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DATING PLEISTOCENE FOSSIL COASTAL SAND DUNES BY THERMOLUMINESCENCE?\*

G. Poupeau<sup>1,2</sup>, J.H. Souza<sup>1</sup>, E. Soliani Jr.<sup>3</sup> and E.L. Loss<sup>3</sup>

<sup>1</sup>  
Centro Brasileiro de Pesquisas Físicas - CBPF  
Rua Dr. Xavier Sigaud, 150  
22290 - Rio de Janeiro, RJ  
Brasil

<sup>2</sup>  
CNRS, Paris  
France

<sup>3</sup>  
Divisão de Geologia Costeira  
Centro de Estudos de Geologia Costeira Oceânica  
CECO/IG-UFRGS  
Rua Gal. Vitorino, 255  
90000 - Porto Alegre, RS  
Brasil

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#### ABSTRACT

It was shown recently that sunlight exposure is able to bleach most of the geological thermoluminescence (TL) of wind transported sediments (WINTLE and HUNTLEY, 1980; WINTLE, 1981; SINGHVI et al., 1982). This property has been used in an attempt to date dunes from the well developed recent quaternary coastal dunes system of Rio Grande do Sul.

Preliminary results presented in this paper show that TL dating of fossil sand dunes from Rio Grande do Sul should be possible in a time range from present to at least 50.000 yr and possibly more than 100.000 yr.

#### RESUMO

Recentemente ficou demonstrado que sedimentos eolicamente transportados perdem a maior parte de sua termoluminescência (TL) geológica quando expostos à luz solar (WINTLE and HUNTLEY, 1980; WINTLE, 1981; SINGHVI et al., 1982). Esta propriedade foi usada na tentativa de obterem-se idades de sistemas de dunas do Quaternário costeiro do Rio Grande do Sul.

Os resultados preliminares apresentados neste trabalho revelam que datações por termoluminescência em dunas fósseis, são possíveis abrangendo o intervalo de tempo compreendido entre o presente até, pelo menos 50.000 anos e, possivelmente mais do que 100.000 anos.

## 1. INTRODUCTION

The numerical dating of quaternary geological units for times much earlier than 40,000 yr, a practical upper limit to the  $^{14}\text{C}$  geochronology, or well outside the time range accessible to the methods based on the disequilibrium of the uranium radioactive series, between  $10^4$  and  $30 \times 10^6$  yr, is still often an unresolved challenge (FAURE, 1977). Recent developments, including either the emergence of entirely new methods, or extensions of older ones to new materials and geologic situations, might soon improve this situation (POUPEAU, 1982). Among the latter group, the thermoluminescence (TL) dating method appears as one of the most promising (WINTLE, 1980, 1982; POUPEAU, 1983).

Thermoluminescence is the light emitted upon heating by solid insulators previously exposed to ionising radiations. Natural minerals may also present some TL. This is because the rocks and minerals are constantly exposed to the  $\alpha$ ,  $\beta$  and  $\gamma$  rays of the natural radioactivity as well as the ionising particles of the cosmic rays. TL is a cumulative property and as a consequence, its intensity in a mineral is a time index. The general principles of TL dating have been given in many books and review papers (see f.i. AITKEN, 1974, 1978, 1981; FLEMING, 1976; POUPEAU, 1983).

The TL method was specifically devised, in the late sixties, for the dating of archaeological ceramics (RALPH and HAN, 1966; FLEMING, 1969, 1979; ZIMMERMANN 1971). It has then been progressively applied to geological materials (WINTLE, 1980), among which quaternary sediments once exposed to the sunlight (WINTLE, 1982). A most recent development in this direction has been proposed in 1982 by SINGHVI et al., with the TL dating of fossilized sand dunes. The work reported by the SINGHVI group had been made on dunes from the Rajasthan desert in India. Although the test TL age measurements (controlled when possible by  $^{14}\text{C}$  or ceramics TL ages) were made on the micron-sized particles, a granulometric choice criticized by PYE (1982), SINGHVI (1982) later precised that similar results were being obtained on larger (100  $\mu\text{m}$ ) quartz crystals.

Following the lines introduced by A. SINGHVI, we started a TL dating project aimed at establishing the geochronology of deposition of the now fossilized beaches and sand dunes which constitute the Coastal Plain of Rio Grande do Sul. For various reasons, it is suspected that these formations, which surrounds the Porto Alegre lagoon and extend over more than 500 km along the coast with up to nearly 200 km width, might have been formed over the last 200,000 to 300,000 yr (JOST and SO LIANI Jr., 1976). It is expected, from both the properties of quartz and typical radioactivity level of sand dunes, that a large fraction - if not all - of this time period might be datable by TL.

We present here some results from a preliminary phase of this project. They have been obtained at the Thermoluminescence Laboratory of the CBPF, on quartz crystals from the 200  $\mu\text{m}$  grain size fraction of various types of sandy deposits: eolian continental dunes, sand beaches, sambaquis sand, from the "estranha" zone, etc... sampled from the north-eastern part of the Rio Grande do Sul Coastal Plain.

## 2. PRINCIPLES OF SAND DUNES DATING BY TL

The natural radioactivity and the cosmic rays produce free electrons in minerals by ionisation. Most of these electrons recombine immediately with ionised atoms, but a small fraction is instead "captured" by lattice defects (chemical impurities, vacancies, etc...) which perturbate the crystalline field. It is these trapped electrons - at least part of them - which are responsible for the TL measured in the laboratory. In effect, giving trapped electrons enough energy by heating allows them to get out of their traps and recombine radiatively or not, with atoms lacking electrons in their outer shells.

Once quartz crystals form within a rock, they accumulate trapped electrons at a rate depending on the ambient temperature, the intrinsic sensitivity of the quartz themselves (depending in turn of the nature and number of lattice defects) and the local radioactivity. Eventually, the TL stored will reach a near equilibrium level, corresponding to the saturation of the preexisting defects, possible additional increases thereafter being a consequence of defect creation by the ionising radiations themselves (Fig. 1). Meteoric alteration may further lead to rock desaggregation, and the liberation of detrital minerals. Among the latter, quartz crystals may lately be transported and accumulate in various types of sedimentary deposits including sand dunes.

During transportation, detrital minerals may be directly exposed to the sunlight. This has for effect (SINGHVI et al., 1982; WINTLE and HUNTLEY, 1980; WINTLE, 1981) to drain most of the geological TL down to a residual level ( $R_0$  in Fig. 1). Once these crystals get buried enough during the process of dune formation they will be again protected from sunlight and re-accumulate TL. For times short enough, of the order of  $2 \cdot 10^5$  yr, the TL growth is linearly related to time (with a possible supra linearity effect in the initial part of the curve), as depicted in Fig. 1, a consequence of the constancy of the environmental radioactivity over such a brief period.

