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THERMOLUMINESCENCE DATING OF PLEISTOCENE SEDIMENTS. A REVIEW AND SOME PRELIMINARY RESULTS ON SAND FORMATIONS FROM BRAZIL*

by

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ABSTRACT

After a short introduction on recent trends in quaternary geochronology, this article focuses on the thermoluminescence dating of sediments, whose principles and present limits and prospects are discussed.

Results are presented for the TL behaviour of sands from various geological contexts in Brazil. They show that the coarse ($\approx$100-200μm) quartz fraction of coastal and intra continental, eolian and fluvial-type deposits, might be datable by TL from the upper Holocene to at least the basis of the upper Pleistocene, with a precision of $\pm$ 10-15%.

1. INTRODUCTION

Gradually, during the last few decades, it has been realized that terrestrial climates change much more rapidly and in a more complex manner than was generally thought before. Drastic climatic changes may occur over time scales of a few thousand or even hundreds of years. These variations influence both the erosional and sedimentary processes, and therefore geological studies of sedimentary series can be used to reconstruct the history of recent climatic variations.

In Quaternary palaeoclimatology, a major problem is raised by the discontinuous nature of the sedimentary record. Sedimentary series are often relatively limited spatially, and lateral facies variations are not infrequent. As a result, stratigraphic correlations are often difficult or even impossible to establish, even on a regional scale. In many instances, the only way to correlate distant lithostratigraphic units is by geochronology or a combination of geochronologic and magnetostratigraphic data. Unfortunately, up to now the few methods available for numerical dating within the last million years applied to quite specific materials. In sedimentary situations, the absolute chronology of Quaternary events $\lesssim$30 000 yr is still almost entirely based on ^{14}C dating. Methods based either on natural rhythms (varve and tree-ring countings, etc...) or thermoluminescence dating of archaeological ceramics (Fleming 1979), although important, have been less extensively applied, and in any case, are limited to about the last 10 000 yr. ^{210}Pb chronology, for ice or lake sediment dating, is confined to the last 100 to 200 yr. The only

classical dating methods extending back in time significantly further than ^{14}C dating are based on the measure of the U-series disequilibrium (Ku 1976). They include the ^{231}Pa - ^{235}U method, dating from 1000 to 180 000 yr, the ^{230}Th - ^{238}U method, dating from 1000 to 350 000 yr, and the ^{234}U - ^{238}U method, dating from 100 000 yr to 1,200 000 yr. These methods were until recently used essentially to date speleothems (Harmon et al. 1975, Schwarcz 1980) and corals; they seem to meet with mixed success in the dating of bones (see Ku 1976). U-series methods have now been extended to the dating of various types of surficial continental carbonate deposits (e.g., caliches: Szabo and Sterr 1978); and with the uranium-trend technique (Rosholt 1978), both soils and continental sediments might be datable with reasonable precision in the age range from $\sim$4000 yr to 700 000 yr (Rosholt 1980, Szabo and Rosholt 1982). Finally, sedimentary layers can also be indirectly dated where they are interbedded with volcanic units, which (depending on their age, chemistry, mineralogy, etc.) can possibly be dated by one or more methods: U-series disequilibrium (see e.g., Allègre and Condomines 1976), uranium fission-tracks (Poupeau 1981), or K-Ar*. For events $\lesssim 10^6$ yr old however, the two latter are only circumstantially applicable (see e.g., the long controversy on the dating of the KBS tuff in Tanzania in Hays 1980, Taieb and Poupeau 1980).

In the late seventies, significant progress in Quaternary geochronology was made in several directions, and prima-

* Another method of dating volcanic rocks, applicable to obsidian, was proposed long ago. Based on the natural hydration rate of obsidian, it was often considered as reliable mostly for relative dating (Goffer 1983). Recent progresses however seem to indicate that it could now be operationnal as an absolute dating method (Michels et al. 1983).

rily in ^{14}C dating. First, with the new design of small proportional counters (see e.g., Harbottle et al. 1979), carbon samples of small size ($\approx$10 mg instead of a few grams) can now be dated, although extended counting times are necessary. This development opens the possibility of dating sites (or sediment core levels) where little carbon is present. Second, two entirely different approaches can now be applied to the ^{14}C dating of samples of any age up to at least 70 000 yr. The isotope enrichment technique, which requires as much as 60g to 120g of C, and involves protracted ^{14}C isotopic enrichment before radioactive counting, has already proven to give reliable ages up to 75 000 yr under favorable conditions (Grootes 1978). The ion counting technique proposed some years ago utilizes cyclotrons or electrostatic particle accelerators of the Tandem Van de Graaf type (Gove 1978, Litherland 1980, Hedges 1981). This high energy mass spectrometry could also be used for samples as old as 70 000 yr and potentially 100 000 yr; the main problem for old samples as for the isotopic enrichment method (see e.g., Grootes 1978) is the proper evaluation of contamination carbon (see Nelson et al. 1978, Fig. 10). The accelerator methods, as compared with the isotope enrichment method have the advantage of consuming very little carbon (1-10 mg C are sufficient); and in any conventional radioactive counting, the benefit of much reduced measurement times. More generally, accelerator dating will also allow routine use, as geochronometers, of a variety of cosmogenic radioisotopes produced in the terrestrial atmosphere by interaction with Cosmic Rays. Thus in addition to ^{14}C, analysis of radioactive isotopes like ^{36}Cl, ^{26}Al, and ^{10}Be, among others (see Gove 1978) will contribute to the geochronology of the variations of our na-

tural environment over the last 10 million years. Accelerator dating, with all these potentialities, thus certainly promises a significant development in the future.

Dating methods using the radiation-damage produced in mineral lattices by natural radioactivity have also been significantly improved and enlarged. In the early seventies, the only dating method in this field was (in addition to fission-track dating) the thermoluminescence (TL) method, and its applications were almost entirely confined to the determination of ages of archaeological ceramics (Aitken 1974). Since then, a newcomer, the Electron Spin Resonance (ESR) method, first proposed some 15 years ago (Zeller et al. 1967), was finally introduced by Ikeya (1978) for the dating of speleothems (Ikeya 1975), bones (Ikeya and Miki 1980), and shells (Ikeya and Ohmura 1981). ESR bone dating appears at present to be the least reliable method, hampered by the same problem as U-series dating, the behavior of bones as systems open to the migration of radioactive isotopes (see e.g., Yokoyama et al. 1981). In principle, carbonates and bones could be dated by ESR for a period covering the last several million years. Application of the ESR method to quartz has also been suggested by Ikeya for the dating of recent fault movements (Ikeya et al. 1981), an application which could be of much interest in neotectonic studies.

TL dating itself has now been extended to non-ceramic materials (Wintle 1980, Poupeau 1983) as burnt archaeological artifacts (Wintle and Aitken 1977, Beltrão et al. 1982, among others) carbonates (Wintle 1978a), volcanic lavas (Gillot et al. 1979) and tephras (Ichikawa et al. 1982), and eolian sediments (Wintle and Huntley 1982). Among the latter, conclusive tests were conducted on oceanic

deposits (Wintle and Huntley 1980), continental loesses (Wintle 1982), and more recently on sand formations (Singhvi et al. 1982). TL dating of carbonates could potentially cover the same time-range as ESR; and for other sediments, be effective over the last 50 000 yr to 300 000 yr*.

Quaternary sandy and silty formations are relatively frequent within continents, either as fluvio-lacustrine sediments or eolian dune systems; and they are the most common type of coastal deposits. Their direct dating therefore would help greatly the establishment of absolute climatic chronologies on continents as well as the ages of recent shoreline level fluctuations. The first attempt of the dating of sands by TL, by Singhvi et al. (1982), was made on the bulk fine fraction (1-10 μm grain size) of dune materials from the Rajasthan desert (India). Although the choice of this grain size is open to criticism (Pye 1982), in this particular occurence it apparently gave satisfying results (see also reply by Singhvi 1982). The same year, the CBPF group initiated a TL investigation of Quaternary sands from various locations and geological contexts in Brazil, in order to evaluate its possible applications. It must be remembered that TL dating of sediments is a relatively new approach yet far from routine, and much work is needed before reaching this stage.

In the following, we review the principles of TL dating of sediments, and give an overview of our TL studies on Brazilian Qua-

*Not mentioned in this short review is the amino-acid racemization (AAR) dating method. In effect, opinions are still divergent about its possibilities in absolute dating of continental deposits, especially bones (compare e.g., Schroeder and Bada 1976 with Von Endt 1979). For instance, evidence for the great antiquity of man in America claimed from AAR dating of human bone fossils between 28 000 and 70 000 yr, were recently dismissed by ^{14}C dating (Taylor 1983). In spite of that, the importance of geochemical and stratigraphical applications of AAR studies to Quaternary research must not be underestimated (e.g.,Schroeder and Bada 1976, Szabo et al. 1981, Wehmiller 1982, among others).

ternary sands. Some results of analysis of sandy formations from the Coastal Plain in Rio Grande so Sul were published elsewhere (Poupeau et al. 1983).

2. THERMOLUMINESCENCE DATING OF SEDIMENTS

2.1 - Thermoluminescence of natural crystals

TL is the light emitted by heating of minerals previously exposed to ionizing radiation. Not all minerals are thermoluminescent, however (McDougall 1968). Thermoluminescence occurs only where specific crystal lattice defects (chemical impurities, dislocations, etc.) are present.

In nature, all minerals are constantly exposed to a flux of ionizing radiations, mostly the α, β and γ rays of the ambiant radioactivity, and to a lesser extent, the hard component of Cosmic Rays. The bulk of their interaction with matter is through ionization, which creates charge carriers: electrons and their positively charged counterpart, or "holes", both moving freely within crystals. Nearly all of the free electrons created in minerals by the passage of ionizing particles recombine almost immediately with holes, but a small fraction eventually becomes captured by defects - or "traps" - of the crystal lattice. This process can be best visualized through the usual band model which describes the energy state of electrons in solids. Peripheral electrons bound to atoms are situated in the "valence band" of energy (Fig.1). In a perfect crystal, ionization would cause them to jump over the forbidden energy band up to the conduction band. The very existence of this forbidden band is the expression of the quantized nature of

electron energy levels. In real crystals, the presence of defects distorts the triple periodicity of the crystalline electric field and creates intermediate energy levels within the forbidden band. It is these energy levels which act as potential traps for electrons and holes, as shown in Fig. 1. Electrons can be captured by an energy level slightly below the conduction band; while holes can become trapped in levels above the valence band, where they are called luminous center.

The electrons in traps are in metastable states: if they acquire enough energy, they get out and recombine with ionized atoms. However, some minerals have deep enough traps for an electron lifetime in them to be of 10^6 yr and up to 10^8 yr at the temperatures usually encountered near the earth's surface. These minerals may then act as natural radiation dosimeters, the number of electrons accumulated in deep traps being a function of time. The reading of such dosimeters can be made with different techniques, using either thermally stimulated processes: e.g., the measure of thermally stimulated exoelectron emission; thermally stimulated currents; thermoluminescence (Zimmerman 1977); or the response to an external excitation, as with electron spin resonance spectroscopy (McDougall 1968, p. 271). To date, only the last two techniques have led to dating methods.

In thermoluminescence dating, the samples are usually linearly heated at 10ºC/sec up to 500ºC (Fig. 2). In these conditions, the natural thermoluminescence, or TLN, of minerals starts to be emitted by recombining electrons (see Fig. 1) at 250ºC and exhibits one to two peaks at temperatures $\geq$300ºC (Fig. 3). It is the high temperature TL peaks which are used in TL dating.

In any given mineral, the total number of electrons traps is a finite number. Therefore, within a rock, the high temperature TLN of minerals, which corresponds to electrons within deep traps, will grow linearly to a saturation level expressing the equilibrium between electron trapping and detrapping (Fig. 4). According to mineral types and the local radioactivity, this saturation level is acquired between $\lesssim 10^5$ yr (e.g., for quartz within a granitic radioactivity environment), to more than 1 million years for carbonates. Because of the short time scale involved in this process, the first part of the TL growth curve is linear, due to the constancy of radioactive energy production rate*. It is essentially the linear part of the growing TL intensity with time which is used in TL dating (for a description of TL dating techniques, see e.g., Fleming 1979, Aitken 1981, Wagner 1981, Poupeau 1983).

2.2 - TL as a dating method for detrital sediments

The TL dating of detrital sediments will be emphasized in this section§. In general, detrital sediments are made of minerals formed well before 10^5 to 10^6 years ago; and therefore their TL at the time of final sedimentation will in general be at saturation levels. However, some possibility exists to date these sediments by TL, where at the time of deposition mechanisms occurred to reset the TL chronometer to zero. At present, two such mechanisms are

* Departure from linearity may occur in recently formed minerals: e.g., Upper Pleistocene carbonate deposits, to allow for the equilibration of the uranium decay series (see Wintle 1978, Yokoyama et al. 1981), see § 4 below.

§ TL dating of recent carbonate formations is therefore not considered here. Discussion of its problems and potentialities may be found in Wintle (1978a) and Poupeau et al. (1984).

known. One is heating above 400ºC to 500ºC in (natural or archaeolo gic) fires, or at contact with hot extruding volcanic material; and the second is exposure to sunlight of individual detrital minerals. Both mechanism lead to dating methods, however, slightly different from each other. In any case, the objective of TL dating is to compare the radiation dose to which a sediment has been exposed since its last deposition, to the annual dose received from its environment; the time (t) elapsed since the geochronometer was last reset to zero being expressed by the ratio:

$$t_{(yr)} = \frac{\text{Total dose accumulated}}{\text{Annual dose}} \qquad (1)$$

TL dating of baked or sunlight-exposed sediments differ essentially in the method of determination of the total dose (or paleodose), as discussed below. The annual dose is obtained by one of several standard methods including either (i) a measurement of radioactive species content of the sediment and the calculation of their annual radiation dose to the sample, or (ii) direct TL dosimetry, or (iii) a combination of both (Aitken and Fleming 1972, Aitken 1978). In any case, in the evaluation of the annual dose one must take into account the relative efficiency of the α rays to β and γ rays to produce TL (Aitken and Bowman 1975).

2.2.1 - TL dating of baked sediments

This method typically is applied to soils underlying lava flows and hot-deposited volcanic ashes. At their contact, soils may be heated at several hundred degrees for a time long enough to erase totally their preexisting TL. Upon cooling, TLN in soil minerals rebuilds linearly with time (if no migration of radioactive

species occurs), as depicted in Fig. 5. In order to date the time of the heating event by TL, one must first check from which temperature on glow curves (such as those in Fig. 3) the TLN has been retained quantitatively. This is the purpose of the plateau-test, in which one compares the natural TL glow curve to the glow curve of a sample aliquote irradiated by a radioactive source in the laboratory (Fig. 6, bottom). The latter exhibits a larger number of peaks than the TLN curve, especially at low temperature. This is due to the fact that laboratory irradiation dose-rates are high enough (up to several million times the natural radioactivity dose-rates) that the rate of infilling of shallow traps largely overcomes its emptying rate. For instance, from laboratory irradiation and TL measurements, it can be shown that the 110ºC peak in quartz decreases within a period of 2 hours*. This means that, if one measures the TLN + laboratory dose 24 hours after irradiation, this peak will be more than 4000 times smaller and consequentely no more observable. On the high-temperature side, the two glow curves on Fig. 6 (bottom) show similar shapes. At each temperature, the ratio R, between the ordinates of these two curves expresses well the above-discussed features; it growths from zero at low temperatures (due to loss of trapped electrons by the sample at the ambient field temperature) eventually to reach a plateau level, where all electrons are retained. It is only at temperatures higher than t_p in Fig. 6 (top) that TL must be considered for dating purposes§.

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* i.e. losses half of its height each 2 hours period.

