

THE ANALYSIS, IDENTIFICATION AND TREATMENT OF AN AMBER ARTIFACT

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This study describes the identification and treatment of an amber necklace, which came into the conservation lab of Conservation Art Service, with an opaque bloom caused by a previous cleaning with a household ammonia cleanser. This paper also includes an overview of amber and its historical use, methods to definitively identify amber, and the identification and treatment of this particular object using infrared spectroscopy.

Amber is a fossilized tree resin, formed through a complex series of steps over millions of years. Its chemical composition varies depending on the origin of the resin, but Baltic amber is synonymous with the chemical name butanedioic acid (C₄H₆O₄), more commonly known as succinic acid, Beck, 1986. Although roughly 80% of all amber samples are Baltic amber, there are other types of amber, not all of which contain succinic acid. It has been theorized that succinic acid may not be contained in the original amber material, and that it may be formed as a product of the aging process through a transition state byproduct, the abietic acid (C₂₀H₃₀O₂) (Rottlander, 1970). Amber objects have been treated with a variety of materials to repair damage or clarify abrasions and imperfections. Few records exist of most of these treatments and few treatments have been published (Beck, 1982).

DEFINITIONS AND HISTORY

Amber has a very rich history of varied uses. It is easily carved and polished, which has made it popular for jewelry and decorative artifacts and a common find at archaeological sites. It was also used medicinally in classical times. The oil of amber has a pleasant, musky scent, and is still used today as a perfume. A more solid resinous stage of the aging process of amber provides the resin that dresses the bow hairs of stringed instruments throughout history and through today. Amber can vary in color from a clear light yellow to a deep red. Samples can also have many inclusions, such as air bubbles, insects, and (in some very rare occasions) vertebrates. Many important scientific and anthropological discoveries resulted from the trapping of insects or animals in amber, allowing them to be preserved for modern study. Amber is rarely formed without impurities, either in the form of inclusions or in the form of fractures or stress lines.

The majority of amber samples today are thought to have come from several different source trees, all of which are believed to be extinct today (Beck, 1986). In fact, there are only two types of trees living that produce a resin capable of becoming amber: the Kauri pine and the legume *Hymenaea* (Ross, 1998). Amber forms when the exuded resin of such trees hardens and the volatile components dissipate, allowing the resin to polymerize into copal, which is the intermediate stage of polymerization between “gummy

resin” and amber. The copal is then incorporated into the earth, where it continues to polymerize and release volatile compounds until it is completely inert, at which point the transformation into amber is complete (Ross, 1998).

Amber that we find today was exuded millions of years ago from the early Cretaceous Period (145-65 million years ago) to the Miocene Period (23-5 million years ago) (Thickett, 1995) and from trees located in many regions around modern-day Europe and the Dominican Republic. The trees in different regions were distinct enough to have recognizable characteristics in the resin they exuded, and thus have chemical differences in their amber forms. The identification of amber is difficult, particularly when one tries to define its provenance, which is important for investigating trade routes in ancient settlements. Sophisticated analytical methods have been developed to distinguish Baltic amber, which contains succinic acid, from other types such as Sicilian or Dominican amber and imitations like Bakelite (a thermoplastic). These analytical methods include Raman and Infrared Spectroscopy, which can recognize the characteristic frequencies at which molecules vibrate to identify specific chemical forms in these materials. These methods are described in more detail below.

Establishing the age of amber can be difficult as well. This is usually estimated from the age of the fossils and sediment deposits found within the sample. Unfortunately, the amber may not have originated at the site where it is found and may have ended up there after a series of erosions. In such cases, dating the amber from the surrounding materials is compromised (Ross, 1998). As such, though the age of the resin is important for dating the species of inclusions trapped inside the amber as it hardened, a method has not yet been confirmed reliable to pinpoint the date of formation.

