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Ancient TL

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"The most beautiful thing we can experience is the mysterious. It is the source of all true art and science."

Albert Einstein

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PRELIMINARY ANNOUNCEMENT - THIRD SPECIALIST SEMINAR

The Third Specialist Seminar on Thermoluminescence Dating will be held July 26-31, 1982 at Roskilde, Denmark. As before, the orientation will be towards those actively engaged in thermoluminescence and a limitation to seventy participants is envisaged.

Those who participated in the 1980 Seminar will be sent details when available; others interested in attending should write to Vagn Mejdahl, Research Councils' Archaeometry Project, Risø National Laboratory, DK-4000 Roskilde, Denmark.

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ON THE LOCAL AND INTERNATIONAL CALIBRATION OF BETA SOURCES FOR TL DATING

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Samples for interlaboratory β -source calibration have been available in the last couple of years from both M. Aitken (Oxford) and E. Pernicka (Austria). Both of these have been used for comparison with the Adelaide source, which had previously been calibrated by gamma-irradiated $\text{CaSO}_4\text{:Dy}$ (Harshaw).

The original calibration was made using CaSO_4 grains in the size range 75-180 μm . After annealing, the CaSO_4 sample (in bulk form as described later) was given 84.6 rad from a ^{60}Co source at the Royal Adelaide Hospital. A graphite parallel plate ionization chamber was used to measure the exposure to an accuracy of "better than" $\pm 2\%$. In order to achieve secondary electron equilibrium the sample was surrounded by a 3 mm layer of selenite (CaSO_4) which lends itself to cleavage into slabs of uniform thickness. Standard calculations such as those described in Attix and Roesch (1968) were then used to obtain the dose delivered to the grains.

There were three main peaks in the CaSO_4 glow curve. The first (i.e. lowest temperature) peak is susceptible to rapid fading and the third peak shows a pre-dosing effect. Hence irradiations with the β -source were aimed at matching the second peak at about 230°C. While this peak also shows a small predose effect on the high temperature side, it is possible to choose a range of 20° covering the top of the peak which is virtually free of predosing. There was no fading over a six-week period. About 3 mg of the irradiated CaSO_4 was sprinkled uniformly on to each 1 cm diameter stainless steel disc. Silicone spray was used to secure the sample. Measurements were made on twelve separate sample discs, with a sample standard error less than 1%. The relative stopping powers of CaSO_4 and SiO_2 suggest that the dose to quartz is a factor 1.02 ($\pm 2\%$) higher than the dose delivered to calcium sulphate at an electron energy of 0.8 MeV. This is about the mean energy from our ^{90}Sr source type SIPI from the Radiochemical Centre, Amersham. For the standard "high-mount" "on plate" source geometry that we use, the dose rate from the 5 mCi β -source was determined to be $22.2 \pm 0.6 \text{ rad min}^{-1}$ to quartz on stainless steel discs.

For the second calibration, eleven discs were prepared from the coarse grain fluorite (90-150 μm) supplied by Oxford and both peaks II and III were used for matching (Murray and Wintle, 1978 and Aitken, 1978). As Oxford does not quote an error for the gamma-irradiation, we assumed that it was 2%. An error of 2% was also used for the uncertainty in the conversion from dose-to- CaF_2 to dose-to-quartz (a factor of about 1.05). For our source geometry the dose to coarse grain quartz on stainless steel discs was determined to be $21.1 \pm 0.6 \text{ rad min}^{-1}$.

The final calibration involved the use of coarse grain (70-150 μm) quartz obtained from Dr. E. Pernicka. Once again, measurements were made according to instructions (Pernicka and Wagner, 1979). At least four discs were prepared from each of the five samples provided, each of which had had a different irradiation. Another four were prepared for each corresponding β -irradiation of the un-irradiated quartz. Our 40 mCi source was used for this calibration, so there is an additional error in the conversion to the 5 mCi source dose rate. The resulting calibration for the 5 mCi source was $21.9 \pm 0.5 \text{ rad min}^{-1}$. Bell (1980) suggests that this value may be about 5% too high. The weighted average calibration for the 5 mCi Adelaide source is $21.7 \pm 0.4 \text{ rad min}^{-1}$ when only the fluorite and CaSO_4 measurements are considered. This would suggest that the quartz calibration does not need to be reduced by 5%, although such a reduction would also be within the error limits.