§ However, in ceramics dating for samples $\lesssim 10^3$ yr, a special property of the 110ºC peak, the pre-dose effect not present in the TLN, can be used to obtain the age of the last firing above 500ºC (Aitken 1974, Fleming 1979).

In equation 1 above, the paleodose is usually determined with the help of the additive radiation-dose method, into which aliquotes of a given sample are given different doses of α, β or γ rays, and their TL glow curve is subsequently determined. A plot can then be drawn, for any temperature $t > t_p$ (Fig. 7, top) of the TL intensity versus laboratory dose. If the array of experimental points is linear and TL grows with dose, then an "equivalent dose" to the paleodose is obtained from an extrapolation of the linear regression line to the data. (The case in which the relation of TL growth to dose is not linear will be discussed below). It is this equivalent dose which represents the paleodose of equation 1 and which is used in TL age calculations.*

Historically, this method of TL dating was the first to be developed, and it has been widely applied since then to the dating of ceramics and other non-ceramic burnt archaeological materials (kilns, hearth stones, burnt artifacts). In the case of geological materials, the method is limited in practice to sediments buried by hot volcanic materials. It works best on cherts and minerals like quartz, calcite, and potassium-feldspars as well as on the highest temperature peak ($\gtrsim 600$ºC) of plagioclases. The silty fraction of ceramics and soils is also widely used in TL dating. However, due to the multi-component nature of the TL-emitting minerals in this grain size fraction, some of which present unstable TL even for high temperature peaks ("anomalous fading": see e.g., Wintle 1978b) special additional caution must be taken before claiming TL ages (e.g., test of fading from irradiated samples kept at ambient temperature for weeks before TL reading).

* Taking into account, as mentionned above, the relative efficiency of α and β rays to produce TL (Aitken and Fleming, 1972).

2.2.2 - TL dating of detrital sediments

This method concerns all detrital sediments whose mineral components were exposed to sunlight just before their ultimate burial. In effect, recent detrital sediments may have their high temperature TL well below saturation level. An example of such a situation is shown (Fig. 8) for the coarse quartzitic fraction of a sand sample from a fixed pleistocene coastal dune in Brazil. Possible source rocks for the quartz in the area are metamorphic rocks last reheated at least 500 million years ago. Clearly, some recent zeroing process must have acted on these quartz crystals to reduce their normally saturated TL to a residual, still to be determined value. It is suggested that the zeroing mechanism is related to sunlight exposure.

Observations like the one presented in Fig. 8 were made by other authors on the 4-11 μm fraction of different kinds of eolian sediments, including oceanic sediments, continental loesses, and fixed desert dunes (Wintle and Huntley 1982, Singhvi et al. 1982). It has long been known that sunlight is able to remove TL from minerals, and therefore direct exposure to solar radiation was suggested as one of several possible bleaching mechanisms in sediments (Morozov 1968, Shelkoplyas 1971)*. Solar heating is probably to be rejected as an efficient zeroing mechanism, as at typical ambient field temperatures of 50ºC, the bleaching of high-temperature TL would require unrealistically long exposure times, up to more than 10^5 yr. On the other side, short exposure to sunlight is very effective on a time scale of hours. For instance, it was shown

*These authors also suggested that crushing may equally deeply alter the geological TL of sediments. They consider that this might be the main factor responsible for zeroing the TL in glacial deposits.

by Wintle and Huntley (1980) that a 20 min. exposure of oceanic sediments to sunlight was sufficient to reduce their TLN by a factor of 5.

Similar effect are observed for the coarse (≈100-200μm) quartzitic fraction of continental sands, as shown in Fig. 9. It appears also in the same figure that the silty fraction of dune sands may require a longer exposure to loose a major fraction of its TLN, though also on a time scale of hours. In all cases, after an initial period of rapid decrease, the TL reduces only asymptotically (Fig. 9). Sunlight, therefore, does not seem to be able to totally remove the geological TL of sediments. Effects similar to sunlight exposure can be produced in the laboratory with ultraviolet (UV) lamps of the sunlamp (mercury vapor) type, with the advantages of better reproducibility and considerable gain in exposure times. The commercially available mercury vapor ("suntanning") sunlamps have an emission spectrum from the visible down to a cut-off at 300 nanometers in the UV. This wavelength also corresponds approximately to the most intense solar radiation.

Sunlight* exposure thus appears to be a reasonable hypothesis to explain the geological TL bleaching in detrital sediments. If so, the TL history of the materials composing such sediments, if once exposed to sunlight, may be summarized as follows (Fig. 10): after an initial period within a parent rock at low

* and especially its most energetic, UV component (Fig. 1).

temperature, which may have lasted for millions of years with a geological TL at saturation, individual crystals are exposed to light as a consequence of alteration and erosion. Their high temperature TL peak height is reduced dramatically to a residual component Ro. After transportation, and eventually several stages of successive burial and sunlight exposure, detrital minerals are definitively covered by sedimentary materials and again accumulate TL at a rate depending both on their number of traps and local radioactivity. To date pleistocene sediments, the task of the present-day geochronologist is therefore to determine both Ro and its paleodose. Two methods are presently in use.* In both, one determines a least square line through experimental points with the additive dose method as for baked sediments. The difference comes from the determination of Ro (taken as equal to zero in baked sediments: see Fig. 5).

In the method proposed by Singhvi et al. (1982), one first exposes an aliquote of the powder sample under a sunlamp for a period supposedly long enough to simulate the hypothesized exposure of the sediment components to sunlight. The residual TL glow curve is then taken into account to calculate a plateau-test curve (see f.i. Singhvi et al. 1982, Poupeau et al. 1983). Then, for any temperature $t > t_p$, one can determine a glow growth curve versus dose. Taking into account the value $R'o$ of the sunlamp exposure residual at this temperature, it is possible to obtain graphically the paleodose to the sample, as the intersect of the TLN+dose curve and a parallel to the dose axis passing by $R'o$ (Fig. 11, top). The basic hypothesis of this approach is that the experimentally determined $R'o$ value is effectively equal to the Ro of the solar

* others were first tested and presented by Wintle and Huntley. They are discussed in their 1982 article.

irradiation. The assumption that R'o = Ro is not a verifiable hypothesis, however.

A second method (Wintle 1982, Wintle and Huntley 1982) does not make any assumption about the level of TL bleaching of the sample. It consists in determining two regression lines, the intersection of which directly gives Ro and the equivalent dose. These two lines are respectively the usual TLN+β doses line, and a line determined from TL measurements obtained after β-dose irradiations followed by a standard exposure time to a sunlamp (Fig. 11). Where these two methods were applied to the same sample, they were observed to give the same value (within 5%) for the equivalent β-dose (Wintle and Huntley, 1982). The second, more general method of calculating ED has now been adopted in our laboratory for the study of brazilian Pleistocene sands.

3. TL STUDIES OF PLEISTOCENE SANDY FORMATIONS IN BRAZIL

Although attempts to date sediments by thermoluminescence were undertaken as early as 1965 in the USSR (for a historical review, see Wintle and Huntley 1982), it is only in 1979 that absolute dating techniques were firmly established by Wintle and Huntley (1979, 1980, 1982) and later by Singhvi et al. (1982), for the fine (2-11 μm fraction) of eolian sediments. In 1982, we initiated an investigation on various types of sand deposits in Brazil in order to test the potential of this method for dating samples of archaeological and/or geological interest.

In order to avoid some of the problems linked to the multi-mineral composition of the bulk fine fraction of sands (variety of

zeroing mechanisms, presence of mineral phases affected by anomalous fading, or of minerals of neogenesis, etc...), we worked on the ≈100 μm fraction of these sands. Before any TL measurement, the samples were first sieved to a convenient size fraction, etched for 1 hour by HF 40% at 25ºC washed in distilled water, and dried at room temperature. This procedure generally allows one to obtain a pure quartz fraction, as checked by X-ray diffraction analysis. Laboratory irradiations were performed with a ^{90}Sr β-emitter source of 50 millicuries, delivering from 570 to 610 rads/mn to the samples, according to the sample holder type (steel or brass) and size. TL readings were made at a linear heating rate of 10ºC/sec on 4mg aliquotes, using optical filters with a maximum transmission coefficient centered respectively at a wavelength of 380 nm and 430 nm.

Exposures to UV were realized with a 275W General Electric sun lamp at a distance of 30cm, a geometry allowing simultaneous exposure of up to 25 sample holders.

3.1 - Sands from coastal formations

The origin and dynamics of coastal dune complexes is controled by geological, oceanographic and climatic factors (Pye, 1983a). In a given coastal dune system, it is not always easy to decipher the relative contributions of these three influences. Moreover, where dune systems occupy wide areas, geological methods cannot resolve all events in their evolution and there may exist difficulties in correlating local stratigraphies. This kind of situation characterises the Coastal Plain which lengthens over more than 500 km in the state of Rio Grande do Sul (Brazil), and continues

northward to the state of Santa Catarina and southwards to Uruguay (Jost and Soliani 1976). This Coastal Plain extends for its major part over a width of 200 km and is partly divided parallel to the coast into two sections by a lagoon system dominated by the Porto Alegre open lagoon and the closed Mirim lagoon.

This vast stretch of mainly sandy formations, testifies of repeated cycles of marine transgression, and therefore of multiple sedimentation-erosion sequences, locally modulated by climatic (wind, rain, etc...) influences. Although geomorphologic and sedimentary studies allow to distinguish some major events recognisable over wide areas, it is not yet possible to reconstitute precisely the history of this formation. Different indexes suggest that the Coastal Plain is a recent formation, which might have formed within the last 300 000 yr. The area therefore seemed of great interest for a test program of TL behaviour in coastal formations both because of the age range involved and the diversity of available sediments, from shallow water deposits to eolian dunes.

A group of 10 samples, representing diverse deposition ages and sedimentary ambiances, were selected. In some samples, the paleodose was determined following method 1 of Fig. 11 (Poupeau et al. 1983). Our procedure is depicted in Figs. 12 to 14. We first determined the shape of the residual TLN after a 15 hours sunlamp exposure as shown in Fig. 12 and used this result for the plateau test in Fig. 13 which shows TLN was quantitatively retained from 300ºC (other samples may have better defined plateau as RMG 04A, see Fig. 9 in Poupeau et al. 1983). The deduction of the

paleodose from a combination of the additive dose method and prolonged sunlamp exposure is depicted in Fig. 14. This procedure has been applied to sample 04A and 05B of table 1. The paleodose to other samples was determined following method 2 of Fig. 11. An example is shown in Fig. 15 with sample RMG 05A. The paleodoses reported in table 2 were all measured from the maximum height of the high temperature peak, at 380ºC. Radioactivity measurements of the samples are not yet completed and thus annual doses cannot at this stage be precisely derived. However, considering that the average sand annual dose rates are of the order of 0.5 rads, our present results are in reasonnable agreement with geological expectations (table 1, column 4). At the moment, the most important results of this study are that (i) all sands from Rio Grande do Sul Coastal Plain (including some not shown in table 2, for which TL measurement are still incomplete) have an unsaturated TL, implying that all have been submitted to a zeroing process, only one being rejected for TL analysis, due to poor measurement reproducibility (table 1); (ii) from essentially the error involved in the correction for the residual thermoluminescence Ro, it appears that samples of ages $\lesssim$ 1 000 yr could be difficult to date with a precision better than $\pm$ 50%, especially when supralinearity (see §4 below) effects are present (Fig. 16) whereas an analytical precision on the age of $\pm$ 10-15% could be reached for older sands; (iii) from the linear behaviour of the additive dose data to the TLN, samples as old as at least 200 000 yr could still be dated by this method.

The basic hypothesis underlying these measurements is that coastal sands before final deposition were efficiently zeroed by the sun. As a test, we measured the TL surficial sand of the Copacabana beach in Rio de Janeiro and other beaches in the

region and effectively verified that their TLN was unaffected either by prolonged sun or sunlamp exposure. We consider this result consequent with the fast solar zeroing rate previously observed (Fig. 9).

3.2 Sands from intra-continental formations

The first attempt of TL dating of intra-continental sands was led on eolian dunes from the Rajasthan desert in India (Singhvi et al. 1982). We are presently working on a series of sand deposits resulting from water transportation.

One site under scrutiny is a sandy terrace situated on the internal side of a meander from the Rio da Cabeça near the city of Rio Claro (state of São Paulo). The Rio da Cabeça is one of numerous small rivers which ensure the drainage of natural waters in the area. The choice of the sampling locality, within the archaeological excavations of the Alice Boer site, was dictated by its acknowledged importance in Brazilian archaeology (Beltrão 1974). Briefly, the site can be described as a recent sandy terrace lying on older, high energy fluvial deposits. Lithic artifacts were uncovered both in the central part of the sandy units numbered 5 to 8 in Fig. 17, as well as on top of the blocky layer 4. From previous ^{14}C (Beltrão 1974) and TL (Beltrão et al. 1982) measurements, it appears that the artefacts in the sands were deposited for a time range extending from ≈2000 yr to at least 14 000 yr BP. Extensive sand sampling was recently made within the four most recent archaeologic excavations (Poupeau) 1984). At all levels in the site, the TLN of sands was found to be

unsaturated and presents a linear growth with dose.The paleodoses to the first samples already analysed were found to increase by a factor of 5 from the top of the cultural sandy layers to their bottom. From a known γ-dose rate in the site of 0.2 rads/yr (Danon et al. 1981) and reasonnable assumptions on the relative abundances of U Th and K in sands, one can deduce a minimum time span for the deposition of the lithic industry extending from $\approx$3 000 yr to $\approx$15 000 yr BP (Poupeau, Zuleta, Souza, Rivera, da Cunha, Danon and Beltrão, unpublished data). Taking into account a $\approx$30% uncertainty in the ages evaluation (mainly due to the still poorly known annual dose rate), these results are in agreement with previously published ages. Radioactivity measurements of the sands, now in progress, will soon allow to get a higher precision on these ages, whose archaeological implications will be discussed elsewhere (Poupeau and coll., in preparation).

Another kind of waterlain sand deposit under study in our laboratory is of the slope and fan-type deposit. The formation investigated is constituted of napes of sands deposited along a quartzitic belt near to the town of Central (Bahia). From a geomorphologic point of view, these deposits are estimated as "recent", although of unknown age. Several samples were taken at different depths within the $\approx$ 5m high stratigraphic column in a single sand quarry. There too, all samples were found with an unsaturated TLN and linear growth with dose. In one of these samples, the paleodose was calculated either from additive β-doses and sunlamp exposures, or γ-doses and sunlight exposure (Fig. 18). Both methods gave same β- or γ-equivalent doses and same level of residual TL Ro, thus experimentally demonstrating the equiva-

lence of our sunlamp exposure procedure and of sunlight TL bleaching. Other comparative measurements of this type on sands from different origins (Souza and Poupeau, unpub. data) confirm this result.

From our present experience on intra-continental and coastal sand formations from Brazil, it therefore appears that a very efficient zeroing mechanism may lower drastically the TLN of coarse ($\approx$100μm) detrital quartz crystals previous to their final deposition. This zeroing mechanism is most probably of a radiative origin, and attributable to the UV component of the sunlight. From the experimental precision in the evaluation of the paleodose and annual dose of equation (1), ages of $\pm$ 10% to 15% could in principle be obtained. However, before TL can be applied as a dating method, a series of problems, some of which listed below, have to be resolved.

4. PROBLEMS AND PROSPECTS

Somewhat arbitrarily one could classify the problems associated to the TL dating of sands into three categories, bearing respectively with the paleodose and annual dose measurements and the geologic accuracy-or meaning- of such TL ages.

