GS 1568

NATIONAL SCIENCE FOUNDATION
WASHINGTON, D.C. 20550

APR 1 0 1967

Dr. Froelich Rainey
The University Museum
University of Pennsylvania
Philadelphia, Pennsylvania 19204

Dear Dr. Rainey:

This will inform you that your proposal has been approved for support by the National Science Foundation under the title "Dating of Archaeological Evidence of Thermoluminescence."

I am attaching some materials relating to the conditions of the grant and our administrative requirements. Please review them carefully; if you have any questions about them, or if any questions arise during the course of the grant, do not hesitate to get in touch with us.

May we wish you the best of success in your research program.

Sincerely yours,

Richard W. Lieban

Richard W. Lieban
Program Director for
Anthropology

Enclosures (3)

Best Ralph

May 2, 1968

Dear Richard:

I am enclosing the report at the end of our first year with National Science Foundation Grant GS-1568 to assist in our development of thermoluminescent dating of pottery. You will note that Ralph and Han have very materially improved in the accuracy of thermoluminescent dating; particularly in the fine-grained pottery where the accuracy of dating compares very favorably with C-14 and we have no doubt now that this will be a very significant technique for archaeological dating.

Of course we have been primarily concerned with improving the method but I hope now to turn toward more dating of unknown periods which will then mean that archaeologists in general become aware of this new technique.

It is a very real satisfaction to report to the National Science Foundation our success in this project and as you know, we also have under separate grants perfected a more efficient magnetometer for archaeological prospecting. For your own information, I want you to know about the tremendous success we have had with the still newer type cesium magnetometer first tested in the Olmec country of Mexico during the past April. There will be a press release coming out on this within the next couple of weeks and I will certainly see that you get a copy of it. So, at least we have succeeded in two of our major attempts at developing new techniques in MASCA.

All the very best,

Froelich Rainey
Director

Mr. Richard Lieban
Program Director for Anthropology
National Science Foundation
Washington, D. C.

FR/jt

NATIONAL SCIENCE FOUNDATION
WASHINGTON, D. C. 20550

APR 1 0 1967

Dr. Gaylord P. Harnwell, President
University of Pennsylvania
Philadelphia, Pennsylvania 19104

GS-1568
Renewal of GS-1028

Dear Dr. Harnwell:

It is a pleasure to inform you that a grant of $40,700 is awarded to The Trustees of the University of Pennsylvania for the support of research entitled "Dating of Archaeological Evidence of Thermoluminescence." This research is to be under the direction of Froelich Rainey, University Museum. It is effective April 1, 1967, for a period of approximately two years.

The Foundation requires that this grant be administered in accordance with the conditions, policies, and procedures stated in "Grants for Scientific Research," June 1963 (as amended December 1963 and supplemented by Important Notice of January 24, 1966), Enclosure R-8 (Rev. 9/66), and the attached budget summary.

Please acknowledge acceptance of this grant under the above terms and include in your acknowledgment a reference to the grant number.

Sincerely yours,

John T. Wilson
Deputy Director

Enclosures

Masca

August 21, 1969

SCURLOCK

GS-2716

Dear Mr. Merritt:

It was a pleasure talking to you on the telephone the other day. I am answering you in reference to Mr. Scurlock's letter to Dr. Rainey of August 15. This is a continuation of a grant for thermoluminescence whose grant number was N.S.F. G.F. 1568, which expired in June of this year. The new grant, which is a renewal of G.S. 1568, becomes active the first of September, 1969. Since in Mr. Scurlock's letter he states that expenditure limitations will not be known until September or October, I am taking the liberty of submitting a budget for the amount indicated in the Foundation's letter to Dr. Harnwell of July 30, of which I am enclosing a copy.

Sincerely,

David Crownover
Executive Secretary

Mr. Anthony Merritt
Franklin Building

DC/c
Enc.

UNIVERSITY of PENNSYLVANIA

PHILADELPHIA 19104

Office of Research Administration

REAGAN A. SCURLOCK, *Director*

Franklin Building
3451 Walnut Street
(Area 215) 594-7293

August 15, 1969

Dr. Froelich Rainey
Director
University Museum

Re: NSF Grant No. GS-2716

Dear Dr. Rainey:

The notice of award of the subject grant for which you are Principal Investigator has recently been received by the University. This grant must be included under the expenditures limitation which has been imposed on the University by the National Science Foundation for the current fiscal year.

The preliminary limitation which has been established represents a reduction in expenditures on existing grants of approximately 42% with no allowance for new and renewal grants that will be awarded during the coming year. It is obvious that this limitation has created a critical financial situation for the University. However, it is anticipated that some relief will be forthcoming when Congress takes final action on NSF's appropriation for F. Y. 1970.

