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THE DOCUMENTATION OF ADHESIVES IN THE WHOLE VESSEL POTTERY COLLECTION AT THE ARIZONA STATE MUSEUM

Julie Unruh, Tara Hornung, and Stephanie Ratcliffe

Abstract

The Arizona State Museum’s whole vessel collection contains ceramics from nearly every cultural group in the American Southwest, including the type collections for two major prehistoric cultures. A project is underway to rehouse the entire 20,000-object collection in a purpose-built storeroom. Because it is the most comprehensive collection of its kind, the project received a “Save America’s Treasures” grant in 2000.

In preparation for the move, the condition of each object is being recorded in a “Pottery Project” database. In addition to identifying conservation concerns, the survey phase of the project includes gathering statistics on features of interest to the conservation lab and other researchers from the 20,000-object sample base.

As a part of the project, all adhesives used in the collection are being identified and documented by staff and students. An assembly-line system has been developed to deal with thousands of adhesive identifications, including ethnographic repair materials (resins and plant gums), adhesives used by archaeologists and collectors, undocumented adhesives used by the Museum, and assembly adhesives used by contemporary potters. The process is primarily low tech, utilizing spot tests, UV examinations, and solubility. Select adhesives receive further instrumental analysis.

A searchable and sortable Access database was designed to collect the data. At the end of the survey, the database will be able to instantly:
- provide a library of adhesives used in the collection over the past 100 years;
- provide statistics regarding the frequencies of usage of specific adhesives within the collection;
- provide a timeline of adhesives usage within the collection;
- identify regional and cultural use of particular ethnographic repair adhesives;
- identify trends in adhesive usage in Southwestern archaeology.

Introduction

The Arizona State Museum holds one of the most comprehensive collections of Southwestern Native American ceramics in existence. The whole vessel collection contains 20,000 complete vessels from nearly every cultural group in the American Southwest, including the type collections for the Mogollon and Hohokam prehistoric cultures. Unfortunately, the collection is housed in inadequate storage with little climate control, and as a result the collection has incurred damage from soluble salts, abrasion, adhesive failure and breakage. Because this collection is a
significant cultural resource, funds were raised to rehouse the collection in a purpose-built storeroom. The project, called the “Pottery Project” by the Arizona State Museum received a “Save America’s Treasures” designation in 2000.

In preparation for moving the collection, every pot is being surveyed and a condition report written. In addition to identifying conservation concerns, the survey phase of the project is an opportunity to gather statistics from the 20,000-object sample base on features of interest to the conservation lab and other researchers. As one facet of the project, conservation staff and students are endeavoring to identify and document every adhesive used in the collection. The adhesives fall into two groups: ethnographic repair materials, applied by the cultural groups who used the pottery; and adhesives used by archaeologists, collectors, and conservators at the Museum and elsewhere. The vast majority of these adhesives are completely undocumented. The Pottery Project has given the Arizona State Museum an unprecedented chance not only to identify these adhesives, but to put them into a searchable, sortable database for easy access by researchers.

Because thousands of adhesives must be identified, out of necessity the identification procedure was designed to be as quick and uncomplicated as possible. It is helpful that the presence of certain adhesives can be anticipated. It is known that cellulose nitrate was used by many archaeologists working in the American Southwest from at least the 1940s onward. Adhesives commonly used by early restorers, such as animal glue, and by later conservators, such as the PVOH derivatives and the acrylic copolymers, were also anticipated. Research revealed that a variety of ethnographic adhesives could be expected, including an animal adhesive made by boiling bighorn sheep horns, cactus gums from a variety of cacti and cacti fruits, piñon pine pitch, natural tar, waterproofing adhesives involving a mixture of sea turtle fat and cactus gum, and “creosote lac”, a lac secretion from a scale insect that lives on creosote bushes.

With these possibilities in mind, an adhesive identification system was developed based on three simple and inexpensive procedures: UV examination, solubility, and microchemical spot tests. Identification can be done quickly in an assembly line fashion. Figure 1 is a chart of the process for eight of the most regularly encountered adhesives.

