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TL age of Upper Pleistocene stratified deposits measured using the additive, regeneration and RΓ methods

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Stratified sands, which are components of the glacial and interglacial Vilkiškės and Tartokai profiles (Lithuania), were used as research material for experimental dating with the TL method. Sieve analyses of samples collected from stratified sands show varying grain-sizes in the samples, with domination by different fractions indifferent samples. Four aliquot of grains of the same diameter were selected from each sample for further research by means of the additive, regeneration and RΓ methods. The results showed some consistent patterns, which suggest a direction that future studies could take on these deposits samples. It is necessary, though, to examine grains which comprise a substantial percentage share in the total sample mass.

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Key words: Lithuania, Pleistocene, thermoluminescence, TL age, laminated lacustrine sand.

INTRODUCTION

According to Malinauskas (1991), the deposits exposed at Vilkiškės in southeastern Lithuania accumulated by saltation of sand in a stream during the Merkinė Interglacial period. Satkūnas (1993), though, suggested that the sands accumulated in a lacustrine basin at the beginning of the Butėnai Interglacial. Detailed structural, textural and compositional study of the Vilkiškės and Tartokai deposits was carried out (Barzdžiuvienė *et al*., 2000). Some investigators postulated a relation between older lacustrine sediments from the Butėnai Interglacial and younger ones from the Merkinė Interglacial (Gaigalas and Fedorowicz, 2002).

The Tartokai and Vilkiškės profiles are natural exposures in the sides of the Neris River valley. These exposures are close to each other (Fig. 1). Both sections are more than 30 metres high. They have a similar geological structure and age (Gaigalas *et al*., 2002, 2005).

In 2001–2003, twenty samples were collected for TL dating purposes from sand deposits found in both profiles: aeolian sand, lacustrine sand and fluvial sand. Figures 2 and 3 show dating results, which have been already published (Gaigalas *et al*., 2002, 2005) and presented at conferences in Vilinius (Gaigalas *et al*., 2002) and Ustroń (Gaigalas *et al*., 2004*a*). With the support of a research grant from the University of Gdańsk (BW 1230-5-0125-3) I undertook additional parallel dating of five Vilkiškės samples using the OSL (optically stimulated luminescence) method in the Gliwice laboratory (see Table 1).

At present the Vilkiškės profile has 41 dates obtained using luminescence methods: TL (in Gdańsk and in Lublin), OSL (in Tallin and in Gliwice) and the ^{14}C method (in Gliwice) — see Table 1. There are no significant differences between TL and OSL dates for the youngest aeolian deposits (depth: 2.15–4.70 m). TL and OSL dates are also relatively consistent in the bottom part of the Vilkiškės profile (below 22.4 m). Disparities between TL and OSL dates are found in the middle part of the profile at a depth interval from 13 to 21 m (Table 1). In this part of the Vilkiškės profile there is laminated lacustrine sand.

These disparities inspired the author to undertake methodological work in samples from horizontally laminated sands of both sections. In summer 2004, Gaigalas collected four samples of these deposits from the Vilkiškės (V) and Tartokai (T)

