Chapter 4 Appendix 7

THERMOLUMINESCENCE ANALYSES OF HIROSHIMA CERAMIC TILE AND NAGASAKI BRICK

USING THE PRE-DOSE AND INCLUSION TECHNIQUES

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Following a small program of diagnostic testing on ceramic tile from Hiroshima[1] the pre-dose and inclusion measurements have been completed on a sample of tile from Hiroshima University (University of Utah reference UHFSFT02, at 1442 m from the hypocenter) and a sample of brick (NA1E06, at 1427 m) from Nagasaki. In this outline report the results of the measurements and also the results obtained from measurements on gamma-ray irradiated quartz supplied by the Oxford Laboratory and samples of gamma-ray irradiated material prepared by the University of Utah to enable a comparison of laboratory beta-particle calibrations are presented.

Sample Preparation

The tile and brick were cut into small blocks using a water-lubricated diamond-impregnated wheel; for the Nagasaki brick 18 blocks were produced and for the Hiroshima tile 6 blocks were produced after removal of the outer 2 to 4 mm of surface material. In the case of the Nagasaki brick, where the variation of dose with depth was examined, the approximate block sizes were $25 \times 25 \times 30$ mm.

Each block was crushed between the jaws of a mechanical vice (all preparations were carried out under red light). Sieved fractions in the size ranges 53 to 90, 90 to 150, 150 to 200, and 200 to 350 μm were obtained; for the Nagasaki material wet sieving techniques were

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employed because of the abundance of fine grain aggregations. The 90 to 150 μm fractions were ultrasonically washed in acetone, dried at 50°C, and passed through a Franz magnetic separator to remove the magnetic clay fraction (the product is referred to as the separated sample). The following treatments were subsequently applied to provide thermoluminescence (TL) samples suitable for each technique.

1. Hydrogen fluoride (HF)-washed (HFW) samples were prepared by immersing (and using ultrasonic agitation) the separated material in 20% HF for 10 minutes, after which a solution of $AlCl_3$ was added to remove precipitated fluorides.[2] This treatment has been used routinely for dating work to disperse any clay minerals that remain after magnetic separation.
2. H_2SiF_6 treated (HSiF) samples were prepared by immersing the separated sample in 40% H_2SiF_6 for 40 hours to selectively remove feldspars (Bowman, personal communication).
3. HF-etched (HFE) samples were prepared by immersing separated material in 40% HF for 45 minutes, followed by the $AlCl_3$ treatment referred to above.

After each of the above treatments, samples were ultrasonically washed in the following sequence: distilled water (×3), calgon, distilled water (×3), methylated spirits and acetone, and finally dried at 50°C. All samples were then reseived and the > 90 μm fraction formed the TL sample. A portion taken from each TL sample was inspected under the microscope to check the condition of the grains and for the presence of fine grains. Because of doubts concerning sample purity that emerged during initial TL testing, the mineral composition of a number of prepared TL samples was also determined using x-ray diffraction. The presence of carbonates, detected before use of any of the above acid treatments, is observed by the reaction of the sample with HCl; none showed a significant reaction.

Thermoluminescence Measurements

TL samples were heated in an atmosphere of oxygen-free nitrogen at 10 or 20°C/s in apparatus that was described previously.[3] TL emission was viewed with a detector system comprising an EMI 9635Q photomultiplier and Corning (5 to 60 for high temperature and 7 to 51 for low temperature emissions) and Chance Pilkington HA3 (infrared reflecting) filters. The sensitivity of the TL detection system is routinely monitored using a ^{14}C doped scintillator which provides a stable weak source of light. Samples were irradiated at 20°C while located on the heater plate using a ^{90}Sr-^{90}Y plaque source that is mounted in a purpose-built irradiator.[4] Portions of 1 to 3 mg were deposited as a monolayer onto 0.5-mm thick stainless steel disks within a radius of about 4 mm. In the case of pre-dose measuerments, the disks were coated with a thin layer of silicone oil (Silspray, Duxe Products, USA) before deposition and for high temperature measurements this procedure was omitted to reduce interference by luminescence emission from the silicone oil (the TL emission from these samples was low compared with that usually found with archaeological samples). All disks were heat treated before use to stabilize their colour.