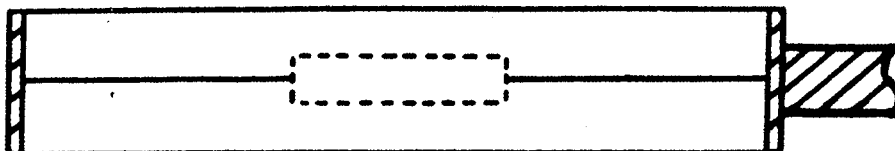
The results are shown in the accompanying table. The three separate calibrations agreed well, indicating that the use of any of the materials should provide an adequate measurement of the β -source dose rate. The Oxford-Adelaide comparison was included in M.J. Aitken's review of dosimetry calibrations at the 1980 Specialist TL Seminar in Oxford.

<u>Calibration By</u>	<u>Place of Irradiation</u>	<u>rad. min⁻¹</u>
CaSO_4	Adelaide	22.2 ± 0.6
Fluorite	Oxford	21.1 ± 0.6
Quartz	Vienna	21.9 ± 0.5

All strengths have been corrected to give the dose rate in February 1980.

In view of the issue raised by Bell above, it is worth enlarging on a couple of points connected with our gamma-ray calibration. Our irradiation cells (figure 1) are flat cylinders, 5 cm in diameter and 9 mm thick. They are made in two demountable halves with a 3 mm cavity in the middle to contain the material to be irradiated and are made of that same material: viz. selenite for CaSO_4 , pure silica sand mixed with a minimum quantity of silicone potting compound for SiO_2 and powdered natural fluorite bound with silicone for CaF_2 . (Our measurements with CaF_2 and SiO_2 are not reported here.) This construction automatically ensures that the sample dose is obtained under the correct conditions of electron-photon equilibrium.

The cells are symmetric and double sided so that they can be irradiated from two sides in turn by a ^{60}Co teletherapy unit. In this way the precise location of the sample with respect to the gamma-ray beam is not critical since the mean of the "up" and "down" readings is independent of small departures from the mean position to second order.



We wish to thank B.W. Worthley for assistance with the gamma-irradiations at the Royal Adelaide Hospital. The fluorite calibration samples were kindly supplied by M. Aitken and the quartz by E. Pernicka. The work was supported by the Australian Research Grants Committee.

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A METHOD FOR THE DETERMINATION OF TH-U CONCENTRATION RATIO

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One of the main problems in calculating the annual dose rate in pottery by thick-source α -counting (Tite and Waine, 1962) is due to the effect of sample reflectivity. Lack of information on the Th/U ratio may be another source of error, mainly in calculating β and γ contributions to annual dose-rates (Sasidharan et al., 1978). This work is intended to investigate the influence of reflectivity and to determine indicatively the Th/U ratio by a new method that needs only two single channel analyzers. The method is similar to that suggested by Pierson (1951) and Sanderson (1979), but we found it to work only by eliminating the influence of reflectivity.

Reflectivity

Huntley (1977) and Huntley and Wintle (1978) showed that reflectivity influences the height of pulses arriving to the photo-multiplier, so that a poorly reflecting sample gives pulses much smaller than a highly reflecting one. The discriminator setting adjusted so that the count rate is 85% of the zero-extrapolated count-rate of a Th sample (or 82% of U sample) could then be affected by reflectivity.

We had a number of Th and U samples with different values of reflectivity. For each of them we plotted the count-rates vs. discriminator setting. The straight lines of Figure 1 are fits of the data by the least square method (all lines are normalized to the zero-extrapolated value of count-rates). The samples are: Th-1 NBL standard n.109 (monazite sand) kindly supplied by Dr. Aitken, 100 ppm Th and 4 ppm U; Th-7 = 7540 ppm Th and 1490 ppm U obtained from Mineralogy Institute of Milano University measured by Ge(Li) spectrometry; U-1 and U-3 respectively 550 ppm U and 2074 ppm U from the Novazza Uranium mine in northern Italy (SIMUR-ENI propriety) measured by chemical analysis and Ge(Li) spectrometry; U-2 = IAEA standart S-8 (pitchblende) 1187 ppm U. U-2 and U-3 were also analyzed by neutron activation by CESNEF (Centro Studi Nucleari Enrico Fermi Politecnico di Milano).

