Dating Uncertainties with Thermoluminescence

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Abstract

Thermoluminescence is the emission of light from a solid material that has received a dose from radiation. This phenomenon can be used to date pottery and volcanic eruptions, since the luminescence increases as it is irradiated with time to the point of saturation. The amount of radioactivity in the vicinity of the sample has to be measured, which can be one of the potential sources of error, since any change in the radioactivity of the past can affect the proposed date. The date obtained is dependent on the environment of the site where the sample is located. Other characteristics of the thermoluminescence process can affect the date obtained. The mechanism of thermoluminescence will be described using an alkali halide, LiF:Mg,Ti, as an example of potential sources of error. Alkali halides have been studied extensively for their use as a dosimeter in the medical radiation field. Careful control of the thermoluminescence can result in good accuracy. The thermoluminescence of quartz will be discussed. The criteria and assumptions necessary for the dating process and the potential problems will be described. The uncertainty of the process will be explained and how it may affect the date. Thermoluminescent measurements, when all uncertainties or the process are accounted for, show dates less than 6000 years. A review of the thermoluminescence process shows that this is a useful area of research for the creation scientist.

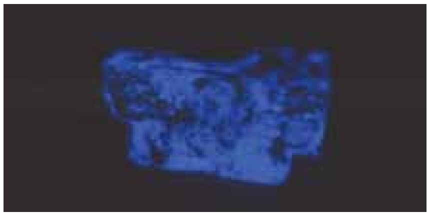

Figure 1. Sample of Limestone Quartz. A *(above)*. Limestone quartz under room light. B *(below)*. Thermoluminescence of same piece of Limestone quartz in darkened room.

Introduction

An introduction and a review of thermoluminescent dating processes, especially for dating of archeological artifacts, are discussed. A brief note of thermoluminescence (TL) was given by Vernon Cupps (2016). Neanderthal and Cro-Magnon humans have pottery artifacts that can be dated to recent times. The determination of accurate dates is de-

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pendent on a number of characteristics of the luminescent material. Generally, variation in processes can be estimated by the use of uncertainty calculations. In the 1990s, the old method of systematic and random errors was replaced by the calculation of uncertainty. These calculations assign parameters for the entire experimental process (Taylor and Kuyatt, 1994). This process will be explained later. Examples of parameters in the TL process that need mention will be illustrated by the use of material, LiF, used for medical dosimetry. Assumptions made for the measurement and the accuracy of the process should be stated. The present manner of doing this estimate to the level of accuracy is called an uncertainty analysis. An explanation of the determination of uncertainty will be given with its application to archeological dating.

Brief History of Thermoluminescence

A brief and incomplete history illustrates that thermoluminescence (TL) has been used in many areas. Sir Robert Boyle was one of the first to observe TL and reported on it in 1663. Madam Curie used TL to measure her radium extractions in approximately 1902. TL was used for dating rock minerals in the 1950s and 1960s, but accuracy for this methodology was not very good because of the environmental changes (mostly in the radiation delivered) that may have occurred for the rock or artifact. This change occurs when the rock is displaced by some external force. In addition, the TL can reach saturation in response to an increase in radiation, which would cause an error in dating. Saturation for the TL process occurs when the signal does not change with increased dosage. Many times, increased dosage beyond the saturation level actually causes damage and thus, a decrease in signal with increasing dosage. The radioactive environment can be measured in a number of ways, e.g. a survey meter, TLD, a spectrometer, etc. Most importantly, one is to measure the in-situ dose rate.

This manuscript will concentrate on quartz dating in archeological artifacts. Farrington Daniels explored dating in 1953 (Daniels et al., 1953). At the University of Oxford, England, Martin Aitken (1985) developed the thermoluminescence dating of archaeological artifacts. Research in this area turned to Optically Stimulated Luminescence (OSL) in latter years. OSL and TL are related processes (see below). TL gained great use in the area of medical dosimetry (measurement of radiation dose using TL). One of the differences between medical dosimetry and dating is the difference in total dosage delivered at the point of measurement. Medical dosimetry involves doses of a few gray each day for 5 weeks, whereas dating usually involves a total dose of thousands of gray, albeit delivered over a greater timespan. The higher dosage can result in saturation of the TL signal. Farrington Daniels, University of Wisconsin (1950s), proposed the use of TL in medical dosimetry, which was extended by John Cameron (Cameron et al., 1968). The author entered the field with Cameron in 1963. A large amount of research has been done in the medical dosimetry field and will be used as an example of consideration for TL in quartz. There have been many books written on TL; one of the first was by Cameron (Cameron et al., 1968). The most recent book is by Chen and Pagonis (2019).

