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THERMOLUMINESCENCE DATING FOR EUROPEAN SCULPTURE: A CONSUMER'S GUIDE

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Abstract

Thermoluminescence (TL) dating is one of many tools used for the authentication and technical study of works of art within the Department of Decorative Arts and Sculpture at the J. Paul Getty Museum. This paper considers the lessons learned through attempts to use the TL technique to date seventy-one European objects attributed to the Renaissance through the 18th century, including: glazed and unglazed terracotta sculpture, faience, majolica, and glazed earthenware, as well as clay-based and plaster-based bronze casting cores. When considering whether or not TL dating should be attempted for a work of art, issues such as material type, object history, sample size, and error limits should be taken into consideration. Examples of European materials that are difficult to date using the technique are given. Recommendations are made for safely taking and handling samples, and for choosing a TL lab. The results have been categorized and illustrate that in certain circumstances, TL dating has proven to be a pivotal aspect of our authenticity studies. Optically Stimulated Luminescence (OSL), a related technique that shows promise for greater precision, is briefly described. In summary, we have found that when approached with deliberation and caution, thermoluminescence dating can be a very useful addition to a broad-based sculpture study.

Introduction

The J. Paul Getty Museum began acquiring Renaissance and later European Sculpture in 1982. Since that time, authenticity studies have been an integral part of every purchase. Although archival, art historical, and provenance research have always been the backbone of these studies, over the years technical investigations have played an increasingly important role in acquisitions. Technical investigations are approached from many directions, including evaluation of whether or not the methods and materials used to construct a work of art under consideration are consistent with the attribution. When the investigator is lucky, reliable period treatises on technique are available for comparison. Alternatively, it is sometimes possible to compare the work we are studying to technical data from other, well-attributed works. Quite often though, little such comparative material is available. For this reason, direct dating methods such radiocarbon dating, dendrochronology, and thermoluminescence (TL) dating can be an important tools in authentication studies.

The author has had the opportunity to use the technique, both for authenticity studies for acquisitions, and for general technical studies of sculpture in both the Getty and other collections. In the last twenty-two years, conservators at the Getty have sampled seventy-one objects for TL dating. The Getty does not have in-house facilities for the technique; over the years samples have been sent to seven different labs located in Germany, England, Italy, Denmark, and the US. TL dating is used for objects that contain clay that has been heated during manufacture, for glazed and unglazed terracotta sculpture, faience, majolica, and glazed earthenware, as well as clay-based and as plaster-based bronze casting cores. Porcelain can also be TL dated (Stoneham 1983), but the Getty has never attempted it due to the large size of the core bit needed for sampling (at least 1/8" outer diameter).

TL dating has a mixed reputation in the art historical community; it is relied upon without hesitation in some quarters, yet completely discounted in others. The Getty has taken the middle road, approaching it with full knowledge of its strengths and possible weaknesses. Because of the complexity of the technique, the fact that it is not done in-house, and the important roll the results sometimes play in the assessment of objects, care has been taken to study the fine details of the methods and procedures. The following is a summary of what has been learned about when and how to most effectively use the technique, including how to anticipate, and sometimes avoid, problems.

The Thermoluminescence Dating Technique

The phenomena of thermoluminescence – in which a material gives off light when it is heated – has been known for centuries. It was used primarily as a method for mineral identification until the invention of the photomultiplier, which allowed measurement of very small amounts of light. In the 1950's, thermoluminescence was first suggested as a method for dating geological and cultural materials; methods for applying the technique to archaeological materials were developed in the 1960's (Aiken 1985).

TL dating of clay-containing materials is possible because crystalline components contained in clay absorb energy over time. The amount of energy that is stored is relative to the number of years since the clay was last fired. The energy that is stored comes from many sources, primarily radioactive elements that naturally occur in the clay (potassium-40, thorium, and uranium at concentrations of a few parts per million). As these radioactive elements in the clay decay, they release energy that is absorbed and stored by neighboring crystalline materials, primarily quartz and feldspar. By the time clay is mined to make a ceramic object, the crystalline minerals in the clay have stored a significant amount of energy. When the ceramic object is fired in the kiln, all of this stored energy is released. From this so-called zeroing-out point, the clay starts absorbing energy again. When a sample of the ceramic is removed for TL dating, the sample is heated, this time in the laboratory, and the stored energy, released in the form of light, can be measured with a photomultiplier. The relationship is linear: the more energy that is stored, the more light that is emitted. With calculations, the amount of light released will indicate how many years have passed since the ceramic was made, and therefore the date it was fired, as shown in the equation in Figure 1:

$$\text{Age} = \frac{\text{Total amount of energy stored since firing or casting}}{\text{Amount of energy stored each year}}$$

Figure 1. Equation showing the relationship between light emission and the age of the ceramic.