The TLN intensity is related to the total radiation dose received by the minerals. If the annual dose they are exposed to is known, then in the linear part of the growth curve, a TL age for the dune formation can be derived as:

$$t = \frac{\text{geological dose}}{\text{annual dose}}$$

The calculation of  $t$  requires the determination of (i) the geological dose, by means of TL measurements, (ii) the annual dose, derived from U, Th series isotopes and K abundances as well as sand wetness, (iii) the level of the sun-bleached residual TL,  $R_0$  in Fig. 1. We refer for points (i) and (ii) to AITKEN and FLEMING (1972) and POUPEAU (1983). SINGHVI et al. (1982) have shown that  $R_0$  could be approximated by exposure of sand grains to a standard sunlamp, whose effect was found to produce within a few hours a TL bleaching  $R'_0$  equivalent within 20% to that of a prolonged exposure to sunlight (Fig. 2). The experimentally determined value of  $R'_0$  is then used to "correct" the TLN (natural TL) of its natural residual  $R_0$ . SINGHVI et al. took as  $R'_0$  value the residual TLN after 1000 minutes exposure time to their sunlamp.

The crucial hypothesis in TL dating of sand dunes is therefore to assume that the  $R'_0$  determined in the laboratory is equal to the natural  $R_0$ , in other terms that (i) the geological TL level was effectively drained to its minimum plateau-value, (ii) that either any sunlamp laboratory simulation of present sunlight illumination have same effects than sunlight exposure at the time of natural bleaching. (Differences in atmospheric transparency to a supposedly constant sun luminosity might induce slightly different effects). It is only further test-programs which will show the validity of these assumptions.

### 3. GEOLOGY OF THE RIO GRANDE DO SUL COASTAL PLAIN

The physiographic region known as the Coastal Plain of Rio Grande do Sul (the Brazil southernmost state) covers all the littoral area from Torres at north to Chui at the Uruguay border (Fig. 3) with extensions to Santa Catarina and Uruguay. It corresponds to the emerged part of a sedimentary sequence possibly accumulated since the Upper Cretaceous, inside an elongated graben-type structure (Pelotas Basin) limited by a series of normal faults oriented preferentially along a NE-SW and secondarily NW-SE axis. The Pelotas Basin is believed to have formed as a consequence of a distension tectonics preceding the separation of Africa and South America.

It appears, from the lithology of the Coastal Plain sediments, that they were deposited in various ambiances testifying of repeated transgression-regression cycles. Temporal variations of sea level do contribute together with other factors (winds, pluviosity, etc...) to influence strongly, even at a very local scale, the processes of erosion and sedimentation and therefore the morphology of coastal deposits. Each transgression - regression cycle can be identified on a local scale by a polarised sequence of deposits, with beach sands passing progressively to transitional lagoon sedimentation and continental eolian sands.

Both the geomorphology and identification of this typical sedimentary sequences offer some clues to the deciphering of the paleogeographic evolution of the Coastal Plain. Thus a number of transgression-regression cycles can be identified, whose deposits are limited by erosion scarps, the oldest sedimentary units occupying the relief heights. From the intrications of successive sedimentary-erosive cycles, it is possible to reconstitute the sequence of events which gave rise to the Coastal Plain. However, it is yet in general not possible to numerically date this sequence, but in specific instances. For recent times, either turfas or archaeological settlements (sambaquis) are good time-markers. Both are datable by  $^{14}\text{C}$  and the latter eventually also by TL (on ceramics or hearth stones). Otherwise, approximate numerical dating rely on the occasional discovery of fossil bones from Pleistocene Megafaunas.

#### Sampling

The material in this work has been sampled in the northeastern corner of the Coastal Plain (A in Fig. 3), between the cities of Cidreira and Torres. This area has been chosen as one of the best geologically known. For each sampling, usual cautions for TL dating were taken: the samples were taken from homogeneous materials, at a depth from surface of at least 50cm. Upon sampling, sands were immediately set in plastic bags hermetically closed (for further wetness measurements) and finally protected from sunlight under a black plastic cover. From the samples collected during a field trip, only 3, samples RMG 05B, 04A and 03D have been studied with some detail up to now.

Samples RMG 03D and 05B come from sub-recent eolian deposits. Their stratigraphical position and relations with an alignment of recent dunes suggest a lower holocene age. Sample RMG 04A is a marine sand deposited in shallow water passing laterally to lagoon deposits with fossil mammals belonging to the Upper Pleistocene.

### 4. RESULTS

For this preliminary study, the 74-250  $\mu\text{m}$  size fraction of sand grains was selected. After sieving, the powder was treated at room temperature with HF 40% for 40 minutes and the purity of the quartz sepa-

rate checked with X-rays diffraction. The TL was observed from room temperature to 500C with a heating rate of 10C/sec, in the violet-blue region of the spectrum (320-470nm). Irradiations were performed with a 50m Cie 90Sr source and sunlamp exposures were given by a 275 W General Electric sunlamp at 30cm above the samples. TL measurements were made on aliquotes of 5mg each, with a reproductibility within  $\pm 10\%$

We first checked the effect of our sunlamp over the TLN of sample RMG 05B, using exposure times between 15 and 2000 minutes. The curve showing the fractionnal remnant TL versus exposure time is shown in Fig. 4. About 70% of the TLN is bleached during the first 2 hours of exposure and only 7% more are erased during the next 15 hours. Comparison with Fig. 2 shows that this decay curve is very similar to that of SINGHVI et al.

In order to test the efficiency of the sunlamp relative to sunlight, we studied a sample of the Copacabana beach sand, assumed to be at the  $R_0$  level (Fig. 1) of TLN. In effect we took some superficial sand supposedly well stirred both by natural and human agents. We found that the TLN of Copacabana beach was within 20% of the TL remnant value after a sunlamp exposure of 15 hours.

#### SAMPLE RMG 05B

The TLN (Natural TL) of this sample presents one peak centered at 403C (Fig. 5). When a  $\beta$ -dose is applied three additional peaks appear at 110C, 230C and 310C, the latter barely visible on TLN + Dose curves.

Preliminary measurements having shown that this sample is far from saturation, a plateau-test was calculated from the TLN and TLN + 6.72 krad glow curves of Fig. 6. Also reported in Fig. 6, is the remnant TLN after 15h of irradiation under the sunlamp. The plateau test was calculated as

$$r = \frac{\text{TLN}}{(\text{TLN} + \text{Dose}) - \text{TLN}}$$

For samples well behaving for TL dating,  $r$  grows from zero at temperatures below  $\sim 200\text{C}$  up to a constant, or plateau value for temperatures where the TLN was quantitatively retained (AITKEN, 1974; POUPENAT, 1983). Two  $r$  functions were calculated, one assuming total solar bleaching and the second a TL solar bleaching as given by the lower curve of Fig. 5. In the first case we get a well defined plateau value of  $r = 0.789 \pm 0.069$  (1 standard deviation) while in the second case  $r = 0.64 \pm 0.11$ . These plateaus were calculated for temperature ranges of respectively 300C-460C and 300C-480C. The larger dispersion in the second one comes from the large sunlamp exposure corrections at  $t > 420\text{C}$ . There are indeed indications from the asymptotic nature of the sunlamp bleaching curve in Fig. 4, that this correction might be excessive at high temperatures (see Fig. 6). Still, if one considers only the more restricted temperature range from 300C to 410C, we get a well defined plateau value of  $r = 0.75 \pm 0.024$ . In both case, the 403C peak, belongs to the plateau.

The geological dose to the sample was calculated with the additive dose method, assuming either total or partial solar bleaching. Results in the former hypothesis are given in Fig. 7 for the first and second glow growth curves. The growth curves have been drawn for the TL emitted at 403C.