Luminescence

TL is one aspect of the luminescence process, including fluorescence and phosphorescence. The time between irradiation and emission is the distinguishing parameter. Times of $< 10^{-8}$ s are termed *fluorescence*; times between irradiation and emission that are longer are termed *phosphorescence*. TL might be considered to be a "frozen in" phosphorescence. When the luminescence is stored in the phosphor to be read out later, it is termed *xluminescence*, where x is the method of read out (e.g. heating the sample gives rise to TL). TL occurs when heat is applied in a readout uniform fashion which results in recombination of electrons and holes to produce light. If, instead of heat, a light (laser) is used to release the phosphorescence, it is termed *Optically Stimulated Luminescence* (OSL); generally, the light is a green laser or infrared laser or just a light source. OSL has certain advantages and disadvantages in its use. TL has been studied much longer than OSL, so I will limit myself to the TL process, which is better known. TL is found in many materials. Figure 1 shows an example of limestone quartz. Figure 1A is the quartz under room light and Figure 1B is in a darkened room with the only light coming from the heating of the sample. One problem with using TL to date rocks is the TL signal may saturate and, therefore, not respond in a linear fashion with dosage. Other problems are mentioned below. Dating a volcanic eruption is not as problematic but still can have problems. Quartz is in almost every archeological artifact, especially pottery made from clay (which contains quartz). The abundance of quartz and feldspar in many objects allows for accurate dating. Other materials used for dating purposes are zircon, calcite, and flint. Many dates are determined from the abundant materials. Feldspar needs special care since it has anomalous fading, that is, its signal decreases with time. The process for quartz is well established. While a review of the glow peaks of quartz has been published (Koul, 2008), many papers ignore some of the competing processes that can affect the determined age of the artifact. The best way to explore the effect of these processes is to develop an uncertainty table for the process. An uncertainty table will be developed after exploring characteristics of a medical dosimeter as an example.

The Dating Process

Pottery has been found at a number of archeological sites and attempts have been made to provide dates. Comparison of TL dating with radiocarbon dating can provide a crosscheck; however, radiocarbon dating has its own complications. For example, exploration in southern China has uncovered pottery fragments that are claimed to date back to 20,000 years, using radiocarbon dating (Wu et al., 2012). Pottery has also been dated by TL techniques. In fact, it has been considered one of the most accurate methods for dating pottery (Khasswneh et al., 2011). A paper exploring TL dating from some of these sites has previously been published (Agrawal et al., 1981). Dates of 765 BC are given, compared with radiocarbon dates of 905 BC. Other dates given in that paper range from 1035 BC to 875 BC. None of these articles give an uncertainty analysis, so it is difficult to judge the accuracy of these dates. Generally, the most common display of an uncertainty analysis should include a standard deviation of measurements. However, uncertainty analysis includes more than just the standard deviation; it should include all aspects of the measurement.

The first step for dating of pottery is done by the artisan when he makes the artifact out of clay, which contains quartz. When the artifact is fired to harden it, the intrinsic luminescence is released and set to zero. Since the heating has removed all the intrinsic TL from the quartz, this date is the beginning of accumulation of TL and establishes the date to be determined. Figure 2 illustrates this firing as the vertical line with the TL signal at zero. The amount of TL increases from there based upon exposure to radiation in the surrounding environment.