High Temperature Procedures. The inclusion technique,[5] incorporating further developments,[6] was used for analysis of the behaviour of the high temperature TL emission. Using a heating rate of 20°C/s, sufficient intensity was obtained and analysis of the glow curve (RT-475°C) was made between 350 and 425°C. Both weight and monitor normalization procedures were used to obtain first (additive) and second glow growth curves; with one exception, the dose evaluations given below were calculated using monitor normalization since it offered better precision. Where monitor normalisation was used to obtain the first glow growth characteristic, laboratory doses were adjusted so that all samples had received the same total dose before administration of the monitor dose. This procedure is necessary where sensitization occurs. Normalization for the second glow growth characteristic was obtained using the intensity of the natural TL signal. In a full dating research program the gradient of the second glow growth characteristic was also checked for samples that had received additive doses (ADD), and this was performed in a number of cases. The dose regions investigated extended to four times the accrued dose (that absorbed between last firing and laboratory testing). Portion weights ranged from 1 to 2.5 mg. Tests for short-term nonthermal fading[7] were undertaken for portions that had received laboratory dose in additon to the accrued dose (AD).

Pre-dose Procedures. The diagnostic procedures that have been developed in this laboratory for archaeological samples[8-10] and applied to modern brick[11,12] were used, together with further developments found necessary for the samples under study. These procedures included:

1. Measurement of thermal activation characteristics (TAC). In view of the limited commissioned time for these tests, full additive dose TAC were ommitted.
2. Ultraviolet (UV) reversal tests to establish the nature of the initial sensitivity.[13]
3. Growth characteristics using the multiple activation (MA) procedure to determine the behavior of the filling of R traps and L centers (following Thompson's model).
4. Growth characteristics using the modified additive dose (modADD) and additive dose (ADD) procedures.

The results of these measurements provide the following information:

1. Whether satisfactory intensity and glow curve shape are obtained using a test dose (0.63 rad).
2. The temperatures and procedures required to achieve maximum sensitivity enhancement and the extent of thermal deactivation beyond that maximum. Alterations in the form and peak position of the TAC for activation of the accrued and subsequent laboratory doses.
3. The level of the initial sensitivity, S_o, usually expressed as a fraction of the sensitivity measured after activation of the S_N. The degree to which it may be reduced by exposure to a source of UV radiation which includes wavelengths in the region of 240 nm.[14]
4. The extent of saturation effects in the filling of L centers and R traps.
5. Whether there is a significant difference in the evaluation of the AD using the MA

and modADD procedures. As demonstrated previously[9] changes in sensitization may occur on first heating in the laboratory leading to a false evaluation in the AD using the MA procedure alone.

If the results of these diagnostic measurements indicate that the sample possesses consistent properties and satisfactory precision has been obtained, the same measurements are repeated on a greater number of portions.

The dose regions investigated for each sensitization characteristic were:

1. R trap chracteristic - at least 1.5 × AD,
2. L center characteristic - at least 2 × AD,
3. ADD and modADD characteristics - at least 5 × AD.

Results

Sample Preparation. Extraction of the TL sample proved to be extremely laborious because of the low crystalline yield of both materials, combined with the hardness of the ceramic matrix in the case of the Hiroshima tile and the tendency of the fine grains to aggregate and interfere with the separation of larger grain sizes in the case of the Nagasaki brick. Approximately 70 mg of the Hiroshima sample and 210 mg of the Nagasaki sample was obtained. The TL sample obtained from the Nagasaki brick blocks were amalagamated to enable dose evaluations from three "slices" (with cuts parallel to the front face) of the following thicknesses, given by distances in millimeter from the front surface of the brick: 3 to 32 mm, 34 to 64 mm, and 66 to 103 mm.

Table 1. Dose Evaluations[a,b]

Sample Number	Inclusion Technique				Pre-dose Technique		
	Q±dQ (rad)	I±dI (rad)	1st:2nd	AD (rad)	AD (MA) (rad)	AD (mod.ADD) (rad)	S_o/S_N
UHFSFT02	73± 2 (9)	4±4 (6)	0.6	77± 6	150±8 (4)	78±9 (14)	0.32
NAIE06							
(3- 32mm)[c]	67± 5 (14)	28±7 (5)	1.08	95±10	103±2 (9)	117±5 (17)	0.27
(24- 64mm)	83± 8 (17)	2±2 (9)	0.97	85± 9	72±3 (4)	84±3 (10)	0.30
(66-103mm)	71± 3 (9)	−1±2 (6)	0.97	70± 9	87±2 (4)	77±3 (11)	0.27
OXCALIB.Q	242±12 (7)	153±8 (7)	0.64	395±22			

[a]All dose rates based on beta-particle source calibration of 140 rad/min to 90-150 μm grains of quartz resting on stainless steel disks.

[b]NAIE06 (3-32mm); Q evaluated using weight normalization.