It can be seen from Figure 1 that the threshold value at which the count-rate is 85% or 82% is different for different samples. After fixing a discriminator setting for a sample, the errors we found for other samples were ranging up to 5%. To eliminate the effect of reflectivity, we interposed between the sample and the ZnS(Ag) sheet, a thin (0.9 mg cm^{-1}) mylar foil covered with aluminium. As a consequence, a reduction of the effect of reflectivity was observed as can be seen in Figure 2.

Th-U ratio

On the basis of the work done using SCA and varying the discriminator setting, we used a MCA to verify whether U samples are different enough from Th ones to obtain, from the shape of the spectra, an indication of Th/U ratio in any pottery sample. As expected from the measures with SCA, the spectra are different depending on the reflectivity of each sample (Figure 3). At first sight, nothing can be said about Th/U ratio. Using again a thin mylar foil covered with aluminium, the effect of reflectivity is eliminated and the spectra of Figure 4 were obtained. All the spectra of U samples are quite similar, and the spectra of Th samples, as well, but there is an evident difference between any spectrum of a Th sample and a U one.

Figure 4 shows also that each spectrum can be divided into two different parts. The first (the shaded area in Figure 4) is quite similar in a U spectrum and in a Th one (when normalized); the second is significantly different in U and Th samples. We can write: $C_T = C_0 + C_1$, where C_0 is the shaded area and C_T is the total area (which corresponds to a well-defined percentage of counts from a Th or U sample that is no more than 85% for the former and 82% for the latter because of the presence of the mylar foil: in our case, this percentage is 44.3% for Th and 43.8% for U but it depends on the mylar thickness. Moreover, the ratio C_1/C_0 will be in a definite relation with the Th/U ratio, in Th samples being surely higher than in U ones. In Table 1 are reported the values of C_1/C_0 ratio for a set of samples whose Th/U ratio was known by γ spectrometry or neutron activation measurements. The lower value of C_1/C_0 for the U-2 sample could correspond to a non-equilibrium in U-Ra chain detected by γ -spectrometry and neutron activation.

