- Moderately effective
- Smooth specimen required a second treatment, but cleaned up well
- No apparent damage to the fossils or the matrix

Results: Acidic Formulation

Moderately effective
formula



Smooth specimen required
a second coat

No apparent damage
to specimen or matrix

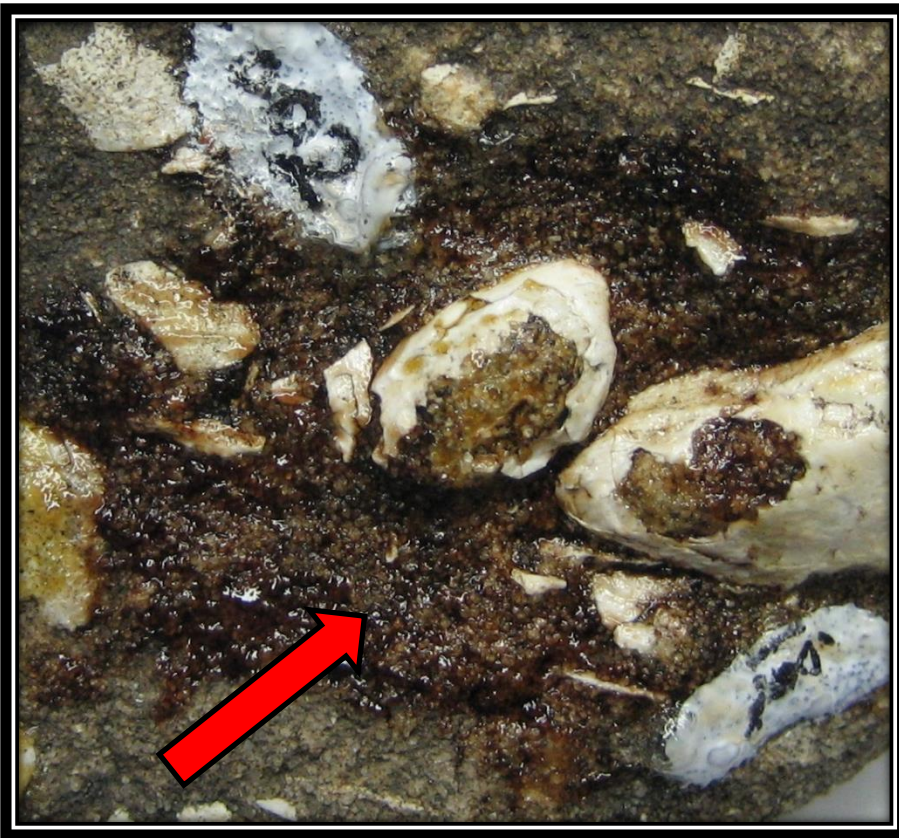


Slide 21

2-Step method (Basic)

- Not very effective
- All 3 specimens required a second treatment
- Some shellac remained in crevices, even after second treatment
- Discolored the matrix
- Was thicker than the other formulations
- More difficult to apply
- Adhered more to the specimens, meaning more residue was left behind

Results: Basic Formulation



Least effective formula

All 3 specimens required a second treatment

Caused matrix discoloration

Most difficult to apply and remove

Leaves a lot of residue

Slide 22

- 2-Step method (neutral-ish)
- Most effective formula
- No specimens in the first trial required a second treatment; due to age and thickness of the shellac, the second trial specimens required a second treatment
- Easy to apply, and easy to remove
- Seemed to remove consolidant effectively, even upon thinner application
- Little residue left behind
- No apparent damage to the fossils or matrix

Final Formulation

- Ethanol: 100 mL
- Acetone: 100 mL
- Xylene: 25 mL

- Ethomeen: 20 mL
- Carbopol: 6 g
- DI Water: 50 mL

**½ solvent of original
formulation**

**Prescribed amount of
gelling and detergent
components**

Clean Removal

**Neutral
pH**

Highly Workable

Slides 23-25

- Before and after of final formulation

Smooth



Rough



Friable



Slide 26

- Shellac, and any markings present, were drawn up into the gel
 - No shell material was observed being removed by the gel
- Despite its effectiveness, there is some concern regarding the acidic formulation
 - Original paper formula had a high pH, and was tested on highly resistant vertebrate teeth
 - Calcite and aragonite shell material is very soft, and susceptible to acidic chemicals
- Is there risk from unnoticeable? Only observation over time will tell