[c]Errors represent the standard error of the mean at the 68% level of confidence. Number of measurements in parentheses.

Accrued Dose Evaluations. The evaluation of accrued doses using the inclusion and pre-dose techniques for Hiroshima and Nagasaki samples are given in Table 1. The inclusion and pre-dose results were obtained from samples that had received HFE and HSiF treatments, respectively. The errors given represent the standard error of the mean value and the number of measurements is given in parentheses. The inclusion of systematic errors is discussed later.

Inclusion Technique. The equivalent dose (Q) and the supralinearity intercept (I) were evaluated by extrapolation to the dose axis of the linear curve obtained by weighted linear regression. The sum of these evaluatons for samples UHFSFT02 and NAIE06 (3 to 32 mm), (34 to 64 mm), and (66 to 103 mm) is given as AD. The ratio of the gradients of the 1st:2nd glow growth characteristics is also given in the table.

Pre-dose Technique. The evaluations of the AD obtained using the MA and modADD procedures are given in the table. In the case of the MA evaluations, the AD represents the mean value of a number of measurements (given in parentheses) where the laboratory dose was within approximately ± 10% of the evaluated AD. These evaluations were made after investigation of the linearity of the filling of L centers and R traps. The modADD evaluations were obtained by extrapolation to the dose axis of the linear curve obtained by weighted linear regression. For both MA and modADD calculations the measured values of the initial sensitivity (given in Table 1) were taken to represent the sensitivity baseline since for both NAIE06 and UHFSFT02 the initial sensitivity was not UV reversible.

Discussion

Because the evaluated doses for these samples are relatively low compared to most archaeological samples, the TL emission was fortuitously high and enabled satisfactory use to be made of the inclusion technique. In the Dating Service a number of ceramics of the last half millennia have been able to be dated using the inclusion technique for which the AD were comparable, but this has been quite rare.

Sample Preparation and Initial Testing. After obtaining evaluations of the AD using the inclusion technique with HFE samples it has been intended to proceed with HFW samples for pre-dose measurements (although the pre-dose technique was successfully used for dating with HFE samples). However, the results (not given here) obtained from pre-dose tests on both Hiroshima and Nagasaki HFW samples indicated MA evaluations of the AD that were significantly higher than those obtained using the inclusion technique and which could not be accounted for by saturation effects. Although at this stage there was no reason to suppose that the pre-dose evaluations were erroneous, further examination of the samples was undertaken by x-ray diffraction which revealed the presence of a plagioclase feldspar in the HFW preparations of the Nagasaki sample and mullite in Hiroshima sample. These two minerals were, as expected, not present in samples that had been HF etched. Little Hiroshima material was available for further acid treatment, but satisfactory pre-dose measurements could fortunately be completed using the modADD procedure (see below). In the case of the Nagasaki sample, where there was a greater quantity of TL sample, the feldspar component was eliminated (confirmed by x-ray diffraction) by use of the fluorosilicic acid treatment described above.

The presence of minerals such as feldspar in ceramics has not previously been considered