Although the *basic* concept of this simplified model is straight forward, in reality many factors complicate the calculations, making the technique particularly labor intensive and therefore costly. This is one reason why, in archaeological settings in which a variety of materials are available for analysis, radiocarbon dating (carbon-14) has often been preferred over TL dating. C-14 dating of the specific materials under consideration in our department is often impractical however, due to the absence of carbon.

Error Limits

Approximately twelve components are included in the calculations undertaken in TL dating. Each of these components contains uncertainties that combine to determine the error limit of the results. The errors have many sources, including random errors due to imprecision of the instruments, and systematic errors that arise when the data is taken from calibration curves, rather than measured directly. The laboratory results are reported to us as a date plus or minus a range of years. This ± value reflects the error limits or accuracy of the date. As stated by Aitken (1989, 156) and observed in the results that we have received over the years, "…in most cases it is possible to determine a TL age to an accuracy of around ± 20%". For example, a faience plaque attributed to the Alcora Factory, circa 1755, was sampled for TL dating as part of a pre-acquisition authenticity study undertaken in 1998 (Fig. 2).

Figure 2. Alcora Ceramic Factory, *Plaque Depicting Jacob Choosing Rachel to be his Bride* The J. Paul Getty Museum, Los Angeles, 99.DE.10

The plaque received a TL date of 1783 ± 22 years (1761-1805). This date indicates that the plaque had been fired 215 years previously, give or take 22 years - a degree of uncertainty of ±10% of the overall age. This date range falls outside of the 1755 attribution. As is standard practice for many labs though, the TL date was reported with one standard deviation, indicating there is a 67% likelihood that the plaque was fired between 1761 and 1805. To increase the likelihood that the actual date falls within the reported date range, the results can be read with two standard deviations. To do this, the plus-or-minus uncertainty must be doubled – in this case plus-or-minus 44 years. By increasing the date range to 1739-1852, there is a 95.4% chance that the date of facture falls within this range. Indeed, in this example, the attribution of 1755 does correspond to the TL date at two standard deviations. This example has been given to make two points: first that it should be clarified with the lab how their results are reported, and secondly that the results cannot be simply accepted as the final word without scrutiny and, at times, interpretation.

Problematic Materials

Certain types of European sculptural materials can be difficult or impossible to date using TL. Light colored French 18th C terracottas are well known to cause problems. This may be due to the very fine grain of the pastes and/or the high calcite content of the clay (Goedicke 2000). We have encountered problems with pieces by Clodion (Claude Michel, French 1738 - 1814) as well as Philippe-Laurent Roland (French, ca 1780 – 1790) (Fig. 3).

Figure 3. This work is one example of a light-colored 18th century French terracotta that is unsuitable for thermoluminescence dating. Philippe-Laurent Roland, *Allegorical Group with the Bust of an Architect* (The J. Paul Getty Museum, Los Angeles, 87.SC.9).

In addition, the light-colored paste of the 16th century lead-glazed earthenware by Bernard Palissy is not suitable for TL dating approximately 50% of the time (Stoneham 1987), as encountered with this Palissy plate in the Getty collections (Fig. 4).

Figure 4. Lead-glazed earthenware by the French 16th C. artist Palissy is often undatable using TL. Bernard Palissy, *Oval Basin* (The J. Paul Getty Museum, Los Angeles, 88.DE.63).

Although the Getty has had considerable success in dating clay-based casting cores (Bassett 2008), many European lost wax bronze casts were made using *plaster*-based cores. As plaster alone cannot withstand the heat of casting, temper is added to the core material. Clay and sand added to the plaster as temper often allow TL dating of the bronzes but occasionally problems are encountered. Dating of ten different bronzes with plaster cores has been attempted; of these, dates were not achieved with three of them. It may be that plaster cores can be difficult to date due to the occasional presence of thermoluminescent crystalline forms of gypsum, and to the fact that standard TL calculations are based on a clay, not plaster, matrix which affects the amount of radioactive materials in contact with the sample during its history (Goedicke 2000).

