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DRICHTER

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"He that would enjoy life and act with freedom must have the work of the day continually before his eyes. Not yesterday's work, lest he fall into despair; not tomorrow's, lest he become a visionary." James Clerk Maxwell

IN THIS ISSUE . . .

INTRODUCTION

Ancient TL is a quarterly newsletter intended primarily to facilitate communications of helpful and practical information between researchers actively involved in thermoluminescence dating. The major subjects of contribution are expected to be experimental techniques and new equipment, TL data of various phosphors and minerals, and data and information on dosimetry and radioactivity determinations. Other topics may include lists of recent publications, announcements of meetings, lists of theses available, and readers' queries. Contributions accepted for inclusion will in general not be reviewed. A general guide for the suitability of contributions is that they should be of interest only to TL practitioners; articles of wider interest and importance should be considered instead for a journal such as Archaeometry. Subscriptions to Ancient TL may be obtained from the editor at a cost per annum (4 issues) of $4.00 (U. S.).

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INSTRUCTIONS FOR CONTRIBUTORS

Contributions should be sent to the editor at the letterhead address. To keep costs and editorial efforts to a minimum, manuscripts should be in camera ready form. To ease preparation efforts, the manuscript rules following have been kept to a minimum:

1. Text:
   Minimum width 6 inches (15 cm).
   Maximum width 7 inches (18 cm).

2. Include title, author(s), affiliation and address. (These will be retyped here for the heading.)

3. References. The format of Archaeometry is recommended. They may be placed at the end of the text, after a centered heading "REFERENCES", as in Archaeometry, or placed within the text. Acknowledgements of previous work in the field which will be well known to active TL practitioners are not necessary.

4. No footnotes.

5. Acknowledgements of funding agencies (if desired) may be placed as the last sentence in the text.

6. Tables and figures should be sent the same size as you wish them to appear in print. If small, you may insert them directly within the text. If large (18 cm maximum width), they may be on separate pages, and will be inserted by the editor in an appropriate place. Figures and tables, if self-explanatory or adequately described in the text, needn't necessarily have captions.

QUESTIONNAIRE RESULTS

A questionnaire to survey interest in Ancient TL was mailed to 67 individuals at 63 laboratories (for large labs, a questionnarie was usually sent only to the head, expecting he would pass on the information to others in his lab). So far, 42 persons from 17 countries have replied indicating they wish to subscribe (1 negative response). Thirty-four indicated they expected to have contributions to make in the next one or two years. The large extent of world-wide interest has been a very pleasant surprise. Topics listed for anticipated contributions included the following:

TL instrumentation
Sample preparation
Spectral measurements
Radioactivity measurements
(alpha counters and other techniques)
Radon
Spurious TL
Dosimetry problems
Source calibrations
Predose measurements
Failed TL dating projects
TL characteristics of teeth, speleothems, $CaCO_3$, feldspars, cherts, quartz, and other materials

EXPERIENCES WITH AN ALPHA COUNTER

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The alpha counter was constructed for determining the alpha particle emission rates from samples for thermoluminescence dating, and for determining uranium and thorium contents.

The basic technique is described by Aitken (1974). The sample is prepared by spreading a covering of the powder on a ZnS screen which in turn is placed in a sealed lucite container on a photomultiplier tube. Each alpha particle causes a large scintillation pulse in the ZnS which is easily detected and counted. Figure 1 shows a diagram of the overall apparatus.

The discriminator setting on the SCA was determined by plotting count rate vs. setting for a 1.02% U sample (Canadian Certified Reference Material BL-3 known to be within 90% of secular equilibrium) and adjusting the setting so that the count rate was 82% of the extrapolated zero-pulse height value. The data for this is shown in Figure 2. The 82% figure is used so that the conversion factors of Aitken and Bowman (1975) can be used later.

The corresponding figure for a sample of USAEC pitchblende No. 6 was determined to be 80.9 ± 0.3%.

In order to determine what kind of effects the upper and lower discriminator may have pluse height spectra were taken for several samples; these are shown in Figure 3. We attribute the differences in the spectra mainly to different reflectivities of the samples, thus a highly reflecting sample will give larger pulses than a poorly reflecting one. It would appear from this that it is necessary to determine what discriminator setting is necessary for each sample (one possible way to do this would be to measure the reflectivity since for most samples counting statistics and an unknown U/Th ratio would make the present method difficult).