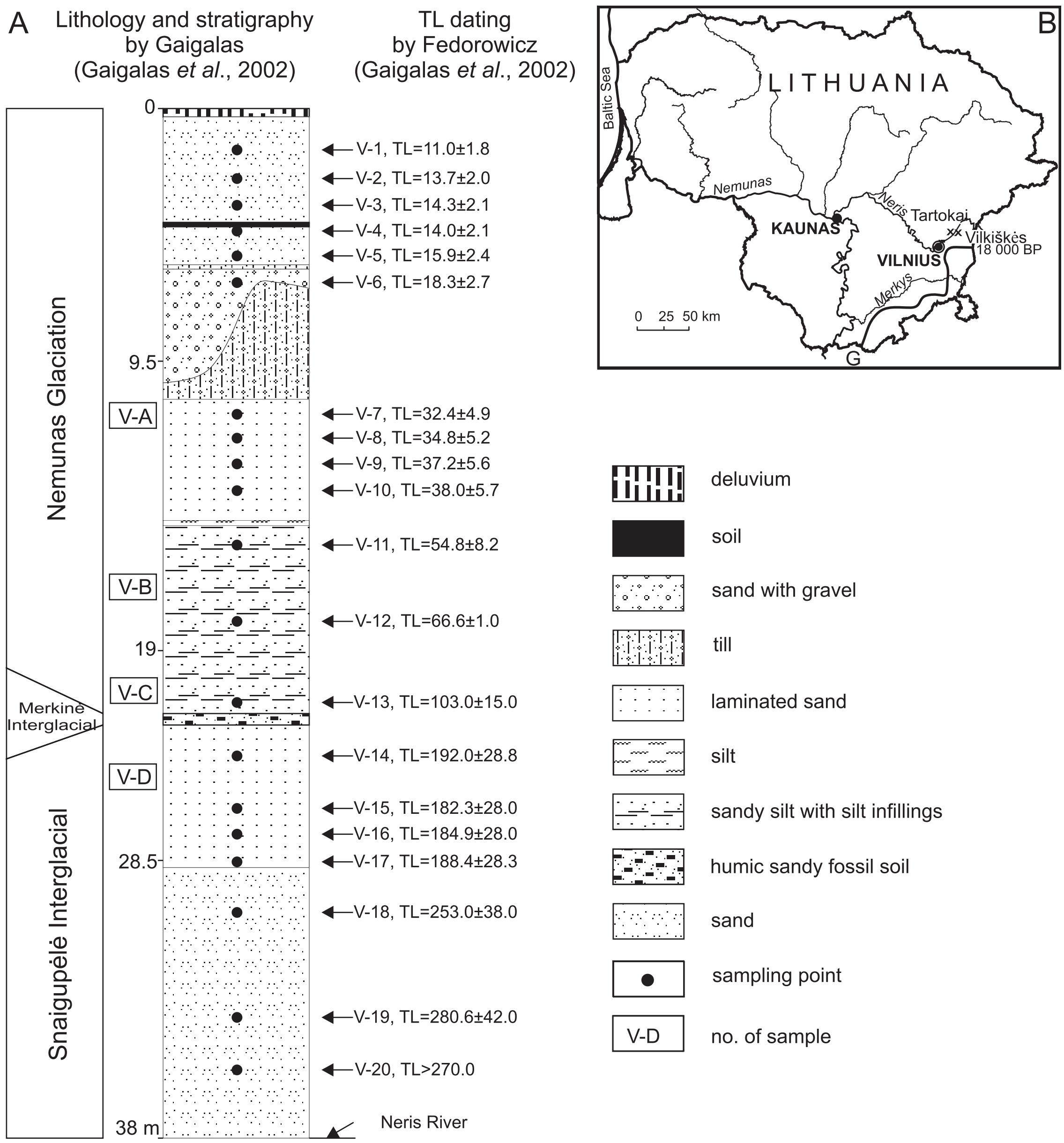

Fig. 1. A —Vilkiškės outcrop with results of TL dating (Gaigalas *et al.*, 2002), B — location of the Vilkiškės and Tartokai outcrops

G — maximum extent of Scandinavian glaciation — Grūda (Branderburgian, Lesznian) stadial of the Late Nemunas Glaciation; TL ages in ka BP

profiles (see Figs. 1 and 2) The grain size composition of near by samples marked A to D are shown in Table 2.

GEOLOGICAL SETTING

The predominant Quaternary deposits in Lithuania are devoid of datable organics. One of Lithuania's characteristic features is the occurrence of deposits from all glaciations known in Eastern Europe, with as many as five interglacials and nine independent till units being found. In the Pleistocene cover of Lithuania there are seven metachronous till formations left during independent glaciations or their major stages (Gaigalas, 1994). These tills are related to advances and retreats of the ice sheets of the Katleriai, Dzǎkija, Dainava, Žemaitija, Medininkai and Nemunas glaciations. The glacial sediments are separated by deposits of normal aquatic (fluvial and lacustrine) sedimentation which was formed during various interglacials: Vindžiǎnai, Turgeliai, Butėnai, Snaigupėlė and Merkinė (Eemian) and interstadials of the Nemunas (Vistulian) Glaciation. There occur lacustrine, boggy and alluvial sands, silts, gyttja, clay, sapropelite with rare mollusc shells (Molodkov *et al.*, 2002). The Vilkiškės and Tartokai sections are composed of Snaigupėlė and Merikinė Interglacial and Nemunas Glaciation deposits. The latter contain till beds (Gaigalas and Fedorowicz, 2002).