A first series of problems is linked to the measurement of the paleodose, due to non-linearity of the high-temperature TL with dose. In the ideal case presented in Fig. 4, the only departure from linearity do occur at high doses, near to saturation of the electron traps. One example of this is given in Fig. 19 on a coastal sand from south-eastern Brazil. In such a case, a backward linear extrapolation to get the β-equivalent paleodose

(as in Figs. 7 and 11) is no more possible. Only a knowledge of the TL saturation function with dose combined with a precise experimental determination of the saturation curve would make this possible. Unfortunately, at the moment, there is not yet agreement on the "best" fitting function for different materials (see f.i. Wintle and Huntley 1982). Another embarassing observation made for the fine-grained (4-11μm) fraction of loess and oceanic sediments is a marked loss of sensitivity at high doses without reaching saturation before very high doses (Fig. 20a), which in old samples might mimic a pseudo-linearity and led to unrealistically high paleodoses. This effect has not yet been studied for sands. From our experience, this do not occur in the quartz size fraction that we measured (see e.g. Figs. 13 and 19). Nevertheless, this effect will have to be considered seriously in the future, for the dating of samples older than $\gtrsim$50 000 yr.

In some young samples, another non-linearity effect might be important in the determination of the paleodose. It is the supra-linearity effect, into which the TL growth with dose is initially faster than linearly (Fig. 20b). Under favorable conditions, it is possible to take into account in the calculation of the paleodose this supra linearity effect (Aitken 1974, Poupeau 1983). The supra-linearity effect, when present, is always limited to an additionnal $\lesssim$0.5 krads dose to the paleodose as determined e.g. from Fig. 7. In other terms, it will in practice be negligible for samples of age $\gtrsim$ 5 000 yr.

Another category of problems is linked to the correct determination of the annual dose rate. In effect, all methods of calculation of this dose rate rely on hypothesis about (i) the cons-

tancy of U, Th and their daughters, and K content with time,(ii) the water impregnation of the material measured. The content of radioactive isotopes in a sediment may have varied with time, with possible alternances of periods of deposition and leaching. The water content of a sediment may have varied with climate modification as well as further sedimentation, tectonic effects, etc... The resulting changes of humidity within a sediment may have influenced the retention or loss of water soluble or volatile (e.g. the gaseous radon 222) products in the radioactive series of U and Th, and in any case, modulate the annual dose rate (water is a good absorber of ionising radiations).

Finally the geological meaning of the TL age of a sediment has to be considered. If the zeroing mechanism in sands has to be attributed to the solar UV (with eventually other effects in the case of glacial deposits as mentionned above), it is not yet known for instance how the TL would be affected by one or several episodes of reworking and resedimentation under water. Neither have post-depositionnal modifications themselves been investigated. These include mechanical (slumping, compaction), biological (bioturbation), mineralogical (authigenesis and cementation) effects, in addition to the weathering effects (see e.g. Pye 1983b) discussed above.

Other parameters to be evaluated in the dating of sediments in general have been listed by Wintle and Huntley (1982) to which we refer for more details.

The complexity of the processes involved in the buildt-up of TLN in "zeroed" sediments preclude their theoretical prevision for individual samples. One of the best approaches presently is

to rely on series of samples whose ultimate deposition age is well known from other methods. This will necessitate for a proper evaluation of the geological accuracy of TL dating of sediments, a close collaboration of the dating laboratories with other specialists in related fields as geology and archaeology.

5. SUMMARY AND CONCLUSIONS

Much progress were realized during the last decade in the geochronology of the quaternary. New methods were proposed, new kinds of materials and events appeared to be datable, over a wider time range than before. The gap between the time range covered by the ^{14}C method and other nuclear geochronometers practically faded away.

One of the most significant progress concerns the dating of sedimentary rocks, which according to their nature, may now become directly datable using either thermoluminescence, electron spin resonnance or accelerator dating. Our own investigation by TL of sand units from various geological contexts in Brazil reveal strong potentialities for the upper pleistocene geochronology. One task of the coming years will now be to proceed to a serious evaluation of these methods, by using series of test samples of well known ages and with supposedly simple history, as well as from a better theoretical knowledge of the TL phosphors themselves. Progress in instrumentation also will no doubt increase considerably our present analytical possibilities.

Acknowledgements

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LEGENDS OF FIGURES

Fig. 1 : Band model for the representation of energy levels of electrons in a solid. In typical thermoluminescent minerals, the forbidden band width is of the order of $\approx$10 electron-volts, while electron-trap depth are down to $\approx$2 eV below the conduction band and the luminous center up to $\approx$3 eV above the valence band.

At its lower energy level in an atom, an external electron belongs to the valence band of energy and is said to be at its ground state (fundamental level). Upon ionisation such an electron can jump through the forbidden energy band (an expression of the quantized nature of electron energy levels in an atom) to the conduction band where it is mobile and can eventually be trapped in a lattice defect within the forbidden band. Its positively charged counterpart, or "electron hole" is similarly mobile within the conduction band and can also be trapped within the forbidden band, where it is called a luminous center.

The trapped electron can be liberated if enough energy is given to him to reach again the conduction band, from which it can recombine radiatively or not with an ionised atom. Electrons in shallow (<1 eV) traps can get out from their traps by simple thermal agitation at ambiant temperature much faster than they fill into. For deep ($\gtrsim$1.4 eV) traps, electrons have lifetime in their traps of $\gtrsim 10^6$ yr. allowing TL dating in the upper pleistocene.

Giving energy by thermal heating (or UV photons) to a thermoluminescent mineral allows electrons in deep traps to be liberated to the conduction band, from which they can recombine with lattice atoms. In the process, some will reach luminous centers as shown in the figure, giving rise to photons in the visible or UV part of the spectrum (alternative electron paths for the production of TL are given in e.g. Bruaux, 1962).

Fig. 2 : Apparatus for TL studies. The sample,usually a powder, is within a sample holder on the heating element of an oven. In order to avoid any spurious light, the oven is isolated from daylight and operates with a neutral atmosphere. The light emitted upon heating is selected by optical filters and collected by a photomultiplicator tube, where each photon is amplified. The heating rate and temperature of the sample are controlled via a thermo-couple in contact with the heating element of the oven. The number of photons emitted as a function of heating temperature is finally plotted on a x-y plotting table.

Fig. 3 : Natural thermoluminescence (TLN) in a) a flint artefact, burned in an archaeological fire some 5 000 years ago. The glow curve presents only one peak with a maximum at 380ºC; b) an upper pleistocene stromatolite sample from the Gregory Rift (Tanzania), with 2 peaks at respectively 280ºC and 320ºC. (Both samples were linearly heated at 10C/sec, their TL being observed in the blue region of the optical spectrum).The lower curve (blackbody curve) refers to the light emitted by the TL oven itself when heated at $\gtrsim$400ºC.

Fig. 4 : Evolution of the high temperature TLN of minerals with dose. The first part of the curve is generally linear (see text). According to mineral species and environmental radioactivity, the high temperature TL even - tually reaches a saturation value (corresponding to the population of all available deep electron traps) within less than 10^5y to more than 10^7 yr.

Fig. 5 : Zeroing mechanism by heating a thermoluminescent mineral at a temperature $\gtrsim$ 500ºC. All electrons traps are emptied.

Afterwards, the TLN builds again linearly with time. This case is typical of ceramics and of soils burned below a hot expanding lava flow.

Fig. 6 : Plateau-test for TL dating. bottom: typical glow curves for a ceramics, respectively for the TLN and TLN + a laboratory β-dose of irradiation. top: the ratio of the two curves, at a temperature $t = t_p$ reaches a constant, or plateau value, where trapped electrons were quantitatively kept in deep traps. It is for the TL emitted at temperatures greater than t_p that TL ages can be determined (except with the pre-dose method, see foot note).

Fig. 7 : Cumulative-dose method for the determination of the palaeodose in burnt sediments. In this method, different laboratory doses of β or γ radiations are given to aliquotes of the sample powder. If for a given $t \geq t_p$, the TL intensity increases linearly with dose, one can extrapolate backwards to a zero intensity level (Fig. 5), to obtain a β-equivalent dose ED. Complications may arise when this glow-growth curve is not linear with dose (see section 4).

Fig. 8 : Glow growth curve of the TL emitted at 380ºC by the coarse (150-200μm) quartzitic component of a fixed coastal sand dune in southern Brazil. The linear growth with dose indicate the TLN is far below saturation.

Fig. 9 : Effect of sunlight exposure on the TL of natural minerals. top: silty (2-11μm) fraction of desert dunes from Rajasthan, India (after Singhvi coll. 1982); bottom, coarse (100-200μm) quartzitic fraction of a coastal dune, Brazil. Note that the time scale to reduce the ini-

tial TL to a residual value is about ten times shorter for the coarse quartz fraction than for the bulk silt.

Fig. 10 : TL history of detrital sediments. 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 t and their high temperature TL drops down to a residual value Ro. III. After ultimate burying under younger sediment layers, the TL builds up again up to the present level. For times from present to $\gtrsim 10^5$ yr, the growth is linear.

Fig. 11 : Determination of the palaeodose for detrital sediments. In both methods one determine the TLN + β-dose curve as in Fig. 7. To account for the residual TL effect Ro, one can either 1) expose for a long time (we used 15h) an aliquote of the sample to be dated under the light of a sunlamp and assume the TL residual R'o residual to be equivalent of the Ro of Fig. 10 above (top), or 2) expose aliquotes which received different β-doses to a standard time (we use 1 hour) under a sunlamp, before TL reading (bottom). The intersect of the TLN+ β-dose and TLN+ β-dose + UV lines gives both the equivalent β-dose to the sample and the TL residual Ro. All experimental points are measured on the TL glow curve at the same temperature $t > t_p$.

Fig. 12 : Glow curves corrected for black body radiation of the TLN, TLN + 6.72 krads β - irradiation and TLN + 15h sunlamp exposure of RMG 05B. All curves are averages over five measured aliquotes (From Poupeau et al. 1983).

Fig. 13 : Plateau-test for RMG 05B, with TLN and TLN + Dose corrected for residual sunlamp intensities Ro. This curve has been established for the TLN peak temperatures at 403ºC from the glow curves of Fig. 4 (Poupeau et al. 1983).

Fig. 14 : Determination of the equivalent-dose to sample RMG 05B according to the method described in Fig. 11 (top). Note that above 12 krads, the TL growth is no more linear with dose and reach a saturation level for doses $\gtrsim$ 20 krads. The TL was measured at 403ºC, temperature of the peak maximum (see Fig. 12).(from Poupeau et al., 1984).

Fig. 15 : Determination of the equivalent β-dose for sample RMG 05A using the method described in Fig. 11, bottom.

Fig. 16 : Determination of the equivalent β-dose of sample RMG 03B with the second method of Fig.11. For this young (1100 $\pm$ 500yr) sample the precision on the paleodose is limited to $\approx \pm$ 50%. Compare with the older sample RMG 05A (Fig. 15) where, with about the same precision on regression lines, equivalent dose can be determined with a precision of $\pm$ 10%.

Fig. 17 : Sketch of the Alice Boer terrace site (Rio Claro, Brazil). Sedimentary units 5 to 8 are made out of diverse units of sands deposited under variable regimes by the now subdued rio at right. The central units 6 and 7 contain a rich lithic industry, while more primitive artefacts are lying atop of layer 4.(from Poupeau 1984)

Fig. 18 : Comparison between sunlamp and sunlight exposure of the same quartzitic fraction (100-125μm) of a sand from the vicinity of Central (Bahia). In one case the additive dose were given by the β rays of a ^{90}Sr radioactive source and in the other, by the γ rays of a ^{60}Co source. Note that the Equivalent Radiation Dose and residual TL values are the same in both methods of TL irradiating and bleaching.

Fig. 19 : Example of a sample near to TLN saturation, sample PR5 (quartz grain size 160-200μm).

Fig. 20 : Non linearity in TL growth. In addition to the idealized TL growth curve with dose of Fig. 4, one can meet with either a) lowered sensitivities well before reaching saturation, or b) supralinearity effect in the initial part of the TL growth curve. See text.

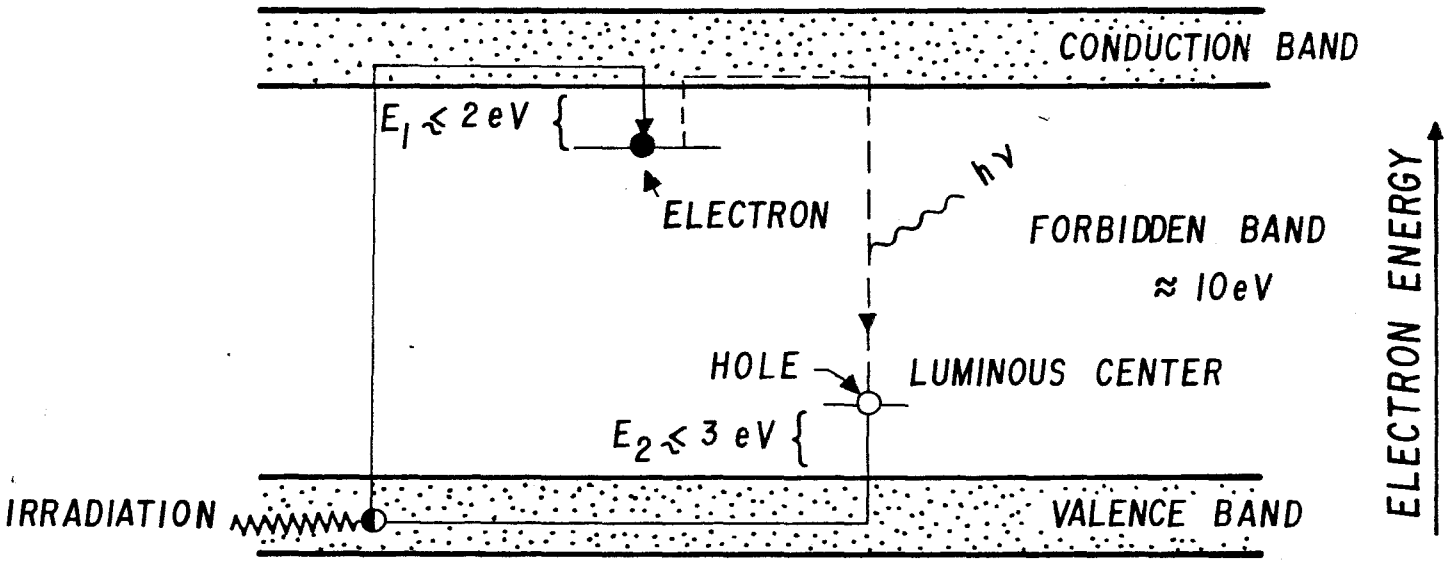
CONDUCTION BAND
$E_1 \lesssim 2$ eV
ELECTRON
$h\nu$
FORBIDDEN BAND
$\approx$ 10eV
HOLE
LUMINOUS CENTER
$E_2 \lesssim 3$ eV
IRRADIATION
VALENCE BAND
ELECTRON ENERGY