All materials testing involves common sense. While a common sense assessment would seem to go without saying, in an assembly line process, there is a danger that the tester will blindly follow the steps without thinking about whether the identification makes sense for the time period and the visual appearance of the adhesive being tested. Conversely, the tester must keep an open mind. In several instances an “ancient” repair turned out to be a modern repair material.

As the beginning of the process, the visual appearance of the adhesives in a group of ceramics to be tested is noted.

The ceramics are then examined under UV light. In some cases, the visual appearance and the fluorescence color together can be enough to allow a good guess as to the identification of the adhesive. In any case, the color of the UV fluorescence determines the next step in the program of testing. UV examination as the “first pass” is additionally helpful in that it reveals adhesives undetected by the naked eye, uncovers multiple campaigns of repair requiring individual testing, and alerts the tester to good sampling locations (Fig. 2).
<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Fluorescence</th>
<th>Solubility</th>
<th>Spot tests</th>
</tr>
</thead>
<tbody>
<tr>
<td>Animal adhesive</td>
<td>bright white</td>
<td>Water (hot water). The older it is, the longer it will take. May smell like animal glue.</td>
<td>Protein: Copper (II) sulfate</td>
</tr>
<tr>
<td>Asphalt (natural tar)</td>
<td>orange</td>
<td>petroleum distillates (petroleum benzine, stoddard's solvent)</td>
<td>none yet</td>
</tr>
<tr>
<td>Cellulose nitrate</td>
<td>bright</td>
<td>acetone</td>
<td>Nitrate: diphenylamine test for nitrates</td>
</tr>
<tr>
<td>Creosote lac</td>
<td>orange</td>
<td>1 M NaOH</td>
<td>none yet look for nitrogen with CaO2 and pyrolysis; look for a positive with the PV(OH) test</td>
</tr>
<tr>
<td>Epoxy</td>
<td>bright white</td>
<td>insoluble; may swell in methylene chloride</td>
<td>none yet look for nitrogen with CaO2 and pyrolysis; look for a positive with the PV(OH) test</td>
</tr>
<tr>
<td>Natural plant resins or gums</td>
<td>none to yellow-green: can be dull, can be bright</td>
<td>ethanol</td>
<td>Rosin: saturated sugar and sulfuric acid</td>
</tr>
<tr>
<td>Pine resin (rosin)</td>
<td>none to yellow-green: can be dull, can be bright</td>
<td>ethanol</td>
<td>PV(OH) derivative: KI/I2 and glacial acetic acid</td>
</tr>
<tr>
<td>PV(OH) derivative</td>
<td>pv(OH) fluorescence</td>
<td>acetone; also may be insoluble</td>
<td></td>
</tr>
</tbody>
</table>

Figure 1. Identification parameters for eight adhesives in the Arizona State Museum collection.

Figure 2. A Tarahumara pot with an ethnographic repair. Under UV, it is clear that there are two adhesives: a green-fluorescing adhesive over an orange-fluorescing adhesive. The color of the fluorescence determines the next testing step. Photograph by Margaret Kipling. Assessing fluorescence color is subjective, but by evaluating enough adhesives, the tester is able to formulate a good individual
sense of color. Though an adhesive reference set is available to the project, it was felt that it had limited use. More useful is experience. Over time, in a collection in which the same adhesives are seen repeatedly, it was found that testers are able to develop a good personal knowledge of the appearance of various adhesives under UV light.

The results of the UV examination determine the next steps in the testing process: solubility and spot testing, often done simultaneously. All of the spot tests used are published in “Materials Testing for Art and Archaeology” (Odegaard et al. 2000). The authors discuss the spot test procedures, chemical reactions, reagent preparation, and safety precautions in detail; accordingly, those topics will not be discussed here. However, some published procedures were streamlined, and those modifications are described below.