As a result of the above, the Vice Provost for Research has decided that the initiation of new NSF projects will be deferred until the final expenditure limitation for the University is known which will probably be in late September or early October. The Foundation has indicated its willingness to extend the period of those projects which are delayed as a result of the expenditures limitation.

Should you have any questions concerning this matter, please do not hesitate to contact us.

Sincerely yours,

Reagan A. Scurlock

Reagan A. Scurlock

RAS:jn

Nov. 66

UNIVERSITY OF PENNSYLVANIA
PHILADELPHIA, PENNSYLVANIA 19104

Title of Proposal

Thermoluminescent Dating of Pottery

Submitted to
National Science Foundation
Washington 25, D.C.

Principal Investigator: Froelich Rainey, Director, University Museum and MASCA

School: University of Pennsylvania Department: University Museum

Starting Date: 1 January 1967 Duration: two years

FUNDS REQUESTED

First Year: $17,701 Second Year: $23,011 Total: $40,712

Corporate Name of University: THE TRUSTEES OF THE UNIVERSITY OF PENNSYLVANIA
(A Pennsylvania non-profit corporation)

Contracting Office: OFFICE OF PROJECT RESEARCH AND GRANTS
3400 Walnut Street, Philadelphia, Pa. 19104

Date:_____ Approved:_____
David R. Goddard
Provost

Approved:_____
Froelich Rainey
Principal Investigator
Director, University Museum

_____
Otto Springer
Dean of the College

Thermoluminescent Dating of Pottery

ABSTRACT

Previous experiments with the thermoluminescent dating of pottery have illustrated that dating within a precision of $\pm$ 300 years is possible. The problem now is to find the causes of this remaining uncertainty and the means to reduce it. Experiments and studies of the effectiveness of the various types of natural and artificial radiations, of the variable susceptibilities of clays to radiation damage, and of the reproducibility of the measurement apparatus are planned. Also, many more samples are to be dated with emphasis upon the earliest pottery obtainable in order to assess the contribution to thermoluminescence of the cosmic ray background and for correlation with C-14 dates in the B.C. millennia.

I. Description of Proposed Research

Experiments with thermoluminescent dating of pottery have been conducted at the University of Pennsylvania since 1958. Until 1962 work was intermittent and devoted to the development of apparatus for the rapid heating of powdered potsherds--now 16^{o} C per second. In our next stage of work, supported with NSF funds, it was found that age correspondence based on thermoluminescent output and rate of alpha particle bombardment was extremely poor or non-existent, and at first was not improved by correction for variable susceptibilities by X-ray bombardment. Subsequent work has been devoted to finding the best way to correct for the variable susceptibilities of clay to radiation damage. Significant improvements in age correspondence has been obtained due to two observations. First, it was found that the identical sample that had been measured for natural thermoluminescence must be irradiated and remeasured for the susceptibility correction. Fortunately, our system of mounting each sample on aluminum foil with silicone oil enables this to be done. Secondly, it was observed that the glow curve of the artificially-induced peak contained unstable low temperature components which decayed within a period of two weeks. After this time, with the age calculation based on the ratio of the upper-half area of the natural and artificial glow curves and corrected for rate of alpha bombardment, we have obtained good age correspondence for series of potsherds of known age ranging from A.D. 300 to 5500 B.C. (Ralph and Han, 1966; Rainey and Ralph, 1966). The uncertainty in dating is of the order of $\pm$ 300 years. The problem now is to find the causes of this uncertainty and the means to reduce it.

Thermoluminescence provides a measure of radiation damage. In the case of pottery, alpha, beta, and possibly gamma particles from the traces of radioactive elements (mostly uranium, thorium and potassium-40) in the clays bombard other constituents and cause electrons to be raised to metastable levels. The firing of the pottery, however, supplies enough energy to enable these electrons

to return to stable levels in this semi-conducting material and as they do so, they emit photons. The thermoluminescent glow curve (light output versus temperature, see Fig. 1) provides, therefore, a measure of the accumulated radiation damage which is proportional to the time which has elapsed since the pottery was last heated, or, in most cases, since it was fired. For age determination it is necessary to measure also the rate of bombardment and to determine the susceptibility of each sample of pottery to radiation damage.

