ADVANTAGES OF Ge(Li) AND PURE Ge DETECTORS IN GAMMA SPECTROMETRY MEASUREMENT OF U, Th, AND K

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Introduction

Direct gamma spectrometry provides a practical method of analysis of natural samples for U, Th and K, and has been used for many decades now in geochemical investigations. The high resolution semiconductor (Ge(Li) or pure Ge) detectors in use in the last 15 years or so have made this method of analysis more precise and less cumbersome than NaI(Tl) spectrometry. Smith and Wollenberg (1973) discuss in detail the U and Th estimation in geochemical samples using Ge(Li) spectrometry. The gamma spectrometric method using NaI(Tl) detectors has also been recently employed in TL applications (Meakins, et al., 1979; Proszynska, et al., 1982).

Advantages of Ge(Li) or pure Ge spectrometry

Compared to NaI(Tl) spectrometry the semiconductor detectors have the following advantages: (i) Due to the high resolution the specificity is excellent. To illustrate, an energy window for U measurement by NaI(Tl) of 1.62 to 1.9 MeV includes 8 gamma lines due to Th-232 (daughters) with an absolute intensity of 5.9% as against the 35.9% intensity for the 2.61 MeV gamma-ray. The K-40 energy window, 1.35 to 1.55 MeV, contains 7 lines (12.2% absolute intensity) due to U-238 and 5 lines (3.8% absolute intensity) due to Th-232. Single peaks specific to either element are easily evaluated by Ge(Li) spectrometry by simple addition of peak channel contents followed by a linear background subtraction. To obtain meaningful peak counts in the NaI(Tl) spectrum one must perform a least squares analysis using the information content of all the channels naturally requiring a computer. (ii) Samples with high Th/U or U/Th cannot be assayed for the lower concentration element (U or Th, respectively) by NaI(Tl) spectrometry. For example a thorium ore sample with Th/U of 30 was easily analysed for the U content by Ge(Li) spectrometry (Smith and Wollenberg, 1973), whereas this is altogether not possible with a NaI(Tl) detector. (iii) The simultaneous estimation of U or Th, in the case of Ge(Li) (or Ge) spectrometry, through photopeaks due to different daughter products provides an internal check as well as information on any disequilibrium in the series. Various gamma energies useful for this purpose are listed in Table 1 along with the nuclide names. It is seen that these spectrometers make possible direct estimation of U-238. Coles, et al. (1975) could detect 0.2 ppm of U-238 by direct estimation using a Compton-suppressed Ge(Li) spectrometer. It is also seen from Table 1 that it is sufficient to calibrate and measure only up to about 1.6 MeV. The lower energy lines from Tl-208 and Bi-214 could be used for Th and U estimation instead of 2.61 MeV and 1.76 MeV. This is advantageous since the detector efficiencies at the lower energies are appreciably higher.

Our system description and detection limits - In our laboratory off-shore core samples were measured using a Ge(Li) detector having a 16.5% relative efficiency and a resolution of 2.2 keV for the 1332 keV gamma of Co-60, coupled to a 1024 channel analyser. TL dating on these samples was done and is reported elsewhere (Sadasivan, et al., 1981). The detector had a lead shield of 7.5 cm thickness on the sides and 3 cm at the bottom. Just above the detector and covering half the top area (48 x 48 cm) there was 10 cm of lead while the remaining area was covered by a 1.2 cm thick steel plate. Typical background counts in relevant peaks are listed in Table 2. The gamma spectrum of a core sample is shown in Figure 1.

The peaks used for analysis are shown underlined in the figure. The detection limits for U, Th and K estimation in this system are given in Table 3 along with those obtained in our earlier system consisting of a 12.4 x 10.0 cm NaI(Tl) detector. The minimum detectable level is taken as three times the standard deviation of the background under the peak, accumulated for the same duration as the sample counting time. For the Ge(Li) system, the backgrounds under U or Th peaks have been taken as that which are obtained in a spectrum of Th standard containing 10 ppm of Th and in a spectrum of U standard having 5 ppm of U, respectively. The sample weights in the Ge(Li) system were about 300g while they were about 2 kg in the NaI(Tl) system. The detection sensitivity in Ge(Li) system is more than adequate for geological and environmental sample analysis. It must be remembered that with high U or Th the quoted detection limit for Th or U has no relevance in the case of NaI(Tl) detector.