Incomplete heating during manufacture can also be the cause of inaccurate results. If the sculpture was never fired or was not heated high enough for a long enough time when it was being made, there may be no zeroing out of the geologic energy dose. The time and temperature necessary to completely zero out a dose will vary according to the size of the object. When such an object is dated, the resulting age will be far too early [1].

Another problem is that objects that have been radiographed will not receive an accurate TL date. X-ray radiation will alter the stored TL dose, increasing the TL age [2]. Although attempts have been made to calculate how much affect a single exposure will have, estimates vary from a single exposure adding five years to the TL date of an object, all the way up to a single exposure adding 50 or 100 years to the age. As x-ray intensity falls off markedly over distance, such factors as the distance from the center of the beam to the sample location, and the amount of attenuation of the beam within the object will affect the amount of radiation absorbed by the sample, greatly complicating an accurate estimate of the date shift due to radiography. Regardless the amount of alteration of the dose, the effect of radiography on early archaeological material will be far less than the effects on younger post-Renaissance material. For this reason, we always sample before an object is radiographed. Because it is occasionally necessary to remove a second sample during the dating process, it is ideal to postpone radiography until a firm date has been achieved. In some instances, during authentication studies for acquisition, objects not yet owned by the museum are not radiographed, even with a firm TL date in hand. Should the museum decide at the last minute not to go through with a purchase, the X-rays will alter any TL dating results attempted by a future owner. Although the results of all studies should stay with the object, once it has left the museum there is no guarantee that this will occur. This is of course, an ideal, as radiography may be an important contingency for acquisition, but it acts as an illustration of the care that should be taken in undertaking such studies.

If an object has been exposed to heat some time after its creation, a sample from the object will yield a TL date of the reheating rather than of its original date of manufacture. The degree to which the accumulated TL dose is damaged through later heating will vary according to the temperature and the exposure time, in general terms the stored TL energy will be damaged at temperatures above approximately 350 degrees Celsius (Fleming 1971). The surface must be carefully examined for signs of exposure to heat in the years after it was first fired, including the object having been in a fire, or restoration steps such as refiring of ceramics, solder repairs, or bronze repatination. Incorrect dates of manufacture will also occur in the case of unfired clay sculpture such as *bozzetti* [preliminary sketches] which have been fired decades or centuries later in order to preserve them.

Sampling for TL Dating

It is essential that specific procedures be followed when sampling for TL dating, both to cause as little alteration as possible to the object, and to ensure that the sample is not damaged or contaminated as it is being removed. The surface, the structure, and the overall condition of the work of art must be well understood. The location must be carefully chosen and documented. The sample site is chosen with many points in mind, and it should be as unobtrusive as possible. Even when drilling on the reverse or bottom of an object, surface features should be avoided such as finger or tool marks that may suggest how the surface was worked. At times, minor surface damages such as old chips or losses may provide an ideal location for drilling. However, it is important to understand the structure of the object, in order to avoid drilling in unstable areas, and to avoid contaminating the sample with restoration or mounting materials. Although samples are often taken from the bottom or reverse of an object, both terracotta sculpture and bronze casts are often hollow, sometimes allowing sampling from the interior.

The sample site is photographed before drilling. Written documentation includes the sample location, the rational for sampling, the drilling technique, type of drill bit, size of sample, date, and person taking the sample. Because bright light will damage the TL signal, drilling should be undertaken in a room that can be darkened completely, using a safelight such as a Kodak lamp with a 6B filter made for darkroom use (Fig. 5).

Figure 5. Sampling for TL dating must be undertaken in the dark using a safelight.