**Procedure 1: Visual assessment: plastic-like, transparent, whitish, yellowish**

**UV fluorescence: bluish, greenish, yellowish, no fluorescence**

By far the most common adhesive in the Arizona State Museum collection is cellulose nitrate. Cellulose nitrate was used by virtually all archaeologists working in the Southwest during the 20th century, and its occasional use continues until the present day. Cellulose nitrate can fluoresce a range of pale colors including blue, green, and yellow; and also has been observed to have almost no fluorescence. However, combined with an assessment of the appearance of the adhesive in visible light, a fairly good guess as to its identification can be made quickly. The guess can be confirmed in seconds with the diphenylamine test for nitrates (Odegaard et al. 2000, 164 -165). The published procedure was adapted in order to do the spot test on a swab. Since cellulose nitrate should be soluble in acetone, a small amount of the sample is picked up with an acetone swab. Note that this is also a solubility test. If the sample does not dissolve enough to be picked up on a swab, then the adhesive is unlikely to be cellulose nitrate. The diphenylamine reagent is dropped directly onto the swab. The characteristic blue color which indicates a positive will develop directly on the cotton.

The diphenylamine test can also give a positive for nitrate salts in the ceramic fabric. However, conscientious sampling techniques combined with an awareness of the potential for salts in the pot allow the tester to be fairly certain that a positive indicates nitrates in the adhesive, rather than nitrates in the ceramic. A control swab, swabbed on the surface of the ceramic rather than on the adhesive, can be helpful.

If the adhesive has a cellulose nitrate appearance and fluorescence but the diphenylamine test is negative, a good second guess is a poly(vinyl alcohol), or PVOH, derivative. This class of adhesives includes the poly(vinyl acetate) (PVAC) adhesives such as the AYA- series adhesives (Union Carbide) used in conservation; PVAC craft glues such as Elmers Glue-All (Borden), and some wood glues. The spot test for PVOH uses two reagents, glacial acetic acid and potassium iodide (Odegaard et al. 2000, 166 -167).

Trials were run to determine whether the PVOH test could be successfully performed on a swab. While it was found that it was possible to produce a positive reaction on a swab, in the end it was decided that, for two reasons, it is better to perform this test in a spot test plate as published. First, PVOH adhesives can be only marginally soluble, or even completely insoluble, in acetone.
If that is the case, an acetone swab may not pick up a good sample, though the test may still be successfully performed on a solid sample taken in the conventional way with a scalpel. Second, the positive reaction is a red color which may be faint, or may form only at the edges of a sample. If so, the reaction is more likely to be visible on a solid sample under magnification rather than on a dissolved smear of sample on a cotton swab.

The PVOH test will also give a positive for starch, and a positive for at least some epoxies. However, if the tester is paying attention to solubility and fluorescence, there should be no confusion with these materials.

If the spot tests give a negative for cellulose nitrate and a negative for a PVOH, the next guess might be that the adhesive is an acrylic adhesive such as Acryloid B-72 (Rohm and Haas). There is no spot test for acrylics, though fluorescence and solubility provide clues for its identification. It was fortuitous for the project that at the Arizona State Museum, the advent of acrylic use was approximately the same time as the advent of good conservation documentation. Consequently, in the Arizona State Museum collection, it has usually been possible to positively identify acrylic adhesives via the conservation documentation. In other situations, documentation will not exist. It is hoped that a spot test to identify acrylic polymers will be developed in the future [1].

Figure 3. Testing flow chart for adhesives which are visually plastic-like, transparent, whitish or yellowish, and which are pale blue, green, yellow, or have no fluorescence under UV light.
**Procedure 2: Visual appearance: transparent to dark brown**

**UV fluorescence: white**

In the Arizona State Museum collection there are two main adhesives that fluoresce white: animal adhesive and epoxy. They are not similar visually. If the tester is paying attention, all subsequent testing after the UV examination should be a confirmation of what is already suspected.

Animal glue is water soluble, though the solubility decreases with age. Nonetheless, solubility can usually be confirmed in seconds with a damp swab. (Hot water may dissolve more adhesive than room temperature water.) The identification is then double checked with the Biuret test for protein using copper II sulfate and sodium hydroxide (Odegaard et al. 2000, 144 -145), which can be done directly on the swab, as for the nitrates test.