Up to a laboratory dose of 8 krad, the first glow curve growth linearly with a sensibility to  $\beta$ -dose about equal (within 8%) to those

of the second glow curve. The latter presents a measurable supra linearity effect which has been taken into account in the geological dose calculation.

Extrapolations from the growth curves of Fig. 8 give respectively  $\beta$ -equivalent doses of 5.95 krads and a supra linearity effect of 0.83 krads. The total geological dose to the sample is therefore estimated (within  $\pm 10\%$ ) at 6.78 krads in the hypothesis of total solar bleaching or 5.1 krads using the sunlamp correction.

The radioactivity measurement of sand RMG 05B is in progress and therefore at the time of writing this article a precise TL age for this sample cannot yet be computed. Still, assuming a typical annual dose rate of 0.7 rads/yr in a quartz sand, the geological dose to sample RMG 05B would correspond to a dune formation age of  $\approx 8000$  yr. Due to the uncertainties in annual dose, we estimate this age to be accurate only to within  $\pm 50\%$ . Even with these restrictions, this preliminary result is in reasonable agreement with the geological estimate as discussed above, as slightly younger than 12000 yr.

#### SAMPLE RMG 04A

The TLN in sample RMG 04B also is unsaturated. When corrected for solar bleaching it presents a good plateau with  $r = 5.5 \pm 1.1$  between 375C and 500C (Fig. 9). Contrarily to sample RMG 05B, sun bleach correction from sunlamp exposure brings a better plateau. This difference in behaviour might reflect in part differences in quartz insulation history. A preliminary determination of its geological dose was made from the TLN emitted at the peak value of 400C. The first and second glow curves grow linearly with dose with significant differences of sensitivity (Fig. 10). The first glow growth curve is linear up to laboratory doses of 20 krads. The second glow growth curve does not present supra linearity within the precision of our present measurements. With a geological dose corrected for solar bleaching of 38 krads and same assumptions for annual dose as for RMG 05B we arrive at a TL age of 54300 yr, i.e. well within the Upper Pleistocene times ( $\approx 10\ 000$  to 100 000 yr) as inferred from geological considerations.

#### SAMPLE RMG 03D

The third sample in this study has also a TLN well below saturation level for the 74-105  $\mu\text{m}$  fraction of the quartz crystals. Unfortunately this sample presents no TL reproductivity. In order to eliminate possible effects of quartz contamination by other mineral phases, prolonged HF etching times - up to 3 hours - were used, without improving this situation. Plateau-tests show  $r$  values monotonically growing with temperature. It is not yet known whether this is due to anomalous fading of TLN - considered generally as insignificant in quartz see AITKEN, 1981 - or incomplete TLN solar bleaching or even other reason.

The homogeneity of dune sediments in terms of solar bleaching as well as deposition history, is not necessarily the same for all grain sizes. We therefore started to study the TLN of the  $< 74\ \mu\text{m}$  fraction, which was found to exhibit a reproducibility better than 10%. Detailed study of the TL characteristics of this grain size fraction for RMG 03D is in progress.

#### 5. CONCLUSIONS

We studied 3 sand samples from Pleistocene stabilized dunes of

the coastal formations of Rio Grande do Sul. From this first approach to date fossil dunes by thermoluminescence we conclude:

1. In at least part of our sampling, the  $\frac{1}{2}$  100  $\mu$ m quartz crystals granulometric fraction has been sufficiently exposed to sun during transportation and up to the dune final sedimentation to erase most of their geological TL.
2. In two samples drained out of their geological TL, the reproducibility of TL measurement was good enough ( $\pm 10\%$ ) to allow a measure of their geological dose.
3. Assuming reasonable values for annual radiation doses to the sand, TL ages for dune formations were calculated. These ages are compatible with geological estimates.
4. From the first glow growth curve of samples RMG 05B and especially RMG 04B it appears that, depending on the materials, dunes TL ages could be determined from present to at least 50.000 to 130.000 yr, with a precision approaching  $\pm 15\%$

Further steps in our work will include refined redetermination of the geological doses of the samples studied here, as well as others not yet reported, including determinations of their saturation dose, internal radioactivity and wetness for a precise annual dose determination, etc... Whenever possible, the age of dunes will be controlled with other methods. For instance, for periods  $\leq$  7000 yrs, sambaquis are expected to help in the evaluation of the method, as many can be dated, in addition to TL on sand grains, by  $^{14}\text{C}$  on bones or charcoal, and TL on ceramics.

#### ACKNOWLEDGEMENTS

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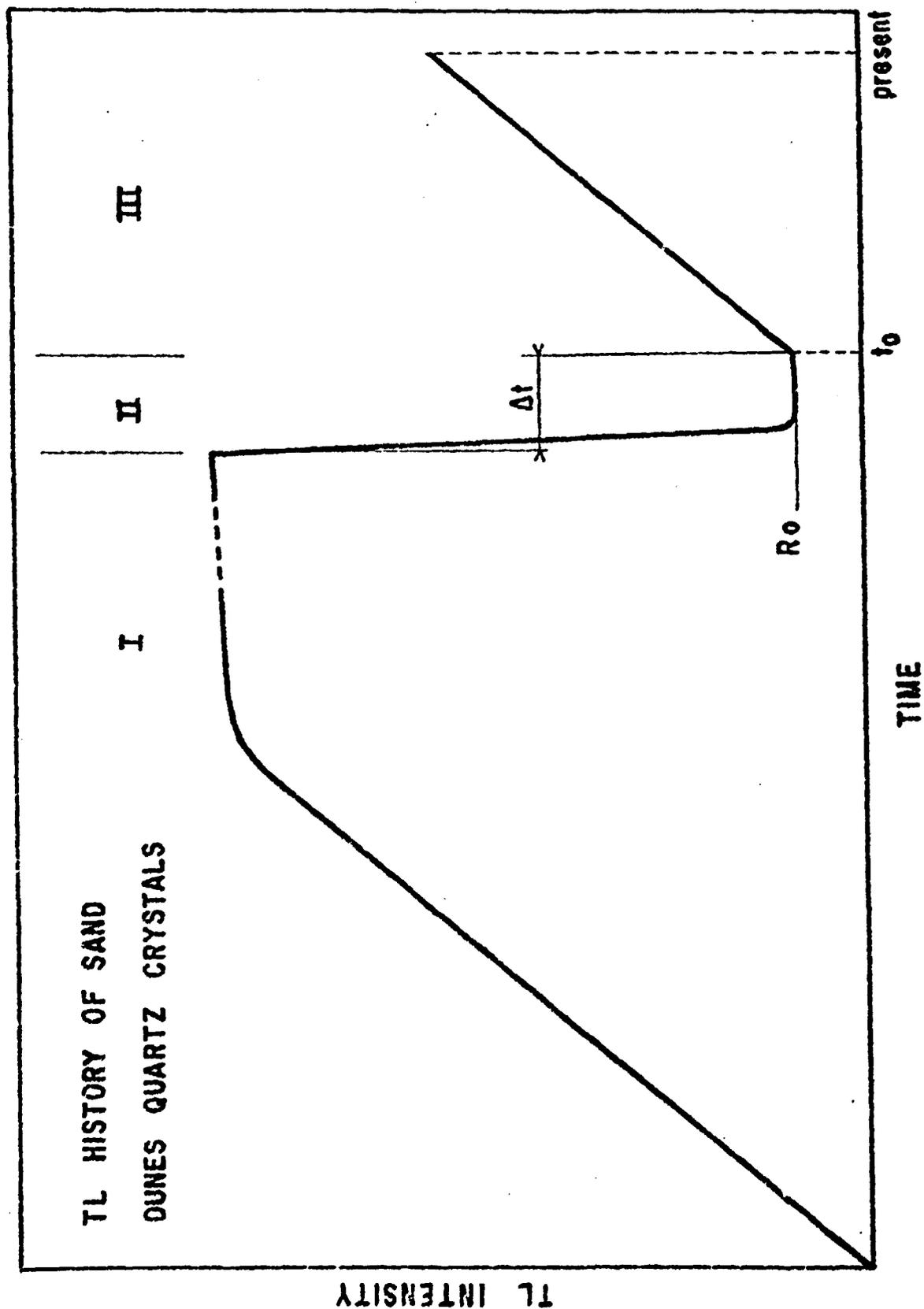