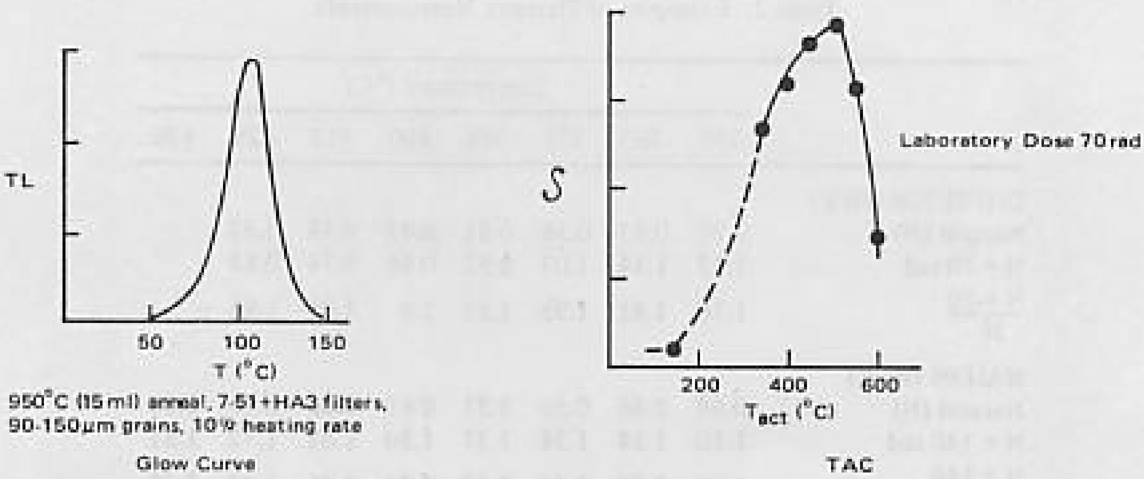


Figure 1. Pre-dose behavior exhibited by annealed samples of geological albite

to present a problem in the evaluation of the AD using the pre-dose technique. Potassium feldspar is known to have a TL peak at about 140°C and this is evident as a shoulder or slightly overlapping peak on the high temperature side of the 110°C TL peak of quartz. The TL properties of mullite in porcelain have been studied at Oxford by Doreen Stoneham and although a shoulder in the mullite glow curve at about 110°C is evident, the 110°C TL peak of the porcelain slice is attributed to quartz. Pure samples of plagioclase feldspar (albite and labradorite) were obtained and both of these samples exhibited a TL peak at about 110°C (10°C/s) that would not be distinguished from the quartz 110°C TL using our detection system (Figure 1). Moreover, both feldspars exhibited pre-dose sensitization similar to quartz, being much stronger and persisting to greater doses in the case of labradorite. It is believed that this has not previously been reported and has important implications in the use of the pre-dose technique, especially in view of the high dose evaluations obtained using the MA procedure with samples containing plagioclase feldspar. Samples of mullite have yet to be tested but it is suspected that they will show pre-dose behaviour similar to that of the plagioclase feldspars.

The dose calculations using HFW samples suggest that the pre-dose properties of the feldspar and quartz present were sufficiently dissimilar to cause significant complications in the form of the growth characteristics and thus to require the removal of one of the components. One possible explanation of the initial results is that a TL component in the sample sensitizes and contributes to the measured sensitivity (S_N) after the first heating in the laboratory. During this thermal activation the mechanism associated with the nonquartz TL component saturates and it is only quartz that contributes in further measurements, giving rise to an erroneously high evaluation of the AD. In the case of the Nagasaki sample, it appears that the component is due to a plagioclase feldspar (the question of whether it is albite or labradorite is presently being examined). Thus the pre-dose results given in Table 1 for the Nagasaki brick were obtained using HSiF-treated samples. In two cases HFE samples were also tested and although the AD estimates were significantly less than those obtained using the HFW samples, they were higher than the inclusion and pre-dose (HSiF) estimates by approximately 25%. In cases where the pre-dose AD evaluations for archaeological samples were compared using both preparations a significant difference has not been observed.

Table 2. Examples of Plateaux Measurements

	Temperature (°C)							
	350	363	375	388	400	413	425	438
UHFSFT06 (HFE)								
Natural (N)	0.70	0.63	0.56	0.51	0.43	0.38	0.33	
N + 70 rad	1.23	1.14	1.09	0.93	0.86	0.74	0.63	
$\frac{N+70}{N}$	1.76	1.81	1.95	1.82	2.0	1.95	1.91	
NAIE06 (HFE)								
Natural (N)	0.44	0.46	0.50	0.57	0.61	0.66	0.71	0.68
N + 140 rad	1.10	1.14	1.26	1.37	1.50	1.62	1.72	1.51
$\frac{N+140}{N}$	2.50	2.48	2.52	2.40	2.46	2.45	2.42	2.22