Fig. 1

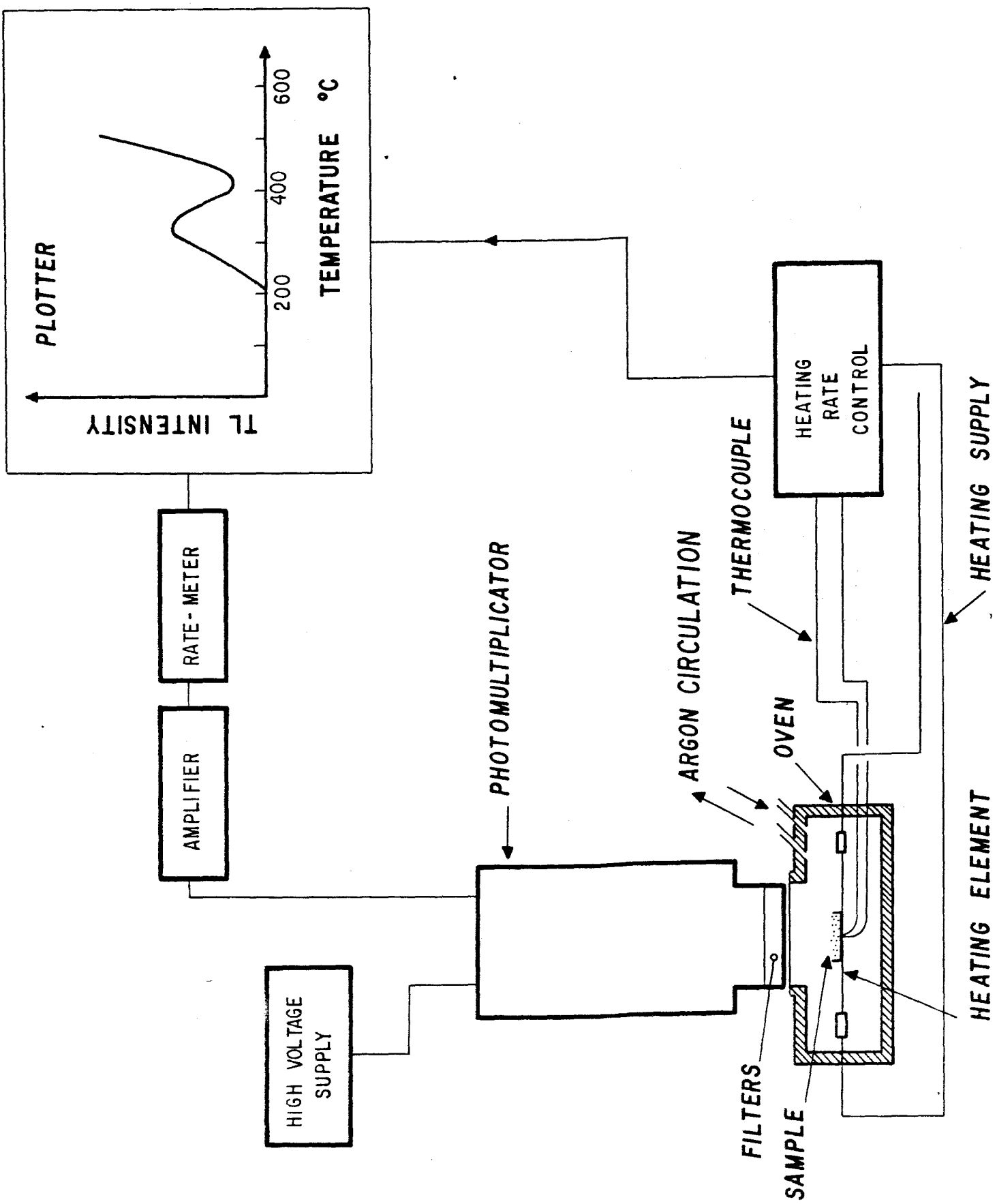
PLOTTER
TL INTENSITY
TEMPERATURE °C
200
400
600
RATE-METER
AMPLIFIER
HIGH VOLTAGE SUPPLY
PHOTOMULTIPLICATOR
ARGON CIRCULATION
THERMOCOUPLE
OVEN
HEATING RATE CONTROL
HEATING SUPPLY
HEATING ELEMENT
FILTERS
SAMPLE

Fig. 2

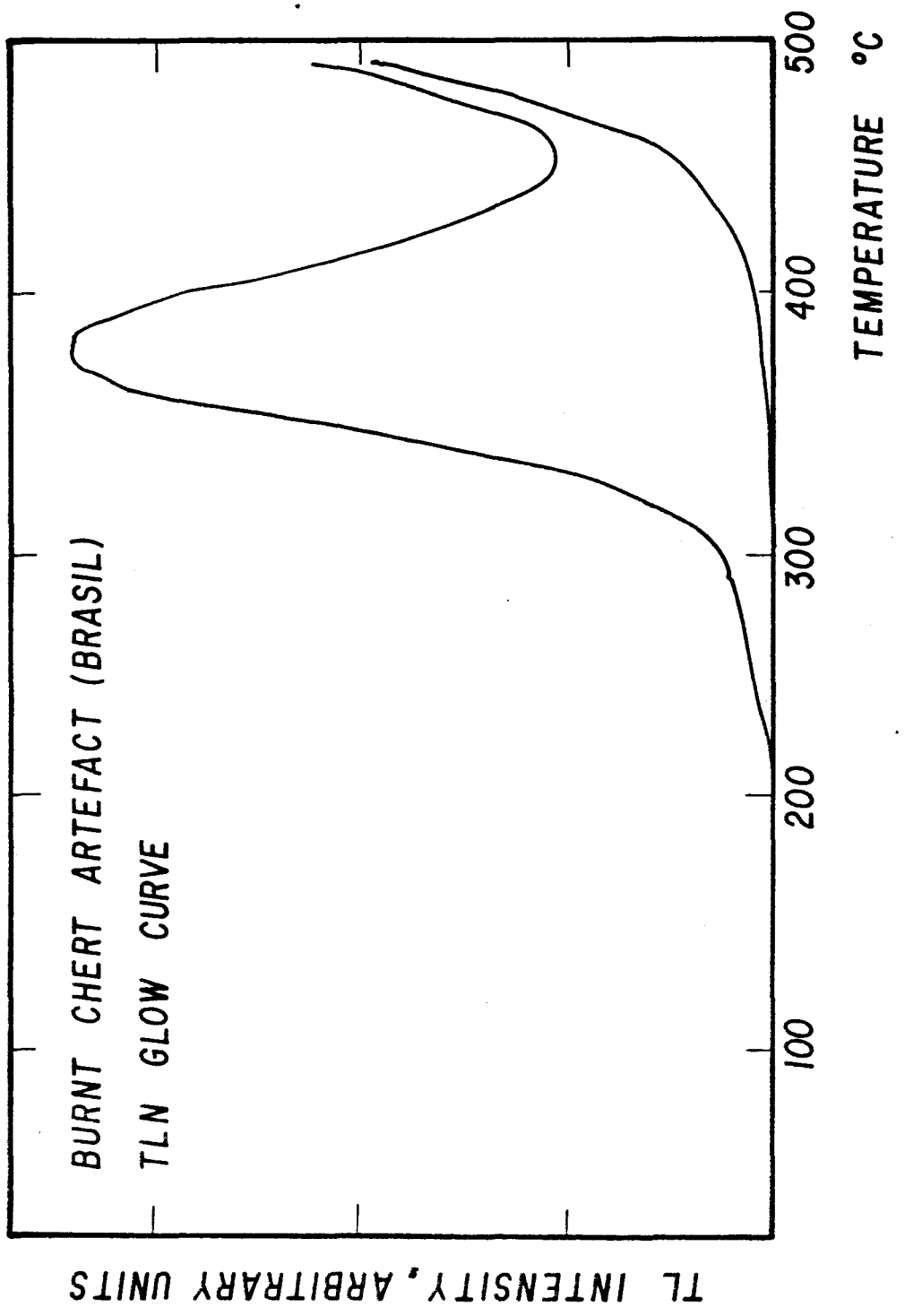
BURNT CHERT ARTEFACT (BRASIL)
TLN GLOW CURVE
TL INTENSITY, ARBITRARY UNITS
100
200
300
400
500
TEMPERATURE °C

Fig. 3a

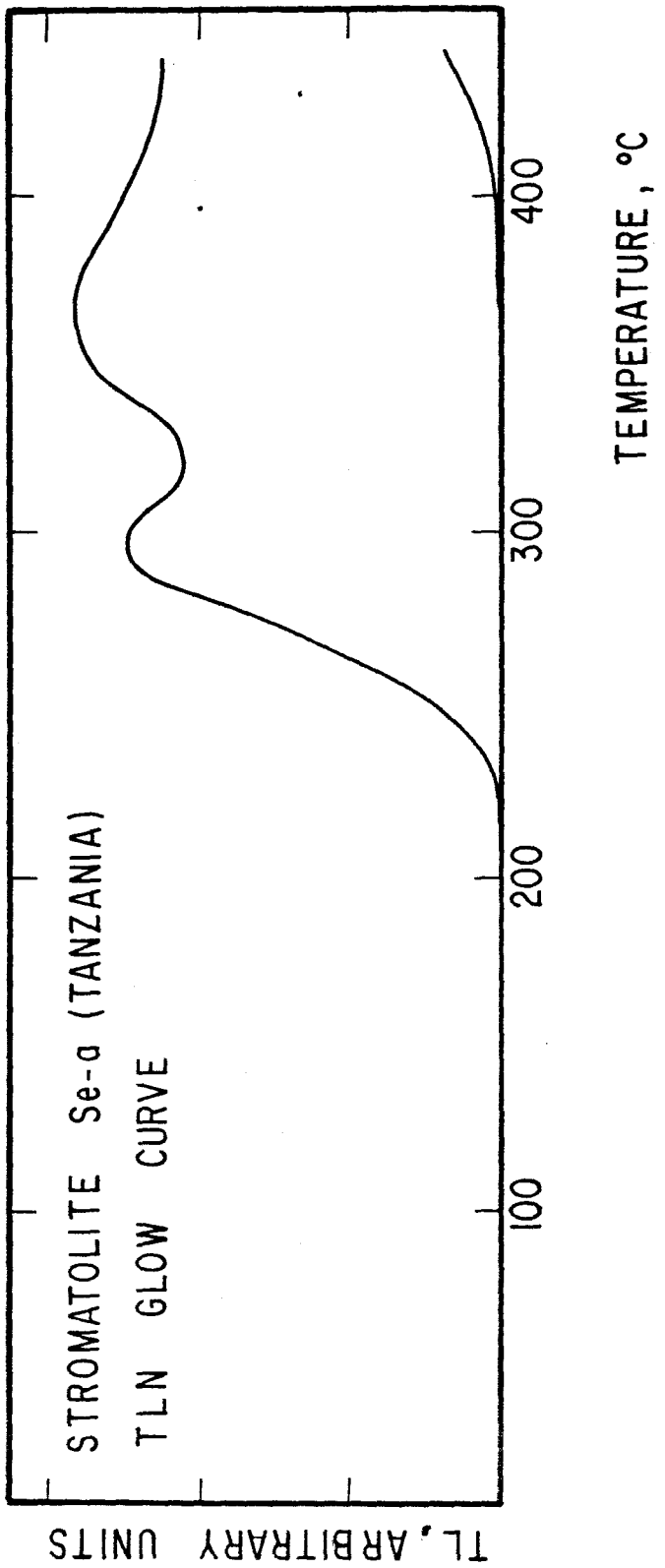
STROMATOLITE Se-a (TANZANIA)
TLN GLOW CURVE
TL, ARBITRARY UNITS
100
200
300
400
TEMPERATURE, °C