The evaluation of the effect of the bombardment in producing radiation damage is complicated by the fact that various types of particles are emitted from the traces of radioactive elements in clays. From the low rates of emission in clays and the relatively short exposure times for pottery, we estimate that the thermoluminescence is a result of the filling of lattice defects or traps rather than from the creation of new ones. For the displacement of atoms in materials, it is known that alphas, neutrons, and cosmic rays are more than a hundred times more effective than betas and gammas (Levy, 1966), but for the filling of traps, less is known about the effectiveness of each type of particle. At the recent NATO-USAF-sponsored Advanced Research Institute on Applications of Thermoluminescence to Geological Problems held in Spoleto, Italy from September 5-16th, 1966, several somewhat conflicting theories were presented. However, the general consensus was that in the decay series of U^{238} and Th^{233} (the isotopes which cause most of the radiation damage in clays), the betas are much more effective in filling traps than the alphas (Aitken, Tite, Fleming and Fremlin, 1966). Since the betas emitted in these series are proportional to the rates of alpha emission, it is still possible to determine their relative rates with alpha counters. But, if the betas are more important, it should be necessary to measure the contribution from K-40 (89% beta emission) and other possible beta emitters in clays. The determination of potassium contents provides a relative measure of the K-40. In the series of pottery from Iran (see Fig. 2),

potassium contents range from 2.14 to 3.29%, but the small deviations found were not consistent with the discrepancies in ages. More comprehensive measurements and a search for other beta emitters are now planned. Studies of ancient glass and other compounds with very low or non-existent uranium and thorium contents, and hence negligible alpha emissions, may help to elucidate these matters.

The uncertainty quoted for each age determination (represented by the vertical lines in Fig. 2) is the standard statistical deviation of the average of approximately 20 single runs of a sample. The correction for artificial thermoluminescence (induced by X-ray irradiation) helps to reduce this uncertainty. An example (shown in Table 1) illustrates that the deviation of the ratio of natural to artificial thermoluminescence is only $\pm$ 5% whereas the individual spreads were $\pm$ 20% and $\pm$ 17% respectively. The X-ray irradiation does, therefore, provide a correction factor for the variations in the susceptibilities of clays to radiation damage, but the uncertainty must be reduced to less than $\pm$ 5% for more precise age determination. At this stage in our progress, we are not sure whether the remaining uncertainty is due to true variations in the clays or to the non-reproducibility of our technique of measurement or to both. Experiments with more homogeneous materials are now in process.

At the conference in Spoleto, the attainment of comparable precision in dating attempts was reported by only one other laboratory--namely, the Research Laboratory for Archaeology and the History of Art, Oxford (Fleming, 1966). Their present technique differs from ours in that they separate out the quartz fraction (which contains most of the thermoluminescence) and they also use a beta source (Sr^{90}-Y^{90}) for artificial irradiation. Too few measurements were reported in Spoleto to ascertain whether or not their precision was better than ours, but interlaboratory collaboration will soon provide the answer. If their results are more precise, then their system should be followed here.

As far as we know from publications, reports at the NATO conference, and personal contacts, research in the thermoluminescence of pottery is being pursued by only two other universities, namely, Oxford and Wisconsin. Preliminary experiments only have been carried out at Wisconsin. At Oxford, the work has been carried on as thesis projects in physics for graduate students. As projects for theses in physics, it has been essential for them to investigate every fundamental aspect of the process even before many of these could be measured, identified, or explained adequately. Our goal has been to develop a workable method of dating pottery to facilitate fundamental archaeological research with the hope that when accurate determinations can be made, many of the basic questions and uncertainties may be resolved.

In summary, experiments and consultations are required for, at least, another year directed toward the improvement in the precision of the method. In the course of these it is expected that more will be learned about the fundamental mechanisms of this type of radiation damage. It is necessary also to date many more samples of pottery to determine whether or not other complicating factors remain. Emphasis will be placed in dating the earliest pottery obtainable (to about 6500 B.C. from Çatal Hüyük and Haçilar, Turkey and from Nea Nikomedeia, Greece). Contemporaneous carbon samples have already been dated in our Radiocarbon Laboratory. As seen in Fig. 2, there is a tendency for the earliest thermoluminescent ages (samples #1, #2, and #3) to be older than corresponding C-14 dates. Only one (#3) is significant at this time, but if the difference is real, it is possible that thermoluminescent dating will provide an extension of the known age time scale beyond the limit of dendrochronology (now 4400 B.C.), and thus a means of determining the magnitude and duration of the long-term change in atmospheric C-14 content (starting about 100 B.C. and going back in time, (Ralph, et al, 1965; Rainey and Ralph, 1966). Some of the differences between thermoluminescent and C-14 ages may be due to exposure of

the pottery to cosmic rays. The contribution from this source has, so far, been neglected but it may become significant for these longer times of exposure. Determinations and study of corresponding thermoluminescent and C-14 ages vs. known ages from 3000 to 4400 B.C., and also of the depths from which the potsherds were excavated may elucidate this matter.

Thermoluminescence has already provided an excellent means for the detection of fakes. Museums and private collectors are frequently faced with the problem of whether an object is what it is purported to be or a modern imitation. Even small cermaic figurines from times and cultures in scarce supply bring very high prices, in addition to the fact that scholars and students do not want to be fooled by studying an imitation. If the object has not been tampered with by recent heating, a minute sample scratched from the bottom and five minutes of laboratory time will reveal the truth.