Odegaard et al. lists a second test for protein using calcium oxide and pyrolysis (142 -143). That test is actually a test for bound nitrogen. Epoxies contain nitrogen. Consequently, if that test is used to identify a protein adhesive, an epoxy can give a false positive (and note that both epoxy and animal glue fluoresce white, further confusing the issue). In fact, this situation is helpful. There is no spot test for epoxies. However, two tests have now been identified which may give a false positive for epoxy: the PVOH test discussed above, and the bound nitrogen “protein” test. If an adhesive does not seem to be soluble in anything and yet gives a positive for nitrogen as well as a positive for PVOH, a good course of action is to see if it reacts to methylene chloride. Epoxies will not dissolve in methylene chloride, but they should swell. (A convenient way to watch a reaction with any solvent is to put a small sample on a slide, cover it with a cover sheet, and wick in the solvent from the edge of the cover. In this way the solvent fumes are contained and less solvent is needed.) Under the microscope, swelling may be seen as movement of the sample rather than a dramatic change of dimensions.

Figure 4 presents the Procedures discussed above as a flow chart.
Figure 4. Testing flow chart for adhesives which are visually transparent to dark brown, and which fluoresce white under UV light.

Procedure 3: Visual assessment: transparent to dark brown
UV fluorescence: yellowish-green, green

Substances that fluoresce greenish and are soluble in ethanol were suspected to be plant gums. In the Arizona State Museum catalog information, often anything that looks dark brown and fairly lumpy has often been assumed to be piñon pine pitch. Accordingly, as a point of departure, we began testing dark brown, ethanol-soluble repairs with the Raspail test for rosin (rosin is pine resin; Odegaard et al. 2000, 158 -159).

The least destructive method for sampling any of the thick resinous substances in the collections was to take a “scratch test”. This sampling method uses a frosted microscope slide, cleaned with acetone to remove contaminates. The frosted end of the slide is swiped across the sample with light pressure, picking up a streak of residue on the surface of the slide. The test is then performed directly on the streaked slide and observed under a microscope. The benefits of this method are that the repair resin remains relatively undisturbed; and that, as for the swab sampling method, samples can be more easily taken from awkward sampling locations without needing to manipulate the ceramics unnecessarily.
The Raspail test for rosin indicates the presence of abietic acid. The sample is saturated in a saturated sugar solution, and then exposed to concentrated sulfuric acid. A positive reaction is raspberry red (a blue-red or cool-red color). The color can take up to 30 minutes to develop with low concentrations of abietic acid (aged samples), and false positives are common due to the warm red color that can develop simply from contact between the sample and the concentrated sulfuric acid. The testers found that it took practice to master the techniques and to learn the subtleties of color development.

While some of the greenish fluorescing ethnographic repairs tested positive for rosin, some did not. Nonetheless, because of the fluorescence, it was still suspected that they were plant adhesives of some sort, and a search began for a way to determine the presence of a plant material.

There is a simple test for carbohydrates using triphenyltetrazolium chloride or TPTZ (Odegaard et al. 2000, 134 - 135). Unfortunately this test it cannot be recommended. It was discovered that this test gave a positive for everything, including a blank, and this test was subsequently abandoned as a possible procedure for the project. A second test in the book for carbohydrates with o-toluidine (132 - 133) is actually a test for polysaccharides. Although this test will not identify every plant product, it should identify most of the gums and resins. Unfortunately, this test also was determined to be problematic: all the tests were either negative or inconclusive.

At this point one of the authors of the book was consulted (Zimmt, 2005). It is believed that the problem with the o-toluidine test may lie in the procedure, rather than in the test, and that there still may be some hope for using a revised version of this test in the process. However, until the problem is solved, this test still cannot be recommended. At present, we are left without a spot test to identify plant gum adhesives, or resins which are not pine resin.

In the end, Fourier transform infrared spectroscopy (FTIR) was used to identify some of the ethnographic adhesives. Obviously FTIR is not a solution for most museum labs. Again, it would be very helpful to have a conclusive test for plant-based adhesives. It is hoped that such a test can be identified in the future.

Figure 5 presents the procedures discussed above as a flow chart.
Figure 5. Testing flow chart for adhesives which are visually transparent to dark brown, and which fluoresce yellowish-green under UV light.