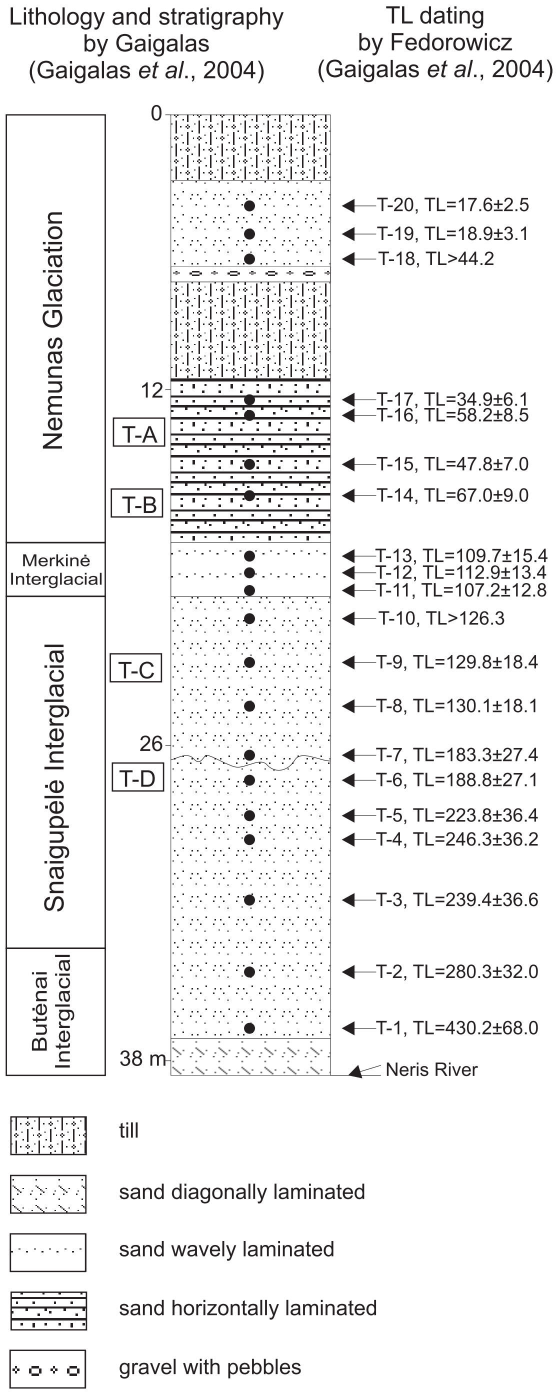

Fig. 2. Tartokai outcrop with results of TL dating (Gaigalas *et al.*, 2004)

For other explanations see Figure 1, TL ages in ka BP

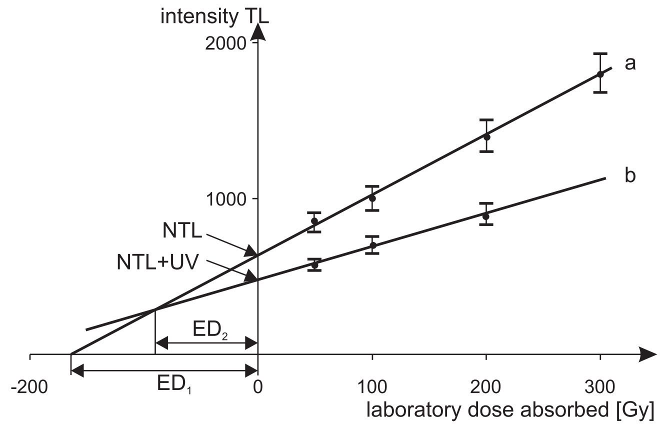

Fig. 3. Illustrations of the additive (a) and RΓ (a+b) methods of equivalent dose determination