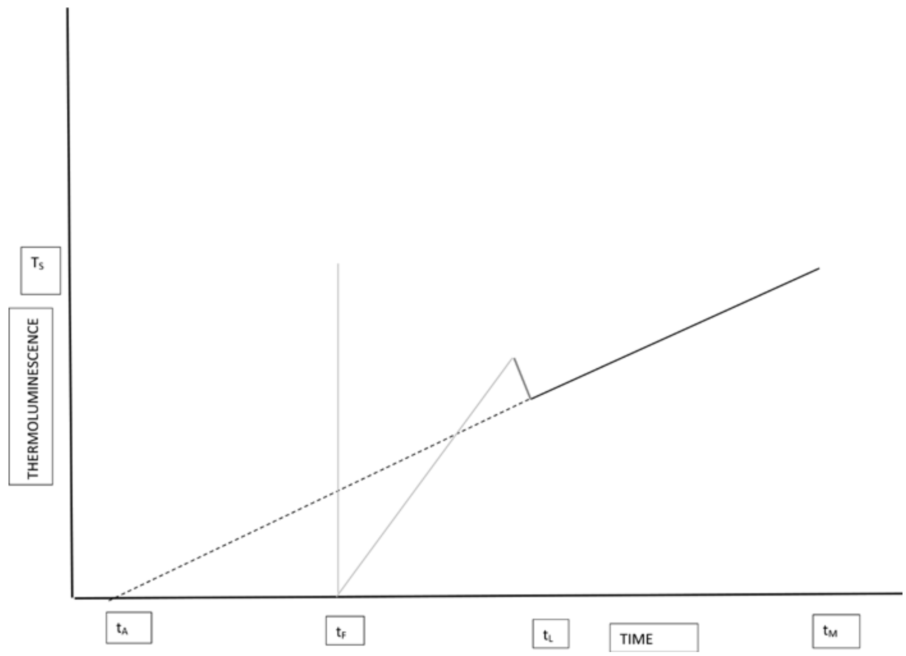

Figure 2. Example of thermoluminescence versus time of a buried artifact. At the point of firing the TL is set to zero. If the artifact is moved, its environment is different and the extrapolated date is wrong.

Determination of the Date

As the artifact (or the shard of the broken piece) ages, radiation from within the artifact and from the surrounding environment increases the stored luminescence (refer to Figure 2). When the shard is recovered after a number of years, a sample is removed from the artifact, and the quartz is heated for the TL. Thus, the TL signal from the artifact is determined, designated as T_S. The radiation rate of the environment (in situ) and the contained radiation is measured. However, the radiation rate of either the environment or the object itself is not necessarily constant and therefore may change over thousands of years. This change in radiation rate is one of the fundamental flaws regarding assumptions concerning TL and OSL dosimetry over long periods of time. The burial dose rate is then determined (dD_B/dt). The radiation emitted is usually alpha, beta, or gamma emissions. Each of these emissions has a different depth of penetration depending on the absorbing material. As an example, alpha particles travel about 1mm, beta particles about 5 mm, and gamma rays are not totally stopped. Thus, the amount of radiation dosage will vary with depth, and estimates will need to be made. In addition, the cosmic ray flux should be considered depending on elevation, latitude, longitude, and burial depth. Obviously, these quantities change over time; often in unpredictable ways and thus can affect the total radiation received. Another conflicting problem is energy dependence. For example, lower energy radiation generally has a greater TL response for a given dose.

There are different methodologies to determine the TL sensitivity of the sample, but all have a means of calibrating the quartz sample for TL response versus the dosage given. The doses given are generally desired to yield TL that would be near to T_S. Therefore, a ratio is formed for the calibrated dosage given, D_C, to the TL response, T_C. Thus, the sample dosage is determined by equation 1.

$$D_S=(T_S/T_C) * D_C \quad (1)$$

The age of the artifact is determined according to equation 2,

$$Age = D_S/(dD_B/dt) \quad (2)$$

where D_S is the dose measured from the sample and dD_B/dt is the burial dose rate.

There are a number of assumptions involved in each of the steps above. The first and most obvious is that the radiation rate was constant during the entire course of burial, and the burial was not disturbed. Since most of the radiation products are long-lived and are in secular equilibrium, the assumption of a constant radiation rate is assumed. This assumption may not be true because of displacement of the artifact and it does not account for any short-lived nuclides that may have affected the dose. In addition, Vernon Cupps (2019) has a new book that contends that radiation decay rates may have been different in the past. Many items of the past have a great uncertainty associated with them. Figure 2 graphically illustrates what may happen during this process. At the time of firing, t_F, the TL signal is reduced to zero. Thereafter, the TL signal of the original quartz in clay is gradually increasing with time, depending on the radiation received. However, the sample could be displaced in some manner (e.g., earthquake) to another site which has a different (lower) radiation exposure. During this displacement, t_L, the sample may lose signal because of heating from the sun or being closer to the surface. This dosage rate at the new site is different so that extrapolation back in time would give an increased age, t_A. The longer the burial, the greater the probability of this occurring. This process is an unknown effect since the background of the sample is not "well controlled." The end result here is extrapolation to a greater age, t_A, than the actual archeological age, t_F. An estimate of the probability of any of these events occurring can be listed in an uncertainty analysis.