It is possible to lower the detection limits for Ge(Li) detector since these are now available with very much higher active volumes (efficiencies) and also by effecting further reduction in background by better shielding. Thus samples of even less than 100g could be conveniently measured for the U, Th and K contents by direct spectrometry. Even with the set-up described above we could measure K in some archaeological specimens of total weight less than 10 grams (Nambi, et al., 1979).

In conclusion, direct spectrometry using semiconductor rather than NaI(Tl) detectors are more suited for U, Th and K estimation especially in TL dating applications where the available sample volumes are small. If gross alpha measurements are inadequate then the Ge(Li) or Ge detectors should be used.

Table 1

Series	Gamma Energy (keV)	Nuclide	Series	Gamma Energy (keV)	Nuclide
U	46.5	Pb-210	U	1001.1	Pa-234
	63.3	Th-234		1764.7	Bi-214
	186.0	Ra-226	Th	583.2	Tl-208
		U-235		3911.2	Ac-228
	352.0	Pb-214		968.9	Ac-228
	609.4	Bi-214		2614.5	Tl-208

Table 2

Peak Energy (keV)	Bkg/50000 Sec.
186.0	220
238.6	985
352.0	212
583.2	350
609.4	229
911.2	112
1120.4	374
1459.1	585

Table 3

Detector:	12.5 x 10.0 cm NaI(Tl)	16.5% rel. eff. Ge(Li)
Sample weight:	2000 gram	300 gram
Element	Detection limit	
U	25.0 μg	60 μg
Th	35.1 μg	270 μg
K	98.6 mg	200 mg

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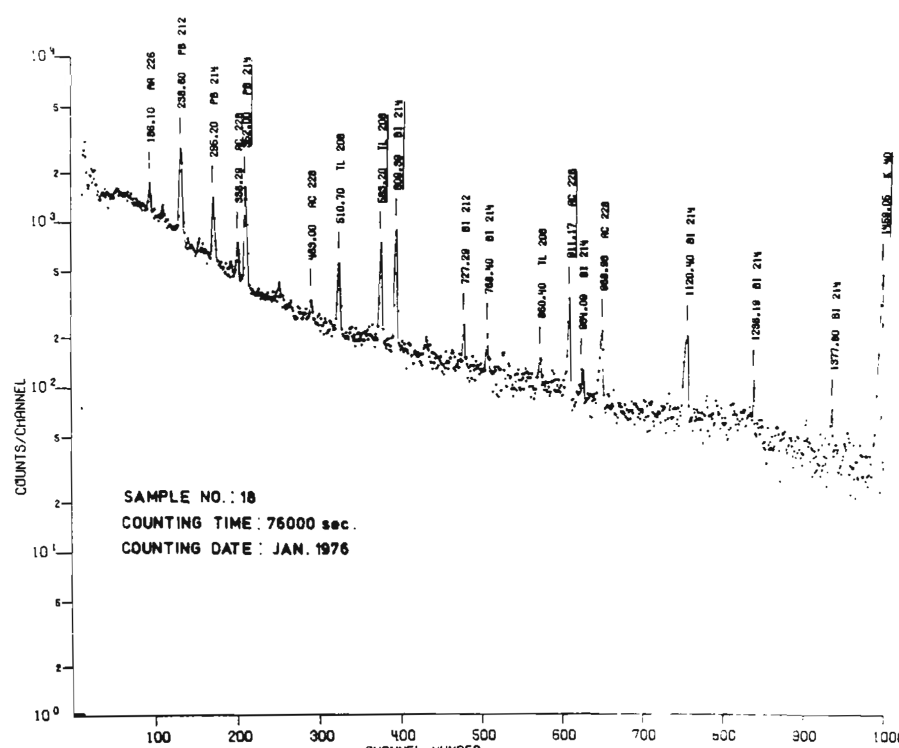