References

Rainey, F. and Ralph, E.K., 1966, Archaeology and Its New Technology. Science, v. 153, pp 1481-1491.

Ralph, E.K., Michael, H.N., and Gruninger, Jr., J., 1965, University of Pennsylvania Dates VII. Radiocarbon, v. 7, pp 179-186.

Ralph, E.K. and Han, M.C., 1966, Dating of Pottery by Thermoluminescence. Nature, v. 210, no. 5033, pp 245-247.

Papers presented at the NATO-USAF Advanced Research Institute on Applications of Thermoluminescence to Geological Problems, held in Spoleto, Italy, September 5-16, 1966:

Aitken, M.J., 1966, Thermoluminescent Dating in Archaeology: Introductory Review.

Fleming, S.J., 1966, Thermoluminescent Age Studies on Mineral Inclusions Separated from Ancient Pottery.

Fremlin, J.H., 1966, The Relative Effectiveness of Different Radiations in Producing Thermoluminescence in Archaeologically Important Materials.

Levy, P.W., 1966, Application of Recent Radiation Damage Studies to Geology.

Ralph, E.K. and Han, M.C., 1966, Progress in Thermoluminescent Dating.

Tite, M.S., 1966, Some Complicating Factors in Thermoluminescent Dating and their Implications.

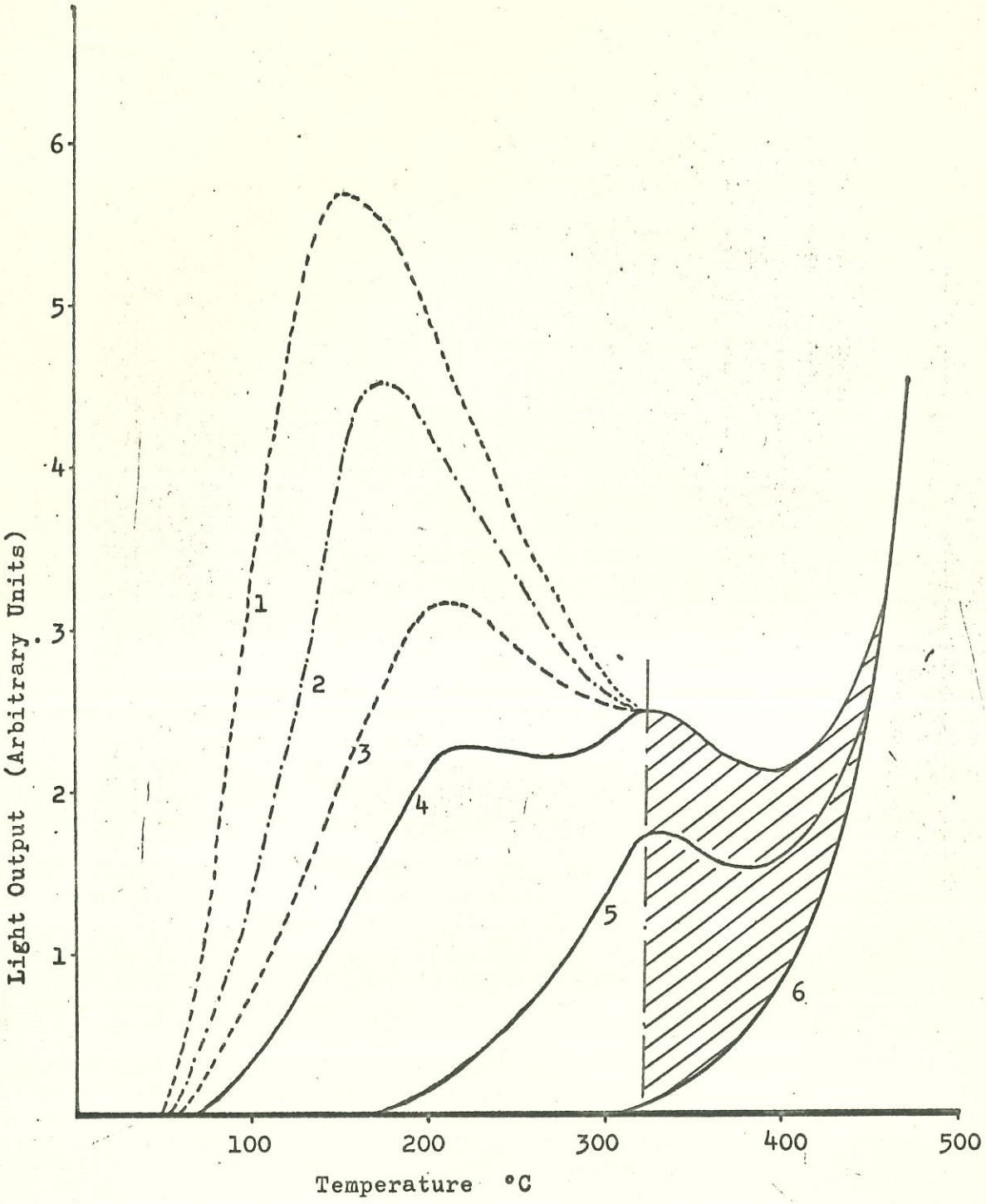

Fig. 1. Typical glow curves.