IDENTIFYING AMBER

As a polymerized resin with many components, amber is difficult to identify. Different tests have been developed to address this problem. Some are more traditional and/or invasive and can be performed with common household materials, while others are more technical and require access to sophisticated equipment. As amber contains a complex combination of organic molecules that vary from region to region, it is very difficult to identify amber with

a high degree of confidence using only one method. Typically, a combination of analytical techniques is employed to distinguish between amber and other synthetic natural resins. For example, it is relatively easy to differentiate between synthetics, copal, non-Baltic, and Baltic amber if the analyst can use a combination of analytical techniques.

	Baltic Amber	Non-Baltic Amber/ Fossil Resins	Copal	Bakelite (phenol formaldehyde resin)	Synthetic Plastics
UV test	Fluorescence occurs (pale blue)	No fluorescence	No fluorescence	No fluorescence	No fluorescence
Hot Needle test	Melts slowly, exudes sweet, resinous/pine odor and sooty fumes	Melts slowly, exudes sweet, resinous/pine odor	Melts rapidly, exudes sweet, resinous/pine odor	Does not melt easily, exudes acrid, chemical smell	Does not melt easily, exudes acrid, chemical smell
Static Electricity test	Becomes electrically charged	Becomes electrically charged	Does not become electrically charged	Becomes electrically charged	Does not become electrically charged
Knife test	Sample crumbles or splinters into small fragments	Sample crumbles or splinters into small fragments	Sample crumbles into small fragments	Sample crumbles into larger fragments	More easily cut without fragmenting
Melting point	287-300°C	287-300°C or 260-270°C	90-130°C	Decomposes rather than melting	Decomposes rather than melting
Solvent testing	Insoluble in acetone, soluble (20-25%) in ethanol	Slightly more soluble than Baltic amber	May become tacky in acetone, ether or alcohol	May dissolve in acetone, ether or alcohol	May dissolve in acetone, ether or alcohol
Density/ Specific gravity test (in salt solution of 1.1 g/mL)	1.050-1.096 g/mL floats	1.058-1.068 g/mL floats	1.03-1.08 g/mL floats	1.2-1.4 g/mL floats	varies varies
Infrared Spectroscopy	Broad absorption around 3450 cm ⁻¹ indicating carboxylic acid, shoulder between 1250-1175 cm ⁻¹ and sharp peak between 1160 and 1150 cm ⁻¹	Same results as Baltic amber except for the Baltic amber shoulder between 1250-1175 cm ⁻¹	Differs from amber with bands of varying strengths/shape around 3050, 1650 and 887 cm ⁻¹	Broad absorption around 3450 cm ⁻¹ , doublet around 2900, peak around 1650 and doublet at 1450 cm ⁻¹	Varies widely

Tab. 1 - List of traditional and instrumental methods to identify amber.

TRADITIONAL IDENTIFICATION METHODS

The following list includes techniques that have proved effective in identifying amber. Some of these are more traditional methods and therefore require little in the way of specialized equipment or chemicals. By their very nature, however, these traditional methods are more limited, are highly invasive, and may be unreliable for a positive identification. Depending on the sample, it may be helpful to use either a combination of traditional methods, or to focus specifically on the noninvasive methods that do not damage the artifact. One might argue that many of these could be seen today as outdated and as inappropriate.

1. Ultraviolet (UV) light test (highly invasive): Expose a freshly cut sample to UV light. Baltic amber (succinite) produces visible fluorescence if the sample has been cut recently. Non-Baltic amber, copal, bakelite, and synthetic plastics will not fluoresce. (Mills and White, 1987)
2. Hot needle test (highly invasive): Apply a hot needle to an inconspicuous test area. If the needle enters the sample easily and gives off an acrid odor, the sample is a synthetic substance such as bakelite. If the needle enters easily but gives off a sweet odor, this indicates copal. If the sample offers resistance (the needle is difficult to push into the sample) and gives off a sweet smell, this indicates amber.
3. Static electricity test (noninvasive): Rub the sample with a wool cloth and hold it next to small strips of paper. Amber and bakelite develop a charge and will draw the paper to them; copal and most synthetic imitation amber will not (Beck 1982, Dahlstrom 1996).
4. Knife test (highly invasive): Cut an inconspicuous test area with a sharp tool such as a scalpel. Amber and copal crumble, bakelite will produce larger splinters (Dahlstrom and Bronst, 1996), and plastics should cut more easily.
5. Melting point test (moderately invasive): The expected melting point of amber is 287-300°C (Stout et al. 1995). The melting point of copal is slightly lower, while bakelite is expected to decompose without passing through a liquid phase (Beck, 1982).
6. Solvent tests (moderately invasive): The solvents used