NTL — natural thermoluminescence level, (NTL + UV) — residual thermoluminescence level; ED_1 — determination by additive method, ED_2 — determination RΓ method; for other explanations see Figure 2

STRATIGRAPHY

The stratigraphic framework of the Lithuanian Pleistocene has been elaborated by Gaigalas and Satkunas (Gaigalas, 1994). Gudelis' palaeogeographic studies (1961), Riškiene's (1979) palaeobotanical data, Kondratienė and Riškiene (1983) and Livrand (1991) contributed significantly to that framework.

Desposits of the last two glacial periods and interglacials are particularly well exposed in Lithuania. These are: the Snaigupėlė Interglacial (=Lublin), the Medininkai Glaciation (=Warta), the Merkinė Interglacial (=Eemian) and the Nemunas Glaciation (=Vistulian).

Snaigupėlė Interglacial deposits have been identified in Lithuania. The TL age (250 000±20 000 and 248 000±25 000 years BP) of the Snaigupėlė Interglacial was determined on the basis of samples collected from fine-grained pale grey lacustrine sand (at a depth of 3 and 9 metres) in the Gvildžiai exposure near Klaipėda in West Lithuania (Gaigalas *et al.*, 2001; Gaigalas and Fedrowicz, 2002). The analysis of Snaigupėlė Interglaciation sections shows that the lake sediments recognized in East and West Lithuania may most likely be attributable to the Drenthian–Warthian ice-free time interval of isotope stage 7. The flora of these deposits has been examined (Livrand, 1991). Palynological examination of the deposits was conducted by Kondratienė (1959, 1996), who concluded that these deposits were younger than the Butėnai Interglacial and older than the Merkinė Interglacial. The deposits were correlated with the 7th MOIS (Marine Oxygene Isotope Stage) level. Medininkai Glaciation sediments are represented by boulder clay in Lithuanian profiles. Lacustrine and boggy sediments of the Merkinė Interglacial are lie above them, i.e. gyttja, peat, clay soil, sand and sand with organic material. Sediments of this interglacial are widespread in Lithuania and reliably identified. Palynological diagrams have been developed for them (Riškiene, Kondratienė). Eemian deposits from Lithuania have been dated using TL, OSL and ESR methods (Gaigalas and Hütt, 1996; Gaigalas, 2000; Molodkov *et al.*, 2002). The dates obtained with these methods indicate the time span of the interglacial as 120–70 ka BP (Gaigalas, 1994).

Thanks to the numerous TL, OSL and ESR (electron spin resonance) dates and palynological studies, it has been possible to prepare a chronostratigraphy for the last interglacial and last glacial phases (Gaigalas, 1994). The Nemunas Glaciation

Table 1

TL, OSL and ^{14}C ages (ka) for samples from the Vilkiškės profile as obtained in different laboratories in 2000–2004