FIGURE:1. Ge(Li) SPECTRUM OF A DEEP CORE SAMPLE. ENERGY MARKINGS ARE IN KeV

(Compare this spectrum with the NaI(Tl) spectrum in Meakins et al.,1979)

APPLICATION OF QUARTZ THERMOLUMINESCENCE TO THE UNDERSTANDING OF SOIL PROCESSES*

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In recent years the luminescence of minerals has found ever growing use in solving several geological problems, such as the diagnostics of minerals and the dating of the physical age of one or another geological complex. We have attempted to evaluate the influence of soil processes on the intensity of the thermoluminescence of quartz, the essential mineral of Estonian soil-forming rocks. It was assumed that as the result of weathering in the soil, even in grains of such a resistant mineral as quartz, the diffusion of impurities along structural channels becomes easier, and may lead to a change in the intensity of the TL of a mineral, as the latter is rather sensitive to structural changes.

For these purposes five Estonian soil sections in fluvioglacial sands were studied. In 4 cases the soil was podzol, and in one case it was brown soil. Samples were collected from all genetic horizons. For the quartz separation we used our own technique (Hütt et al., 1977): a fraction 0.1-0.16 mm was obtained by sieving and from this fraction minerals with density 2.61-2.62 g/cm^3 (light fraction) and 2.63-2.67 g/cm^3 (heavy fraction) were separated by heavy liquids. In order to get a monomineral quartz sample and to remove the contaminated surface layer the grains were etched in HCl and HF. The above treatment eliminated the influence of the surface layer and the TL was thus associated with internal processes due to the defects in the quartz lattice.

We studied the heavy and light fractions of quartz in order to explain the occurrence of the great amount (up to 40% from the bulk sample) of light quartz, although according to the literature quartz with a density below 2.63 forms only 0.1-4% of the quartz in magmatic rocks and Phanerozoic sandstones (Katz and Simanovich, 1974).

The results obtained permit the following conclusion to be made. Light and heavy fractions are indistinguishable according to their TL properties. It does not show that they are of the same origin, however, it eliminates the possibility that the light quartz might be of soil origin.

In soils the intensity of the TL of quartz is subjected to regular changes - in the upper part of the section it is usually much weaker than in the lower part (see fig.). An abrupt change is observed at the upper boundary of the BC horizon. The BC horizon serves as a transitional area between the part of the section where the soil-forming rocks have undergone great changes due to soil processes, and the part of the section where no changes have taken place in the soil (C horizon, curves 32, 33, 81). This change is distinct in better developed soils whereas in less differentiated soils (curve 31) it is almost unnoticable. The 6th section serves as an exception, where the TL intensity shows an abrupt increase in the upper part of the A2 horizon.

*[Editor's note: this article is a translation of the Russian paper originally appearing in Esti NSV Teaduste Akadeemia Toimetised. 31. Koide Geoloogia (1982, Nr3, 117-118). Special thanks are extended to Helle Kukk for translation and D. J. Huntley for editing.]

The lm homogeneous layer of soil-forming sediments is practically of the same age. In the control-section comprising lm layer of fluvioglacial deposits untouched by soil processes no changes in TL intensity were found.

The content of radioactive elements is constant throughout the whole section: y-spectrometric studies by A. Molodkov (Inst Geol Acad Sci ESSR) showed the constancy of U, Th and K content in the samples. Such factors as cosmic radiation etc. may cause some increase of TL in the near-surface part of the sediments.

Thus, the decrease in TL intensity of quartz in the near-surface part of soil-forming rocks may be ascribed to soil processes, taking place in the soils studied in an acid medium with pH 3.4-4.4., and resulting in various degrees of weathering of the quartz.

The data obtained show that the TL intensity of quartz associated with specific quartz lattic defects may serve as an additional criteria for the evaluation of the intensity of the effect of soil processes on minerals. Microscopic methods which have been used so far for the study of the changes in the grain surface do not always reflect the actual degree of weathering of minerals.