1. Artificial glow immediately after irradiation with X-Rays.
2. Artificial glow 24 hours after irradiation with X-Rays.
3. Artificial glow one week after irradiation with X-Rays.
4. Artificial glow two weeks after irradiation with X-Rays.
5. Natural glow curve.
6. Background (heat radiation only) curve.

The hatched regions are the significant areas for both the natural and artificial thermoluminescence.

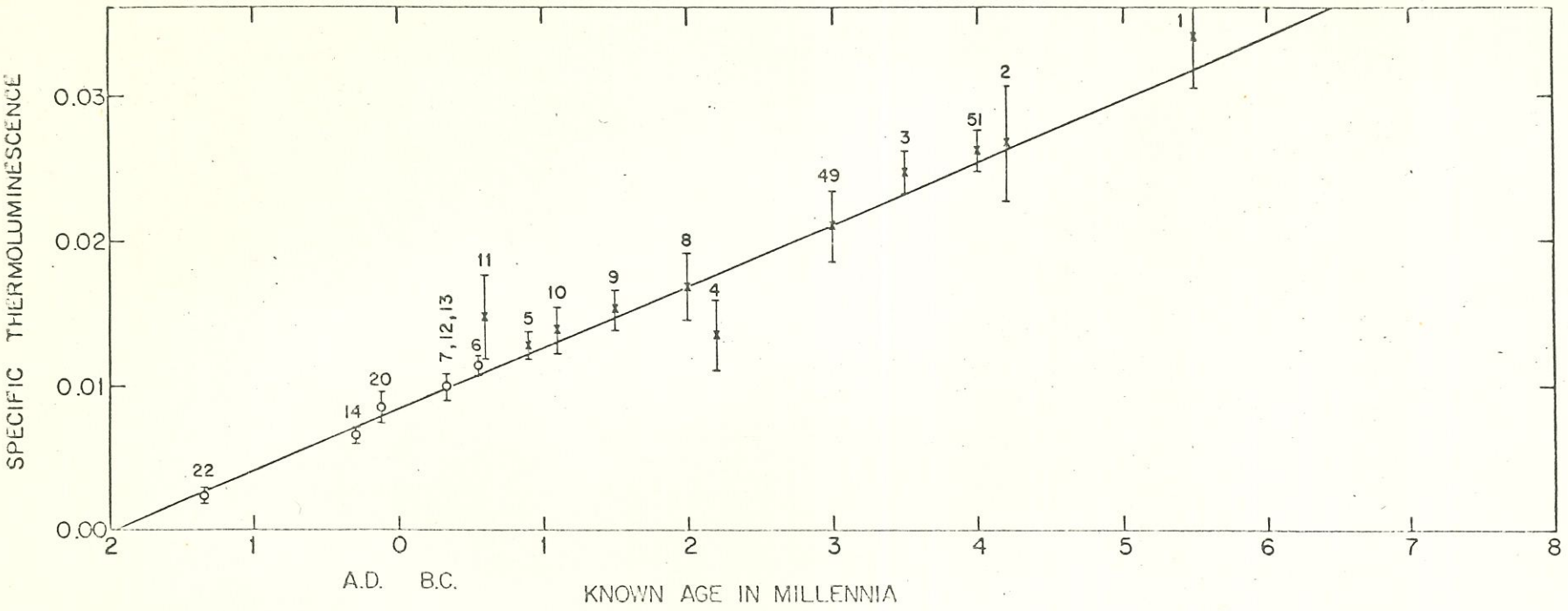

Figure 2: PLOT OF THE SPECIFIC THERMOLUMINESCENCE VERSUS DATED SAMPLES

Sample Provenience

1, 2, 3, 4, 5, 9, 10, & 11 : From Hasanlu, Iran

49 & 51: From Susa, Iran

8: From Baluchistan

6 & 7: From the plain of Sybaris, Italy (Excavated from 4 meters below the water table)

12, 13, & 14: From Torre Mordillo, near plain of Sybaris, Italy

20: From Le Muraglie, near plain of Sybaris, Italy

22: From Pecos, Southwest, USA

Notes

The slope of the line is based on the average specific thermoluminescence per year for the more firmly dated samples (nos. 6, 7, 12, 13, 14, 20, and 22), and are represented with circles. The other samples, identified with X's are dated by associated C-14 samples.