Fig. 1 - TL history of quartz crystals from sand dunes. I: in parent-rocks the crystals accumulate TL up to saturation of preexisting traps. II. After rock alteration detrital quartz are exposed to sunlight during a time  $\Delta t$  and their TL drops down a residual value  $R_0$ . III. Once buried definitively within the dune at to the TL builds up again up to the present level. For times from present to  $\approx 10^5$  yr, the growth is linear.

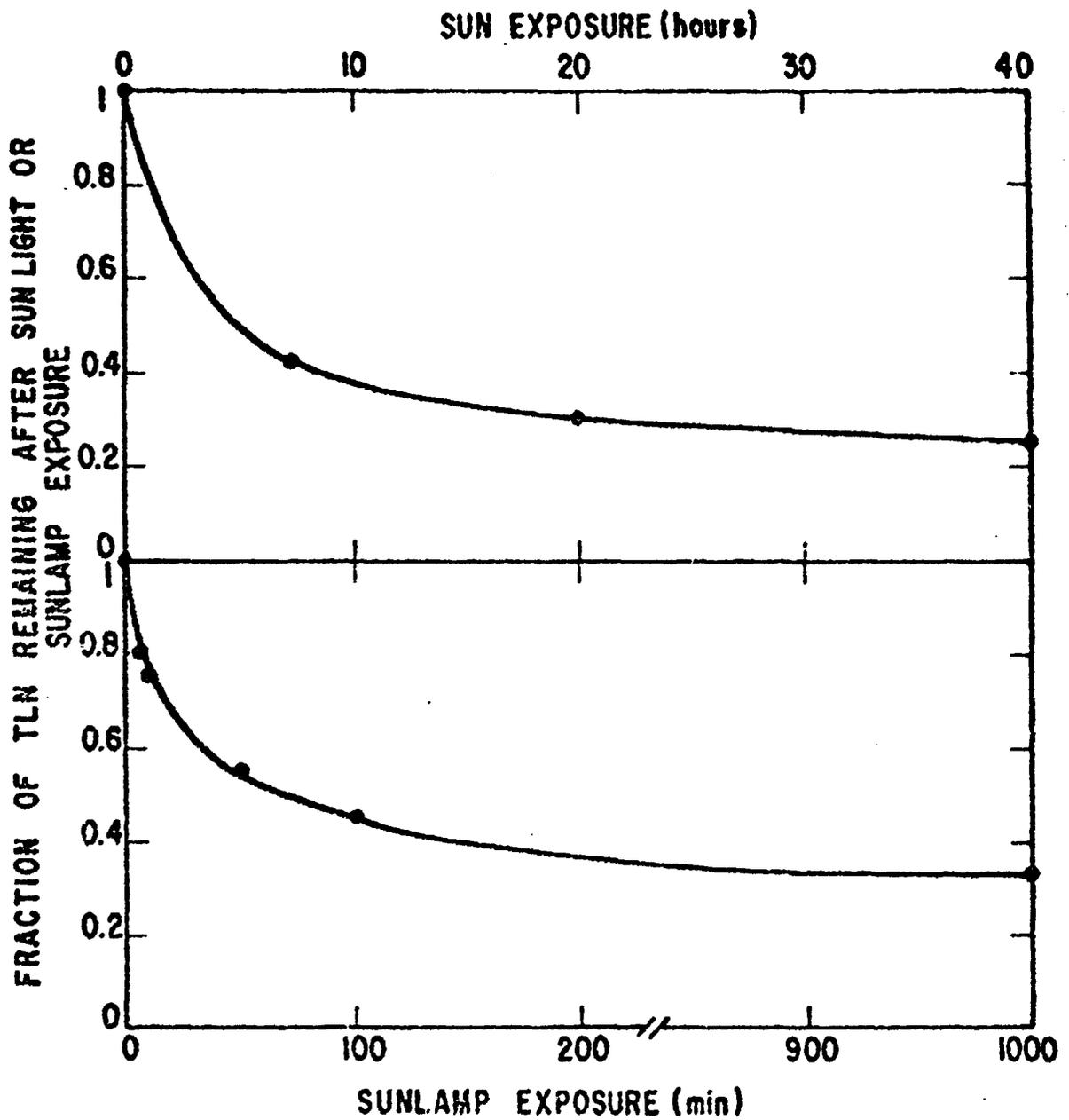


Fig. 2 - Comparison of the effect of sunlamp and sunlight exposures on the fine grained (1-8 $\mu$ m) fraction of fossilized sand dunes from the Rajasthan desert (from SINGHVI et al., 1982). The TL intensity was taken at 320C. The residual TL after 250h sunlight exposure was found to be equivalent within 20% to the value measured after 1000 mn sunlamp irradiation.