Determination of Accuracy of Artifact Dating

The above determination of TL with radiation dosage at the burial site is not the only problem for determining an accurate date of an archeological artifact. There are a number of factors that can affect the age determination, as reflected in the following quotes from the literature:

> …thermoluminescence is at the most about 15% accurate. It cannot be used to accurately date a site on its own. However, it can be used to confirm the antiquity of an item. (Wikipedia)
>
> This technique (Thermoluminescence Quartz dating)… is only accurate on objects 300 to 10,000 years in age. (RationalWiki)

These quotes illustrate the necessity of considering the uncertainty of the whole process involved. Other TL characteristics that can affect the dating process can be explored by examining some characteristics of a controlled and well-studied phosphor in medical dosimetry, namely LiF:Mg,Ti. The characteristics (such as supralinearity, etc.) that can affect the TL signal can be extended to quartz as well in an attempt to determine the uncertainty involved in the TL process. The parallel between the two processes will be explored by looking at the medical dosimeter, which is well controlled, versus the artifact TL, which is not as controlled. The basic TL process will be explored for this purpose.

Basic Thermoluminescence

All the luminescent processes involve the solid-state band structure, which includes empty energy levels called the *conduction band*, filled energy levels called the *valence band*, and an energy gap, which is the separation between the two bands. If the conduction band and the valence band overlap (no energy gap), the material is a metal with an ease concerning conduction. The magnitude of the energy gap determines whether the material is a semi-conductor or an insulator. Luminescent processes generally occur in insulators as a result of traps (energy levels located in the energy gap from impurities). The luminescent process results from defects and impurities causing the electron, hole traps, and the recombination center, which is another defect, where electrons and holes recombine. The energy from the absorption of the radiation is stored in traps, or defect centers. The electrons are released when the material is heated, and the electrons recombine with the holes (positively charged), generally at a recombination center. This process results in the release of light with wavelengths characteristic of the recombination center. This process is represented schematically in Figure 3. OSL is the same process except that electrons are released by optical stimulation. These defects or traps and recombination centers are generally caused by impurities in the crystal lattice. The impurities cause color centers, and the TL can vary greatly depending on the amount of impurities (e.g., in LiF:Mg,Ti, the best TL occurs with 200 ppm of Mg and 20 ppm of Ti). A simple color center is the F center, which is the absence of a fluorine atom with an electron taking its place. A more complex one with an impurity (a dipole) is in Figure 4 (see DeWerd and Stoebe, 1972). Dipoles and combination of dipoles (dimers and trimers) are responsible for the traps in LiF:Mg,Ti.

Since defects and impurities are so important for the TL process, we need to consider the effect in quartz. The impurity level and type of impurity in