Sample No. 6. From: Plain of Sybaris, Italy

Age: 550 B.C. Alpha activity: 32.5 ± 1.2 cts/hr. ±(3.7%)

Run No.	Area of Natural Glow in cm^2 Nat. Th-L ± σ_2	Area of Artificial Glow in cm^2 Art. Th-L ± σ_4	Area Ratio $\frac{\text{Nat. Th-L}}{\text{Art. Th-L}} \pm \sigma_5$
1	17.3	46.5	0.372
2	14.0	37.5	0.373
3	14.9	43.5	0.343
4	17.8	51.0	0.349
5	17.0	48.0	0.354
6	20.3	57.0	0.356
7	21.5	54.0	0.398
8	13.0	39.0	0.333
9	18.8	49.5	0.380
10	22.0	57.0	0.386
11	16.0	44.4	0.360
12	26.0	69.0	0.377
13	16.1	46.5	0.346
14	20.3	51.0	0.398
15	25.0	63.0	0.397
16	23.5	61.5	0.382
17	23.0	60.0	0.383
18	16.5	45.0	0.367
Average	19.1 ± 3.8 ±(20%)	51.3 ± 8.6 ±(17%)	0.370 ± 0.019 ±(5.1%)

Calculation:

Specific Thermoluminescence = 0.370/32.5 = 0.0114

Percentage deviation of the Specific Thermoluminescence = $\sqrt{(5.1)^2 + (3.7)^2}$ = 6.3%

6.3% of 0.0114 = ±0.0007

Result: Specific Thermoluminescence = 0.0114 ± 0.0007

Table 1. Example of replicate runs and calculation of deviations for one sample. The uncertainties for each column represent $\sqrt{\frac{\sum(n_i - \bar{n})^2}{N - 1}}$

II. Facilities

During the past four years the work with thermoluminescent dating has been carried out in the laboratories of the Applied Science Center for Archaeology, (ASCA), University Museum, by Mark Han under the direction of Elizabeth Ralph. Funds are requested for the purchase of an X-Y recorder to replace the present one which has been borrowed, and other components as they become obsolete as well as supplies and materials. Also, funds are needed for the salary of the research chemist, and an assistant in the second year.

III. Personnel

Curriculae vitae of the following persons who are engaged actively in this program are attached:

Dr. Froelich Rainey, Principal Investigator, Director of ASCA

Miss Elizabeth K. Ralph, Faculty Associate, Associate Director of ASCA

Mr. Mark C. Han, Research Chemist in ASCA

IV. Budget

First Year: January 1, 1967 - December 31, 1967.

Salaries	Proposed NSF Funds	University Contribution	Total Project Funds
Froelich Rainey, Principal Investigator (10% time - 12 months)		$2,500	$2,500
Elizabeth K. Ralph, Faculty Associate (10% time - 12 months)		1,200	1,200
Mark C. Han, Research Chemist (100% time - 12 months)	$8,500		8,500
Total Salaries	8,500	3,700	12,200
Employee Benefits (8.3% of Salaries)	706	307	1,013
Overhead (37% of Salaries)	3,145	1,369	4,514
Equipment			
X-Y Recorder	2,200		2,200
Expendable Equipment and Supplies	2,000		2,000
Services			
Machine shop	250		250
Shipping, Printing and Duplicating, etc.	150		150
Travel			
To conferences and other research centers	750		750
TOTAL BUDGET -- First Year	$17,701	$ 5,376	$23,077

Second Year: January 1, 1968 - December 31, 1968.

Salaries	Proposed NSF Funds	University Contribution	Total Project Funds
Froelich Rainey, Principal Investigator (10% time - 12 months)		$2,600	$2,600
Elizabeth K. Ralph, Faculty Associate (10% time - 12 months)		1,250	1,250
Mark C. Han, Research Chemist (100% time - 12 months)	$9,000		9,000
Research Assistant (Half-time academic year, full-time summer months)	3,600		3,600
Total Salaries	12,600	3,850	16,450
Employee Benefits (8.3% of Salaries)	1,049	320	1,365
Overhead (37% of Salaries)	4,662	1,425	6,087
Equipment			
Component Replacements such as amplifier, voltage supply, etc.	1,500		1,500
Expendable Equipment and Supplies	2,000		2,000
Services			
Machine shop	250		250
Shipping, Printing and Duplicating, etc.	200		200
Travel			
To conferences and other research centers	750		750
TOTAL BUDGET -- Second Year	$23,011	$5,595	$28,602
TOTAL BUDGET -- TWO YEAPS	$40,712	$10,971	$51,679

V. Current Support and Pending Applications

A. Current Support

1. Field Expeditions

   a. F. Rainey--directs the expeditions to Sybaris and other sites in Italy which are supported by privately raised funds.