Fig. 3b

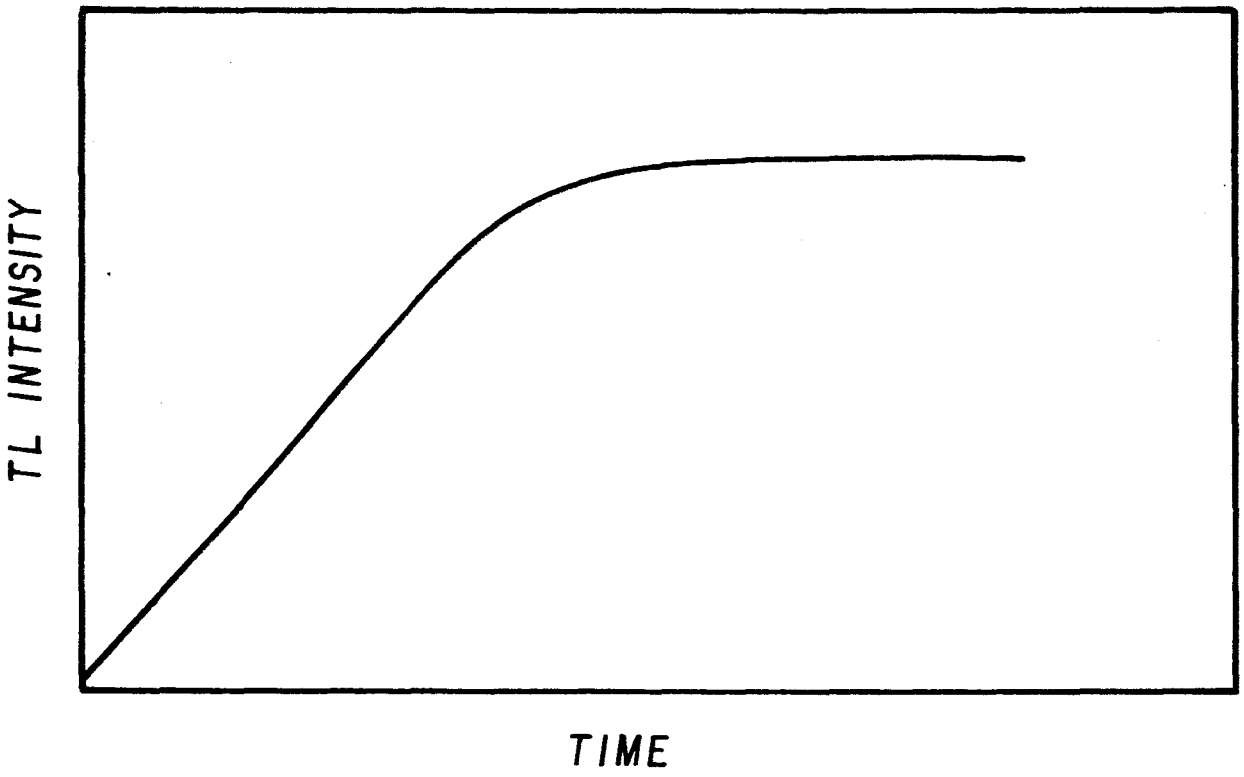

Fig. 4

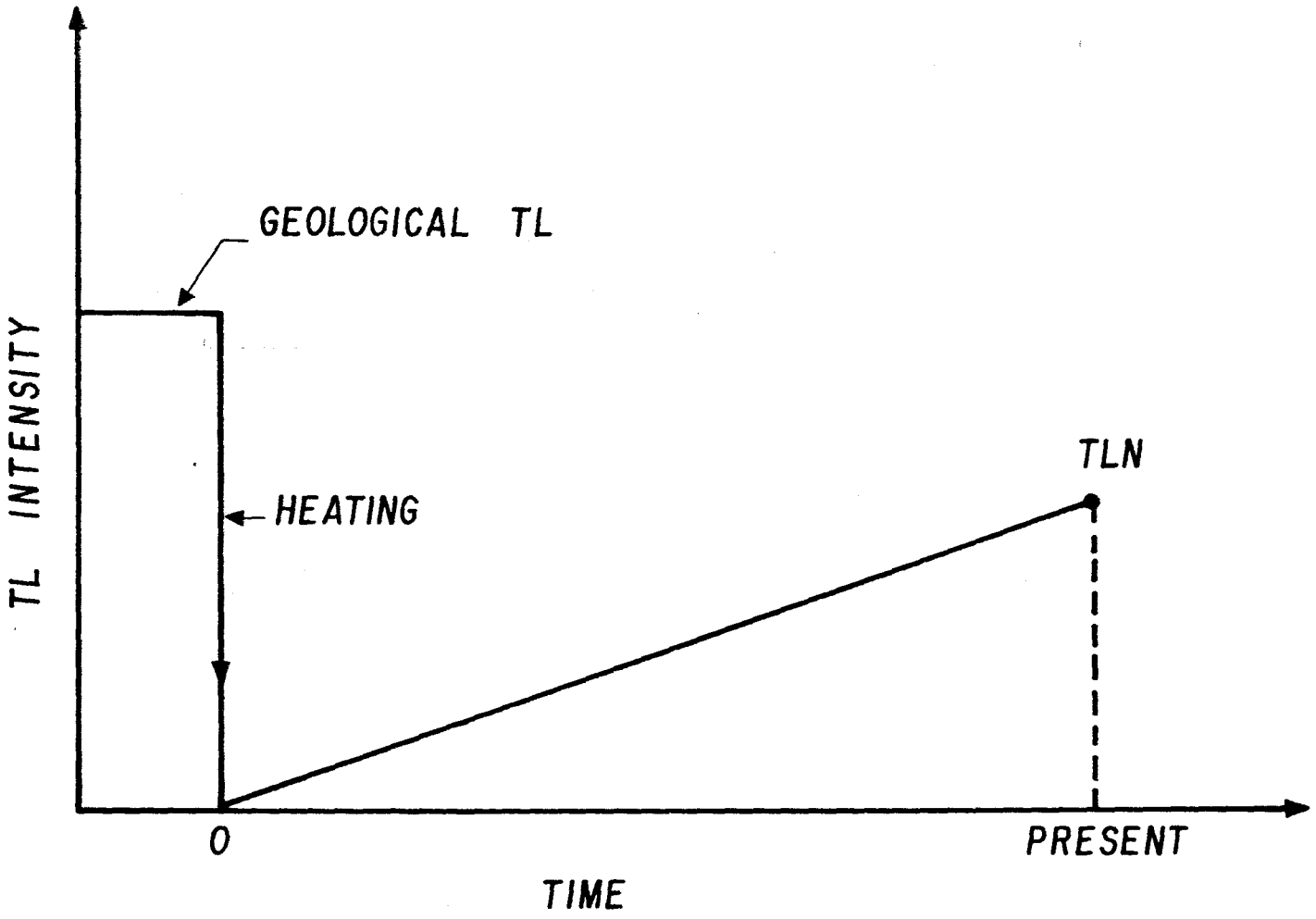

Fig. 5

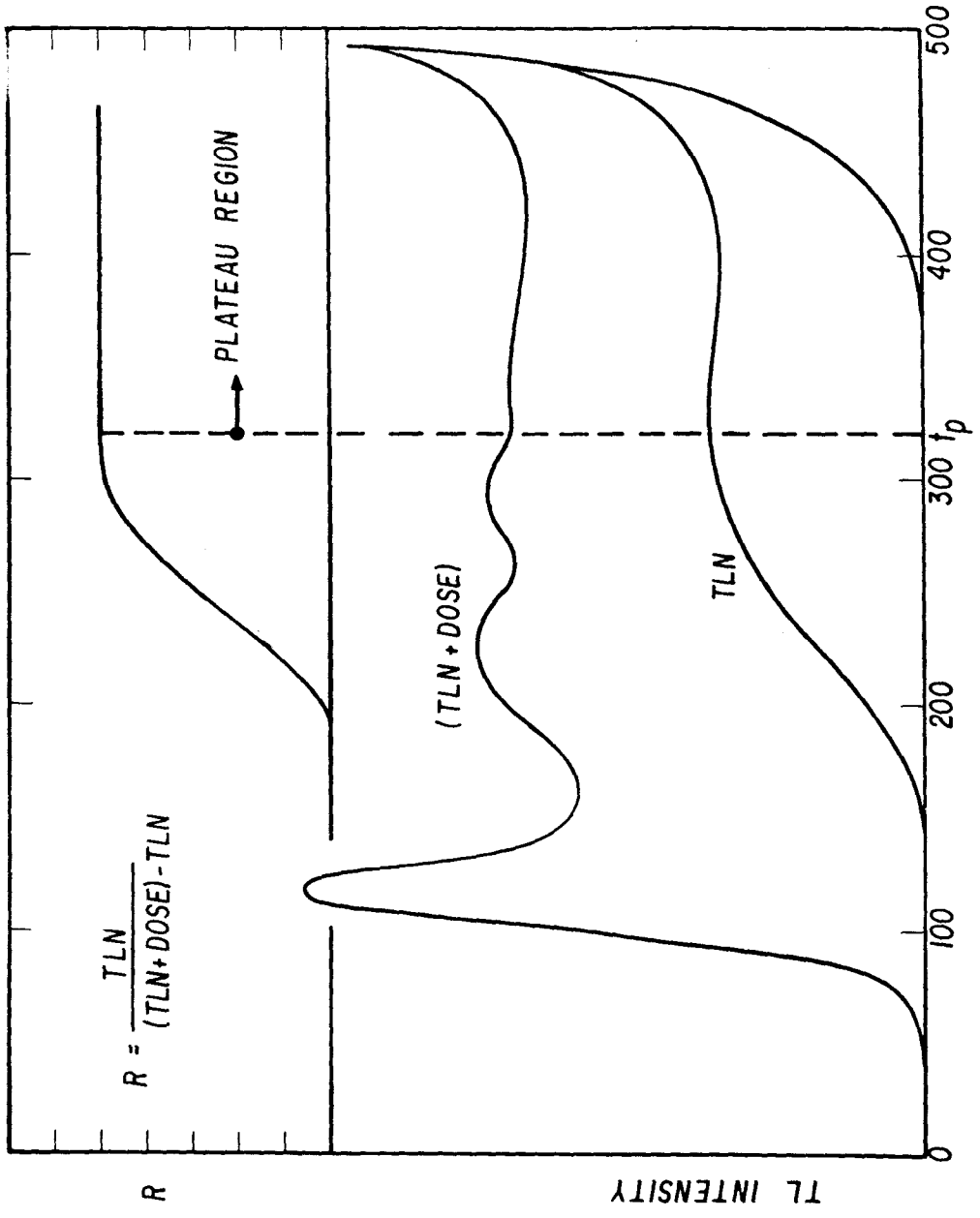
R = TLN / (TLN+DOSE)-TLN
PLATEAU REGION
(TLN+DOSE)
TLN
R
TL INTENSITY
0
100
200
300 t_p
400
500
TEMPERATURE , °C

Fig. 6

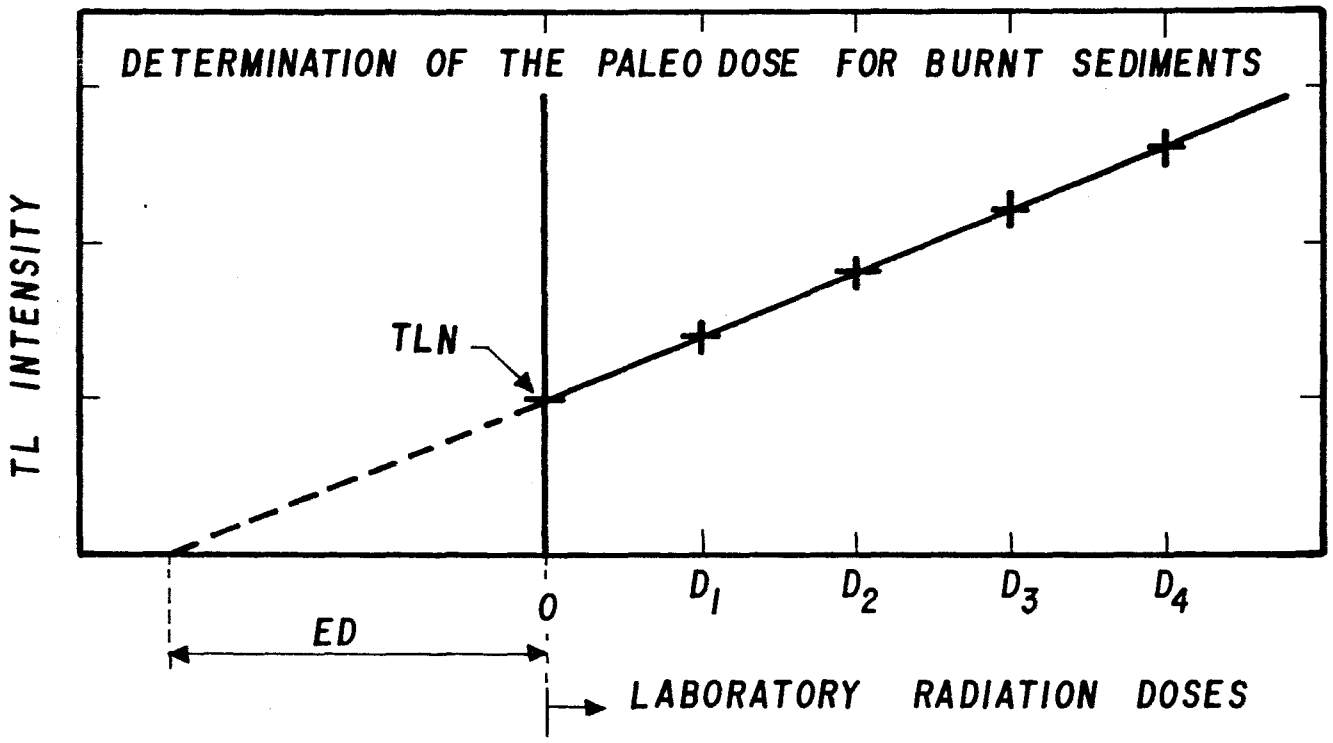
DETERMINATION OF THE PALEO DOSE FOR BURNT SEDIMENTS
TL INTENSITY
TLN
ED
0
D_1
D_2
D_3
D_4
LABORATORY RADIATION DOSES

Fig. 7

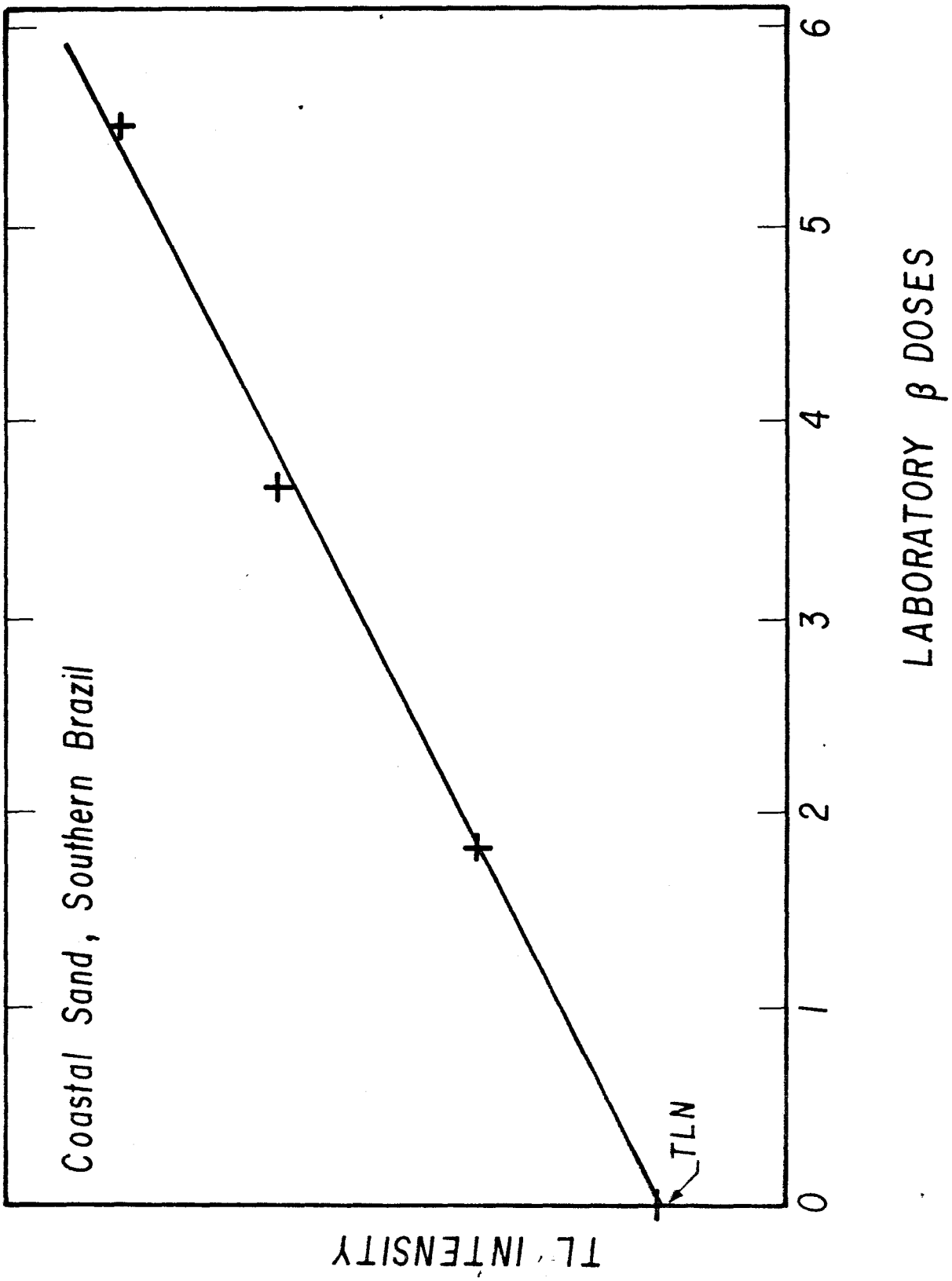
Coastal Sand, Southern Brazil
TLN
TL INTENSITY
0
1
2
3
4
5
6
LABORATORY β DOSES

Fig. 8

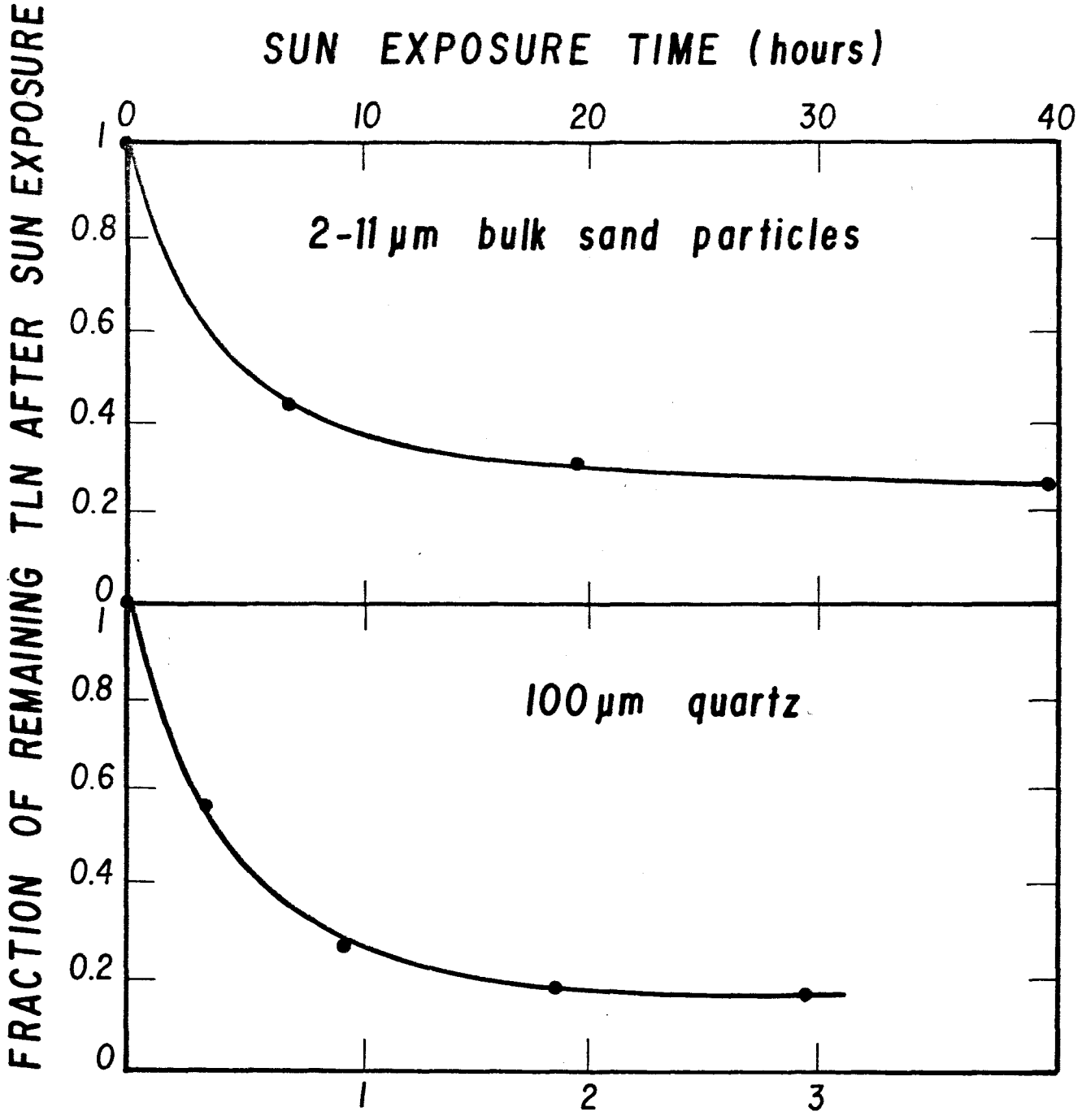
SUN EXPOSURE TIME (hours)
0
10
20
30
40
FRACTION OF REMAINING TLN AFTER SUN EXPOSURE
1
0.8
0.6
0.4
0.2
0
2-11 μm bulk sand particles
100 μm quartz
1
2
3