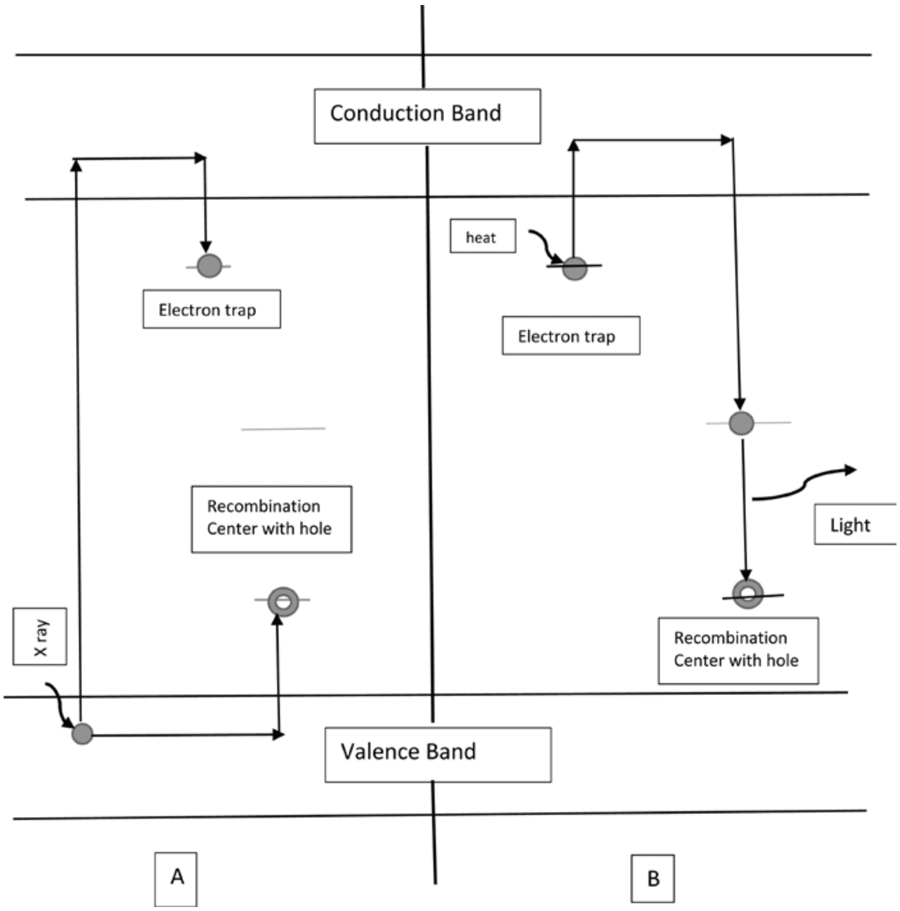

Figure 3. Energy level diagram for thermoluminescent materials. A: X-ray creates electron and hole which get trapped. B: Energy level diagram showing application of heat and recombination of hole and electron to produce light.

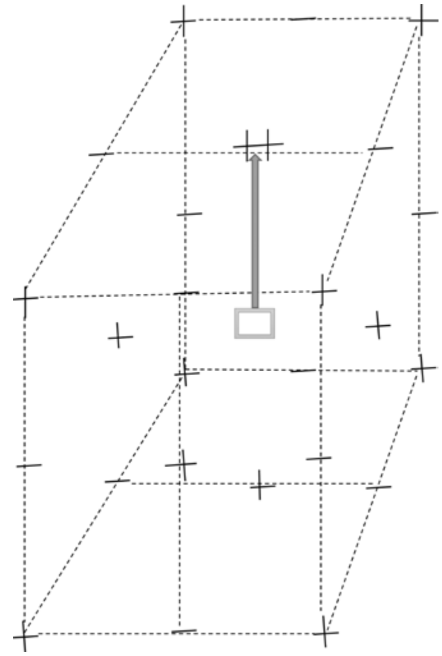

Figure 4. Diagram of a Z-center, an impurity electron trap center (see DeWerd and Stoebe, 1972). Note the dipole of this center. The double + is Mg in LiF.

quartz can affect the TL greatly and result in orders of magnitude variation in luminescent intensity. The trapping centers are also not completely known; the impurities can be Al centers, Si vacancies, or Ge vacancies. For the dating process, the impurities of the quartz should be determined and given when the resultant TL is presented. Generally, the impurities affect the glow curve or the spectrum. It is difficult to always determine the impurities without destroying the sample. In all cases, the TL intensity vs. calibration dosage needs to be given which is part of the determining factor for the age.

Glow Curves

The signal resulting from TL is called a glow curve. As the electron traps are heated, greater numbers of electrons are released until few filled traps are left. This results in a non-symmetric peak. If there are a number of traps in the phosphor, there can be a number of peaks at different temperatures as shown in Figure 5 for LiF:Mg,Ti. The numbered peaks correspond to different trap depths. At room temperature, these peaks decay away with time (DeWerd and Stoebe, 1972). Peak 1 is gone within 10 minutes after irradiation, and the 105°C peak has a half-life of 10 hours. The 190°C peak has a half-life of 80 years. The temperature rate of the readout can affect the overlap of the glow peaks. For quartz, the temperatures of the glow peaks at a 15°C/s heating rate are 110°C, 325°C, and 375°C. The decay of the low temperature trap can add electrons to the higher temperature traps, which when read-out can change the age determination, making the age greater than directly determined. This is also true if a preheat is applied when read-out but not when calibrated. The 325°C peak is decreased by solar light, so it must be kept in the dark to be used. The 375°C peak is the one usually used.