The author wishes to thank G. Hütt for scientific discussions and E. Sheremet for conducting the TL measurements.

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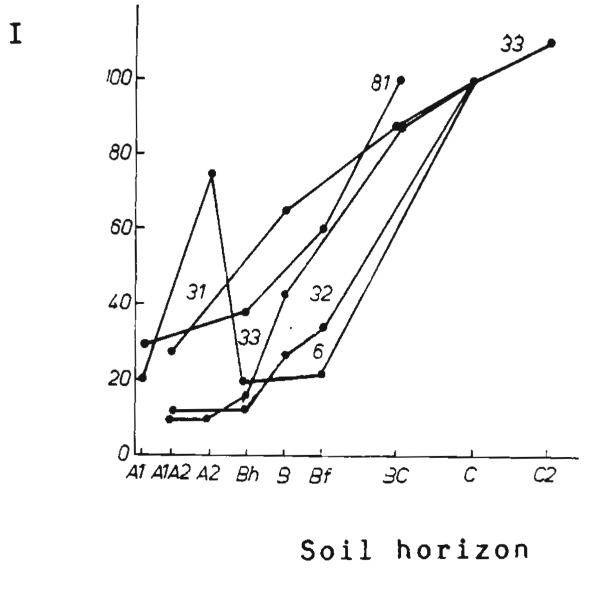

Figure: Change in the intensity of quartz thermoluminescence (I) in sandy soils from fluvioglacial sediments. from Estonia. 6, 31, 32 and 33 - podzols, 81 - brown soil.

TL DATING SERVICE AT THE DURHAM TL LABORATORY

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We have established a commercially based TL ceramic dating service with the aid of a grant from the Nuffield Foundation and expect it to be fully operational this summer. The service work will proceed in parallel with existing dating research in the laboratory and may incude TL research work not specifically related to archaeological ceramics. The Service has been initially structured to cater for the needs of British archaeologists, although we think that the fundamental approach would be appropriate to most TL dating laboratories. Two levels of accuracy are offered as part of a two tier service. The first, <u>survey dating</u>, will offer limited accuracy in the region of $\pm$ 20% (overall accuracy, 1σ), intended to provide chronological markers for a site and to test the suitability of samples for further TL measurements. Subject to satisfactory survey results, samples from a site may be considered for a <u>dating programme</u>, which offers the currently accessible range of accuracy of between $\pm$5 - $\pm$10% (overall accuracy, 1σ) for single dates. A minimum number of sherds from levels that cover the range of occupation of the site will be recommended for such a programme. We hope that the availability of low cost dating within a short processing time - we expect to better one month for survey dates - will prove attractive. For a limited period we will be offering three survey dates for L100; thereafter L60 for single survey dates. It will provide us with the opportunity to rapidly screen samples for high accuracy dating and provide a convenient first stage at which to halt work should problems be evident. While the cost of survey dating will be within normal excavation budgets, the cost of a dating programme will be beyond most. However, an important element of the Service will be to assist the archaeologist with a grant proposal for a research dating programme based on survey results and its relevance discussed in the light of archaeological evidence.

We consider that a two tier system is necessary to achieve adequate turnover, and thus commercial viability. However, it also has the potential to provide TL data on a much wider variety of ceramics than would be the case for specialized research projects. Survey dating is not intended to serve solely as a prerequisite for dating programmes and TL dates for early, middle and late phases of a site would provide a useful aid to the interpretation of site chronology.

If you would like further details of the Service, please write to us.

DURTL THERMOLUMINESCENCE DATING & RESEARCH SERVICE

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THERMOLUMINESCENCE RESEARCH IN INDIA: A REVIEW OF APPLICATIONS TO ARCHAEOLOGY, SEDIMENTS AND METEORITES

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Although several groups in India specialize in research on thermoluminescence as a phenomenon, only three institutions have active applications programs. Three more institutions have definite plans to initiate research on thermoluminescence application. This review is intended to provide a glimpse of activities of these institutions.