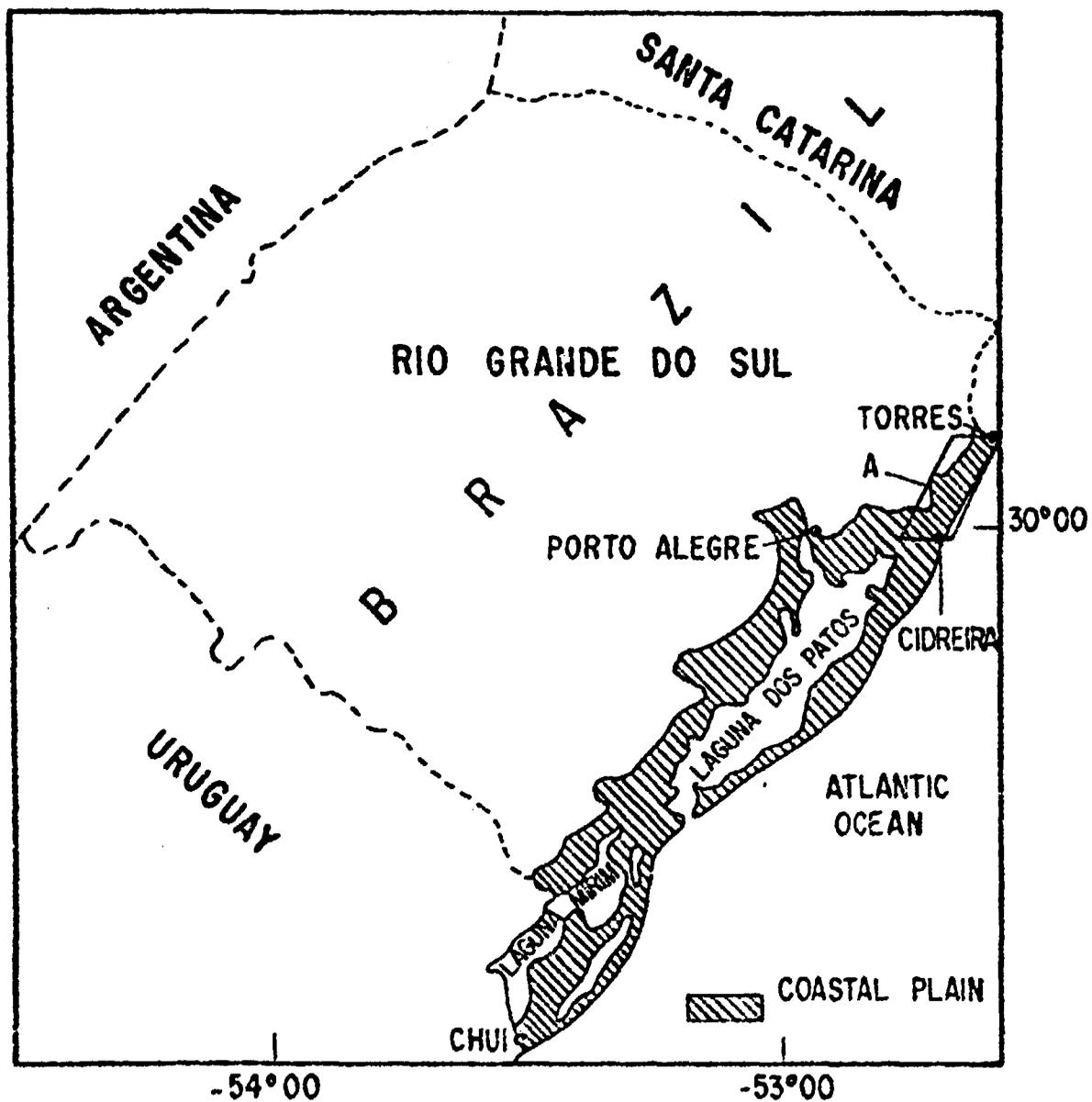


Fig. 3 - Map of Rio Grande do Sul, showing the coastal plain and the sampling area (quadrilateral marked by letter A).

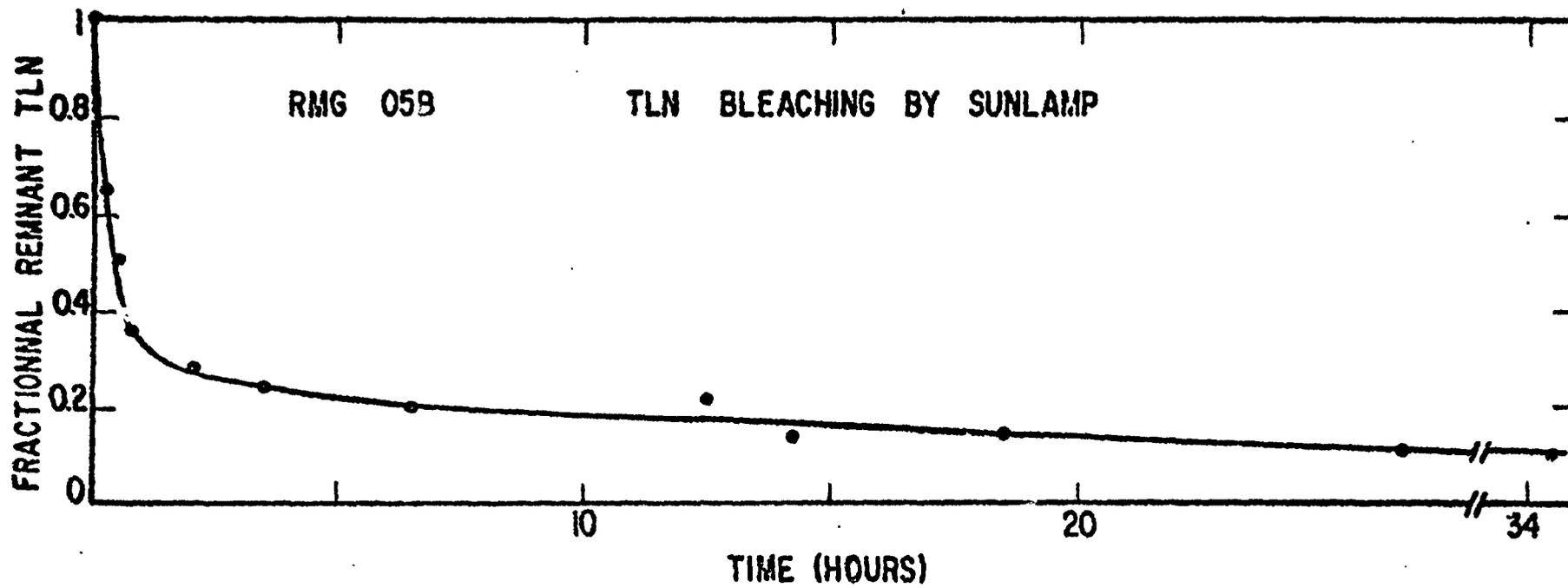


Fig. 4 - Fractional decrease with time of the TLN from the 74-250  $\mu$ m fraction of quartz crystals from sample RMG 05B. (measured from the TL emitted at the peak temperature of 403C).