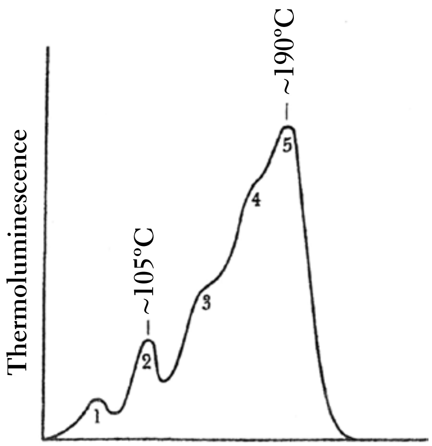

Figure 5. TL vs. temperature for LiF: Mg,Ti, with numbered peaks.

The 110˚C peak has also been used but with special manipulations (Koul, 2008).

Supralinearity

Another effect of impurities can relate to the recombination center and the TL response. Doped Lithium fluoride (LiF:Mg,Ti) has an increase in response above linearity at doses above 5 Gy; this is termed *supralinearity*. An example for peak 5 from Figure 5 with dose is shown in Figure 6. The curve marked LiOH has been grown with additional OH ions. Note that the supralinearity is gone, but the sensitivity of peak 5 is also decreased. Thus, the control of impurities affects the characteristics of the TL process, including supralinearity. Quartz samples are not as well controlled as LiF:Mg,Ti, and thus, can exhibit variation in these processes. Supralinearity also occurs in quartz. For example, Figure 7 shows supralinearity of the 110˚C peak in quartz. The data points are from Martini and Fasoli in Chen and Pagonis (2019). Note how an error could occur if the calibration was done only at the lower doses. If the TL/dose was extrapolated from the lower values, the dosage expected from the TL would be about 250 Gy, as opposed to 200 Gy. This would be a 25% error resulting in an increased age. This difference is indicative of the problem when the standard assumption is made that dosage is given uniformly with time and the response of the TL material is uniform. A knowledge of the dosage response, including supralinearity, is important when calibrating the TL material. A linear fit can result in significant errors (Gruen, 1996). Gruen considers the error that could result by the failure to account for supralinearity and saturation region. Supralinearity must be accounted for in the dating process.