Bhabha Atomic Research Centre, Bombay (Dr. C.M. Sunta/Dr. K. S. V. Nambi)

The Health Physics Division, Bhabha Atomic Research Centre, Bombay, has a strong group on thermoluminescence research, active for almost two decades. The primary mandate of the group is personnel radiation dosimetry, survey of natural radiation environment and development of new TL phosphors for gamma and mixed-field dosimetry. The group has provided an excellent survey of natural radiation field on the monazite rich Kerala Coast (SW-India) having analyzed TLD data on 2,500 dwellings and 10,000 persons. The group has now initiated a TLD survey of natural radiation fields throughout the country. Another important contribution from this group has been the detailed studies on the role of rare earth impurities doped in a variety of phosphors ($CaSO_4$, CaF_2, etc.). Research in TL applications have included archaeological dating, depositional rates of beach sands, dating of authigenic minerals such as gypsum, and mineral prospecting. A particular mention may be made of a new approach developed by this group for estimating the firing temperature of materials based on temperature dependence of predose sensitization.

Indian Institute of Technology, Kharagpur (Dr. I. K. Kaul)

Another institution, where TL application was also initiated more than two decades ago is IIT, Kharagpur. The group has to its credit, one of the first ever TL dating results, where they reported dating of smoky quartz crystal formed within a radioactive mineralized zones. Owing to doubts on the validity of TL in dating long term events, the group has since concentrated in studies on minerals and on factors cardinal to dating long term events. The group has also proposed use of TL sensitivity and glow curve shapes as an indicator of the diagenetic fabric in the case of deep sea carbonate oozes.

Physical Research Laboratory, Ahmedabad (Dr. A. K. Singhvi)

Installed during 1978-79, the activities of this group are (1) Archaeological dating, (2) Dating of sediments, (3) Thermal and irradiation history of meteorites. Of the eight archaeological sites dated by the group the dating of Ochre Colour and Pre-Northern Black polished ware pottery from Sringaverapura, has helped assign a date to the antiquity of Indian epic Ramayana to 750BC. Similarly, dating of Megalithic pottery to 1120 BC is significant since they ascribe the megaliths significantly older antiquity than believed hither-to. In another study, it was shown that, in marbles, low temperature (76^{o}K - Room temperature) TL characteristics and their annealling behavior are correlatable to their provenance.

In the area of sediment TL, the group has reported two new applications of thermoluminescence in, (1) the dating of dune sands and (2) glacier dust and has plans to extensively apply TL to the understanding of the dynamics of dunes wind regimes and the antiquity of Indian Thar Desert. The group has also dated several loessic horizons in Kashmir valley.

The research on Lunar and Meteorite TL has been aimed to understand the ablation mechanisms, orbital parameters, etc. of a meteorite body and the group has described a new mechanism for the assymetric TL depth profiles in some meteorites, based on their oriented passage through the earth's atmosphere.

Among those who have recently initiated research in TL the emphasis of the Department of Physics, Kurukshetra, will be towards solving some of the archaeological problems and that of the M. S. University, Baroda, will be to apply TL in quaternary studies.