Sample no.	Depth [m]	TL Fedorowicz (2001) Gdańsk	TL Kusiak (2002) Lublin	OSL Hütt (2000) Tallinn	OSL Bluszcz (2002) Gliwice	OSL Molodkov (2004) Tallinn	^{14}C Pazdur (2002) Gliwice
V-1	2.15	11.0±1.6	–	–	–	–	–
V-2	3.20	13.7±2.0	–	–	–	–	–
V-3	4.40	14.3±2.1	–	–	12.7±0.5	–	–
V-4	4.50	14.0±2.1	23.0±4.0	–		–	–
V-4a	4.60	–	–	–	–	–	10.8±0.16 7.7±0.16 10.3±0.1
V-5	4.70	15.9±2.4	21.0±2.1	–	–	–	–
V-6	5.20	18.3±2.7	–	–	–	–	–
V-7	12.45	21.4±4.9	–	–	–	–	–
V-8	13.50	34.8±5.2	–	–	–	–	–
V-8a	14.00	–	–	–	–	136.7±8.8	–
V-8b	14.30	–	–	–	–	147.1±9.4	–
in0V-9	14.50	37.2±5.6	–	–	129.0±24.0	–	–
V-10	15.50	38.0±5.7	–	–	–	–	–
V-11	17.80	54.8±8.2	–	–	150.7±5.8	–	–
V-12	19.50	66.6±10.0	–	–	–	–	–
V-13	21.05	103.0±15.0	–	–	–	–	–
V-14	22.40	192.0±28.8	–	–	–	–	–
V-15	24.10	182.3±28.0	–	–	–	–	–
V-16	25.70	184.9±28.0	–	–	211.0±16.0	–	–
V-17	27.00	188.4±28.3	–	–	–	–	–
V-17a	27.50	–	–	250±17.4	–	–	–
V-18	28.60	253.0±38.0	623.0±145	>250.0	–	–	–
V-18a	29.50	–	–	–	–	230.2±16.2	–
V-19	33.00	280.6±42.0	–	>250.0	127.9±6.3	273.3±16.0	–
V-20	33.75	>270.0	443.0±71	>250.0	–	–	–
V-21	34.50	–	–	–	–	307.1±17.4	–

Gdańsk (Fedorowicz) — TL (multiple-portion regeneration method), Lublin (Kusiak) — TL (RΓ), Tallinn (Hütt, Molodkov) — OSL (RΓ), Gliwice (Bluszcz) — OSL SAR (single regeneration method), Gliwice (Pazdur) — ^{14}C

was divided into three parts: an early (70–55 ka BP), middle (55–30 ka BP) and late interval (30–10 ka BP). Initially, the Nemunas interval was nonglacial in Lithuania. The Scandinavian ice sheet moved into Lithuania as late as during late Nemunas time (Gaigalas, 1994), the latter being represented by two stages: Grăda and Baltija.

METHODS

The samples collected from the profiles were dried and then sifted (Table 2). Genetic interpretation is taken from an earlier study (Gaigalas *et al.*, 2002, 2004).

Vilkiškės outcrop:

V-D (depth 22.50 m): fine-grained sand with admixture of coarse-grained silt (lacustrine origin, lateral zone);

V-C (depth 19.60 m): coarse-grained silt, sandy (lacustrine origin, slope zone);

V-B (depth 17.70 m): fine-grained sand with admixture of coarse-grained silt (lacustrine origin, lateral zone);

V-A (depth 12.50 m): fine-grained sand, silty (lacustrine origin, lateral zone);

Tartokai outcrop:

T-D (depth 27.00 m): fine-grained sand, silty (lacustrine origin, lateral zone);

T-C (depth 22.80 m): mixture of fine-grained sand and coarse-grained silt (lacustrine origin, transition zone from lateral to slope or beach zone);

T-B (depth 17.50 m): coarse-grained silt, sandy (lacustrine origin, slope zone);

T-A (depth 13.50 m): coarse-grained silt, sandy, with admixture of coarse-grained sand (lacustrine origin, slope zone).

Table 2

Results of grain-size sieve analysis of samples from the Vilkiškės (V) and Tartokai (T) profiles

Fraction [µm]	V-A [%]	V-B [%]	V-C [%]	V-D [%]	T-A [%]	T-B [%]	T-C [%]	T-D [%]
>250	2.0	2.2	2.1	0.8	0.8	1.8	0.2	5.3
160–250	45.3	15.2	38.7	14.8	25.5	30.0	10.5	32.2
125–160	28.3	10.4	36.0	51.4	26.7	12.9	2.8	1.1
100–125	8.8	13.6	7.9	11.3	9.6	6.8	17.4	10.2
80–100	9.1	48.8	8.9	14.0	29.3	38.9	57.8	41.0
63–80	5.1	6.2	5.1	6.9	5.9	8.0	9.6	7.4
<63	1.4	3.6	1.3	0.8	2.2	1.6	1.7	2.8

Percentage (mass) share of quartz grain fraction in the total mass of the sample

The results of grain size analysis (Table 2) show that it is possible to differentiate one or two dominant fractions in all the samples. These fractions refer only to segments in which the grain diameter falls within the range of 80–250 µm. Grains of this size are most frequently used in the luminescence dating procedure. Polish laboratories analyse grains of the following diameters: 80–100 µm (Gdańsk), 125–200 µm (Gliwice), and 45–63 µm (Lublin).