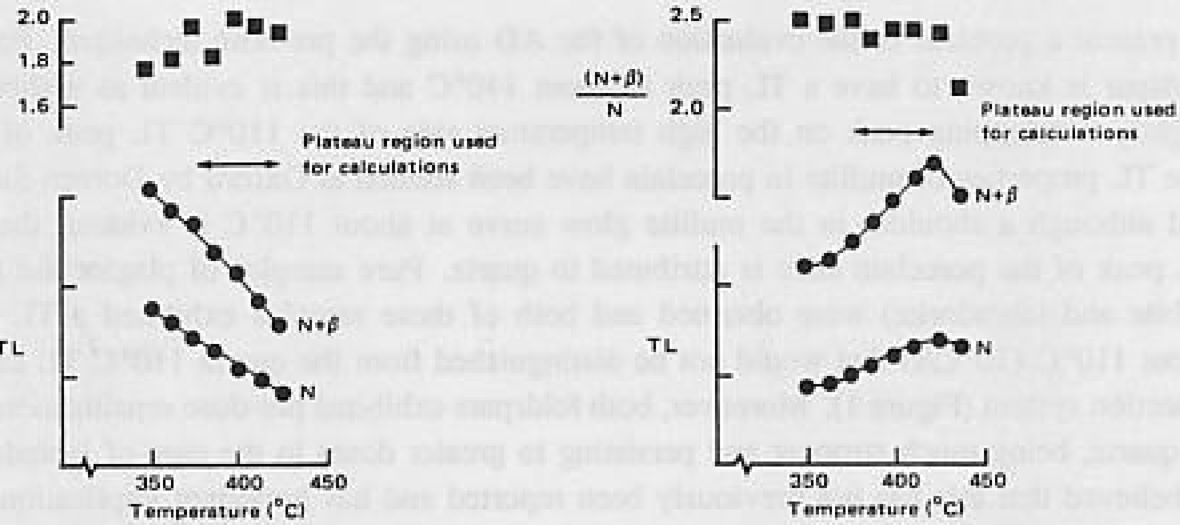


Figure 2. Examples of plateaux data obtained from Hiroshima (UHFSFT02) and Nagasaki (NAIE06) samples using the inclusion technique

Inclusion Technique. Satisfactory plateaus were obtained from all samples, although they were generally narrower for the Nagasaki samples, where a lower temperature peak dominated the glow curve (Table 2 and Figure 2). It is to be noted that a larger value for the intercept, I, was obtained in the case of NAIE06 (3 to 32 mm) where weight normalization was employed, compared with values of I obtained for the deeper slices (34 to 64 mm) and (66 to 103 mm). With one exception (UHFSFT02), the ratio of the gradients of 1st:2nd glow growth characteristics is within acceptable limits to meet the parallel slopes criterion. There are however cases where, using monitor normalization, this criterion will not be met, yet the correct AD may be evaluated in the normal manner.[6] In the case of the Hiroshima sample, the convergence of the growth characteristics (1st:2nd = 0.6) is the result of sensitization in the high temperature region of the glow curve. By calculating the degree of sensitization that occurs between the measurement of the natural glow curve and the monitor glow curve (a specific pre-dose intervenes), the corrected ratio 1st:2nd slopes is 0.9 and therefore con-

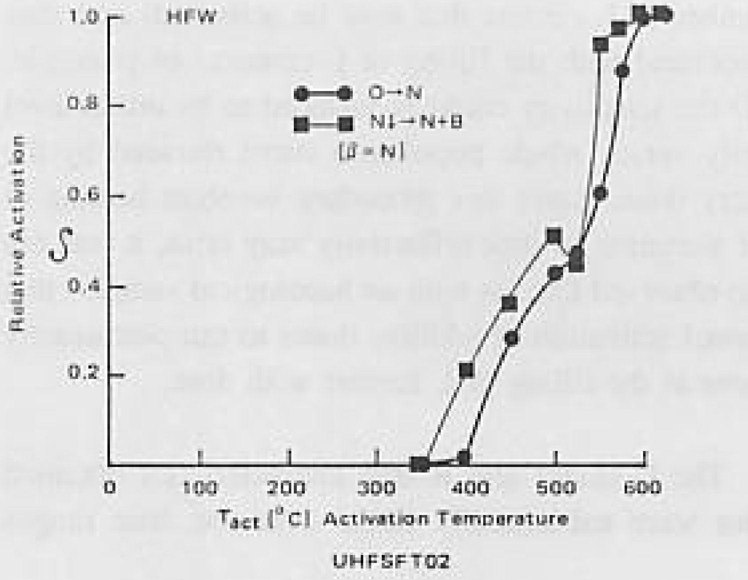


Figure 3. Thermal activation characteristics for Hiroshima tile UHFST02; HFW preparation of sample