Procedure 4: Visual assessment: dark brown, black
UV fluorescence: orange

In the Arizona State Museum collection, two adhesives have been identified which fluoresce orange. Surprisingly, neither one is shellac. One is natural tar or asphalt, and the other is creosote lac. There is no spot test for either of these substances. However, the UV and solubilities are so distinctive that there seems little chance for confusion. Neither is soluble in ethanol, which would indicate shellac. If the adhesive dissolves in one of the petroleum distillates such as petroleum benzine, then it is identified as tar. If not, solubility in 1M sodium hydroxide indicates creosote lac. Creosote lac is, like shellac, a lacquer which is secreted by an insect – in this case one which lives on creosote bushes. The creosote lac insect is related to the cochineal insect. Interestingly, when creosote lac dissolves in sodium hydroxide, a startling fuchsia color is usually, though not always, evolved. This distinctive color may be related to the cochineal dye, and the presence of the color or not may have to do with the purity of the lacquer.
Figure 6 presents the procedures discussed above as a flow chart.

![Flow Chart](image)

Figure 6. Testing flow chart for adhesives which are visually dark brown or black, and which fluoresce orange under UV light.

**Composite materials**

As should be expected, many ethnographic repair materials are composite materials. In some cases, a mixture of fluoresces can be seen in one material under UV light. The individual ingredients cannot always be sorted out. This has been the case with a mysterious repair material which was recorded as “Mexican lacquer” in the catalog information. The material had a characteristic fluorescence under UV light, simultaneously green and orange, implying two different substances mixed together. The same distinctive fluorescence had been observed on repairs on several of the vessels from the Tohono O’Odham and Seri cultures in southern Arizona and northern Mexico. All of these repairs tested negative for pine resin.

“Mexican lacquer” is commonly used to refer to a type of folk art from Mexico created by applying colored varnish layers to wood or gourd. Multiple recipes exist for the varnish, which is called by a number of names, including “aje.” Mills and White (1999, 118) classify “aje” as an insect “resin” (their quotes) and report that aje is a boiled insect extract mixed with seed oils, but that “nothing is known of its chemical nature”.

A resinous sample in the conservation laboratory at the Arizona State Museum, collected in the Chiapas region of Mexico, was labeled “Mexican lacquer.” It was not further identified. It had
the same distinctive green and orange fluorescence under UV light, and also tested negative for pine resin. The sample was only slightly soluble in ethanol, swelled in acetone and was insoluble in Stoddard solvent. It was, however, completely soluble in sodium hydroxide.

FTIR analysis was inconclusive in identifying either component of our “Mexican Lacquer” sample, or even in determining whether either of the distinct fluorescences could be matched to a plant or animal product. However, ceramic repair materials with the same orange and green fluorescence did have an FTIR fingerprint similar to that of our sample, and they also proved to be soluble in sodium hydroxide.

Many clues pointed towards a component similar to creosote lac. (Note that creosote lac is not a boiled insect extract, but rather an exuded lac product.) One of the ceramics which produced a similar FTIR spectrum to the “Mexican Lacquer” sample even was described on the catalog card as “sealed with csipz (xsipx), a lac of creosote bush”. However, the dual fluorescences, the slight solubility in ethanol, and the inconclusive FTIR spectra convinced us that what we have is a mixture, very possibly the insect and drying oil mix described by Mills and White. The substance has yet to be positively identified.

In other cases, the documentation alerted us to the fact that the repair material is a mix. This was the case for a Seri material described as organ pipe cactus gum boiled with turtle or pelican grease (a repair material made for caulking boats).

An attempt was made to verify the catalog information. The gum proved impossible to conclusively identify, due to the unreliability of plant gum tests, as reported above. However, we were successful in determining the presence of a fatty acid, using a procedure developed by Werner Zimmt (2005), reported in Appendix A.

It is unlikely that we would have thought to test for the presence of a fat component had the catalog information not informed us of its presence. This is a warning sign that many of the ethnographic repairs may have constituents that are going unnoticed and untested. Supporting this suspicion is the fact that many of the FTIR spectra are inconclusive. They appear to indicate mixtures of materials; mixtures are not easily identified with FTIR. It must be emphasized that much work remains to be done with identifying ethnographic repair materials. It will be important to build a reference library of FTIR spectra for those materials.