Table 1

	Th/U	C_1/C_0
Th-1	25	1.70
Th-7	5	1.57
U-1	0	1.02
U-3	0	1.03
U-2	0	0.86

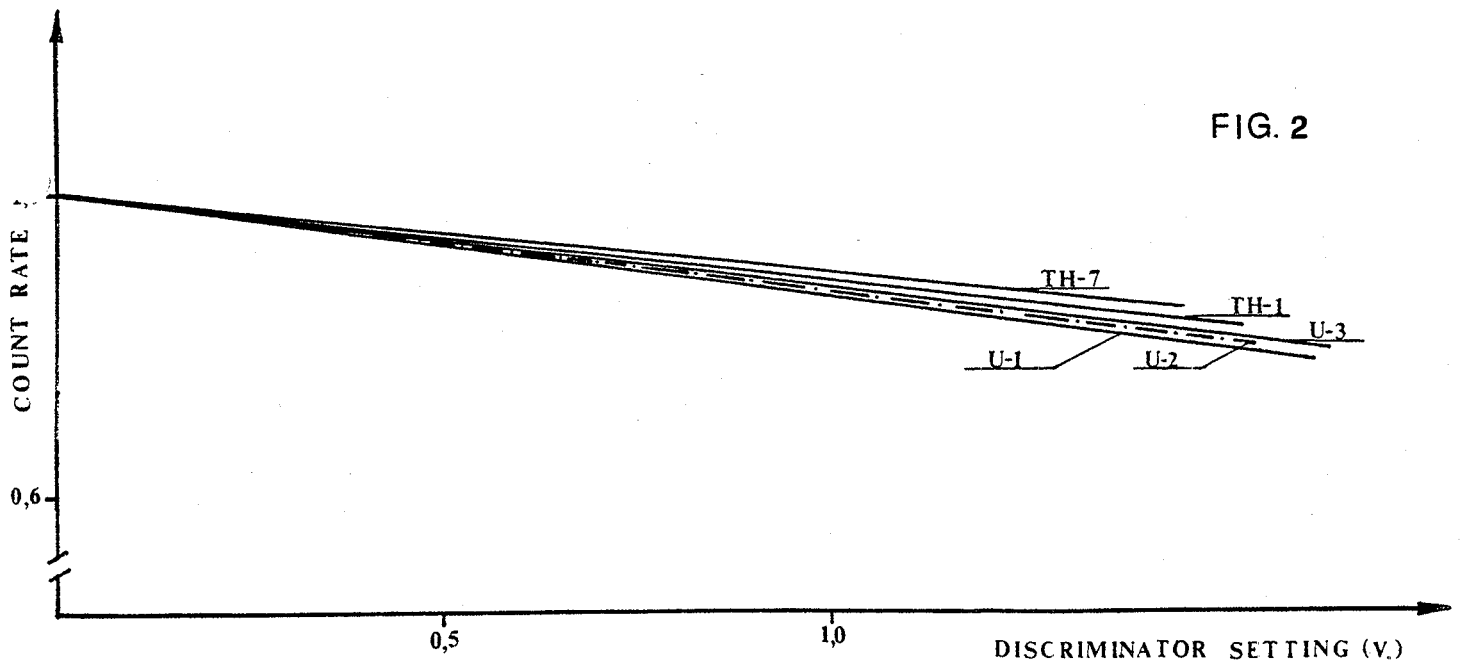
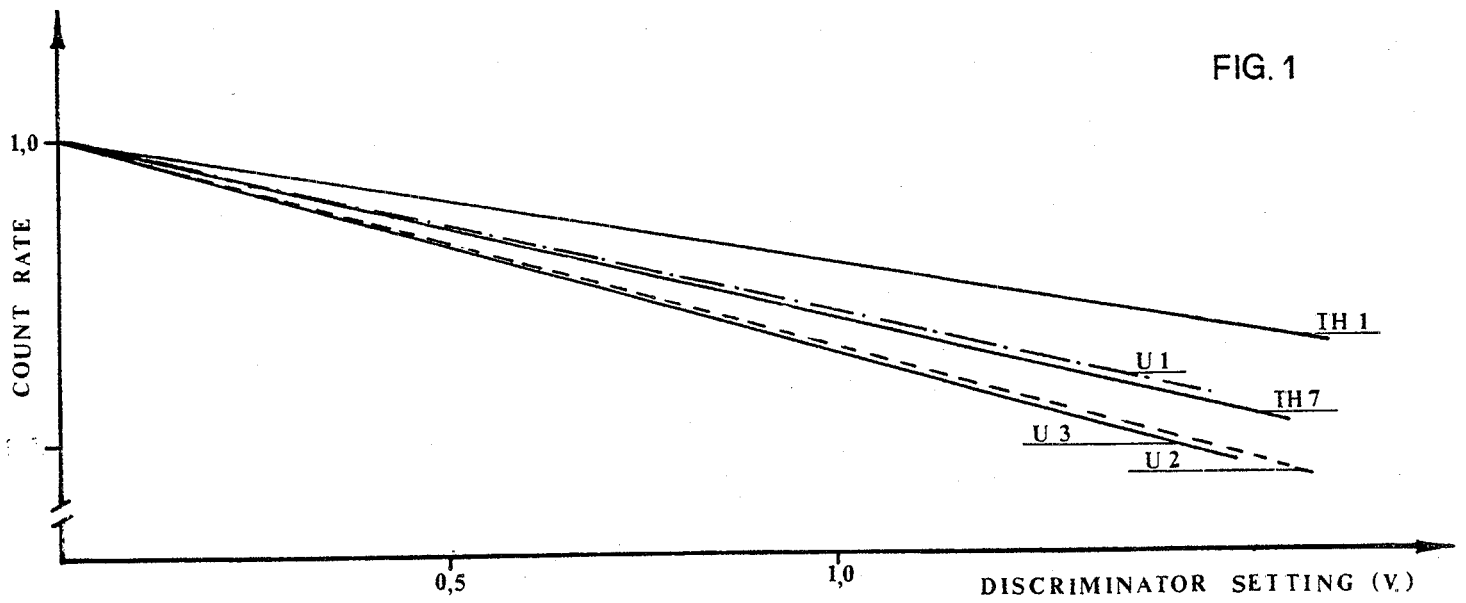
Conclusions