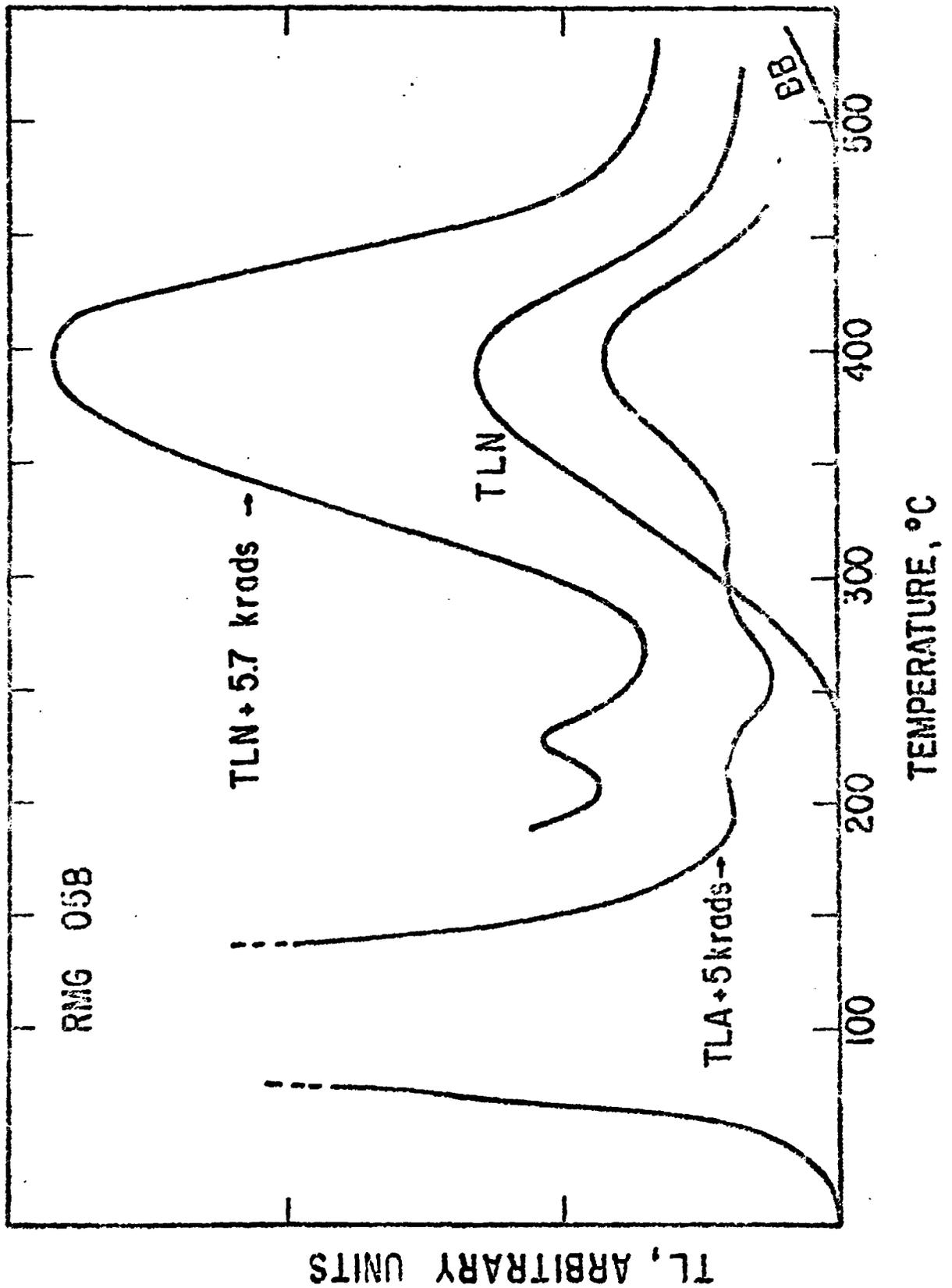


Fig. 5 - Glow curves of the TLN, TLN + 5.7 krad and 5 krad TLA (Artificial TL obtained after TLN emptying and a subsequent  $\beta$ -irradiation) of sample RMG 05B. BB = black body curve.

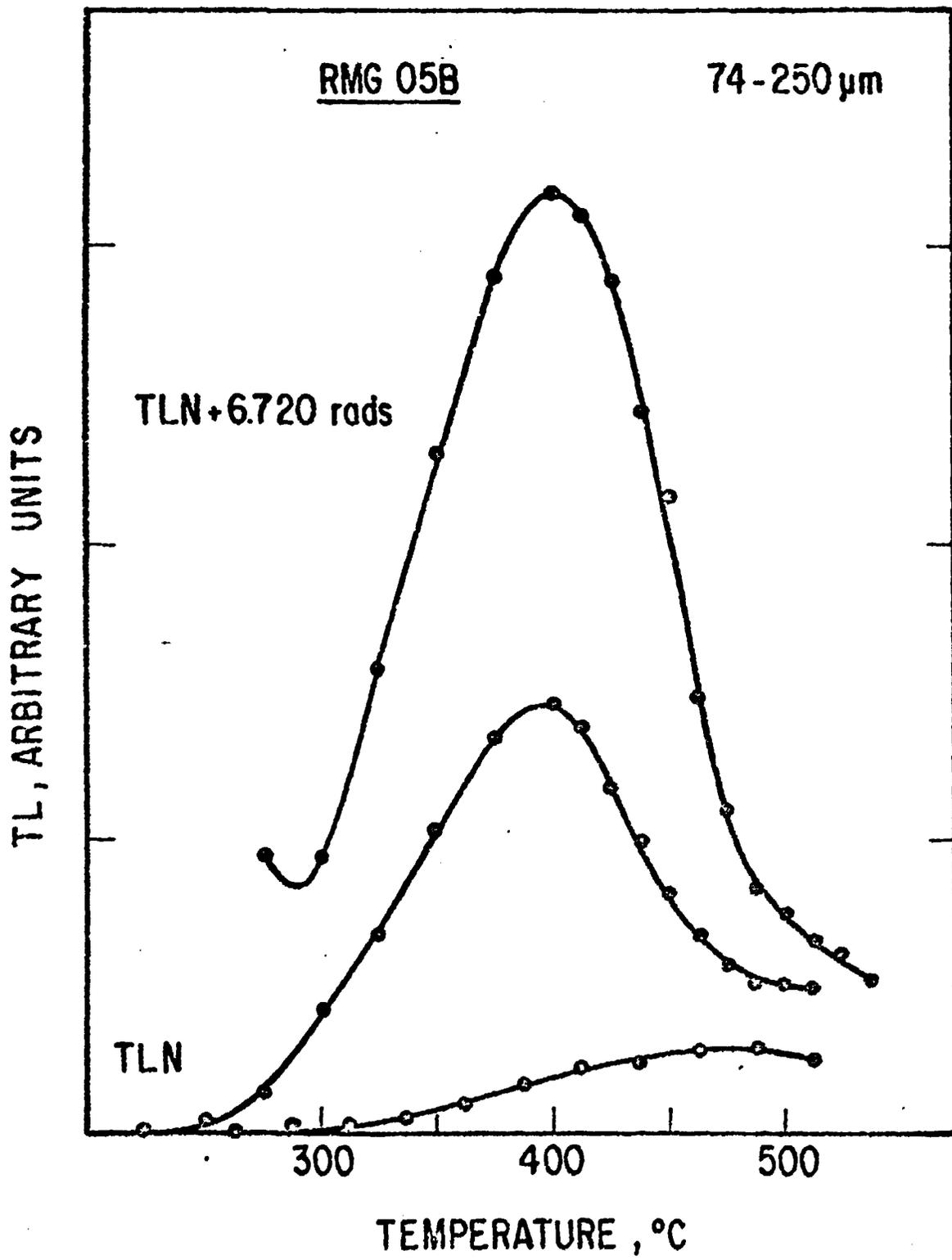


Fig. 6 - Glow curves corrected for black body radiation of the TLN, TLN + 6.72 krad B - irradiation and TLN + 15h sunlamp exposure of RMG 05B. All curves are averages over five measured aliquotes.