To establish whether or not the counter was working as expected count rates were determined for several samples of known composition and converted to U and Th contents using the conversion factors of Aitken and Bowman (1975). These results were then corrected for variations of alpha-particle range with atomic number (A) using the Bragg-Kleeman rule (range $\propto \sqrt{A}_{eff}$, $\sqrt{A}_{eff} = (\Sigma Y_i/\sqrt{A}_i)^{-1}$ where Y_i are the weight fractions, Evans, 1955).

The results are shown in Table 1 where it is seen that for the two Canadian ore standards the agreement between the stated values and those we determined is excellent. For the U. S. standard which has a very high U content there is a discrepancy which we attribute to non-uniformity of the U distribution and/or inadequacy of the Bragg-Kleeman rule.

Table 1 also contains results from two obsidian samples which happened to be available. For one the results are satisfactory while for the other there is a disagreement, the cause of which is unknown.

I find it very pleasing that without any arbitrary adjustments one can

construct such a simple device for determining U and Th contents so accurately.

Dr. M. J. Aitken gave much useful advice which is very gratefully acknowledged.

REFERENCES

Aitken, M. J., 1974, Physics and Archaeology, 2nd ed., Oxford University Press.
Aitken, M. J. and Bowman, S. G. E., 1975, Archaeometry 17, 132.
Evans, R. D., 1955, The Atomic Nucleus, McGraw Hill, pp. 652-3.
Laidley, R. A. and McKay, D. S., 1971, Contr. Mineral and Petrol 30, 336-42.

TABLE I.

SAMPLE	Stated U	Stated Th	Measured Sample Count Rate	Measured Pair Count Rate per ks-cm^2
BL-2	0.453±0.005%	16 ppm.	572±4	---
BL-3	1.02 ± 0.01%	15 ppm.	1261±7	---
DL-1	41 ppm.	83 ppm.	7.58±0.04	0.44±0.01
#6	45.4%		60,200±1400	---
NCCC	5.6 ppm.	14.2 ppm.	1.10±0.03	.023±.004
NCWE	5.4 ppm.	13.6 ppm.	1.43±0.02	.021±.002

SAMPLE	Calculated U (uncorrected)	Calculated Th (uncorrected)	A_{eff}	Corrected U%
BL-2	0.471±.004%	---	21.56	0.454±0.004
BL-3	1.038±.005%	---	21.74	0.996±0.005
DL-1	≥38±2 ppm.	81±6 ppm.		
#6	49.5±1%	---	43.6 30.4	33.5 (A from stated U) 36.5 (A self-consistent)
NCCC	≥5.3±0.3 ppm.	12.7±2.0 ppm.		
NCWE	≥8.7±0.6 ppm.	10.3±1.5 ppm.		

NOTES FOR THE TABLE

BL-2, BL-3, DL-1 are Canadian Certified Reference Materials obtained from the Mines Branch, 555 Booth Street, Ottawa K1A 0G1 who also supplied detailed composition data for calculation of A_{eff}.

BL-2 and BL-3 are stated to be within 90% or better of equilibrium. The equilibrium status of DL-1 is not known.

No. 6 pitchblende is from the New Brunswick Laboratory of ERDA, Box 150, New Brunswick, New Jersey, 08903, U.S.A. This was assumed to have A_{eff} = 20 apart from uranium oxide.