Quartz grains were separated from the sediment. First, they were treated with 4% HCl, organic substances and carbohydrates being cleared within 24 hours. Next the grains were treated with 2% NaOH solution for a similar time. After multiple rinsing with distilled water, the grains were treated with 40% HF solution. This acid removed the external envelope of grains and dissolved grains of other minerals (Bluszcz, 2000).

Portions of grains were prepared from the treated material to measure equivalent doses with three methods: additive method, regeneration method and partial bleach method (RΓ).

The Vilkiškės section samples were dated in five laboratories in the period 2000–2004 (Table 1). The research was based on luminescence (TL and OSL) and ^{14}C methods. Different types of grains of various diameters were used for luminescence dating. The equivalent dose was measured by a variety of means. The TL method was employed in the Gdańsk laboratory, which analysed quartz grains with the multiple-portion regeneration method. The Lublin laboratory used the polymineral fraction which was examined with the partial bleach method. Applying the OSL method, the Gliwice laboratory used quartz grains and the single-portion regeneration method (SAR). The laboratories run by Hütt and Molodkov in Tallin used feldspar and the RΓ method.

The additive method is the oldest TL procedure. It was used by the Lublin laboratory several years ago. It is not used nowadays; however, it provides a basis for the construction of the "growth" line for the RΓ method. Figure 3 illustrates the determination of the equivalent dose with the additive method (line "a") and the partial bleach method — RΓ (lines "a" and "b").

The construction of "a" growth line consists in marking the natural thermoluminescence (NTL) level on the chart. This level is determined by the energy stored in grains separated from the sample under analysis. Points on the growth line are derived from the results of "artificial thermoluminescence" (NTL+γ). The γ radiation values are selected by the researcher. The Gdańsk laboratory exposes samples to cobalt bomb rays. These are measurements of energy obtained by the grains as a result of additional gamma radiation with known doses. The equivalent dose (ED_1) is selected as an intersection point of the "a" line with the dose line.

The "a" line is an element for the read-out of the equivalent dose in the RΓ method (Wintle and Huntley, 1980). In order to determine it, it is necessary to construct the next growth line "b" (Fig. 3). For this purpose all grain portions prepared for the measurement with the additive method should be exposed to a short-term simultaneous treatment with an ultraviolet lamp (UV). This will reduce the energy stored in the grains. The "b" line is constructed in the similar way to the "a" line. The resulting ED_2 value is an intersection point of the "a" and "b" growth lines.

In the regeneration method the equivalent dose is specified by measuring the thermoluminescence of grains exposed to a few hours' exposure to the ultraviolet lamp (UV). This process is called optical bleaching. It results in the maximum reduction of energy in the grains to the "residual thermoluminescence level" (NTL+UV). During growth line construction, the results of residual thermoluminescence and natural thermoluminescence (NTL) are first placed on the chart. Next, thermoluminescence measurements of grains which were earlier bleached and radiated with γ radiation doses of known values (NTL+UV+γ) are represented in the chart. In this way it is possible to reconstruct the signal which was originally contained in the grains analysed (NTL). The growth line (linear or exponential function) allows one to reconstruct the value of energy stored in the grains. The dose absorbed by the grains under laboratory conditions is assumed to be the ED (Fig. 4).