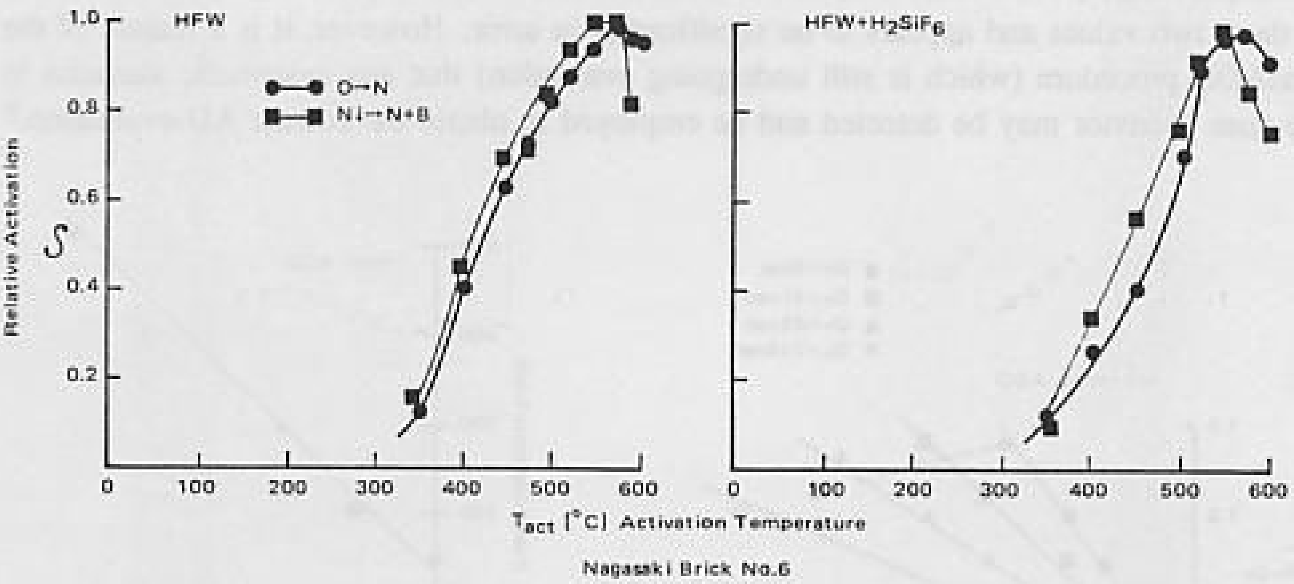


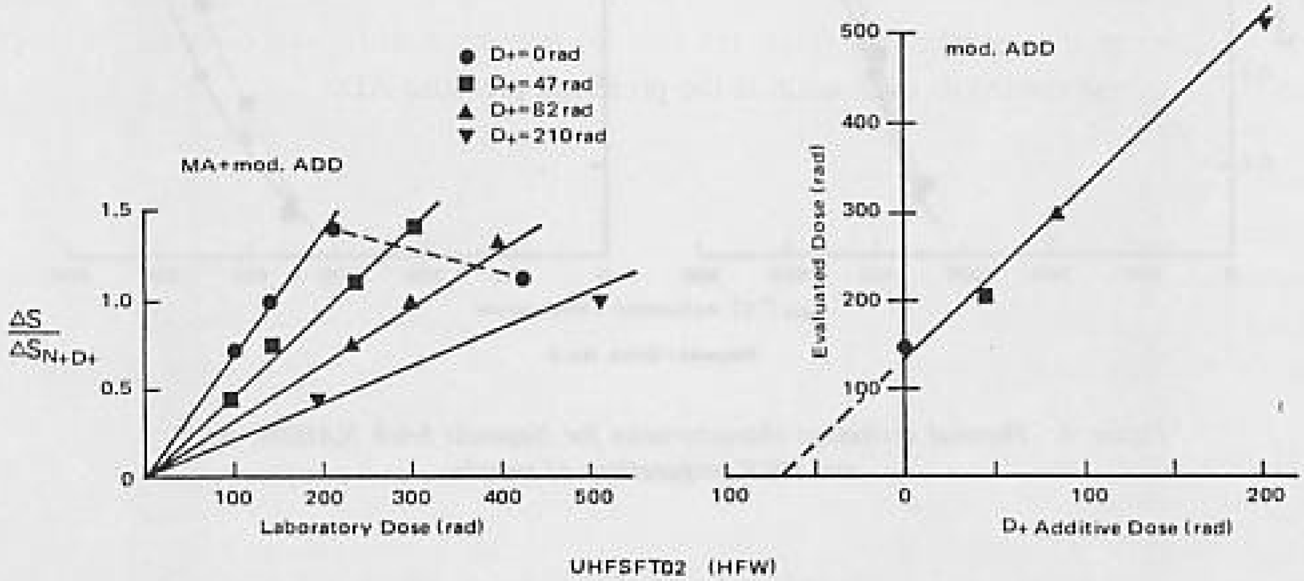
Figure 4. Thermal activation characteristics for Nagasaki brick NAIE06; HFW and HSiF preparations of sample

sidered acceptable. Nonthermal fading was not detected over a period of one week within experimental limits of 5% using portions from both samples. We note the decrease in mean values of AD evaluations as a function of depth from the front surface, although only the front and back AD evaluations are distinguishable at the 68% level of confidence.