Samples can be removed as chunks (only practical for core material) or they can be removed with a drill. Tungsten-carbide bits should be used; diamond drill bits must be strictly avoided as diamonds may contaminate the sample and are highly thermoluminescent. The sample size for TL dating is quite large; at least 100 mg are needed, sometimes more. To limit the size of the entry hole, a narrow, deep hole is often preferred. A typical 100 mg sample will result in a hole that is approximately 0.25 cm wide x 1 cm deep. A test can be run on a non-artifact fired clay material (such as a flower pot), to determine approximately how deep the hole will have to be in order to get the required amount of sample for the diameter of drill bit being used. The depth of the hole can then be measured and tape placed on the sampling bit to help indicate when the appropriate depth has been reached. Although a cordless power drill offers the most control when removing a sample, the smaller size of a hand-held rotary tool (Dremel) is sometimes preferable. Two other tools have been particularly helpful in taking samples from the interior of sculptures with restricted access. When there is just enough room for a hand to pass into the inside of the sculpture, a pin vice has proven useful (Fig. 6).

Figure 6. A tungsten bit held in a pin vice can be used to hand drill the inside of a hollow terracotta.

A second tool has been used in two instances to remove core material from inside of bronzes with severely restricted access. The tool consists of a hollow tube with rough teeth cut into the end. The relatively soft core is cut by the teeth. The sample remains inside of the hollow tube and is trapped by a solid rod attached to the bottom end (Fig. 7). Alternating sections of hollow and solid rod can be attached to one another, extending the length of the sampling tool. Using this system, it has been possible to remove core samples remaining in small pockets at the top of nearly life-sized figures through small access holes in the feet.

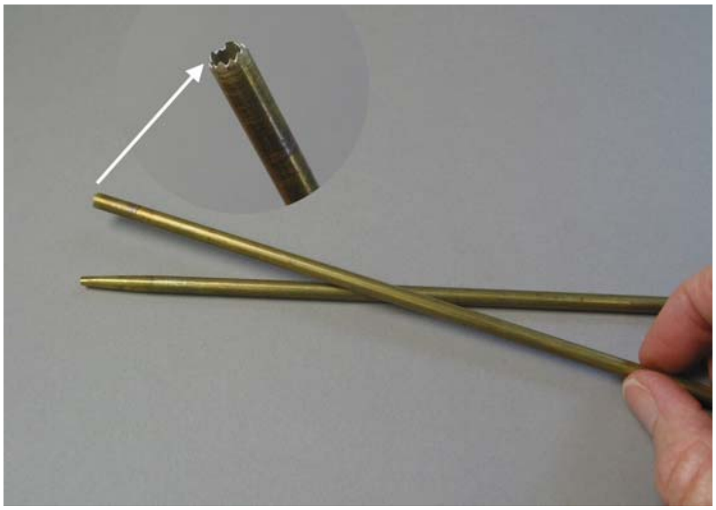

Figure 7. A sampling tube can be used to remove core material from bronzes with very limited access to the interior.

Light-tight sample vials are then prepared. Glass bottles with snap-top lids are covered with foil and slipped into plastic film canisters to ensure that they are protected from the light (Fig. 8). The empty vial and canister are then weighed.

Figure 8. Light-tight sample vials are labeled and weighed before drilling begins.

The work of art must be secured in the dark room at an angle such that it can be safely drilled and the sample contained. Catching the sample may be very straight-forward or quite tricky. Folded weighing paper is most often used to catch the samples. When working in a restricted area, “envelopes” can be carefully configured so that they will stay taped in place deep inside of a cavity during drilling, yet can be removed when needed without spilling the sample (Fig. 9). When the layout is tricky, a practice run for sample removal, with the lights still on, is recommended.

Figure 9. A folded envelope attached inside a hollow sculpture will catch the sample as it is drilled.

The surface that is first removed when drilling is contaminated by light exposure and must be discarded. Working under safelight, the light-exposed surface is pre-drilled to a depth of approximately 2 mm, using a bit of a slightly larger diameter than the one used for sampling. This pre-drilled material is then set aside for elemental or other analysis. The larger pre-drilled hole gives some extra room to keep light-exposed material out of the sample, should there be minor chip-outs or should the bit wander a little at the entrance hole.

The object is then drilled to the depth marked on the bit. The removed powder is placed in the vial, placed in the light-tight film canister, then weighed.

Once the desired sample weight is achieved, the canister is placed in a fully addressed inner envelope marked *DO NOT OPEN IN THE LIGHT*. This inner envelope is then placed in an outer rigid shipping box that is similarly labeled. To date, samples have been shipped to the lab using DHL. It is likely that x-ray screening of packages would affect the samples, but as yet there is no indication that DHL [3].