(acetone, diacetone alcohol, turpentine and trichloroethylene) should be chosen based on their known solubility effects on amber and its imitations (Stout et al. 1995, Anon. 1937). For instance, because copal is not fossilized (i.e., completely polymerized and cross linked), it is soluble in acetone, while the others are not (Poinar and Poinar, 1999 p.192). Beck (1982) also suggests the moistening of broken amber surfaces with potassium hydroxide and pressing them together as both a repair method and a means of authenticating amber.

7. Density test (noninvasive): Determine the mass of the sample and the volume of the water it displaces. The density of the sample is its mass divided by its volume. This technique is only truly useful if there are not many inclusions in the sample. The recorded value for the density of amber is 1.058-1.096 g/mL (Stout et al., 1995).

These methods were applied to our amber artifact after discussion with the owner. The UV test indicated Baltic amber. Our sample produced static electricity when rubbed. Solvent tests indicated Baltic amber. Density and specific gravity tests indicated Baltic amber also. None of the destructive tests were undertaken as they seemed subjective given the non-destructive methods available. It was decided to reserve destructive testing for IR.

AMBER IDENTIFICATION BY INSTRUMENTAL METHODS INSTRUMENTAL METHODS TO IDENTIFY AMBER

A number of more sophisticated instrument-based methods have been used to identify amber. These are predominantly based on the use of Fourier Transform Infrared spectroscopy (FTIR) (Poinar and Poinar 1999; Angelini et al 2005; Shashova et al 2006; Guilianoa et al 2007; Zhu and Xing 2008, Teodor et al 2009; Wolfe et al 2009;), although some investigators have also used FTIR in conjunction with Gas Chromatography/Mass Spectrometry (GC/MS) (Virgolici et al 2010; Shedrinsky et al 2004) and Raman spectroscopy (Teodor et al 2010). Advantages of these methods are that they are more accurate, provide more information about the artifact, and are some less invasive and some can be noninvasive. The major disadvantage is that they usually require much more expensive equipment, involve complex data analysis, and are typically inaccessible to many researchers. For those who have access to such tools, however, these methods are superior and preferable. So far, these techniques can distinguish amber from synthetic imitations, and can differentiate Baltic amber from amber of other origins. A variety of claims of origin have been made, especially regarding the use of X-ray diffraction (Frondele, 1968). Though more recently, IR techniques were shown to identify the provenance of the amber beyond the basic Baltic/Non-Baltic distinction (Shashoua, et al. 2006).

FTIR: Grind a small (~50 micrograms) amount of sample, mix with KBr to prepare a pellet, and record an IR spectrum of the sample in the region of 4000-400 cm⁻¹. The most relevant area is between 1300 and 800 cm⁻¹. Baltic amber has a characteristic horizontal shelf or shoulder in the IR spectrum between 1250 and 1175 cm⁻¹, followed by a sharp peak between 1160 and 1150 cm⁻¹ (Beck, et al. 1965 : 103). This peak has been associated with the spectrum of diethyl succinate, the ester of succinic acid (Beck and Langenheim 1965 : 52-3). While succinic acid is found in more than just Baltic amber and can be the result of an aging process (Rottlander, 1979b in Beck 1986), Baltic amber will always contain succinic acid (Beck 1986 p.74). For this reason, FTIR is not effective at differentiating between different types of non-Baltic amber except in the context constructed by Shashoua, et al., 2006.