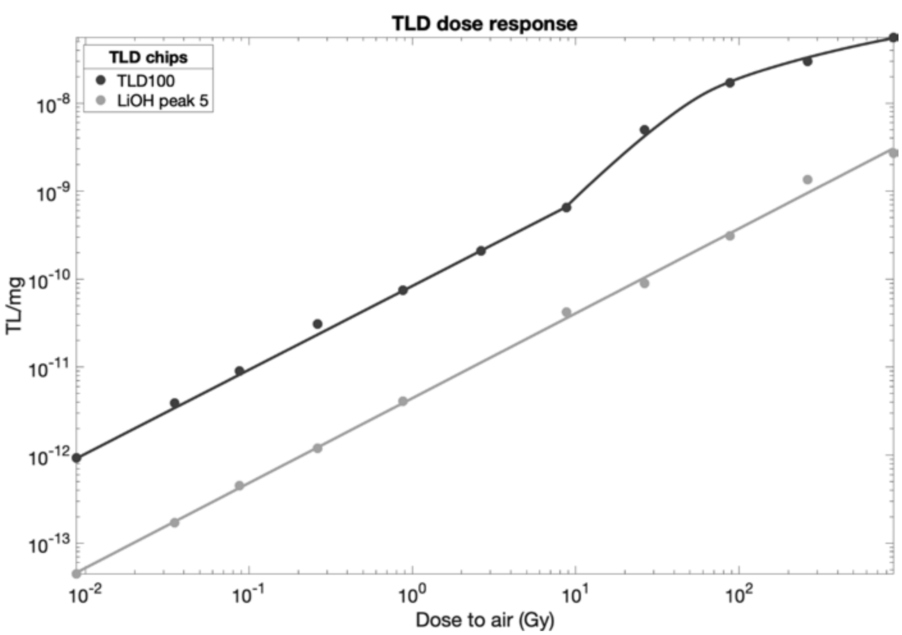

Figure 6. TL dose response for peak 5 in TLD100 (LiF:Mg,Ti) showing supralinearity. The curve designated LiOH peak 5 is the addition of OH ion to LiF:Mg,Ti. Note the decrease in sensitivity and the lack of supralinearity.

Uncertainty Determinations

In the past, scientific measurements were expressed with random and systematic errors. Aitken and Alldred have considered the error limits of TL dating in the past (Aitken and Alldred, 1972). At present, the methodology no longer uses "errors" but uncertainty expressions which involve the entire experimental process. This Uncertainty determination includes all aspects of the experiment, including an estimate of the assumptions used. For example, the assumption that the artifact received constant radiation can be expressed in terms of a probability of being displaced or not receiving constant radiation. The determination of the uncertainty is important regarding the characteristics of the TL process for dating since many investigations only include the standard deviation of the measurements and not estimates of the other influence quantities. The question arises as to the certainty of the dates, which should include an analysis of the uncertainties involved. Information on the process of determining the date and the resulting uncertainties are not reported in many of the articles dating artifacts. Thus, it is difficult to adequately determine the accuracy of the date. The discipline of determining the uncertainty of measurements will be discussed first, using the medical dosimeter as an example.

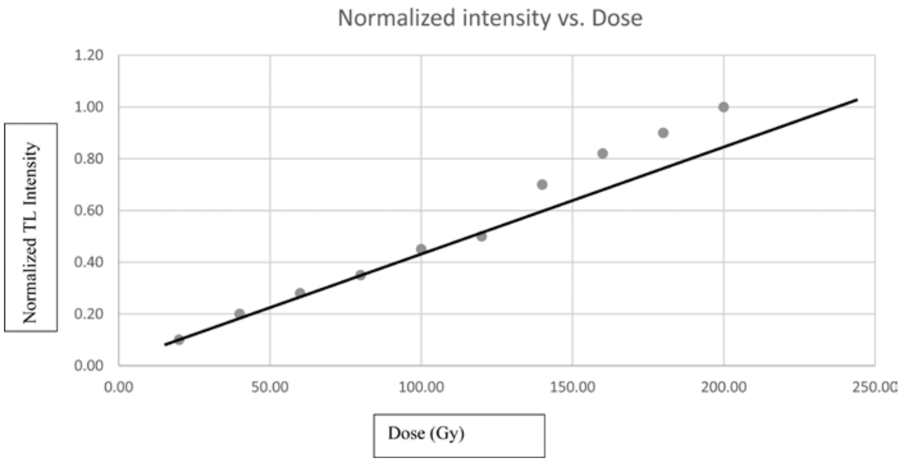

Figure 7. Supralinearity of the 110°C peak in quartz. Data from Martini and Fasoli in Chen and Pagonis (2019).

The Uncertainty Process

The following is a brief explanation of the entire process for uncertainty determinations as given in the National Institutes of Standards and Technology (NIST) technical note publication (Taylor and Kuyatt, 1994). The first distinction is the difference between accuracy and precision. Accuracy is how close the value is to the conventional true value–the absolute correct value. This absolute correct value is generally traceable to a national primary laboratory, e.g. NIST in the U.S. Precision is how close the values are to each other (reproducibility). The uncertainty process is to determine the accuracy of the measurement. This is accomplished through two quantities, termed *Type A* and *Type B uncertainties*, from historical terminology. Type A uncertainty is estimated by the standard deviation of the mean value. These are measured results and generally are the only value given as a standard deviation in many publications. Any valid statistical method for treating data can be used to express Type A uncertainties. Type B uncertainty cannot be estimated by repeated measurements (standard deviations). Type B is based upon scientific judgment but using that which applies to the measurement. Generally, Type B uncertainty is based on a confidence interval. Type B is based on scientific judgment using all relevant information available. From these quantities an uncertainty table is constructed. Uncertainty tables and determinations are important to give a representation of the goodness of the measurements. Determination of uncertainty gives an indication of how close you would expect your measurements to compare to other similar measurements and how close they are to the correct value. It also indicates how close the data is to the conventional true value. Finally, an uncertainty table is constructed to provide a coverage factor; this factor is generally calculated at the 66% level termed k=1. All the values are added in quadrature and then the square root is determined. After the table is constructed, the last line is to be multiplied by 2 for a coverage factor of k=2, which covers 95% spread of the entire experiment. This value is called the expanded uncertainty and would cover 95% of the values in the experiment.