Choosing a TL Lab

The choice of which lab to use is an important one. Many factors should be considered.

1. *Communication*: It is important to work with a lab that welcomes inquiries, including questions regarding the procedures, questions about sampling (particularly problematic pieces), and – if necessary – further explanation of the results.

2. *Experience*: Only a small number of labs offer the technique. The lab must have experience with *authenticity dating of artifacts*, rather than geologic dating. It is ideal if the lab has experience with the general time frame and the type of object under investigation.

3. *Sample size*: For some objects, sample size is of primary importance. Each lab has a sample size that they require to achieve a result, which can range from 100 mg all the way up to 1 gram. Although some of the labs carry out more steps with a larger sample, resulting in a more precise date, this is not always the case.

4. *Available techniques*: TL dating involves numerous steps that vary according to the characteristics of each sample. Two steps that can be useful for European sculpture -- *thermal pre-treatment* for certain types of clay behavior and the *pre-dose technique*, a method for measuring the amount of energy stored over time that is particularly useful for recent objects such as those in consideration in our laboratory – are both particularly time consuming techniques. For this reason, not all TL labs offer these steps; labs that offer both are preferred [4].

5. *Turn-around time*: It has taken anywhere from two weeks to twelve months to get a single result. This is not always an issue in choosing a lab, but it can be very important for acquisition studies.

TL Results and Discussion

Figure 10 shows the distribution of the eighty-two results from seventy-one objects that the Getty has sampled for TL dating (as explained below multiple samples were taken of some objects). When the TL dates are compared to the curatorial attributions, the results received over the years fall into four categories. Forty-eight of the results - the vast majority - correspond to the curatorial attribution. Of the eighty-two total results, five samples received TL dates that were even older than the attribution dates, and fourteen samples received dates that were more recent than the attribution. Over 20% of the samples (fifteen of them) received no date using the technique. The fifteen samples that could not be dated fall into three categories:

1) Problems with the samples occurred seven times (either the sample was too small, the lab refused to date it as the object had been radiographed, or, as occurred most often, the sample yielded a “geologic dose”. The latter results indicate either insufficient heating at the “zeroing out” point when the piece was manufactured, or that the sample was taken of material other than the original fabric).

2) In six instances, the lab reported that the material of which the samples were composed was not suitable for TL dating, including light-colored French clays and plaster casting cores.

3) In two examples, the lab never responded with a result.

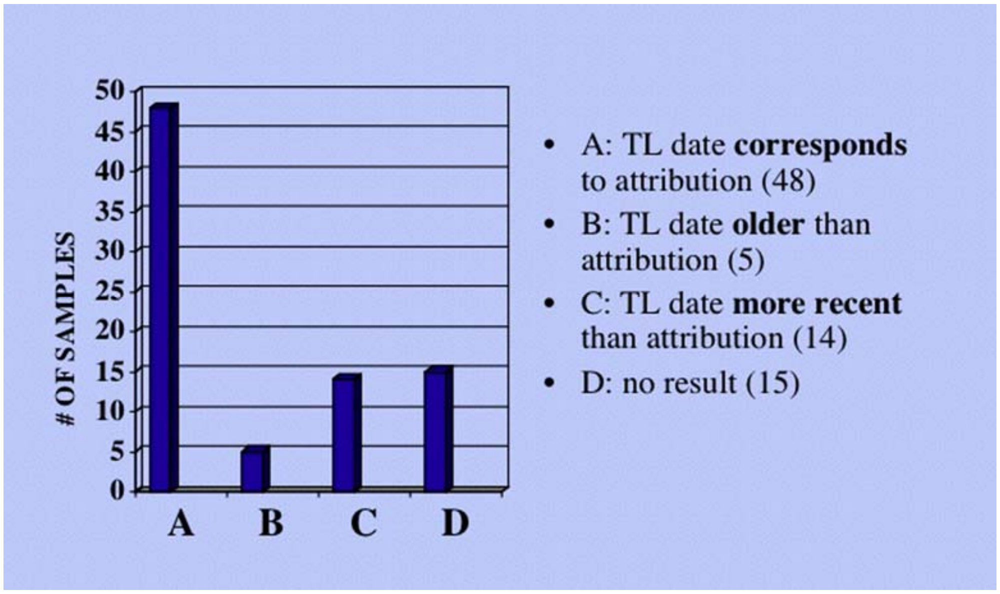

Figure 10. TL results compared to attribution. Decorative Arts and Sculpture Conservation, JPGM, 1985-2006.