FTIR is currently the most accurate and cost-effective method for identifying amber. FTIR instruments are fairly accessible (standard equipment in most academic chemistry departments), and Beck and his colleagues at the Amber Research Institute have provided numerous reference IR spectra of different types of amber (Personal Communications). However, FTIR methods are not foolproof and early work was often confused by the fact that impurities can interfere with the spectrum and produce biased results (Beck et al. 1965: 108). Improvement of FTIR techniques has reduced some problems that plagued results in the 60s and 1970s. (Teodor, et al., 2009). Amber fakes, especially those where the amber is true and the inclusion is false, will not show up as false based solely on FTIR results.

Conservation treatments performed on amber to prevent degradation have been particularly detrimental to future identification (Beck 1982: 106). Amber is vulnerable to environmental decay such as oxidation, the exposure of the sample to oxygen and light (Beck 1982; Beck et al. 1971). In the past, methods used to prevent this process, such as applying a coat of wax or synthetic resin, were invasive and contaminated the sample. These “signatures” of preservation methods show up in IR spectra and often complicate the identification of amber (Beck et al. 1971: 236). Currently, the preservation of amber today involves creating a non-oxidizing environment, with a consistent temperature and relative humidity, and minimal exposure to light. Treatments have included having samples immersed in water with various preservatives (Beck 1982: 105). More recently, they have been embedded in bio-plastics (Poinar and Poinar 1999: 191) or paraffin, which do not absorb in the crucial Baltic amber region (Beck 1982 : 106). Amber oil has been used to produce possible fakes to fool instrumentation as well as restoration effects (Beck, 1982).

THE AMBER NECKLACE: ITS TREATMENT

The amber necklace (Figure 1) contained a number of individual pieces of amber that were rounded, varying greatly in size and shape, and had been bored into to create a passage for stringing. The amber necklace (Figure 1) contained a number of individual pieces of amber that had these holes for stringing. They uniformly displayed a haze or cloudy nature on the surface. This was presented to us as the result of an earlier attempt to clean dirt off the surface. Some minor scratches appeared on the individual pieces and obvious areas of wear or abrasion.

Materials and Methods

Treating the sample in question required first authenticating and proving its general origin. With respect to the sample's integrity, a small amount of amber material was taken for FTIR analysis causing as little damage as possible to a discreet area of the necklace, from the area near the hole for stringing. This sample was ground down using an agate mortar and pestle. Several samples were prepared using both KBr pellet and nujol mull sample preparation methods in order to obtain a useful IR spectra. The analysis was performed on an Nicolet model DXB FTIR instrument running OMNIC software in the Department of Chemistry and Biochemistry at San Francisco State University.

RESULTS

After comparing the samples prepared with KBr pellet and nujol mull, a good IR spectrum was obtained using a KBr pellet. The sample was identified positively as Baltic amber. As the literature predicted, identification was confirmed by the broad absorption around 3450 cm^{-1} indicating the presence of carboxylic acid of succinic acid (Chart1).



THE PROOF OF GENERALORIGIN

was provided by the “Baltic shelf” between 1250-1175 cm^{-1} , followed by a sharp peak between 1160 and 1150 cm^{-1} . The IR spectrum also shows the presence of both water and carbon dioxide, as evidenced by H₂O peaks above 3600 cm^{-1} and between 1500 and 1600 cm^{-1} , and CO₂ peaks around 2350 cm^{-1} . These substances are present in the air background of the sample cell of the FTIR instrument, and did not adversely effect the positive identification of the sample as Baltic amber.

IR spectrum of suspected amber sample in the form of a KBr pellet. Note the broad absorption around 3450 cm^{-1} indicating the presence of the carboxylic acid of succinic acid..