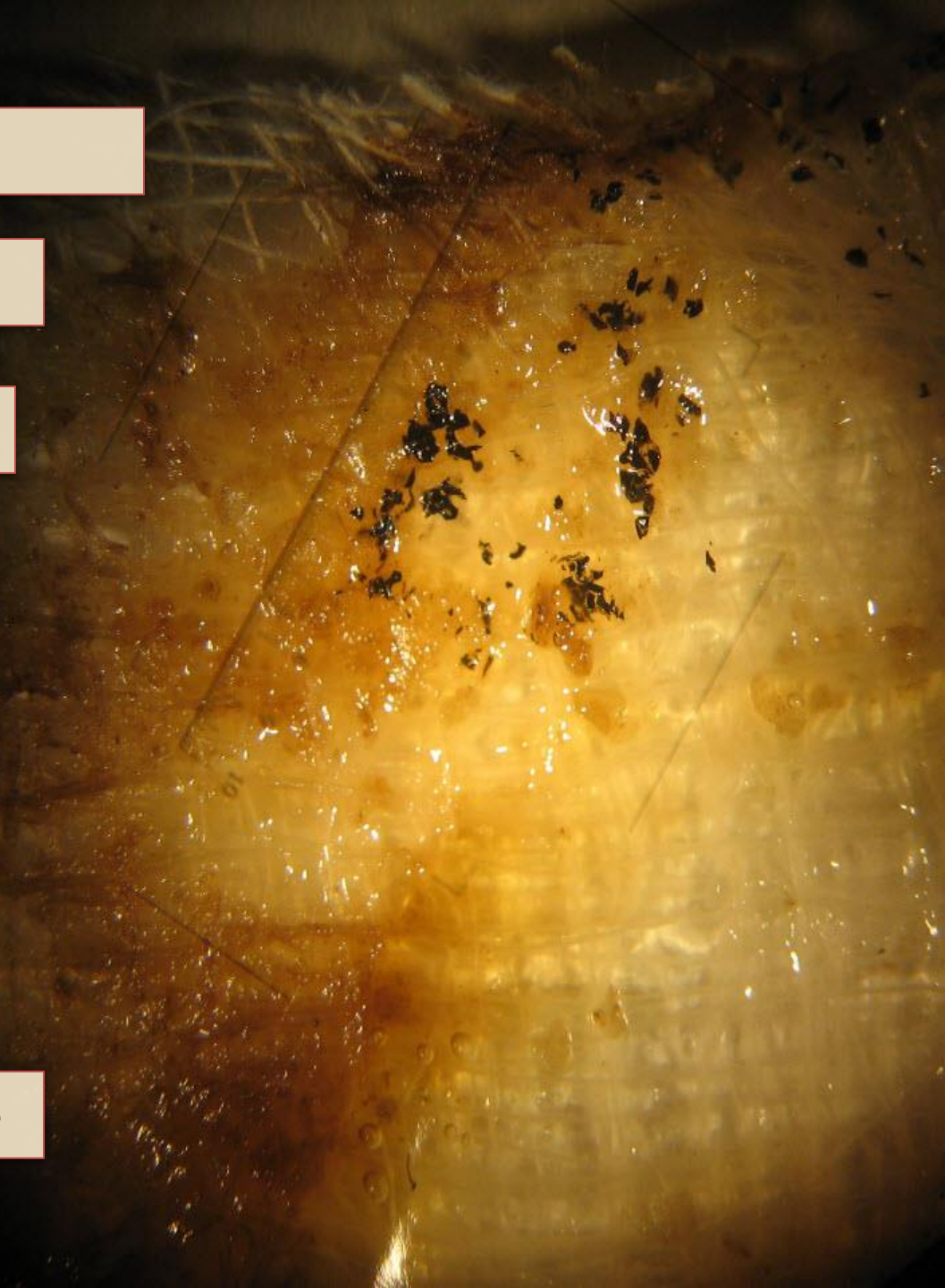
Logistics of procedure

Destabilizing specimens

Markings drawn up into gel

Long term conservation
plan

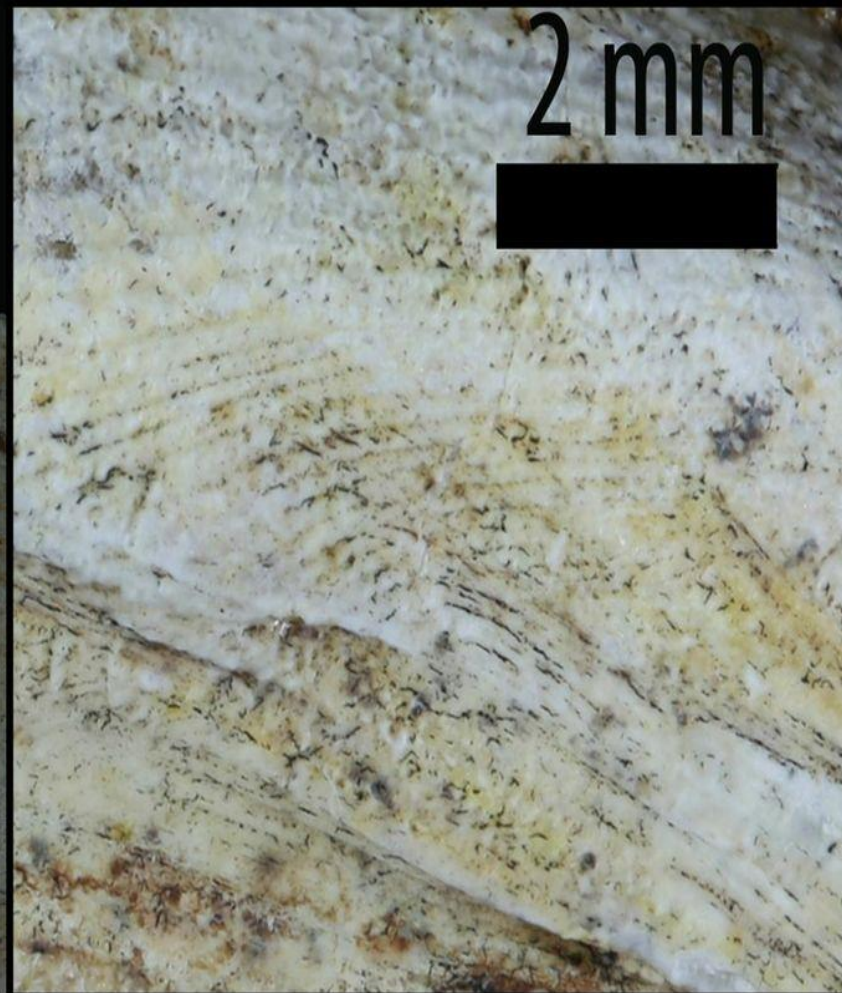
Ageing effects of residue



Slide 27

- Close up of residue

How will this residue look in 50 years?



Slide 28

- With our study, we've been able to isolate 3 risk categories
- Lower risk- Smooth surfaces
 - clean removal
- Not recommended-Rugose surfaces
 - Too much residue was left behind.
- Getty institute tested this formulation for restoring oil paintings, they found the volatile compounds of the solvent evaporated quickly, and the remaining residue was equivalent to touching the surface of the oil painting 10 times with an ungloved hand.
 - The effects of the gel residue aren't fully understood in the context of porous fossil material, and the treated specimens are currently under observation.
- Conditional- Friable visible cracks.
 - However, no damage to the underlying fossil has been observed in the short-term, indicating this consolidant removal is a safer alternative to leaving the consolidant on, as far as physical damage to the specimen is concerned.
 - Remove consolidant in quadrants, re-consolidate using modern materials.



Lower risk

- Smooth surfaces

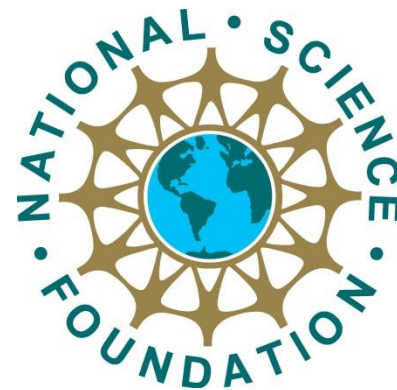
Conditional

- Friable, visible cracks

Not Recommended

- Rugose surfaces

A special thanks goes to Todd Ellis of Texas State University for all his help getting this project started, and Matthew Brown and Ann Molineux for guidance and editing. We would also like to thank Julian Carter for the spectrometry analysis, and Azko-Noble, who have provided samples of Ethomeen® C/25 and Lubrizol for supplying samples of Carbopol® EZ-2.