Fig. 9

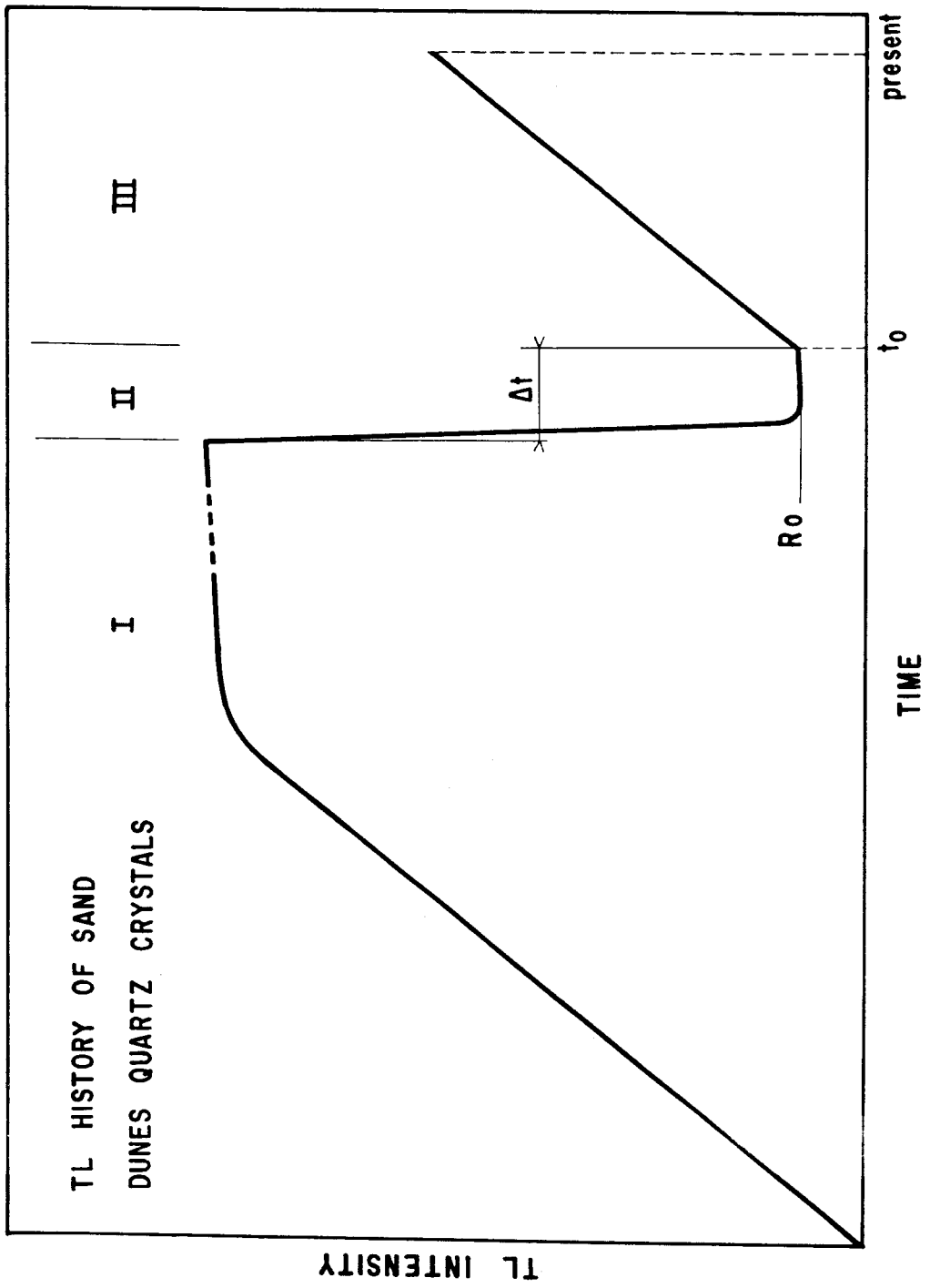
TL HISTORY OF SAND
DUNES QUARTZ CRYSTALS
I
II
III
Δt
Ro
t_o
present
TIME
TL INTENSITY

Fig. 10

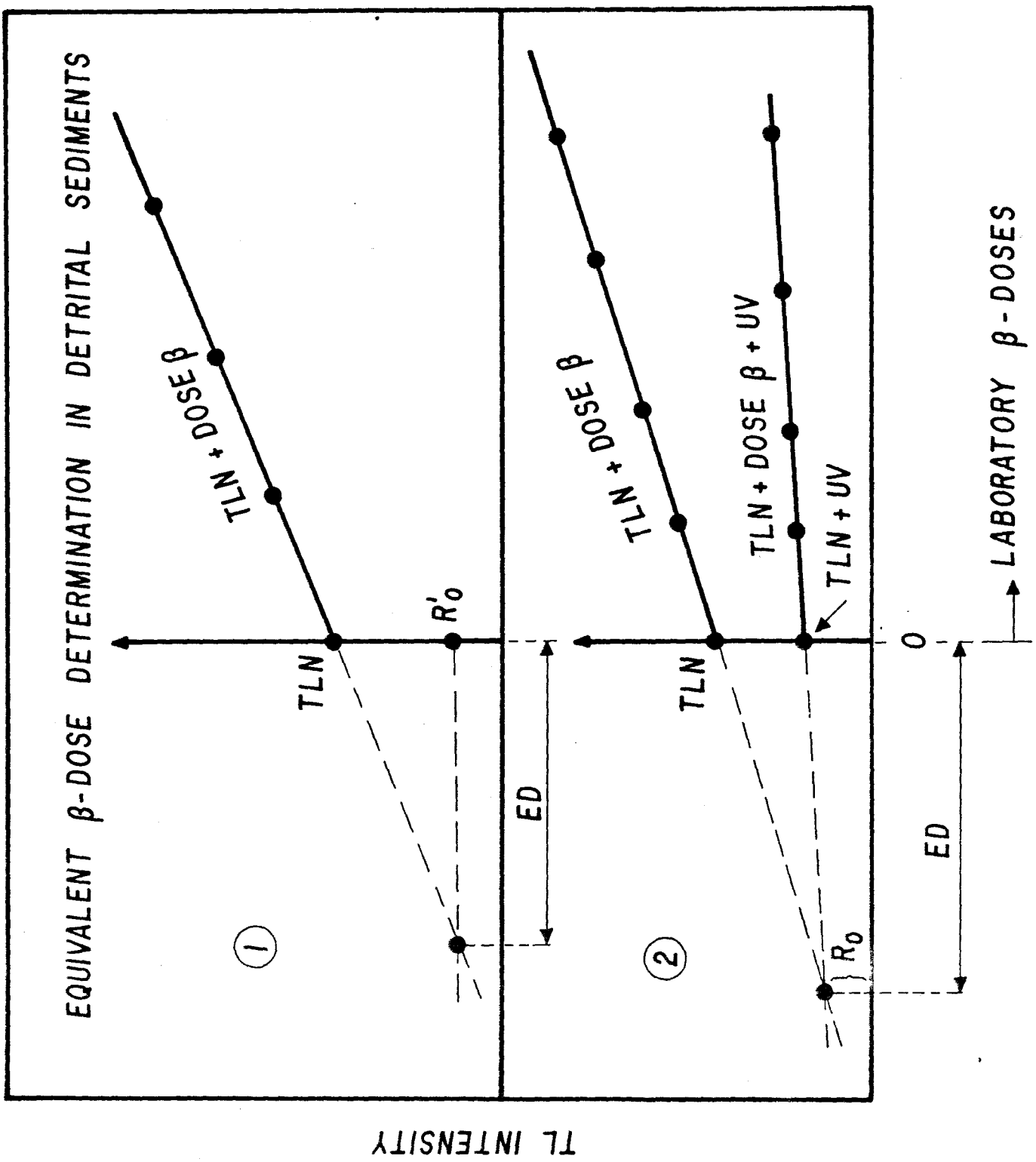
EQUIVALENT β-DOSE DETERMINATION IN DETRITAL SEDIMENTS
TLN + DOSE β
TLN
R'o
ED
①
TLN + DOSE β
TLN + DOSE β + UV
TLN + UV
TLN
0
ED
Ro
②
LABORATORY β-DOSES
TL INTENSITY

Fig. 11

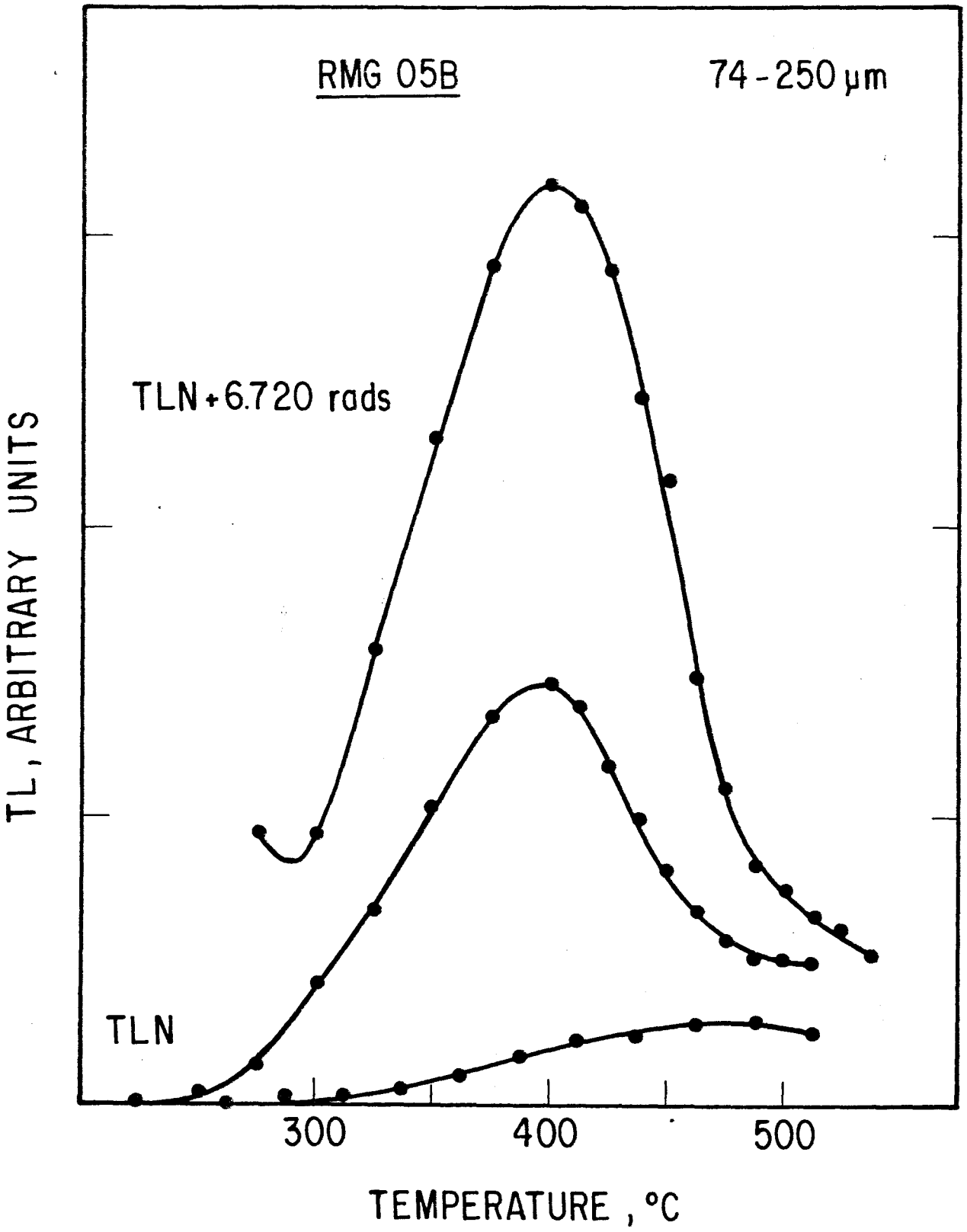
RMG 05B
74 - 250 µm
TLN + 6.720 rads
TLN
TL, ARBITRARY UNITS
300
400
500
TEMPERATURE , °C