2. Radiocarbon Laboratory

   a. University of Pennsylvania annual support = $31,200. This includes salary of E. K. Ralph.

   b. C-14 measurements of Known Age Samples, Dec. 1964 to 1966, NSF GP-3778, $24,950 annually (E. Ralph, Principal Investigator).

3. ASCA

   Continuation of Dating of Pottery by Thermoluminescence, January 1, 1966-December 31, 1966, NSF GS-1028, $16,200. (F. Rainey, Principal Investigator, E. Ralph, Faculty Associate, M. Han, Research Chemist).

4. Department of Metallurgy in collaboration with ASCA.

   Research in Metallurgy and Archaeology, August 1965-1967, NSF P-17186, $12,800 annually (R. Maddin, Principal Investigator, E. Ralph, Faculty Associate).

B. Pending Applications

1. Thermoluminescent Dating of Pottery, 1966-1968, to be submitted to NSF, $51,679 requested (F. Rainey, Principal Investigator, E. Ralph, Faculty Associate, M. Han, Research Chemist). (Present proposal)

2. C-14 Measurements of Known Age Samples and Experiments with Thermoluminescence and Electron Spin Resonance for C-14 Detection, April, 1967-1969, to be submitted to NSF, E. Ralph, Principal Investigator.

These proposals are not being considered nor will they be submitted to other possible sponsors while they are being considered by the NSF.

9/24/68

MASCA BUDGET

1 YEAR

	Present Support	New Funds Required
Thermoluminescence NSF-GS-1568	$20,350	NSF Grant expires 4-1-69
Second Thermoluminesence apparatus for routine dating and authentication		$15,000
*Instrument Surveys-Archaeological Prospecting		
Winter, 1969, San Lorenzo, Mexico,		
Varian Associates personnel		$14,300
University of Pennsylvania personnel		$ 5,000
Spring, 1969, Turkey and Greece		$ 3,000
Lockheed Solid Domain Magnetometer		$33,000
Remote Sensing incl. aerial photography		$10,000
*Information Center - Harrison Fund	$ 6,000	$ 7,000
Conservation *Salaries-Discretionary Fund	$10,000	$15,000
Equipment & Supplies-Women's Committee	$ 2,350	$ 4,000
Chemistry Laboratory- Museum Budget	$ 7,100	$ 3,000
Radiocarbon Laboratory		
University of Pennsylvania	$28,550	
*University Museum	$ 3,100	$ 7,000
NSF-GA-993	$15,000	(NSF Grant expires 5-15-69)
Equipment for 3rd counter		$30,000
Additional Space-sealing and conditioning of half of BW8		$16,240
Total Funds Required	$92,450	$162,540 plus two NSFgrants if not renewed

*Supported now with emergency funds

NSF GRANT GS-1568

Dating of Archaeological Evidence of Thermoluminescence

Revision of Budget for 1968-1969 with 20% Reduction

Grant balance as of 6/30/68	$22,468
Less 20%	4,494
	$17,974

Expenditures

A. Salaries	
(3) Research Associate, Mark Han 12 months	$ 9,000
Employee Benefits (8.3% of salaries)	747
B. Permanent Equipment	1,500
C. Expendable Equipment and Supplies	2,000
D. Travel	750
E. Other	450
Sub-total	$14,447
Indirect costs (37% of Item A)	3,330
Total	$17,777

Please note:

From the balance of $22,468, approximately $3,500 has been spent since July 1, 1968. Therefore, if 20% is taken from this grant, there will be a deficit which may be as much as $3,000.

NSF GRANT GS - 1568

Thermoluminescent Dating of Pottery

FIRST YEAR REPORT

by

Froelich Rainey, Principal Investigator

Elizabeth K. Ralph, Faculty Associate,

and Mark C. Han, Research Chemist

April 25, 1968

The most significant laboratory improvement in the technique of dating pottery by thermoluminescence during the past year was the installation of a linearly programmed heating control system. With this, the heating cycle for the glow curve measurements is completely reproducible and the peak heights, representative of their true temperatures, may be identified. Also, because of the linear control, the deviation among replicate runs of single samples has been reduced significantly. The average deviation of the ratio of natural peak heights to artificial is now ±5%. The samples of known age which were plotted in Figure 2 of the grant proposal have now been measured, and a more precise calibration curve for the measurement of samples of unknown age has now been established.

The uncertainty in the age determination of fine grained pottery from modern times to 700 B.C. is now of the order of ± 100 years or less, but for coarser ware, mostly in earlier

time ranges, the discrepancies between thermoluminescence and carbon-14 dates of some samples continue to be much greater. Investigations of the reasons for this are now being carried out. The intrinsic problem is that most of the thermoluminescence is due to the quartz component of clays whereas the inherent radioactivity is contained to a large extent in the other components. With a few samples of coarse grained pottery, we are now doing magnetic separations of the components to learn more about these basic problems.