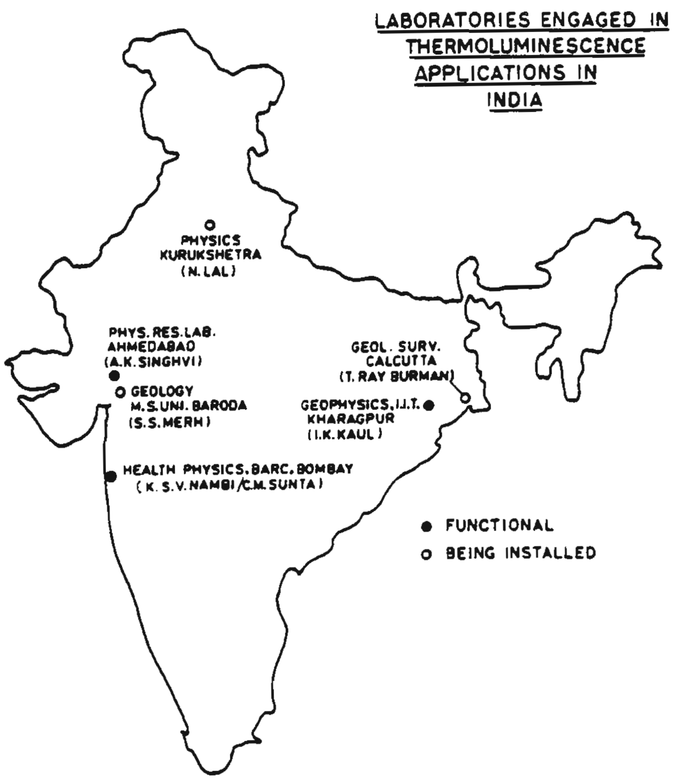
Much of the information on Indian TL work can be obtained from the following sources:

1. Proceedings of the National Symposium on Thermoluminescence and its Applications, Bhabha Atomic Research Centre, Bombay, 1975.

2. Bulletin of Radiation Protection: Special issue on Thermoluminescence: Dosimetry and Applications, Vol. 2 (4), 1979. (Ed. K. S. V. Nambi, Published by Indian Association for Radiation Protection, BARC, Bombay 400 085).

3. Proceedings of 1st, 2nd and 3rd Specialist Seminars on TL/ESR dating held at Oxford (1978), Oxford (1980), and Helsingor (1982).

4. Natural Radiation Environment (Ed. K. C. Vohra et al 1982) Wiley Eastern Ltd.



[Editor's note: The preceding is a condensed version of the original poster paper appearing at the XV Pacific Science Congress, Dunedin, N.Z.]

PONTIFICIA UNIVERSIDAD CATÓLICA DE CHILE TL DATES, 1983(II)

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The TL dates on pottery from two sites: "Chiu-Chiu 200" (Second Region) and "Parque La Quintrala" (Santiago, Metropolitan Region), were measured in 1982. The pottery was crushed and washed 3 hours with HCl at 50°C. The quartz was sieved to obtain grains with 80-120 microns diameter. The details of the experimental apparatus have been reported in an earlier paper (Concha et al., 1980). Beta irradiation was carried out with Sr-90 source, 10mCi (nominal) calibrated in 1981 with LiF powder samples, irradiated and non-irradiated, supplied by Dr. Gilmartin (Brookhaven National Laboratroy, Upton, New York). The annual dose was determined using the revised data of Bell (1979). The Th, U and K_2O concentration values, both sherd and soil (stone-free), were measured by the Neutrónic Activation Department of the "Comisión Chilena de Energía Nuclear". Santiago. Saturation water content was calculated for sherd and soil of the Parque La Quintrala and no moisture content was considered for the Chiu-Chiu's sherds (desertic zone). 15 mrads/yr was used as the cosmic radiation dose-rate. The alpha dose-rate was assumed zero. The equivalent dose is the average of the values obtained by extrapolating first and second glow growth curves. Anomalous fading was not studied in any case. The error estimates were made using the method proposed by Aitken (1976), but the uncertainties σ_3 (due to the stone content of the soil) and σ_7 (radon emanation) were assumed zero. All the dates are in B. P. and 1980 was used as the base year. Errors are given within parenthesis.

Acknowledgements

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lisis crítico comparativo del cementerio Parque La Quintrala. Revista Chilena de Antropología, 3, 41-56.

ARCHAEOLOGIC SAMPLES

A. CHIU-CHIU (Province El Loa, Second Region, 22°20′S, 68°39′ W), Chile.

This site corresponds to the so called "alfareria temprana" period (Chilean Northern region). The context date is about 2800 years B.P. (Department of Anthropology , University of Chile, Santiago)

UC-TL-6: 2950(---, ±240) 970 B.C.

Pottery: Ch-200-I, pulido negro, depth: 0.30-0.50 m.