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Open Access: Conservation, Digitization and interoperability of the Historic Non-vertebrate Collections of the Texas Natural Science Center

Any opinions, findings, and conclusions or recommendations expressed in this material are those of the authors and do not necessarily reflect the views of the National Science Foundation.

In keeping with the spirit of Open Access, we are proud to make available all lab notes, additional images, observations, and tests on our website.

www.utexas.edu/tmm/npl/projects/conservation/shellac



Thanks Todd!

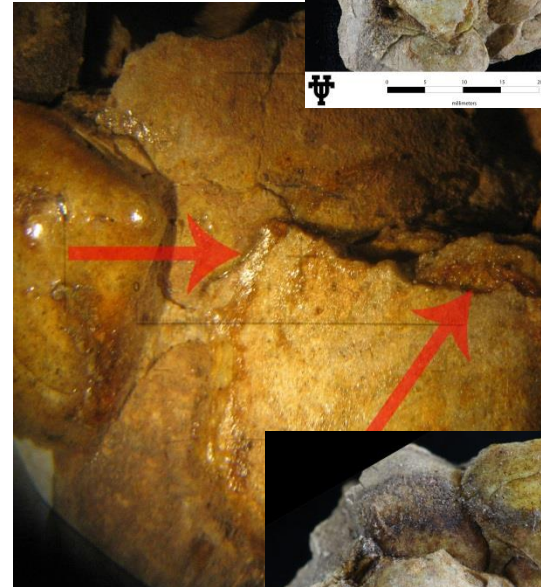
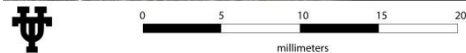
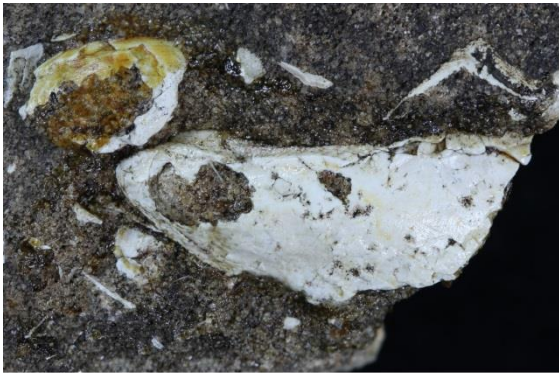


Thanks Matt!

Formulation 1



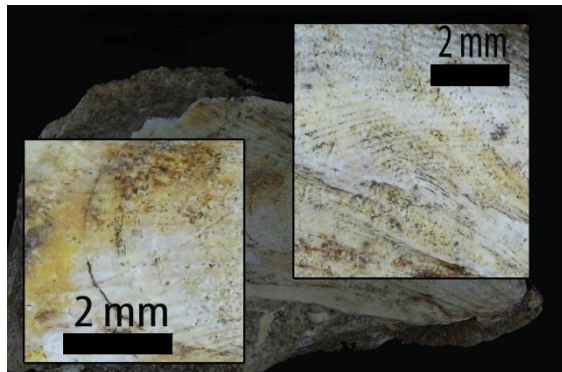
Treatment 1



Treatment 2



Formulation 2



(the smaller one)



Matrix discolored



Lab Notes

Solvent Gel

Composition

200 ml ethanol

200 ml acetone

50 ml zylene

20 ml Ethomeen C/25

6g Carbopol EZ2

50 ml DI water

-Alt. composition (shellac <2yrs)

450 ml ethanol

20 ml Ethomeen C/25

6g Carbopol EZ2

50 ml DI water

[We didn't test this formulation, we wanted to use the general formula- which is what we would be using for specimens that we don't exactly know the consolidant used]

Method 1

Sprinkle Carbopol EZ2 powder onto Ethomeen and stir until you get a paste

Add remaining solvent, stir

Add water while stirring continuously

Screw lid on and shave vigorously

Method 2

Sprinkle Carbopol powder into water while stirring. Stir until smooth paste forms

Pour in Ethomeen and stir until transparent paste forms.