Fig. 12

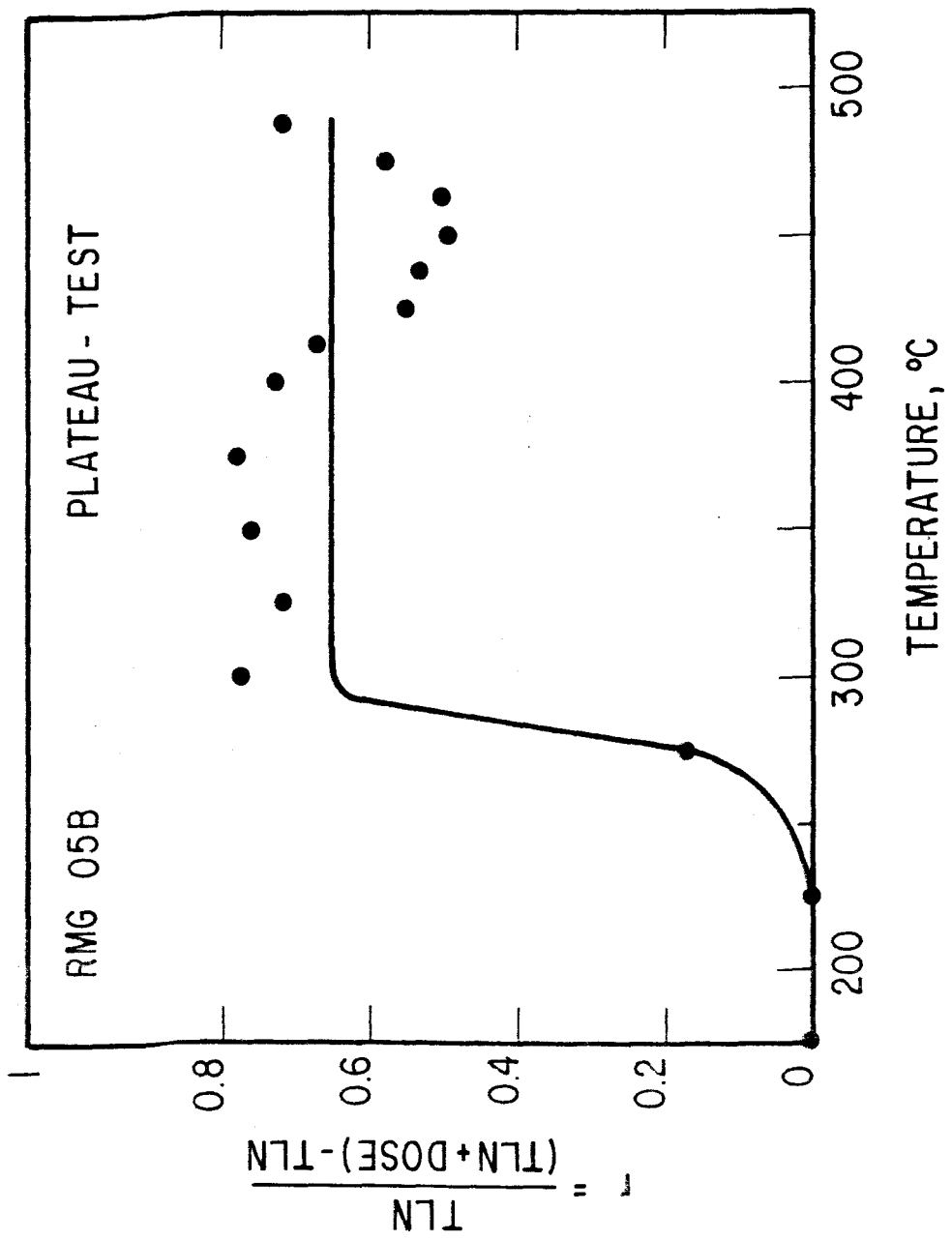
RMG 05B
PLATEAU-TEST
TEMPERATURE, °C
200
300
400
500
0
0.2
0.4
0.6
0.8
1
f = (TLN+DOSE)-TLN / TLN

Fig. 13

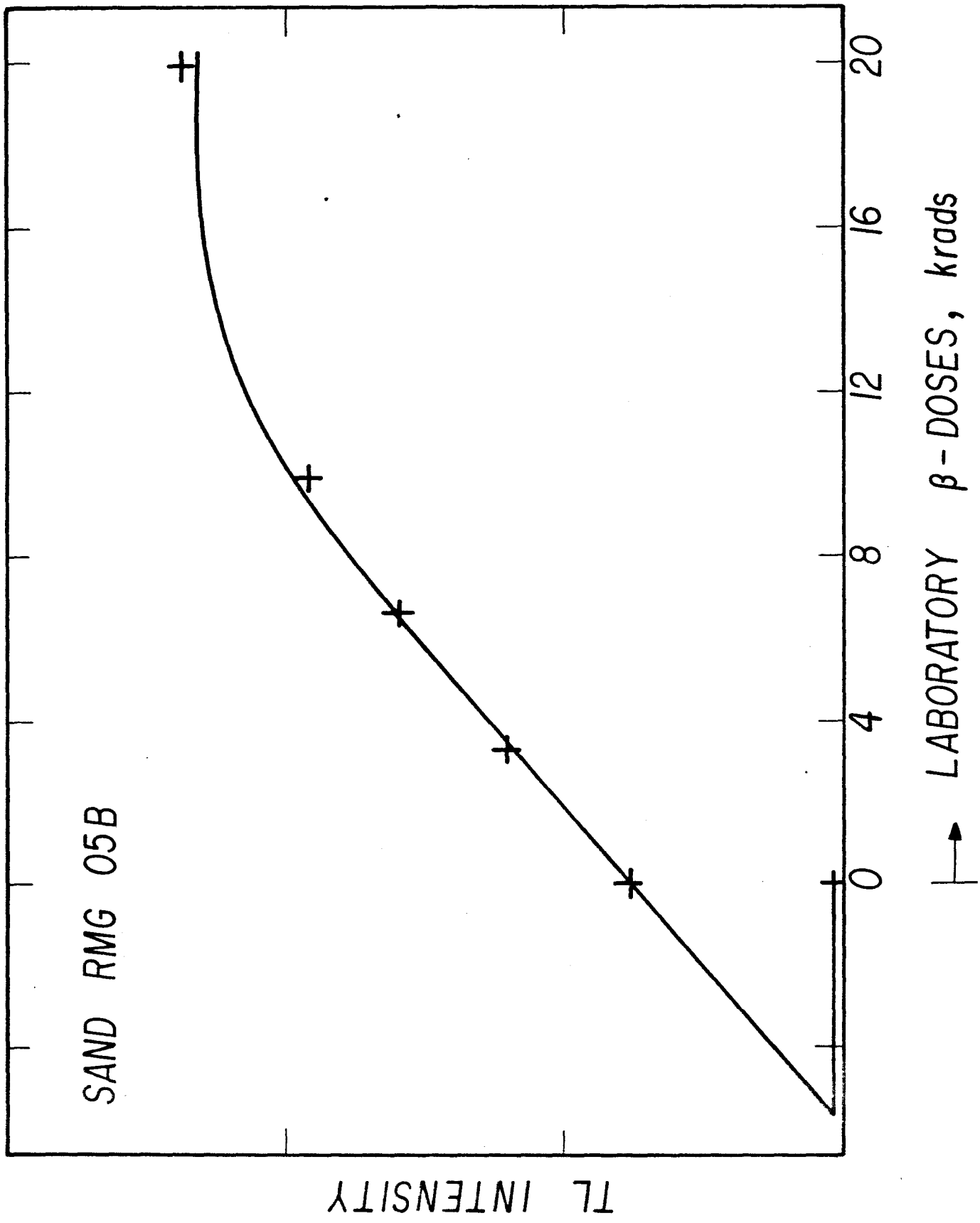
SAND RMG 05B
TL INTENSITY
0
4
8
12
16
20
LABORATORY β-DOSES, krads

Fig. 14

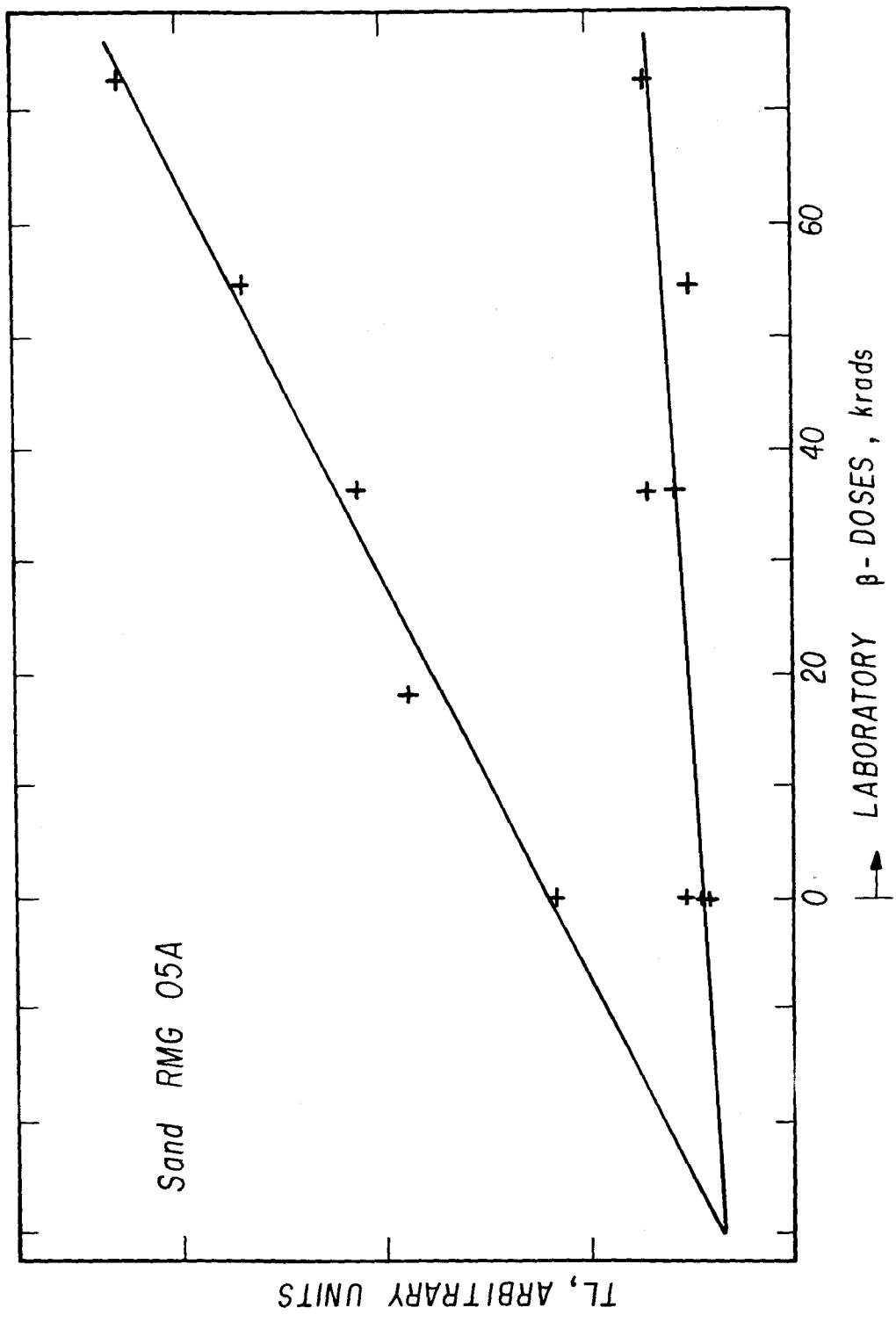
Sand RMG 05A
TL, ARBITRARY UNITS
0
20
40
60
LABORATORY β-DOSES, krads

Fig. 15

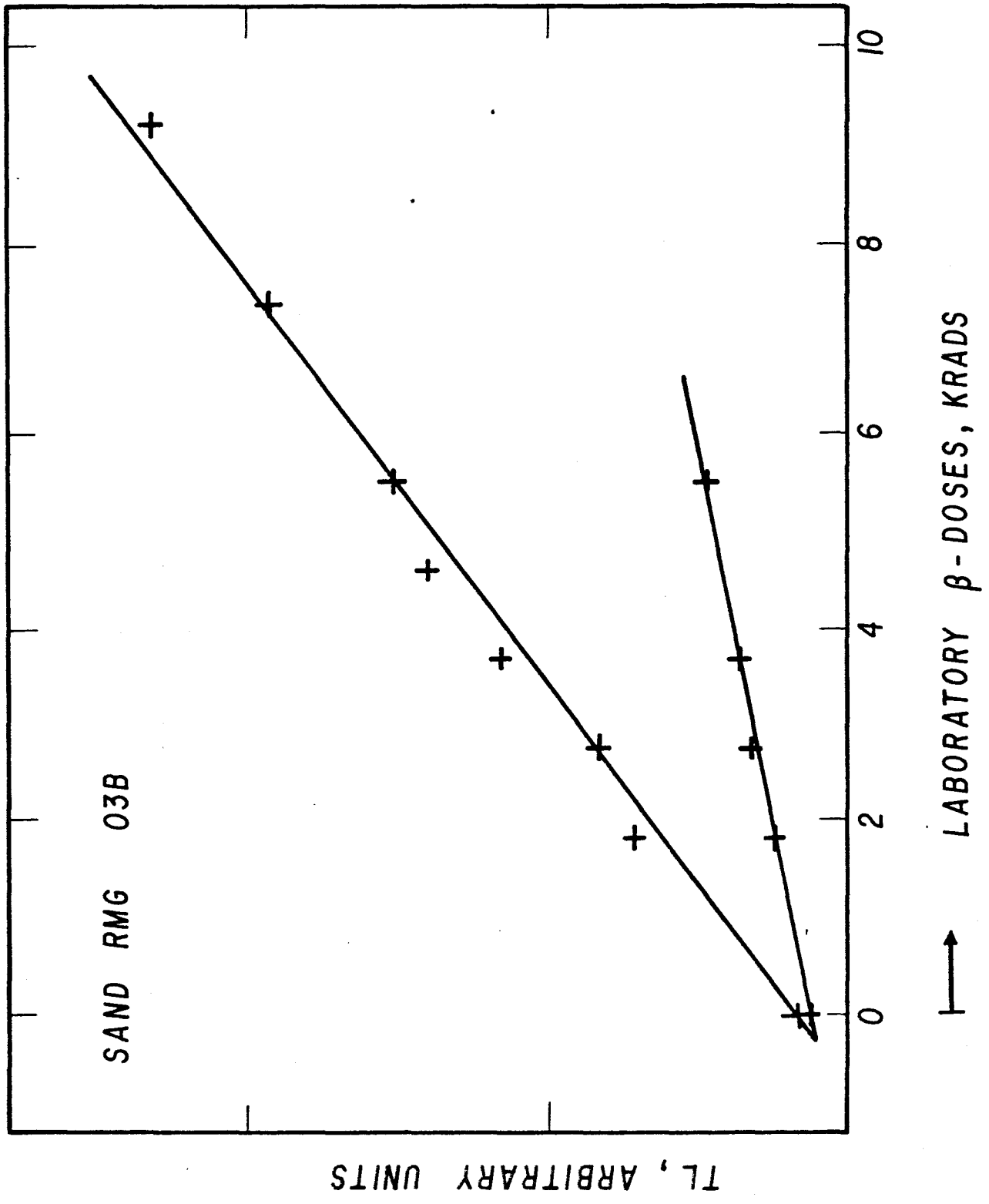
SAND RMG 03B
TL, ARBITRARY UNITS
LABORATORY β-DOSES, KRADS
0
2
4
6
8
10