In order to specify the dose absorbed by the grains in the Vilkiškės and Tartokai samples they were radiated with cobalt-bomb rays of 8 values: 50, 100, 150, 200, 250, 300, 350

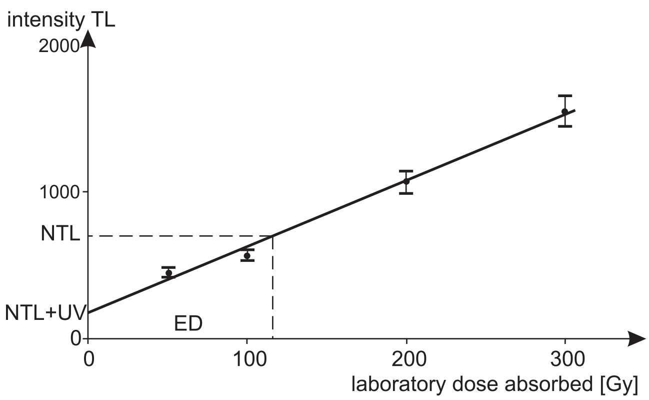

Fig. 4. Illustration of the regeneration method of equivalent dose determination

For other explanations see Figure 2

and 400 Gy. The same values were used to construct the growth line for all the measurement methods used in the research. The upper value of 400 Gy was selected on the assumption that it would be approximately twice as high as the one stored by the oldest grains. Results of equivalent doses (ED) were divided by dose rates (Dr). The result of this quotient is a TL date. The values of the dose rate Dr (Gy/ka) and TL dates (ka BP) are shown in Table 3.

The annual dose was measured with a *MAZAR* spectrometer (Fedorowicz, 2003). The ^{40}K, ^{226}Ra, ^{228}Th concentrations were converted into α, β, γ radiation doses with the use of coefficients specified by Aitken (Aitken, 1983). Adjustments were also made to account for deposit humidity (Aitken and Xie, 1985).

Table 3

TL dates of samples from the Vilkiskės (V) and Tartokai (T) profiles obtained with the following methods: A (additive method), R (regeneration method) and RΓ (partial bleach method) for various diameters of quartz grains

Sample index	Depth [m]	Dose rate Dr [Gy/ka]	Method	TL date for grain fraction [µm]			
				160–250	125–160	100–125	80–100
V-A	12.50	1.13	A	160.3±18.2	61.3±8.2	56.8±6.6	58.8±7.3
			R	140.2±16.5	46.4±6.9	43.4±5.4	50.3±6.9
			RΓ	130.4±15.5	60.4±7.1	50.2±4.8	53.1±6.8
V-B	17.70	2.41	A	>145.2	>145.2	>103.7	>103.7
			R	>124.5	>124.5	>103.7	>83.0
			RΓ	NR	NR	NR	NR
V-C	19.60	1.86	A	190.6±22.0	160.2±18.4	171.1±24.9	89.4±9.9
			R	146.9±19.5	150.8±17.2	146.2±24.2	70.4±8.6
			RΓ	159.9±18.4	150.2±16.9	137.7±21.6	81.4±9.2
V-D	22.50	1.21	A	206.3±23.2	208.3±22.	250.3.4±26.6	260.1±28.3
			R	188.6±22.6	180.4±19.6	230.4±24.9	183.3±19.9
			RΓ	178.4±19.5	186.4±21.1	248.2±27.4	233.1±25.8
T-A	13.50	1.34	A	96.4±11.3	176.3±28.4	86.4±9.0	77.9±8.7
			R	83.4±10.5	148.8±24.9	80.5±9.1	73.1±8.8
			RΓ	82.6±11.0	152.2±27.7	93.3±8.8	62.7±5.7
T-B	17.50	1.21	A	201.4±22.4	>206.6	196.6±21.3	162.2±18.5
			R	149.6±18.4	>247.9	140.4±15.1	131.4±14.6
			RΓ	154.4±17.9	NR	163.6±18.2	145.2±16.0
T-C	22.80	1.03	A	280.4±31.4	>194.2	>242.7	>242.7
			R	>242.7	>194.2	>194.2	270.4±34.6
			RΓ	250.3±17.9	NR	NR	NR
T-D	27.00	0.96	A	290.4±17.7	268.4±29.1	260.4±28.2	286.6±30.1
			R	206.0±25.5	211.3±23.2	199.2±20.9	201.4±22.4
			RΓ	228.8±26.3	214.6±24.7	223.3±24.8	236.6±25.7

NR — (no results) means that it was not possible to specify the age with the R method because the growth line drawn for the additive method showed saturation

RESULTS

Table 3 gives 12 results of TL dates for each sample analysed. The ED was specified for each of the four grain size fractions with the additive (A), regeneration (R) and RΓ methods.