A few other technical and feasibility experiments which have been performed are as follows:

1) Tests of a few samples of obsidian indicate that obsidian does exhibit natural thermoluminescence. More experimentation is needed, but these preliminary tests indicate that it may be possible to date some of the volcanic eruptions from which obsidian was formed. This, in turn, may provide a method, supplementary to trace analyses, for determining the sources of obsidian artifacts. It may also be useful in geological studies of some of the comparatively more recent volcanic eruptions.

2) Preliminary experiments with glass containing high uranium contents showed measureable natural thermoluminescence glow curves. This confirms the early report by A. Liener of the University of Bern. These tests have been done in collaboration with R. Brill of the Corning Museum of Glass. Samples tested included late Roman, historical U.S.A., and synthetic

glasses. Further study is needed to determine the possibility of dating this type of material by the thermoluminescence method.

In spite of the many requests which we have received for dating samples of unknown age, most of these have been deferred in preference to the calibration and experimental studies during this past year. A few exceptions are the dating of:

P-T-101 Tile from Paul III's cupola in the Church of Santa Maria in Aracoeli on the Capitoline Hill.
Thermoluminescence date: 1700 ±100 A.D.

P-T-116 Pottery cup from Hacilar, Turkey.
Thermoluminescence date: 5100 ±600 B.C.

P-T-115 A piece of tile from a kiln at Porto Cheli, Greece. This tile showed abnormal susceptibility to radiation damage and could not be dated.

P-T-126 A piece of pottery found in a shipwreck underwater off the Bahama Islands that may be from the Santa Maria.
Thermoluminescence date: 1475 ±100 A.D.

Other exceptions are the testing of a few objects which have been suspected as fakes and for which curators of Museums have urgently requested our help. The testing of a statuette believed to be Etruscan from this Museum is described in an article entitled "Ultimatum to Terracotta Forgers" by E.L. Kohler (Expedition, Vol.9, no.2, 1967), of which four copies are enclosed. In this article reference is made (page 21) also of the testing of the large Etruscan

warrior from the Metropolitan Museum of New York and of its recent manufacture as determined by thermoluminescence. Another object which was found to be only 100 years old is the Diana from the St. Louis Museum of Art.

Three articles by Ralph and Han pertinent to this project are now in press, and it is anticipated that they will be published in time for submission with the final report.

NSF GRANT GS - 1568

Thermoluminescent Dating of Pottery

FINAL REPORT

by

Froelich Rainey, Principal Investigator,

Elizabeth K. Ralph, Faculty Associate,

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The emphasis in the first year of this two-year grant was upon the refinement of the technique of dating pottery by thermoluminescence. Considerable improvement was reported last year as the result of the installation of linearly programmed heating equipment and of various tests of pottery of known age. Also, experiments with obsidian and glass were reported, as well as a few surprising results of tests of authentication.

In the second year, our rate of production has been increased by the installation of two additional alpha counters. Since the inherent radioactivity of the pottery samples is so low, each one must be counted for, at least, 48 hours. This does not require much personnel time, but now with three counters, it will be possible to keep the alpha determination in pace with the glow curve measurements of the natural and artificial thermoluminescence.

During the past year 37 sherds of unknown age have been dated and 34 are in process. The latter have not been completed due to the previous backlog in counting their inherent radioactivities.

The samples dated include sherds from Germany, South Africa, Thailand, Nigeria, Turkey, etc. - almost all from archaeological sites for which no charcoal was found for C-14 dating. Therefore, for most of these, thermoluminescence is providing the only means of dating in support of the archaeological stratigraphy.

We have continued also to date series of sherds of known age, particularly in the earlier millennia B.C. A number of unresolved problems and discrepancies in ages remain to be solved, especially, with early coarse wares.

In collaboration with Dr. Paul W. Levy of Brookhaven National Laboratory, comparative measurements of thermoluminescence versus ESR (electron spin resonance) have been initiated. These experiments are two-fold. The first purpose is to determine the feasibility of measuring trapped charges in pottery by ESR as a possible means of age determination. The second is to find out if thermal annealing (a necessary step in the thermoluminescence, but not in the ESR measurement) affects the subsequent determination of A-TL (artificial thermoluminescence) in pottery. This latter determination is essential to correct for the varying susceptibilities of clays to radiation damage. To accomplish the second phase of this experiment, duplicate pairs of samples have been prepared - one of each to be used for ESR, and the other for TL.

Since no reprints are available, four xerox copies of the article by Ralph and Han entitled "Progress in Thermoluminescent Dating of Pottery" (published in Thermoluminescence of Geological Materials, D. J. McDougall, ed., 1968) are enclosed. Three other articles are still in press.