Shake vigorously

Test for pH- should be between 7.0 and 8.0.

Add Ethomeen to increase pH

Add Carbopol to reduce it

Mix remaining solvents in a second bottle

Cut in the Carbopol/Ethomeen gel gradually. If it turns cloudy or a sticky residue forms, add more water

A gel of pH 8.5 should be the result

Application

Brush loose dirt from surface

Apply >8mm coat of gel to surface

Stretch plastic wrap over surface (Saran wrap)

Let sit for 15-20 min.

Use wrap to aid in removal of gel, properly dispose of both

Use wooden tool to tease away blistered shellac, and water or ethanol and a soft brush to remove remaining gel

Notes:

- **Formulation 1**

pH too low. Ethomeen added -5 ml

pH still low. Another 5 ml Ethomeen added

pH still a little low, using it anyhow

Instead of Saran wrap, gel will be applied followed by 4 layers of cheesecloth and a piece of a cut up clear plastic bag

Bag curled up, not effective

After removing gel with a short sturdy brush, specimens were rubbed down with acetone to remove residue. [It smears residue around more than it removes it] [Specimens looked pretty good after a treatment, some required 2nd treatment to get rid of the residue, although this may have been more due to us not removing the residue properly]

Formulation 2

Mixed water and Carbopol at half quantities (25 ml water, 3g Carbopol) [We let it sit for a few days]

Collected remaining ingredients at half quantities

Added 10 ml Ethomeen to Carbopol /water mixture, mixed vigorously.

Resulting gel is thick and yellow, not clear (due to the type of Ethomeen used?)[Probably not, the C/25 A, which we thought stood for 'Amber' is not significant, per AkzoNobel rep]

A small sample was broken down in a little water so we could test the pH

pH was tested. 2 dilutions were tested- one highly dilute and one less dilute. Undiluted gel too thick to test on a pH strip.

- pH seemed to stay around 5.0, so a small mixture of water and Carbopol is being tested for pH, also testing the Ethomeen.
- Strips were functioning correctly. Ethomeen and Carbopol are their expected pH.

Adding additional Ethomeen to increase pH

Adding 5 ml

Adding another 5 ml

Adding another 5 ml

Adding another 5 ml

pH of ~7 reached. Total extra Ethomeen = 20 ml

Remaining solvents (acetone/xylene/ethanol) were mixed in a separate beaker

Carbopol, Ethomeen/Water gel was then cut into the solvent mixture and stirred.

A yellow gel formed upon mixing in the original Ethomeen gel

End of mixing, pH was ~5.5

Adding 5 ml Ethomeen

Another 5 ml

Another 5 ml

Another 5 ml

pH ~8 achieved. Left the gel to sit [covered, in a fume hood] and fully disperse

end of day 1- pH 7.3

end of day 4- pH ~8

Adding more Ethomeen- we are looking to achieve pH 8.5, per original paper
Ethomeen added in 5 ml quantities in order to get the correct pH
-added 15 ml. before 8.5 was achieved

2 pH readings were taken at each 5 ml interval. One shortly after adding mixing in Ethomeen and one after about 10 min., after Ethomeen is allowed to disperse in the gel

After 15 ml. [total] of Ethomeen, no significant change to pH was observed, pH is staying ~8. No additional Ethomeen was added

A thick layer (~8mm thick) was applied to 3 different shell specimens, one smooth one rough one friable

Each was covered in cheesecloth and placed into a plastic bag, which was tightened around them and allowed to sit for 15 minutes

After sitting for 15 minutes, cheesecloth was removed and a combination of a fine tipped brush and a frayed tongue depressor were used to remove excel gel

Most, but not all of the shellac was removed. A soft toothbrush was used to attempt to remove some excess shellac, to little avail.

- A second treatment of gel was applied to all 3 specimens
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- The second treatment was wrapped more tightly than the first
- -most of the remaining shellac was removed, but some remained, particularly in the pits and crevices (due to uneven pressure being applied?)
-
- Discoloration noted on matrix of friable specimen.