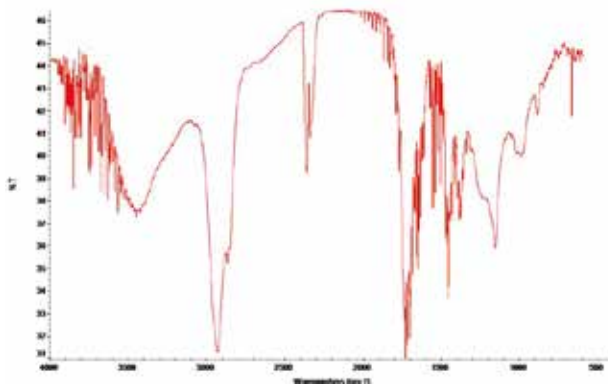
CONSERVATION TREATMENT

Mitigating the opaque bloom of the sample proved difficult to research. The literature available on the treatment of amber is very limited, and most available articles do not discuss thorough treatment options, but rather are limited to housing, consolidation and documentation of amber samples. Thickett and his colleagues wrote a comprehensive article on amber conservation that focuses on consolidation and investigates the properties of various consolidants, including age testing, solubility, and interference with FTIR (Thickett 1995). They also discuss retreatability of materials in consideration of analysis such as IR. The success of treatment and retreatment depends on the durability of a treatment and materials used need to be monitored over time (Caldararo, 1997). In the case of our treatment, the owner reports no change.

Solvent testing was performed on the sample to the end of developing a cleaning methodology, using acetone, diethyl ether and ethanol (Stout, et al., 1995). These solvents had little mitigating effect on the blooming issue. After a suggestion from amber scholar Curt Beck, a dilute solution of 1% hydrochloric acid was tested and proved effective and safe to the amber. This method was used to resolve the blooming using a cotton swab under magnification.

CONCLUSION

The sample in question has been confirmed by FTIR as Baltic Amber. The Amber Research Institute and Curt Beck have provided very helpful references for IR spectra of amber and related materials against which, an expected and confirmed identity of the sample were measured. However, if the sample had been any other type of amber besides Baltic, it would have proved more difficult to narrow the results. Now that FTIR is more available to researchers, more testing of amber and related materials such as thermoplastics and copal will only provide researchers with better tools and information. Furthermore, the availability of FTIR technology should allow researchers to avoid older



IR spectrum of suspected amber sample in the form of a KBr pellet. Note the broad absorption around 3450 cm^{-1} indicating the presence of the carboxylic acid of succinic acid.

and more invasive testing methods. Figure 2 Shows the “after” condition of the segments of the necklace.

The central purpose of this paper is to report the results of evaluations of methods in the literature for addressing problems with the appearance of amber. The ability of conservators to extract information from earlier studies, both in terms of treatments (given the variation of material treated and reagents used and operator skill) can be useful in treatment outcome. Comparison of numerous results of samples provides a background to understand amber variation and may help establish a foundation for distinguishing not only amber origin in geographic terms, but also variation in aging, inclusion effects and composition. The means of using the literature for designing treatments has been discussed in regards to other objects (Caldararo, 2008). Such evaluations should become more common so that practitioners can be confident that current methods are not unique outcomes but reflect the body of knowledge of the field.

ABSTRACT

This study describes the identification and treatment of an amber necklace, which came into the conservation lab of Conservation Art Service, with an opaque bloom caused by a previous cleaning with a household ammonia cleanser. There was no information given its origin available from the owner. Prior to the conservation intervention, it is accepted practice to identify correctly the material the beads were made of and, depending on the results, research the past treatment of the amber to identify the most appropriate means to remove the opaque bloom. A review of the literature of both the amber conservation treatments and identification of false amber indicated little useful published work on these topics in conservation journals, particularly in the form of a single, comprehensive review. Treatment and retreatment issues have become a significant subject in conservation in the past two decades (Caldararo, 1997; Caple, 2000). The goals of treatment or “no treatment,” must be clearly discovered and developed in each case. This prompted us to assemble a list of relevant articles on various methods for testing and identifying amber. This paper also includes an overview of amber and its historical use, methods to definitively identify amber, and the identification and treatment of this particular object using infrared spectroscopy.

KEYWORDS

CONSERVATION; FTIR; AMBER

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