The numbers in Figure 10 total more than seventy-one, as occasionally more than one sample was taken from an object. Twice initial attempts were made to achieve a date using smaller samples than the lab requested; both times it was necessary to go back into the holes to drill more material in order to get a result. It is now clear that either a full sample must be taken, or dating should not be attempted. In six other instances that were considered particularly important or difficult acquisitions, samples were sent to more than one lab. In all six instances, the results were in agreement with one another. In one of the six examples, a potential acquisition arrived in the conservation lab with results from an earlier TL test that corresponded with the Renaissance attribution. This earlier TL work had been done by a lab we did not know. To be careful, we sent further samples to our trusted labs, all of which came in with a 20th century date, and the acquisition was declined.

Figure 11 illustrates the end result for the thirty-two *potential acquisitions* that have been dated using TL. Of the thirty-two results, nineteen objects received TL dates that correspond with the attribution. Of these, the vast majority of objects were acquired (sixteen) and three were not, even though they received the anticipated TL date. When the TL date was older than the attribution, the results were mixed: three objects were purchased and one was not. When no TL result was possible, the majority of the pieces were purchased regardless, without confirmation of the date by TL. These three categories - a) date corresponding to the Curatorial attribution, b) dates older than the attribution and d) no date received - indirectly illustrate that most often TL dating is only one of many factors taken into consideration for acquisition. In category c), however, in which the TL date came in more *recent* than the attribution, *all* of the acquisitions were declined, suggesting that in these instances, TL dating has proven to be a pivotal aspect of our authenticity studies.