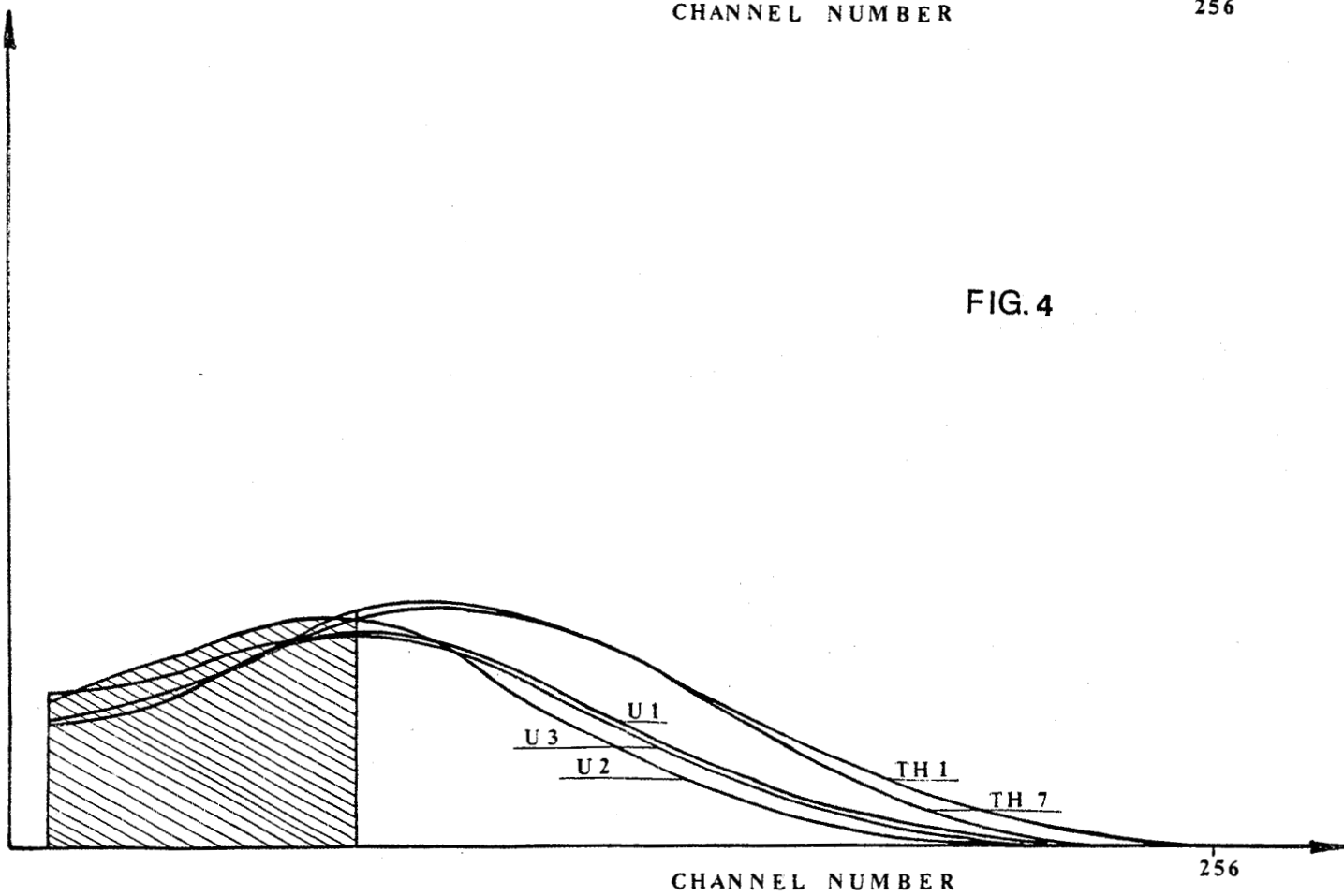
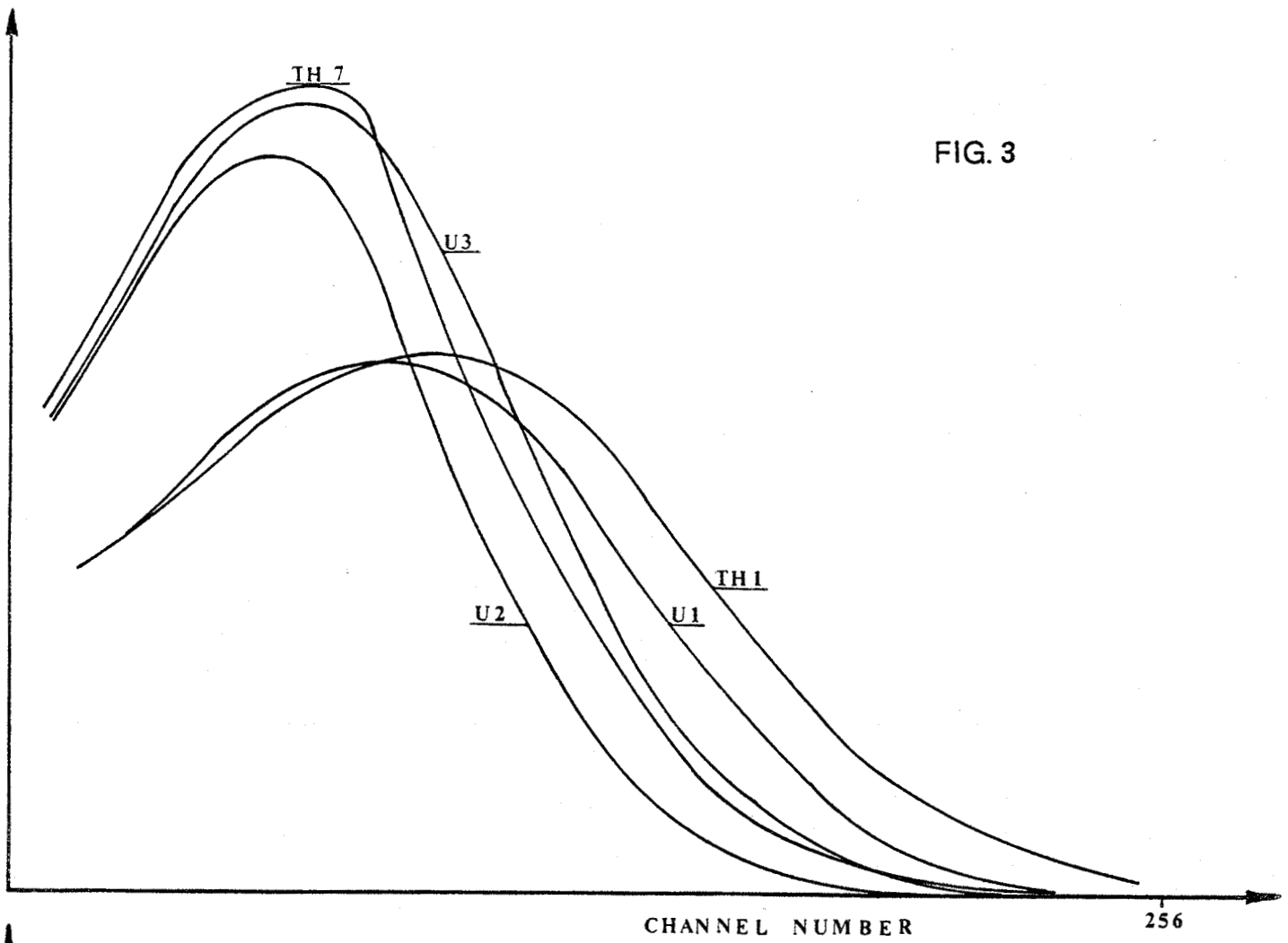
The possibility of having an indication of Th/U ratio by only two count-rates on the same sample seems to be promising, avoiding long measures and expensive instrumentation. As a matter of fact besides the usual instruments needed for α -counting on thick samples, this method requires only an additional SCA and an additional counter. Even if the calculated Th/U ratio obtained by this method has only a qualitative significance, it could be very useful in determining the β -contribution to the annual dose-rate of the sample and the γ dose-rate when only a small amount of soil is available. It is also useful to evaluate γ dose-rate when only an α -counting system is available.