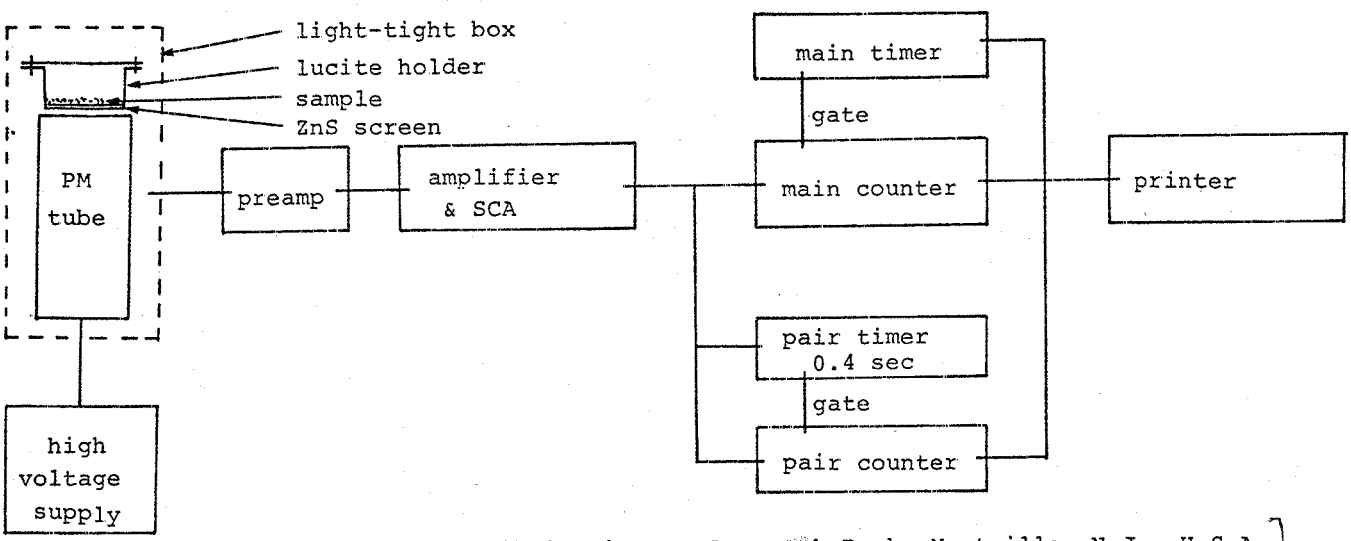
NCCC and NCWE are obsidian from Newberry Caldera, Oregon, Cindercone and Western East Lake flows respectively. U and Th contents are from Laidley and McKay (1971).

Uncertainties quoted are associated with counting statistics and sample areas; no account is taken of the (unknown) uncertainties in the conversion factors.

The pair count rate measured includes a significant portion of random pairs; the Th count rate is determined by subtracting these and multiplying the remainder by 26.9. The latter figure is calculated allowing for geometry, the random emission time, a random (up to 0.1s) delay time inherent in the 719 timer, and the 6 α particles in the Th chain.

A measured background count rate of 0.11±0.02 c/ks for the 13.6 cm^2 ZnS discs has been allowed for; this was measured with a clean lucite disc laid on the ZnS screen. Without this disc the rate was five times larger, presumably due to radon in the air of the chamber.

When a sample is not determined to be in equilibrium only a lower limit on the U content can be calculated and this is so indicated.



components and settings used

ZnS screen	Wm.B.Johnson, Research Park, Montville, N.J., U.S.A.	
HV power supply	Hewlett Packard 6515A	(900v)
PM tube	EMI 9656B	
Preamp	Ortec 113	(C=0)
Amp & SCA	" 490A	(gain=3x4, disc LL=0.4, UL=10)
Main timer	" 773	
Pair timer	" 719	(0.4s)
Counters (2 of)	" 772	
Printer	" 777	
Nim Bin	" 701/2	