Pre-dose Activation Procedures. The activation procedures were selected with the aid of the information provided by the TAC (Figures 3 and 4). In most cases three thermal activations were employed to determine the maximum sensitization; a peak or plateau was obtained by heating to 25°C below, at, and 25°C above the temperature required to achieve maximum sensitization. Improved linearity in the filling of the L centers was obtained by additionally heating to either 625 or 650°C to deliberately reduce the sensitivity by thermal deactivation. This, according to interpretations suggested previously, reduces the number of activated L centers (but not the total number of L centers that may be activated) and thus delays the onset of saturation effects associated with the filling of L centers. In principle, this means that after activation of the AD the sensitivity could be reduced to its initial level by thermal deactivation and the sensitivity versus whole population curve retraced by the application and activation of the laboratory dose. Since this procedure involves heating in excess of 650°C, where complications of alteration of disk reflectivity may arise, it was not pursued routinely for these tests. It is also observed that, as with archaeological samples that have been tested, the application and thermal activation of additive doses to samples usually gives rise to improved linearity of response in the filling of L centers with dose.

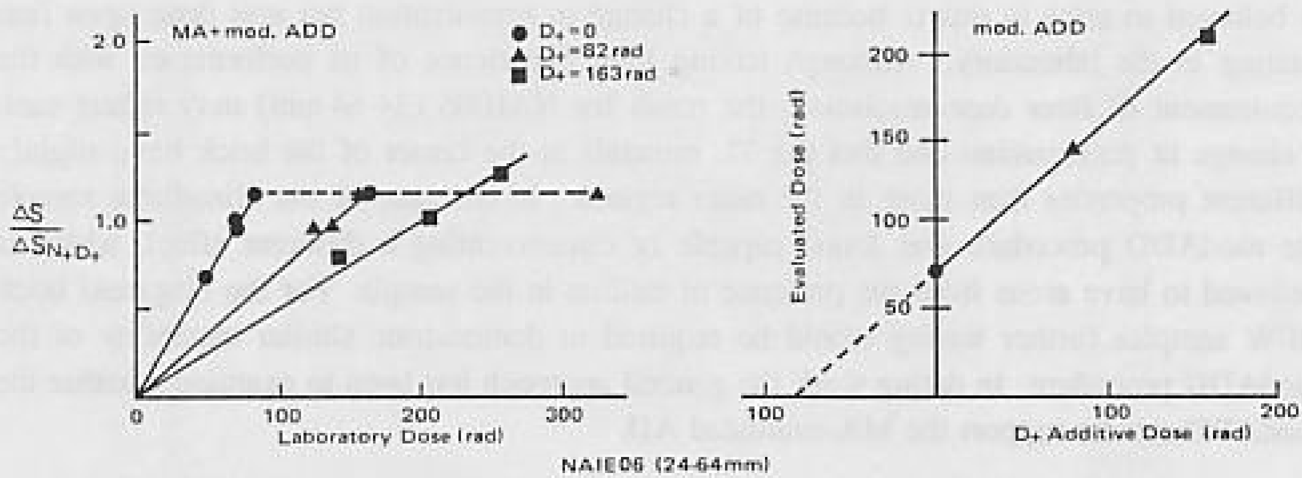
Pre-dose Accrued Dose Evaluation. The L center and R trap characteristics obtained for the Hiroshima and Nagasaki samples were substantially linear over the dose ranges investigated (Figures 5 and 6).

The modADD-evaluated AD (78 ± 9 rad) for UHFSFT02 agrees well with the inclusion technique result (77 ± 9 rad). The MA-evaluated AD (150 ± 8 rad) is roughly double either of these two values and appears to be significantly in error. However, it is a feature of the modADD procedure (which is still undergoing evaluation) that any systematic alteraton in pre-dose behavior may be detected and be employed to obtain the correct AD evaluation.[6]



In plot (i) the ordinate represents the ratio of sensitivity change (ΔS) measured after (the second) thermal activation of a laboratory dose to the sensitivity change measured after (the first) thermal activation of the natural or natural plus additive laboratory dose (ΔS_{N+D+}). Each data point represents a different portion (with the exception of the point linked by dashed line). The MA ($D_+=0$) plot shows linearity in the filling of L centers and R traps to 200 rad, although in the case of the Hiroshima tile, onset of nonlinear effects is indicated by further activations (dashed line). Linearity is demonstrated in the case of portions that have received additive doses. For the mod.ADD procedure (plot ii), the 'Evaluated Dose' ordinate values are obtained by finding the 'Laboratory Dose' corresponding to an ordinate value of 1.0 in a plot (i) for each additive dose used ($D_+=0$, β, 2β, etc). The AD is evaluated by extrapolation to the abscissa.