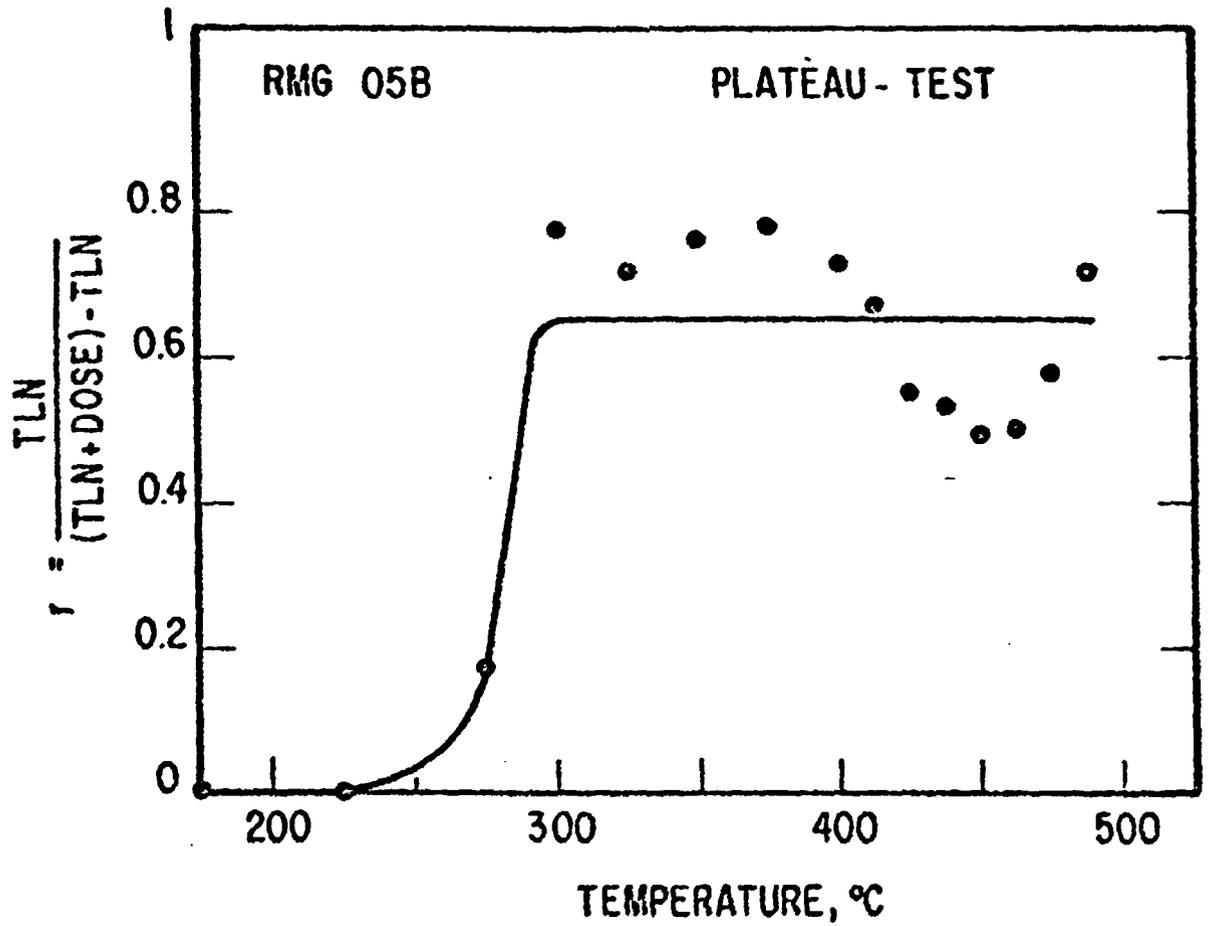


Fig. 7 - Plateau-test for RMG 05B, with TLN and TLN + Dose corrected for residual sunlamp intensities  $R_0$ . This curve has been established for the TLN peak temperature at 403C from the glow curves of Fig. 4.

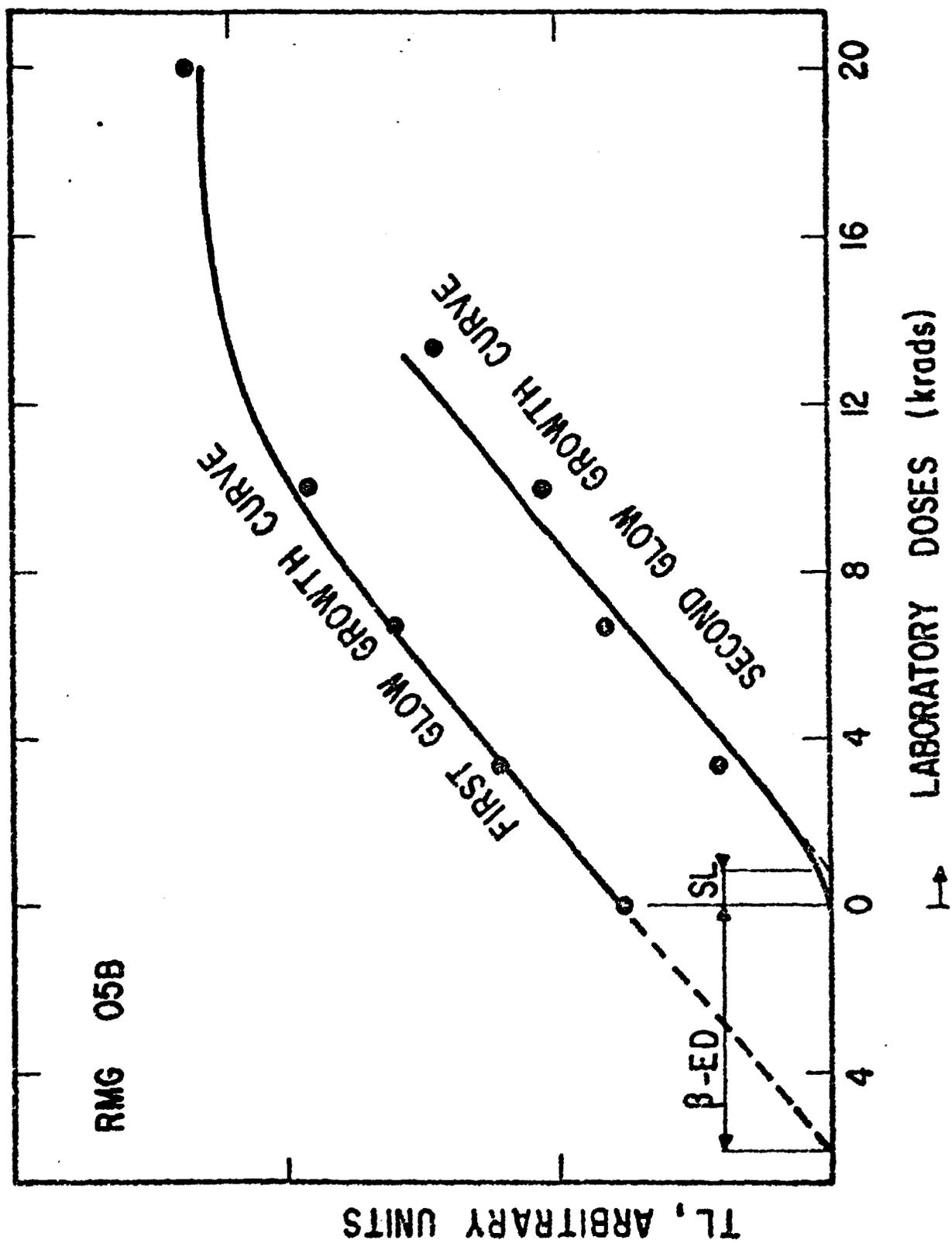


Fig. 8 - TL growth curve for sample RMG 05B for TL emitted at 403C.  $\beta$ -ED and SL, respectively  $\beta$  - equivalent dose and supralinearity dose correction (uncorrected for solar bleaching).