The Pottery Project adhesives database

A database should not just be a file of records. It should be a tool for looking at data in various ways. If the database is designed correctly, the data can be organized as needed to answer particular questions. The Pottery Project adhesives database was designed to:

- provide a library of adhesives used in the collection over the past 100 years;
- provide statistics regarding the frequencies of usage of specific adhesives within the collection;
- provide a timeline of adhesives usage within the collection;
- identify regional and cultural use of particular ethnographic repair adhesives;
identify trends in adhesive usage in Southwestern archaeology.

All information collected for the Pottery Project, including the adhesives identification, is entered into one Microsoft Access database. From the main survey form, as each pot is surveyed, the adhesives information is input into an embedded Repairs “subform” (Fig. 7). The database is designed so that as many Repairs subforms as are needed may be linked to the main record for each pot. If there are multiple campaigns of repair, each repair is recorded in an individual record. The result is that all repairs, not just all pots, can be individually sorted and queried.

![Figure 7. Repairs subform as it appears on the computer screen.](image)

The database is able to sort by any of the fields in the “Repairs” form, and also by any of the fields in the related record for the pot. For example, if the database user asks the question: “What materials were used for ethnographic adhesive repairs?”, he can request that the database return a list of all records for which the “cultural repair” box is checked in the Repairs record, and for
which an entry appears in the “adhesives” field. Thousands of records will be instantly
organized into a useful list. The records can be sorted by date of repair, producing a timeline of
usage; or by the cultural group entered on the related main record, producing an inventory of
adhesives used by that group, and so forth.

If the database user wants to ask the question, “what adhesives are archaeologists in the
American Southwest using?”, he can request that the database return a list of all repairs made by
archaeologists. If it is sorted by the date of the repair, it will be a timeline of adhesives usage in
Southwestern archaeology. If it is sorted by archaeological site location (which is entered in the
related main record), it will be a catalog of all adhesives used at a particular site. That
information could also be further sorted by date, and so forth.

Any of this information could be downloaded to a compatible software program. For example, a
list of adhesives and the dates of their usage can be downloaded into Microsoft Excel, a
spreadsheet program. Excel could then graph the repair date data as an actual timeline, or graph
the incidence of adhesives usage as a pie chart, or graph them both together as scatter graph
showing the incidence of use over time. As we expect many thousands of records at the end of
the project, the automatic graphing facility will be able to accomplish what would not have been
otherwise possible: easily making a large amount of data comprehensible and usable.

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Stout Fund.

Endnote

1. In our testing, HMG brand B-72, an acrylic copolymer adhesive, tested positive for nitrates
with the diphenylamine test. This unfortunate fact was discovered when a ceramic known to
have been repaired with HMG B-72 consistently tested positive for nitrates. Two tubes of HMG
B-72 subsequently also tested positive. At present, no attempt has been made to identify the
source of the nitrates in this adhesive.

Suppliers

Chemical reagents used in the spot testing:
Fisher Scientific, 1 Reagent Lane, Fairlawn, NJ 07410, (201) 796-7100, Fax: 201-796-1329,
(www1.fishersci.com/index.jsp)

Hand held UV lamp:
several models available from Cole-Parmer Instrument Company, 625 East Bunker Court,
Microsoft Access database software, included in Microsoft Office Suite software packages: (www.microsoft.com/products)

References


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Appendix A: test for the presence of a fatty acid

Procedure for determining the presence of fatty acid:

- Add a drop of concentrated hydrochloric acid to a small sample in a spot test plate. Place in an oven until dry. This step hydrolyzes the sample.
- Dissolve the dry, hydrolyzed sample in a drop of Stoddard’s solvent.
- Filter this solution through fluted filter paper in a funnel to remove solids. Transfer the remaining liquid with a pipet onto a clean microscope slide.
- Evaporate the liquid on a hot plate.
- Because the fatty acid is hydrophobic, if the material is a fatty acid, a drop of water will bead on the residue.
- Finally, a drop of 1M sodium hydroxide will dissolve the residue by neutralizing the acid.