Fig. 16

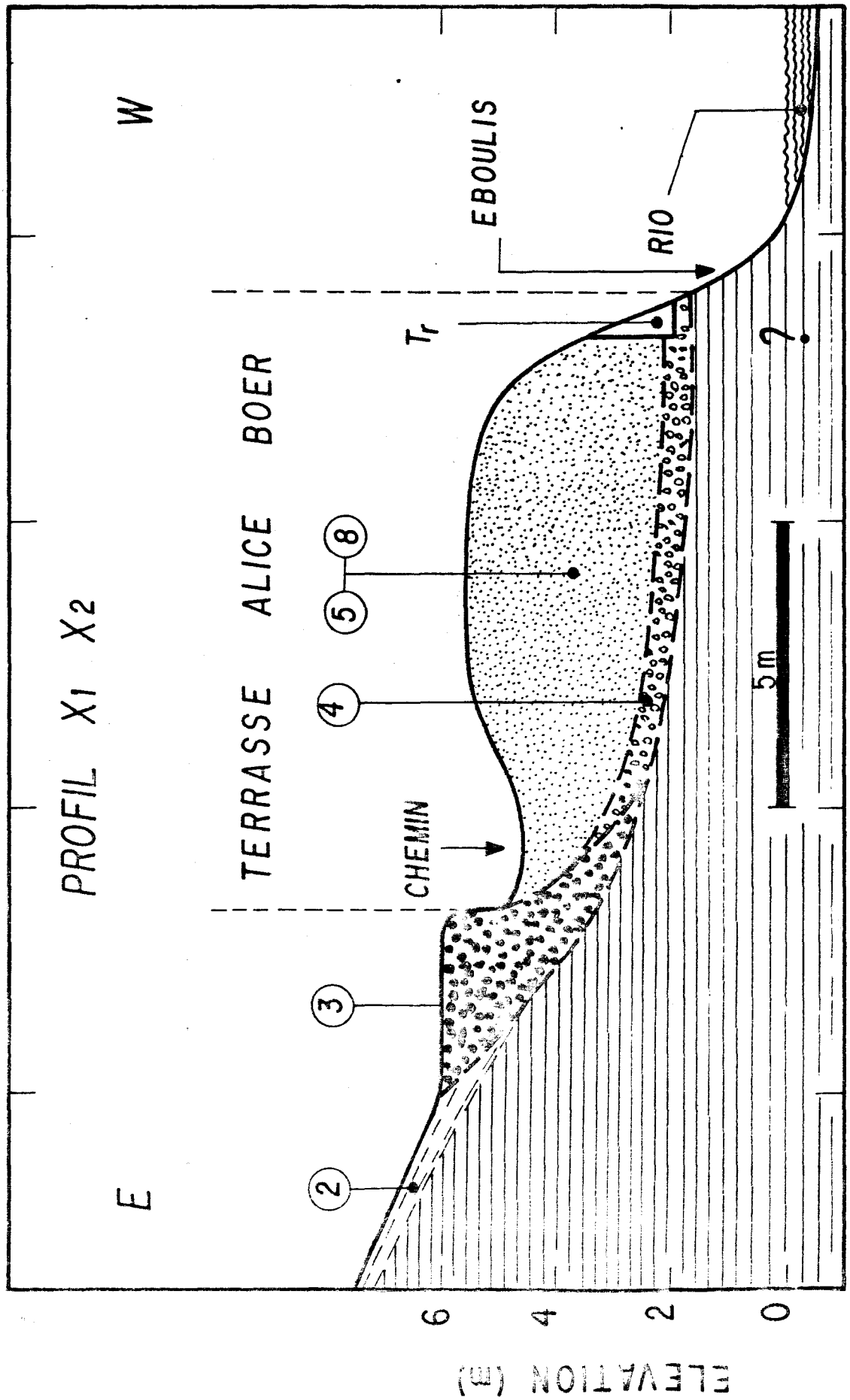
W
E
PROFIL X1 X2
TERRASSE ALICE BOER
EBOULIS
RIO
Tr
CHEMIN
2
3
4
5
8
5m
6
4
2
0
ELEVATION (m)

Fig. 17

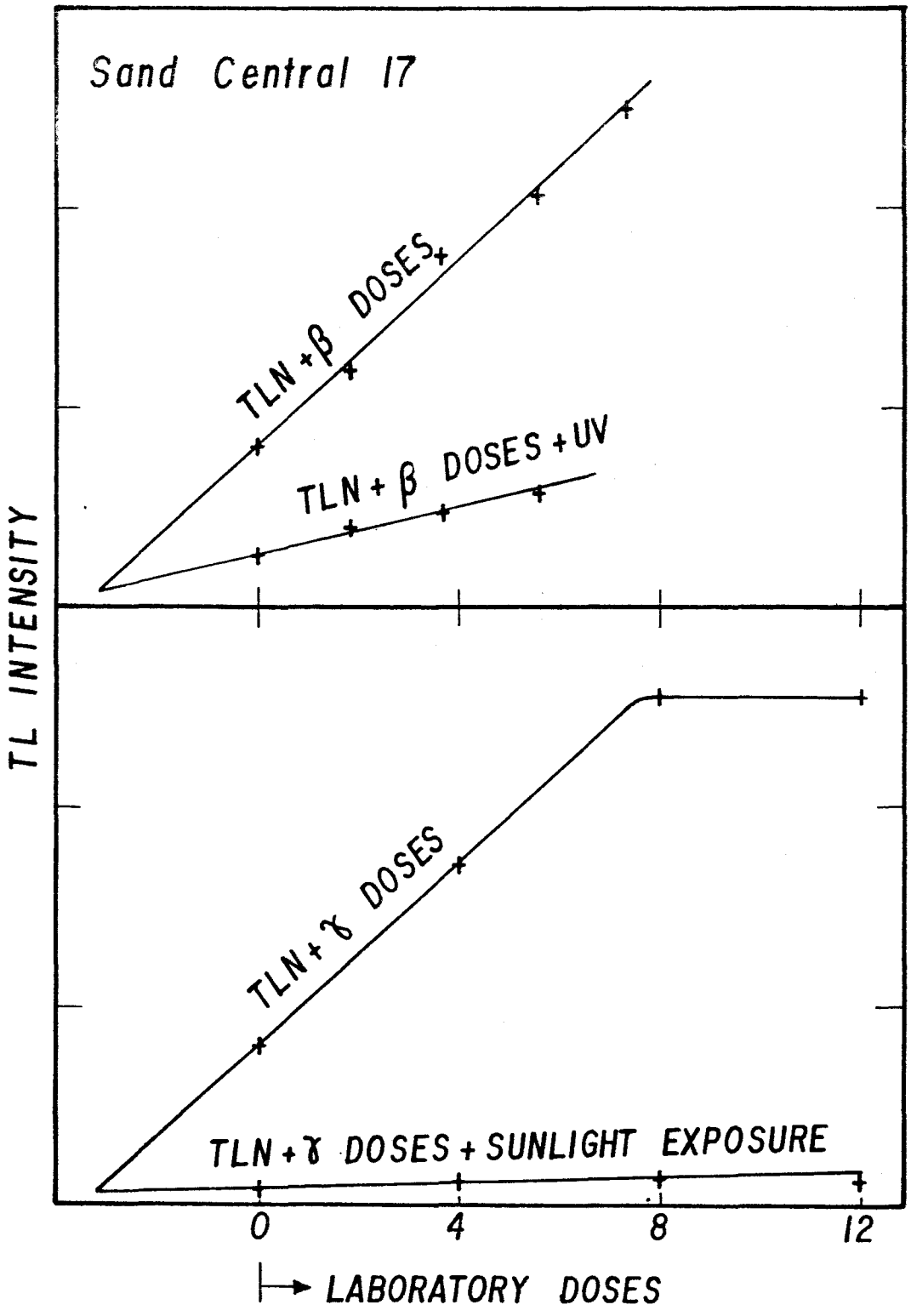
Sand Central 17
TLN + β DOSES
TLN + β DOSES + UV
TL INTENSITY
TLN + γ DOSES
TLN + γ DOSES + SUNLIGHT EXPOSURE
0
4
8
12
LABORATORY DOSES

Fig. 18

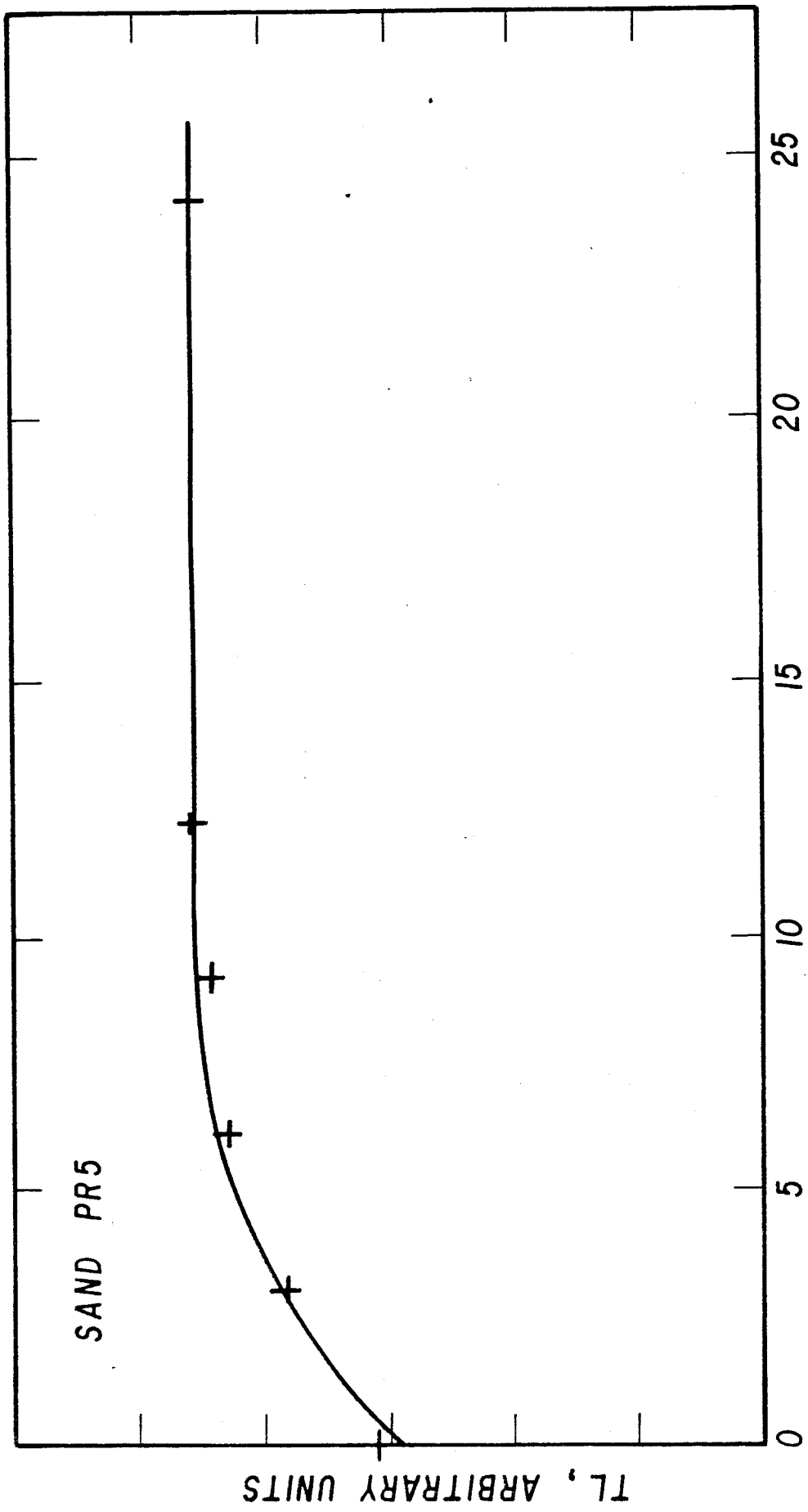

Fig. 19

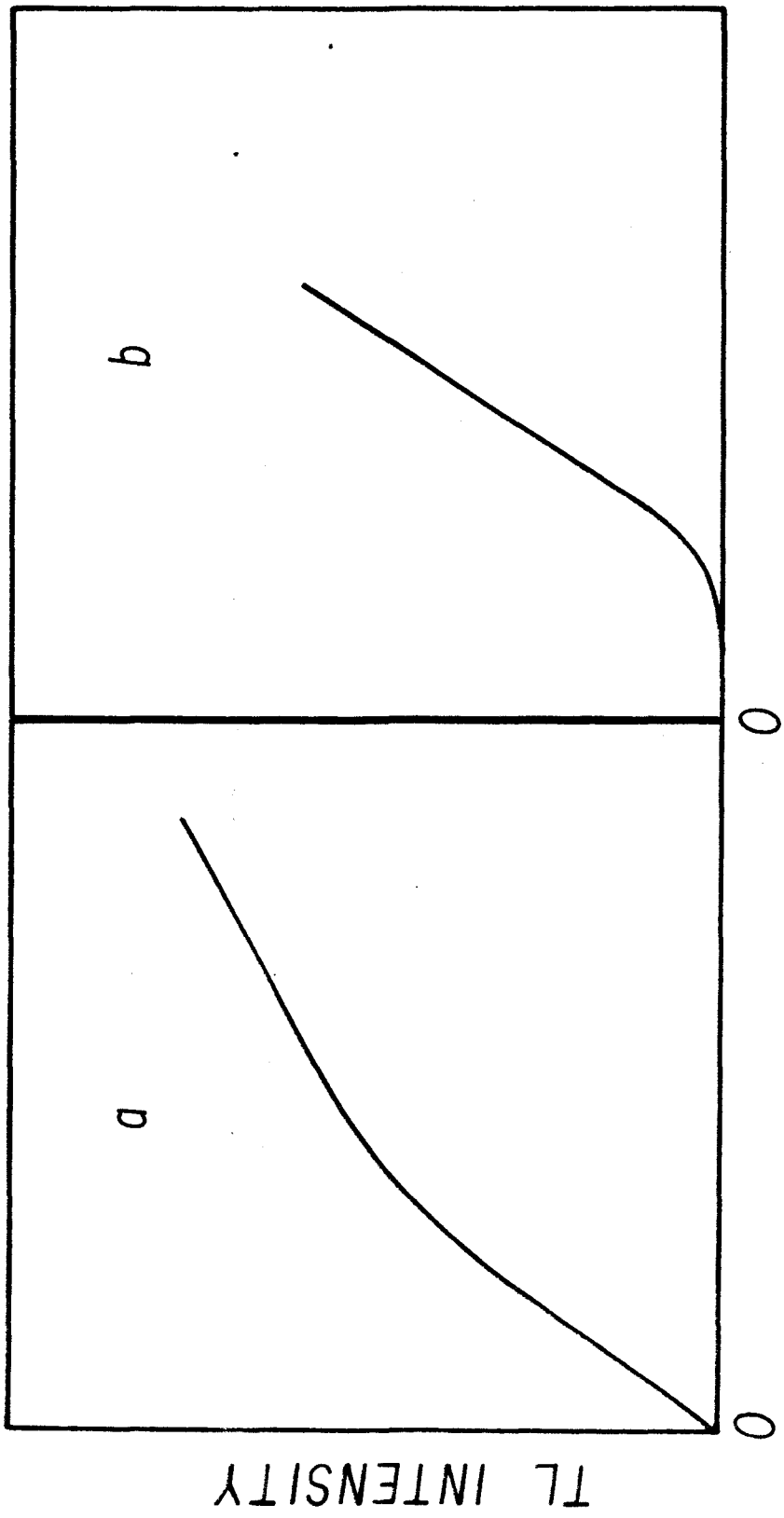

Fig. 20

Table 1

Thermoluminescence data on Upper Pleistocene sands from Rio Grande do Sul Coastal Plains

Sample	Paleodose[+] method	ED	Model Age[§] (yr BP)	Expected Age	Ref*
RMG 03 B	2	0.23	332	Sample taken at same level as archaeological ceramics within a fixed dune. Presence of this ceramics implie a deposition age <1300yr BP	(2)
RMG 04 A	1	38	54000	Sample 04B lies directly on 04A. The latter, from fossil evidences, is an intratidal zone, supposedly buried by 04B at time of sea level lowering. Both of them are thus expected to have similar (upper pleistocene, from fossil record), ages	(1)
RMG 04 B	2	36	51000		(2)
RMG 05 A	2	30	43000	Sample RMG 05B is a white sand overlying 05A. From stratigraphic position and dune alignment, 05B is estimated to belong to the lower holocene ; 05A is a reddened and indurated unit estimated, from its geomormologic and topographic position, as much older than 05B	(1)
RMG 05 B	1	5.1	8000		(2)
RMG 07	2	0.11	152	Sambaqui with archaeological ceramics thus age must be <1300yr BP	(2)

[+]Methods 1 and 2 for paleodose measurement refer respectively to the top and bottom methods of Fig. 11; ED = equivalent β-dose.

[§]Model ages were calculated using an annual dose rate of 0.7 rads, a value expected to be within ± 50% of the actual dose rate (Poupeau et al. 1983). Measurement of the annual doses rates to these samples is in progress.

*References: (1) Poupeau et al. (1983); (2) Poupeau, Souza, Rivera, Faria and Soliani, unpublished data

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