The author is grateful to Dr. N. Cuomo for several useful discussions, to Mr. G. Ferrandi for technical assistance, and to Miss M. P. Pisoni for carrying on many of the measurements. He is also glad to thank Prof. G. Spinolo for his continuous interest and advice.

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ISOLATION OF FINE GRAINS BY DILUTE AQUA REGIA

Li Hu Hou

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In the fine grain technique of thermoluminescence dating the sample contains several kinds of minerals other than quartz. Hence the glow curve is composite, made up of TL peaks from several different minerals. For such samples, the glow curve often has a flat top as shown in Figure 1. To try and work with a single mineral component, we have devised a new method of sample treatment:

1. A 200 mg sample containing grains less than $74\ \mu\text{m}$ is obtained by sieving and put in the bottom of a dry 100 ml beaker. Approximately 20 ml of 15% aqua regia is added slowly. The mixture is stirred with a glass rod and then allowed to stand at room temperature for 10 minutes.

2. The sample is then centrifuged for 5-10 minutes. The supernatant liquid is discarded and the sample washed three times with distilled water. It is then washed with acetone and the samples are then prepared in the usual way.

After such treatment, the glow curve is dominated by the 375°C quartz peak, Figure 2. The change in the glow curve shape is thought to be due to the removal of certain minerals. This will be investigated further.

This method can also be applied to samples which have a low yield of fine grains when they are prepared in the conventional way by washing with acetone after crushing. In these samples, aggregates of quartz grains are formed due to the presence of binding agents such as aluminates, aluminiferous, ferroferric compounds and silicified tuff. This can be observed under a microscope. When aqua regia is applied, the aggregates break up because the binding agents are dissolved. Thus, the fine grains of quartz are released and may be measured in the usual way.

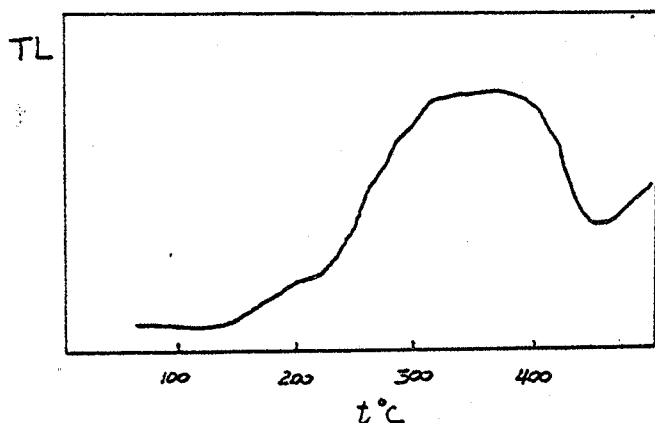


Figure 1. The original glow curve, the sample is prepared without aqua regia.

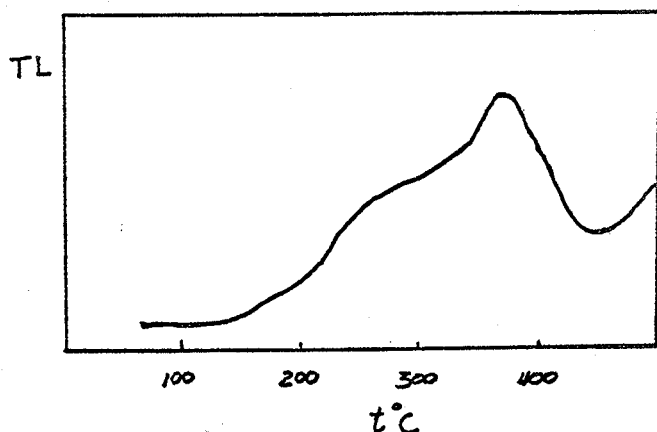


Figure 2. The glow curve, which the sample was treated by the aqua regia.

[Editor's note: Mention should be made here of other proposed acid treatments of fine grain samples. For example: 2% acetic acid to remove CaCO_3 (Huxtable, J., 1978, "Fine grain dating", PACT, 2, 7-11); Conc. Nitric/Conc. Sulphuric/Perchloric, mixed 1:1:2, used to remove clay grains in fine grain samples (Fleming, S. J., 1979, "Authenticity analysis using thermoluminescence", PACT, 3, 360-361).]

PHYSICAL RESEARCH LABORATORY TL DATES - 1981 (I)

D. P. Agrawal, N. Bhandari,
B. B. Lal* and A. K. Singhvi
Physical Research Laboratory, Ahmedabad 380 009, India

Following are the results of the first TL dating project undertaken by the Physical Research Laboratory. The samples dated were from a Ramayana associated site, Sringaverapura (Allahabad). The dates were assayed using fine grain technique. The site and experimental details will be published elsewhere (Agrawal et al., 1981).

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D. P. Agrawal, N. Bhandari, B. B. Lal, and A. K. Singhvi, Proc. Ind. Acad. Sci. (E) in press, 1981.