Figure 1
The Alpha Counting Apparatus

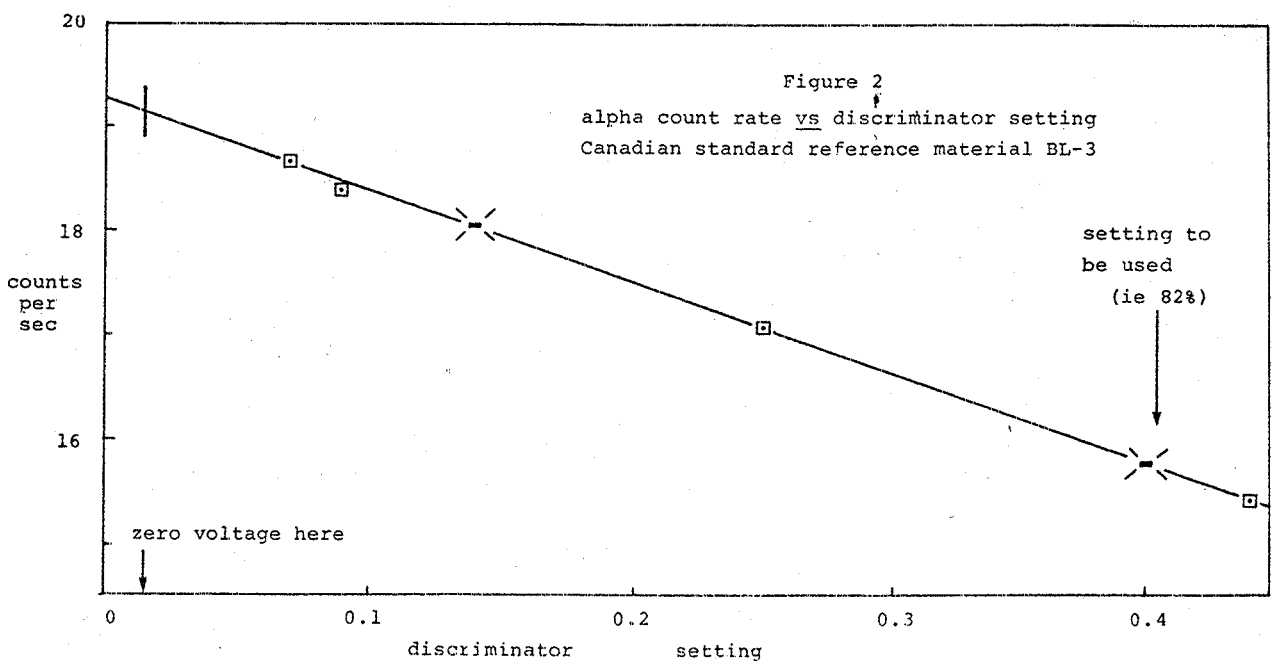
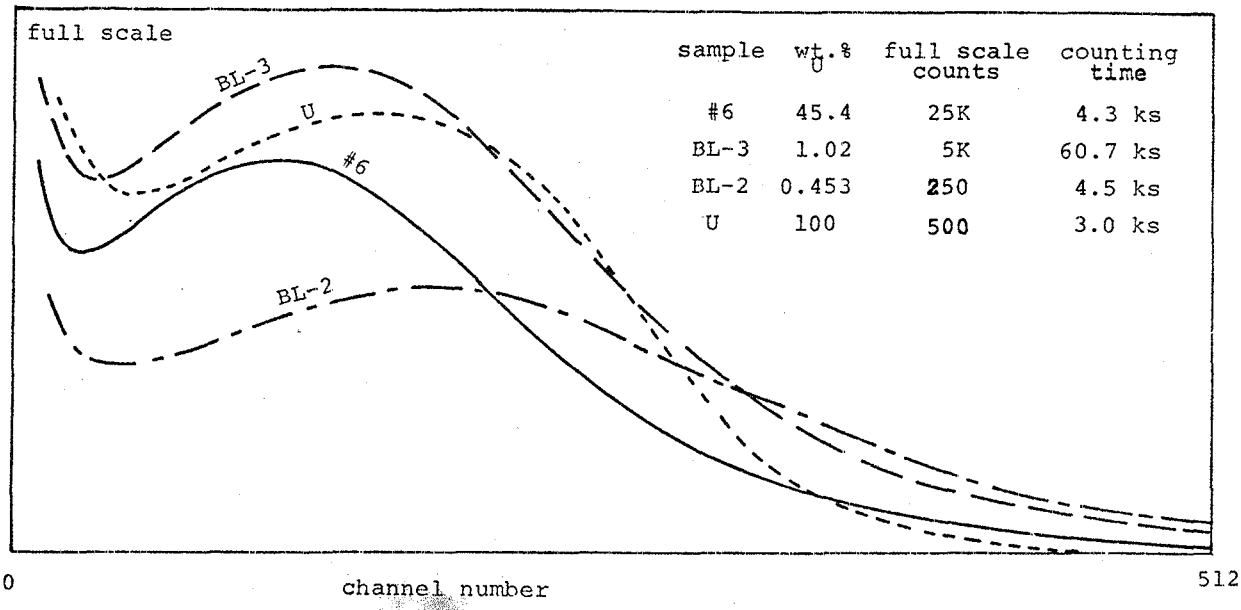


Figure 2
alpha count rate vs discriminator setting
Canadian standard reference material BL-3

Figure 3: Pulse-height analysis



CLEANING QUARTZ GRAINS

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A simple method of cleaning quartz grains using HF acid is described in the following note (see also, Langmyhr and Sveen, Anal. Chem. Acta, 32, 1, 1965). This technique is quite useful as it allows one to use only a single beaker, thereby reducing the chance of losing crystals. Furthermore, when etching is completed, addition of an $AlCl_3$ solution pacifies the harsh HF solution and eliminates the problem of precipitated flourides.