Uncertainty of TL of LiF:Mg,Ti

Experience with the medical dosimeter of LiF:Mg,Ti will be used because of the major amount of research done on this phosphor. An uncertainty table can be constructed (see Table I) for a brachytherapy application of determining the dose rate constant using TL as the dosimeter. Gerhart et al. (2000) published an uncertainty table for this quantity with a final expanded uncertainty (k=2) from this publication of 7.7%. Using the same material, LiF:Mg,Ti, but with increased care, the uncertainty of these determinations has been decreased as given in Table I (DeWerd et al., 2009, 2011). The TL response was determined to be within 1.5% as opposed to 4.6% in the Gerhart publication. The expanded uncertainty for Table I is 4.6% at k=2, whereas the total expanded uncertainty at k=2 for the Gerhart publication is 7.7%. Note the importance of stating all procedures with an uncertainty given to maintain the lowest value of uncertainty for the experimental procedure. It falls on the researcher to do these estimates of uncertainty to complete their experimental procedures.

Estimated Uncertainty in the Quartz Dating Process

Using the information as given in this publication, an estimate of the uncertainty of the dating process using the TL from quartz can be determined. The steps would include estimates of the probability of the assumptions made. Table II is an attempt to consider all the parameters that would be involved in the TL dating process. Individual experiments certainly can be less than given in Table II, but these values are estimates for a maximum uncertainty. Note that the conclusion would be, using these estimates, that an age of 20,000 years could really be an age between 2,900 to 10,000 years. This discrepancy indicates that an honest evaluation of the uncertainty involved in the dating experiments must be done, rather than

Table I. Example of determination of the experimental uncertainty using TL for brachytherapy dose rate constant.

Parameter	Type A Uncertainty	Type B Uncertainty
TL Reproducibility	1.5%	
Dose Calibration		1.0%
Energy Dependence Correction	0.5%	0.5%
Positioning of TL		1.0%
PMT Linearity		0.5%
Sum in Quadrature	1.58%	1.58%

Total Uncertainty k=1	2.24%
NIST uncertainty	0.5%
Combined Uncertainty	2.30%
Expanded Uncertainty k=2	4.60%

Table II. Estimates of uncertainty involved in the quartz dating process.

Quantity	Estimate of Uncertainty at k=1
Environmental considerations–possibility of changes	10%
Probability of radiation not being constant	10%
Measurement of radiation in the environment	5%
Amount of impurity and TL process, including sensitivity	10%
Energy response from different emissions	20%
Saturation of TL	15%
Calibration of dose—if supralinearity not accounted for	25%
Reproducibility of reading	5%
Match of PMT to emission spectra	10%
Calibration Dose	5%
Traceability to NIST (particulate radiation)	10%
Combined Uncertainty k=1	42.72%
Expanded Uncertainty k=2	85.44%

just claiming a date because it is what the scientist would expect. All of the dates determined using TL are generally less than 10,000 years. However, this age is probably an overestimate based on the uncertainties involved.

Summary

It would appear that the most accurate dating method for artifacts would be via the TL process. An important exercise for the TL dating of artifacts would be to perform an uncertainty analysis to determine the accuracy of the dating process. An uncertainty analysis is very important for understanding the accuracy of the measurement. Researchers must be familiar with the entire process and characteristics of material they are using. Generally, the expectation of the whole process would require that dates be given an uncertainty analysis to determine the accuracy or the variation from the conventional true value. If results are analyzed and all uncertainties taken into account, the ages of the TL dating of artifacts would be expected to be younger than 10,000 years. The age of the pottery sherd would expect to agree with young ages.

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