Figure 5. Examples of sensitization characteristics obtained using multiple activation and modified additive dose procedures for Hiroshima tile UHFSFT02



See footnote Figure 5

Figure 6. Examples of sensitization characteristics obtained using multiple activation and modified additive dose procedures for Nagasaki brick NAIE06

In this case the results suggest that, in the absence of gross saturation effects (confirmed), either of the following have occurred: (a) a halving of the pre-dose-sensitization per unit dose on first laboratory heating, or (b) a TL component of the sample is present exhibiting pre-dose properties in the first, but not subsequent, thermal activations. Since the occurrence of (a) is in the opposite sense to the previous observations, and, as discussed above mullite was detected in the sample, it is suspected that (b) may account for the behavior observed and thus that the modADD evaluation is the preferred pre-dose AD.

The AD evaluations for the NAIE06 samples show a decrease with depth from the surface similar to the inclusion results, but offering greater precision for each determination. It is noted that, using the standard error limits at the 68% level of confidence, the modADD-evaluated AD are higher than the MA-evaluated AD by approximately 15% for the two slices nearest the surface of the brick (3 to 32 mm and 34 to 64 mm), yet the reverse occurs for the rear slice (66 to 103 mm).

In principle, the modADD procedure is susceptible to yielding an overestimation of the AD compared with the AD evaluation since linearity of sensitization over a greater dose region is required. However, it was found that the linearity of the filling of R traps and L centers is often improved after the thermal activation of an additive dose. In these cases it is in the evaluation of the AD using the MA procedure where any nonlinear effects can be more pronounced, and if not taken into account (for L centers in particular), may lead to an overestimate of the AD. This result will in turn lead to a greater proportional increase in the modADD evaluated AD since it is obtained by extrapolation to the dose axis of the growth characteristic, the zero ADD point of which is provided by the MA data. Thus, at this stage, care should be taken to assess potential sources of systematic error before drawing conclusions from comparison of the two sets of pre-dose results. It may be more appropriate to take the mean of the pre-dose AD evaluations (3 to 32 mm, 110 ± 6 rad; 34 to 64 mm, 78 ± 4 rad; and 66 to 103 mm, 82 ± 4 rad) which compare well with the inclusion technique evaluations using the quoted precision limits. On the other hand, it is worth noting that the modADD procedure has been successfully used to detect and circumvent gross underestimates of the AD that may occur using the MA procedure alone and which is believed to arise in quartz because of a change in sensitization per unit dose upon first heating in the laboratory. Although having little experience of its performance with the requirement of finer dose resolution, the result for NAIE06 (34-64 mm) may reflect such a change in sensitization and that the TL minerals at the center of the brick have slightly different properties than those in the outer regions. In the case of the Hiroshima sample the modADD procedure was found capable of circumventing a different effect, which is believed to have arose from the presence of mullite in the sample. For the Nagasaki brick HFW samples further testing would be required to demonstrate similar capability of the modADD procedure. In dating work the general approach has been to examine whether the modADD results support the MA-evaluated AD.

Conclusion. Taking an error of ±6% associated with the calibration of the ^{90}Sr source used in these measurements (which is considered to be appropriate)[3] and calculating the average of the pre-dose and inclusion results (with the exclusion of the MA-evaluated AD for reasons given above), gives the AD for each sample given in Table 3.

Table 3. Final Results for the Accrued Doses to the Samples

Sample	Accrued Dose
UHFSFT02	78 ± 12
NAIE06	
3- 32 mm	110 ± 9
24- 64 mm	78 ± 6
66-103 mm	82 ± 6

Calibration Intercomparison

Oxford-prepared Quartz. The AD (delivered by a gamma-ray source) was evaluated using the inclusion technique and the growth characteristics obtained by monitor normalization. The results are given in Table 2. The AD of 395 ± 22 compares well with the stated administered dose "in the region of 400 rad".

National Bureau of Standards (NBS)-irradiated TL Material. As discussed with the University of Utah it was found that the samples contained significant amounts of plagioclase feldspar (known to exhibit anomalous fading in the high temperature region of the glow curve) and after HF etching very little sample remained. Unetched material exhibited fading in the high temperature region of the glow curve. It was thus considered to be unsuitable for pre-dose and inclusion measurements on the same sample. However, using a sample that had received the HSiF treatment described above, it was possible to obtain a pre-dose evaluation (MA) of 42.5 ± 0.3 rad for the equivalent dose.

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