Sieved grains (up to a few hundred milligrams) are placed in the bottom of a dry 100 ml Nalgene polypropylene beaker. Approximately 5 ml of 49% HF acid is slowly added. The mixture is carefully swirled and then allowed to sit at room temperature for 30 minutes. At the end of this time, approximately 50 ml of 25% $AlCl_3$ solution is added. (This solution is easily prepared by placing the contents of a standard one pound jar of $AlCl_3.6H_2O$ crystals in a one litre bottle and filling it with distilled water.) After swirling, the beaker is placed in a boiling water bath and the solution is slowly stirred using a Teflon covered stirring bar with a hot-plate stirrer.

After a few minutes the cloudy solution will become clear (sometimes slightly yellow), and the cleaned grains can be retrieved by filtering with a glass frit and vacuum apparatus. When the last of the solution has been drawn off, the crystals are washed twice using distilled water and acetone. After drying, the grains can be loosened and removed from the frit by gently tapping over a piece of weighing paper.

The procedure may be changed for larger or smaller samples by simply scaling the listed quantities.

HINTS FOR THE REDUCTION OF SPURIOUS TL

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The use of ovens that can be evacuated and then filled with "oxygen-free" inert gas for the TL heating has generally reduced spurious TL levels to negligible levels. However, some fine-grain samples from ceramics are still found to be obviously spurious (indicated by the nonexistence of a plateau and the characteristic gradual increase in TL with increasing temperature above ~ 300°C). And frequently one wonders if a given sample is emitting a few per cent spurious TL, not quite enough to upset the plateau but nevertheless introducing perhaps as much as a 10% error in the date. Recently, we made some measurements with a low noise system (less than 5 cps black body plus dark count at 350°C) and found spurious signals of 5 to 50 cps from all of the following:

1. 1 mg samples of "clean" 100 μm grains of quartz and zircon
2. Mica
3. Aluminum, brass, nichrome, and silver

We have had success in reducing these spurious levels by increasing the purity of the atmosphere in the oven. This was accomplished by means of two filters; one on the vacuum line to stop back diffusion of pump oil vapor into the TL oven, and one on the inert gas (argon) line to scavenge oxygen and water vapor from the inert gas (see Figure 1).

The coaxial trap is from Veeco Instruments, Inc., Terminal Drive, Plainview, N. Y. (with offices on all continents except Africa), Model VS-120, $138.00. The trap's metallic absorbent removes pump oil very efficiently and is claimed to work maintainance free for many years. The trap slightly increased pumping time to ~ 100 microns (because of reduced pumping speed), but below 100 microns evacuation was more rapid than without the trap, indicating that considerable oil diffusion had been occurring. After installation of the trap, we also found it possible to remove the beaker of P_2O_5 desiccant from the oven with no apparent change in evacuation rate.

The oxygen/water-vapor trap housing is a molecular sieve trap (also from Veeco, Model TR-101, $175.00). It is filled with a mixture of molecular sieve beads (supplied with the trap) which absorb water vapor, and a reagent "Ridox" from Fisher Scientific Co., U. S. A. (with branches in Germany, Switzerland, Mexico, and Puerto Rico) No. R-30, which scavenges oxygen. The trap housing contains a heater for activating the molecular sieve material at 200°C. The Ridox regent is activated simultaneously at the same temperature by flowing a regeneration gas (4-6% hydrogen in an inert gas, e. g. helium, a non-explosive mixture) through the housing. The product data sheet from Fisher gives the details for calculating the oxygen reduction to be expected and regeneration procedure. For our particular trap, holding 30cc of Ridox, a flow rate of 1 ℓ/min, and assuming the gas initially contains 5 ppm oxygen, 95% or more of the oxygen should be removed for a flow period of 80 hours. Then the unit is regenerated.