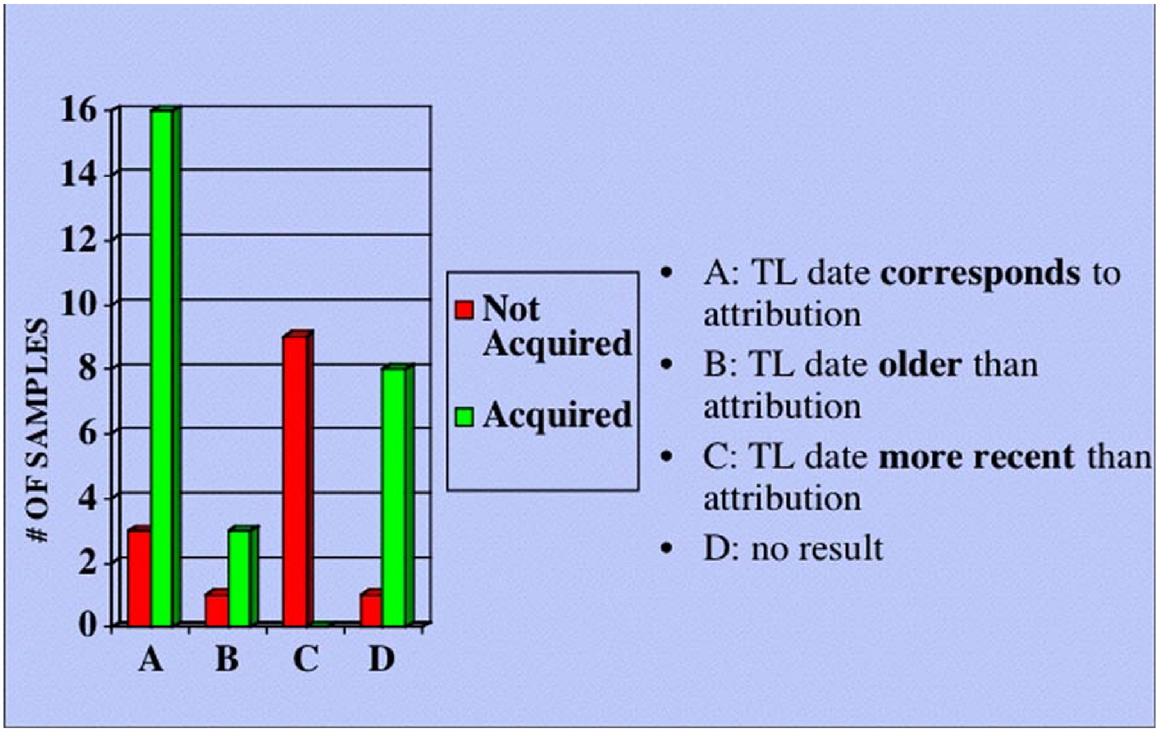

Figure 11. Potential acquisitions: TL dated sculptures, JPGM, 1985-2006.

Optically Stimulated Luminescence

A technique related to TL shows great promise for authenticity dating. Referred to as Optically Stimulated Luminescence (or OSL), it offers many advantages over TL. OSL is similar to TL except that the stored energy from the decay of radioactive elements is released by exposure to light, rather than heat. Given the same sample size as that used for TL, the technique offers greater accuracy and less error. In addition, it is more reliable than TL for dating very low-fired materials, and can be used for dating unfired materials such as mortar, stucco, gesso, and earthen walls (Feathers 1995). At least two labs are working on the adaptation of the technique to the dating of artifact materials; hopefully their research will prove fruitful.

Summary

The advantages of the thermoluminescence dating technique are significant:

1. It is one of only a few direct dating methods available for cultural materials.

2. There are independent laboratories that will test single samples, some of which have a considerable amount of experience in authenticity dating of artifacts.

Yet the potential problems with the technique are not insignificant:

1. A large sample is needed (at least 100 mg).

2. Relatively high error limits (generally around 20%).

3. Some clays are not suitable for TL dating. Lack of suitability cannot always be predicted beforehand.

4. The technique involves many steps and is therefore expensive.

5. The sample must be taken and handled with care as the TL signal can be damaged with exposure to light, heat, and ionizing radiation (such as x-rays).

One of the most important steps when considering TL dating is to identify the questions it is hoped to answer. Due to error limits, this may be particularly important for Renaissance and later material. For instance, when TL is used for authentication, it is important to know when copies or outright forgeries would have been made to determine if there is enough time between authentic and copy to get a clear answer. Distinguishing a late-17th century original from a mid-19th century reproduction should be possible by TL. Separating the work of father from that of the son is not.

In closing, the Getty has found that when approached with deliberation and caution, thermoluminescence dating can be a very useful addition to a broad-based sculpture study. It is considered an important tool in our authentication and technical studies, and the museum looks forward to the further development of OSL as a dating option.

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This paper is dedicated to Christian Goedicke who recently retired after thirty-two years in the research labs at the Rathgen Forschungslabor in Berlin, specializing in thermoluminescence dating of cultural materials. Over the last thirteen years, Dr. Goedicke has generously explained different aspects of the technique and results, patiently offering invaluable support and advice. Victor Bortolot and Doreen Stoneham have also kindly offered their time and advice. The author would also like to thank the many staff and interns of Decorative Arts and Sculpture Conservation at the Getty who over the years who have sat in the dark puzzling over how to balance the need to do no harm with the need for information. Abby Hykin and Julie Wolfe have contributed greatly to the sampling procedure. The author would also like to thank Brian Considine, Arlen Heginbotham, Katrina Posner, and Julie Wolfe for their advice in preparing the original presentation, and Brian Considine for his keen editing as well as his continued encouragement of this research.

Endnotes

1. Theoretically, a *plateau test* undertaken during TL dating should indicate whether or not an object's geologic dose was fully "zeroed out" at the time it was made (Aitken 1989, p.150).

2. There is increasing concern that artifacts may be intentionally exposed to x-rays in order to artificially create a false TL date. It may be possible to determine that an object has been exposed to x-rays by examining the behavior of the TL curves. See, for example, Lo 2004; 24.

3. An investigation of the effects of x-rays from airport security scanning has shown a measurable influence on the TL signal of quartz included in slag matrix (Haustein et al. 2003). The author would like to thank Mark Rasmussen for directing her attention to this article.

4. Thermal pre-treatment and the pre-dose technique are succinctly described in Aiken 1989, p. 155 – 156.

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