Comment- Natural dose: 1150 rads (I=10), δQ=5%, plateau ≅ 70°C.
Annual dose: 0.39 rads/yr, sherd water = 0.

UC-TL-7: 2850(---, ±230) 870 B.C.

Pottery: Ch-200-III, pulido café, depth: 0.30-0.50 m

Comment- Natural dose: 940 rads (I=150), δQ=5%, plateau ≅ 70°C.
Annual dose: 0.33 rads/yr, sherd water = 0.

UC-TL-8: 2950(---, ±260) 970 B.C.

Pottery: Ch-200-IV, alisado negro, depth: 0.30-0.50 m.

Comment- Natural dose: 945 rads (I=85), δQ=4%, plateau ≅ 70°C.
Annual dose: 0.32 rads/yr, sherd water = 0.

B. PARQUE LA QUINTRALA, LA REINA (Province Santiago, Metropolitan Region, 33°25′S, 70°35′ W) Chile.

This site corresponds to the so called "agro-alfarero temprano" period (Chilean central region). ^{14}C dates on associated charcoal from the similar sites are between 180 B.C. - 430 A.C. (Thomas et al., 1980).

UC-TL-9: 1760(---, ±200) A.D.220.

Pottery: PLQ-VIII, alisado café, layer 13, depth: 2.00-2.16 m

Comment- Natural dose: 335 rads (I=55), δQ=9%, plateau ≅ 80°C.
Annual dose: 0.19 rads/yr, sherd wt.sat./dry=1.14.

UC-TL-10: 2000(---, ±150) 20 B.C.

Pottery: PLQ-I, pulido negro, layer 7, depth: 1.10-1.15 m.

Comment- Natural dose: 360 rads (I=60), δQ=3%, plateau ≅ 80°C.
Annual dose: 0.18 rads/yr, sherd wt.sat./dry = 1.14.

UC-TL-11: 1780(---, ±160) A.D.200.

Pottery: PLQ-III, alisado café, layer 6, depth: 0.95-1.10 m.

Comment- Natural dose: 160 rads (I=30), δQ=6%, plateau ≅ 70°C.
Annual dose: 0.09 rads/yr, sherd wt.sat./dry = 1.14.

UC-TL-12: 1600(---, ±130) A.D.280

Pottery: PLQ-II, pulido café, layer 13, depth: 0.80-0.95 m.

Comment- Natural dose: 160 rads (I=40), δQ=5%. plateau ≅ 80°C.
Annual dose: 0.10 rads/yr, sherd wt.sat./dry = 1.14.

All the sherds were collected by Maria A. Benavente and Carlos Thomas from the University of Chile, Santiago.

* * * * * *

7th INTERNATIONAL CONFERENCE ON SOLID STATE DOSIMETRY
Ottawa, Canada - September 27-30, 1983

The seventh in the series of International Conferences on Solid State Dosimetry will be convened in Ottawa, Canada at the Federal Government Conference Centre from September 27 to 30, 1983.

The scientific sessions will cover advances in a variety of fundamental and applied topics concerning solid state dosimeters and dosimetry including: basic physical mechanisms; ionizing and UV radiation dosimetry; exoelectron and track etch techniques; new materials; instrumentation; personnel, environmental and medical dosimetry; and standardization.

PROCEEDINGS

The proceedings will be published as a distinctive issue of *Radiation Protection Dosimetry*, Volume 6. Each participant will receive a copy of the proceedings.

TECHNICAL EXHIBITS

Provision is being made for a limited number of technical exhibits. For details contact Morgan Cox, Harshaw Chemical Company, Crystal and Electronic Products Department, 6801 Cochran Road, Solon, Ohio, 44139 U.S.A.

PROGRAM (ABSTRACTS)

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LOCAL ARRANGEMENTS

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PRE-DOSE DISCUSSION

Ian Bailiff has proposed an informal discussion during the conference for those involved with pre-dose measurements. Those interested should contact him at the address given on page 7 of this issue.

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