1. The age of sample portions shows a positive correlation with the grain diameter (in 11 out of 18 cases).

2. Most frequently, the date specified for a given grain diameter shows the largest values using the additive method and the lowest values for the regenerative method.

3. The majority of results for a given grain diameter obtained with the three methods do not differ significantly. This shows that the grains did not change their sensitivity.

4. It was not possible to date the samples with the RΓ method in 8 cases because the calibration curve drawn for the additive method showed saturation. In particular, the saturation condition was shown by the V-C sample. The dose rate was the highest for this sample compared to the others and it amounted to 2.41 Gy/ka.

5. The results obtained for the grains whose percentage was minimal in the entire sample are marked by the high measurement uncertainty, e.g. grains of the 125–160 µm fraction in the T-D and T-C samples. In respect of the T-D sample, the age departs sharply from the remaining results and it is significantly higher (*ca*. 17%). In respect of the T-C sample, it was not specified precisely.

One of the samples (V-C) showed a saturation condition for all the methods and for each fraction.

DISCUSSION AND CONCLUSIONS

The measurements presented in this article were conducted on samples collected from stratified deposits, the age of which should not exceed 250 000 years. This limitation was to eliminate reservations concerning the upper limit of the equivalent dose, i.e. 1200 Gy, for the applicability of the regeneration method (Frechen *et al*., 1997).

The eight samples, the results of which are presented in Table 3, produced nearly one hundred results broken down according to the measurement methods and quartz grain fractions. Minor differences in the age of a single fraction analysed with the three methods show that there is no change in the sensitivity of samples whose age does not exceed 250 ka BP.

The equivalent dose did not exceed 400 Gy for all the samples. Hence, it was three times lower than the one indicated by Frechen (1997) as the limit of regeneration method applicability. The Table 3 results do not show a tendency of consequent age underestimation if the regeneration method is applied to samples older than 80 ka BP (Berger *et al*., 1992). It is difficult to discuss the results of V-B and T-C samples due to indefinite age values. The remaining results show that the selection of grain fraction for luminescence dating should not be accidental.

TL age results are usually subject to measurement uncertainty which does not exceed 15%. Sometimes differences between the results of one sample divided into four fractions (Table 3) substantially exceed these values. According to the author, prior to the commencement of luminescence dating, a sieve analysis should be the first activity conducted during the examination of sediments of water origin. The second activity should be to eliminate such fractions from further research that have a minor percentage representation in the entire sample. Further examination should focus on the predominant sample in the fraction. The results in Table 3 show that if the sample has two dominant fractions in terms of their percentage share, their results are very similar.

The growth of age along with the increase of a sample grain diameter (see Table 3) may be explained as follows. Larger and heavier quartz grains go down faster than smaller and lighter grains. For this reason, grains of a larger diameter might have had a shorter exposition to solar radiation which was reducing the energy stored in them earlier. Thus, the age of larger-size grains may be longer than that of smaller grains.

It is loess that is the most frequently used material in luminescence methods. The above cited reservations concerning the applicability of regeneration method also apply to loess. The world literature most frequently quotes loess dating results. The largest number of methodical works and studies concern loess. Other types of deposits enjoy much lower interest. Stratified (lacustrine and fluvial) deposits are not always highly regarded as a luminescence dating material by researchers who conduct dating. Dating is most frequently carried out by physicists.

This methodical work was inspired by long discussions of the author (physicist) with the geologist (Gaigalas). The physicist conducted dating and prepared the results. The geologist will determine their value. The grain-size analysis and geochemical analysis were already conducted for the Vilkiškės and Tartokai sections. As a result, six lithocomplexes were distinguished, which allowed for the correlation in both outcrops (Bardžiuvienė *et al*., 2000). Maybe future interdisciplinary discussions will generate new ideas. According to the author, the development of luminescence methods stem from the collaboration between naturalists and physicists.

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