SRINGAVERAPURA (Dist. Allahabad, 25°31'N., 82°E.)
Pottery: Loc. Tr. SVP-1, Sq. YA-3, Qd-3

Sample No.	Layer	Culture	TL date (B.P. years)	Archl. estimate (B.P. years)	Radiocarbon dates (B.P. years)
PRL:TL:21	17	Red Ware - Pre-NBW	2660 \pm 280	2700-3000	-
PRL:TL:24(1)	17	Black and Red Ware - Pre-NBW	2769 \pm 400	2700-3000	-
PRL:TL:24(2)	17	Black and Red Ware - Pre-NBW	2690 \pm 280	2700-3000	-
PRL:TL:29	18	Black Slipped Ware - Pre-NBW	2743 \pm 300	2700-3000	2700 \pm 130 (PRL-669)
-	19	Black Slipped Ware	-	-	2855 \pm 200 (PRL-670)
PRL:TL:33(1)	19E	Ochre Colour Pottery - OCW	2900 \pm 380	Pre-3000	-
PRL:TL:33(2)	19E	Ochre Colour Pottery - OCW	2855 \pm 270	Pre-3000	-
PRL:TL:38(1)	19E	Ochre Colour Pottery - OCW	3015 \pm 280	Pre-3000	-

*Radiocarbon dates are based on $t_{1/2}=5730$ years and 1950 as the base year, while TL dates have the base year 1980. Thus there is a systematic difference of 30 years between TL and radiocarbon dates.

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READERS' CLUES AND QUERIES

A Possible Correlation between the Alpha Efficiency and the Anomalous Fading Characteristics

There appears to be some correlation between the alpha efficiency (α) and the anomalous fading characteristics of a phosphor. Thus quartz and $\text{CaF}_2:\text{nat}$ with low α value usually show no anomalous fading whereas $\text{CaF}_2:\text{Dy}$, $\text{CaSO}_4:\text{Dy}$ and K-feldspars with α values typically in the range (0.2-0.5) are known for significant anomalous fading characteristics. Correlation between the luminiscent mineralogy, the glow curve shape and anomalous fading characteristics have already been reported (A. K. Singhvi and D. W. Zimmerman, 1979, *Archaeometry* 21, 73) and it may now be useful to investigate, whether fine grain discs with low alpha efficiency indicate the absence of anomalous fading. I would like to investigate this in detail, and would appreciate receiving the following information from scientists involved in fine grain TL and TL dosimetry.

1. Sample details (site location); (In case of phosphors, please indicate preparation procedure)
2. Alpha efficiency (please specify α or k)
3. Anomalous fading with assessment procedures
4. Typical natural beta-induced and alpha-induced glow curve

A. K. Singhvi
Physical Research Laboratory
Ahmedabad 380 009, India

Research Position Available

A position is available in the TL laboratory at Simon Fraser University. The position is that of post-doctoral Fellow or Research Associate. Those without Doctoral degrees or those on leave from their present position are included in the latter category. Experimental experience with TL dating or with optical properties of solids is desirable. Please send applications, including a c.v. and names of two persons willing to provide references, to:

D. J. Huntley
Physics Department
Simon Fraser University
Burnaby, B. C., Canada V5A 1S6

Research Position Available

Physicist (Ph.D. or strong M. S.) with experience in Archaeological thermoluminescent dating techniques (predose and quartz inclusion) required to establish a well-equipped, experimental TL dating laboratory. Initial research emphasis will be the examination of individual components of the predose dating process with the aim of implementing a field dosimetry project for the estimation of cumulative external gamma-ray doses received by residents downwind of the Nevada Atomic Test Site. Duration of the project will be contingent upon the outcome of the pilot studies. Salary range \$20,000-\$30,000 depending upon experience and qualifications. Apply in writing to: Division of Radiobiology, Bldg. 351, University of Utah, Salt Lake City, Utah 84112, Attn: Dr. E. H. Haskell. Phone (801) 581-6600.

SOME RECENT BIBLIOGRAPHY

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TL LABORATORY SURVEY

To aide subscribers in visiting TL laboratories during their vacation/holiday time (or any other time for that matter), a survey of TL laboratories is being conducted to be published in the newsletter. The preliminary laboratory list below has been compiled merely to include labs from various geographic areas and is not intended to be a complete listing. In order to complete the list as well as obtain some indication of laboratory interests, a questionnaire has been attached to each subscription invoice. I would like to encourage each TL laboratory to take just a few seconds to fill in the questionnaire and return it along with their subscription remission. A tabulation of the responses and a final version of the map will be included in a subsequent issue of Ancient TL.

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A MAP SHOWING THE LOCATION OF SOME TL LABORATORIES
(NUMBERS REFER TO THE LIST ON THE PRECEDING PAGE)