The benefit of the oxygen trap was measured to some extent by use of an oxygen meter (Teledyne, P. O. Box 70, San Gabriel, Cal., 91776, Model 311). Although in principle capable of measuring oxygen levels to ~ 0.1 ppm, the particular meter we were using was limited to a ~ 1 ppm by a small leak in the intake manifold. Without the oxygen trap, a level of 3.5 ppm was measured directly from the argon tank, and the same with the meter connected on the outlet of the TL oven (after first evacuating the oven, then refilling with argon). (Incidentially, the argon tank came with a guarantee of <0.5 ppm oxygen.) With the oxygen trap in place and activated, the output from the oven read~ 1 ppm, and as mentioned above was believed to be the limit of the meter. No improvement was seen in oxygen level by flushing the gas, as opposed to simply filling the oven after evacuation. Even heating a fine-grain disk to 450°C, with a static gas, the oxygen level still measured 1 ppm. The importance of evacuation was demonstrated by flushing the oven at 3 ℓ/min for 5 min. from room pressure without first evacuating the oven; giving an oxygen level of 3000 ppm.

The effect of the two traps on TL from a 1 mg sample of our "spurious standard", a finely ground limestone sample, is shown in Fig. 2. The traps appear to have given a sustantial reduction in the spurious level. However, they are not a cure-all; some spurious TL still remains. Similar effects were seen with fine-grain samples from a potsherd. The spurious levels from 1 mg samples of quartz and zircons were reduced from ~ 30 to < 3 cps.

As a final hint, small spurious signals (~ 30 cps) from aluminum pans were eliminated by replacing them with pans made from gold foils. Attempts to gold plate aluminum and brass pans were not successful; after heating to high temperatures the gold coating disintegrated, although a more sophisticated plating might be more successful.

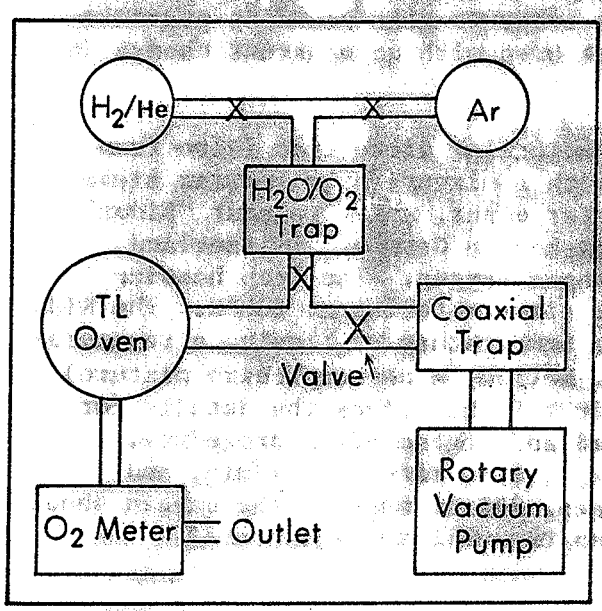

FIGURE 1

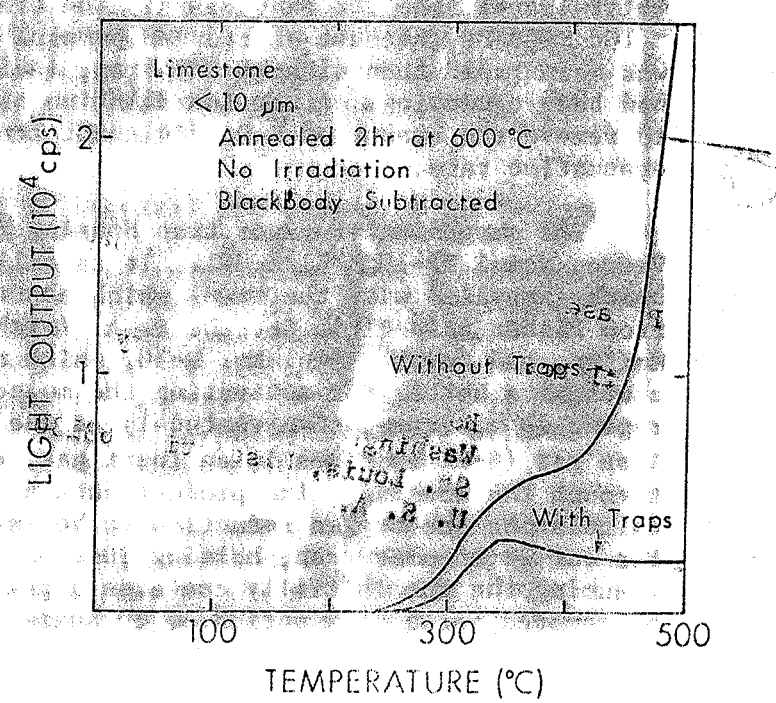

FIGURE 2