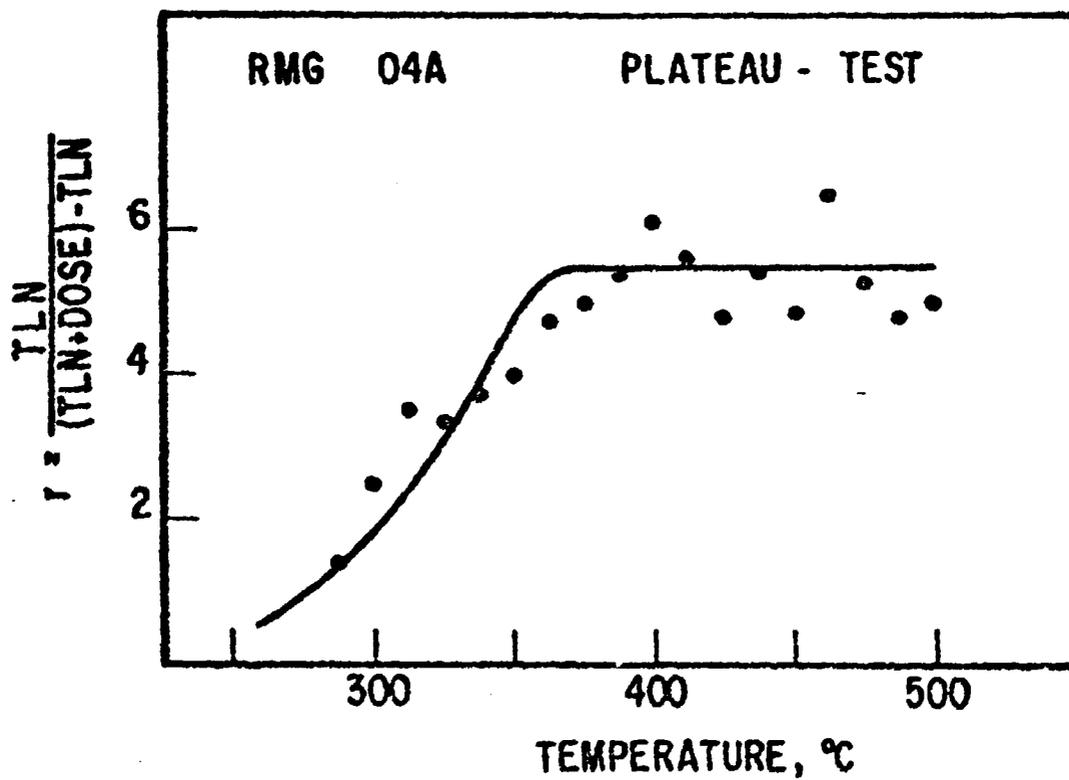


Fig. 9 - Plateau-test for RMG 04A corrected for sunlamp bleaching.  
 Curve calculated for the TL emitted at 400C.

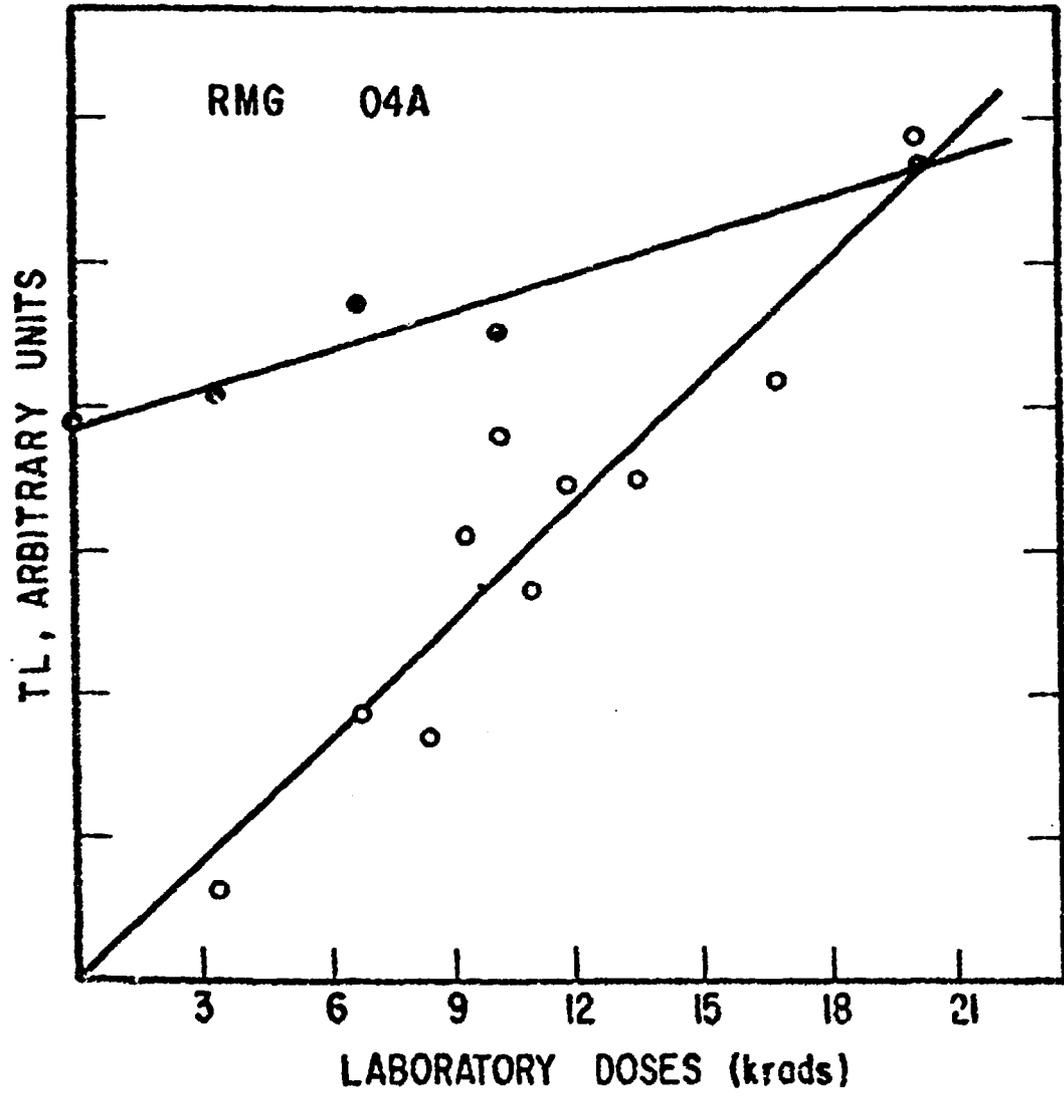


Fig. 10 - TL growth curves for sample RMG 04A for the TL emitted at 400C (uncorrected for solar bleaching).