

## TESTING THE POTENTIAL OF OPTICALLY STIMULATED LUMINESCENCE DATING METHODS FOR DATING SOIL COVERS FROM THE FOREST STEPPE ZONE IN TRANSYLVANIAN BASIN

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**Abstract:** In the area of the expositional forest-steppe in the Transylvanian Basin there are many larger or smaller islands of fossil Clinostagnic Phaeozems and Chernozems covered by Luvisols. This work aims at testing the potential of state of the art optically stimulated luminescence dating methods for obtaining an absolute chronology for these soil covers. As novel techniques are used, they are being described in detail. The ages obtained (~ 10 ka for the Phaeozems, respectively ~ 4 ka for the Luvisols) can be regarded as evidence that the chernozem soil cover was a stable feature before the ascent of human activities and that these soils suffered a degradation in the a late Holocene that reflects a climatic transition. Thus, this is at least partial evidence that these paleo-pedological structures from Transylvania consisting of a fossil mollic horizon of a Phaeozem covered by an albic horizon of an actual Luvisol can be considered a marker resuming the climatic change associated with the Subboreal-Subatlantic transition which began around 4ka BP.

**Key words:** optically stimulated luminescence dating, absolute chronology, chernozem soil cover, forest-steppe, Transylvanian Basin, climatic transition.

### 1. INTRODUCTION

The drier colder climatic period of the Holocene known as the Subboreal favored the expansion of the Boreal forests and steppes / forest-steppes in Western Eurasia. In the late Holocene a climatic transition from this climate to milder wetter conditions (Subatlantic) has taken place (Speranza et al. 2000). Wetter conditions favored the expansion of the nemoral and subtropical forests in the same geographical area. Subsequently the steppes and forest-steppe contracted under the pressure of the nemoral forest in the western part of the Holarctis and the former vast grasslands were progressively replaced (Kremenetski, 1995).

In the area of the expositional forest-steppe in the Transylvanian Basin there are many larger or smaller islands of fossil Clinostagnic Phaeozems and Chernozems covered by Luvisols. Though intuitively posed in connection with the climate change associated with the Subboreal-Subatlantic

climatic transition, no such a pedological structure was dated until now in Transylvania.

Having an accurate and absolute chronology of these soils is of major importance in order to confirm whether these formations are indeed paleo-pedological relicts that can be interpreted as a reflection of a late Holocene climatic transition.

Luminescence methods are now widely used in the dating of geological sediments such as aeolian, marine and fluvial sand or loess (Stokes 1999, Duller 2004, Roberts 2008, Wintle 2008 a, b). After a first successful application of OSL dating for loess deposits in Romania (Timar et al 2010), this work tests the potential of applying state of the art optically stimulated luminescence dating techniques on quartz grains extracted from Chernozems and Luvisols from Transylvanian Basin.

### 2. STUDY AREA

There are three fundamental types of forest-steppe areas derived from the extant literature which

deals directly with this type of landscape (Sakalo 1961, Karamysheva and Khramtsov 1995).

The typical forest-steppe is a mosaic of forests and mesic/mesoxeric grasslands (called “meadow-steppes” - very different from the ones in the steppe zone) which has a pattern dictated by the intimate relationships between the two fundamental types of ecosystems. This type is now encountered only in central-western Transylvania in the southern part of the region called ‘the Transylvanian Plain’ and in the Secaselor Tableland. Here the meadow-steppe grasslands and their associated Haplic and Luvis Chernozems and Phaeozems (IUSS WRB 2006) are placed on the floodplains, plateaus and shaded slopes and the nemoral forests of oak and the associated Luvisols exist only as tiny islets. The precipitations are below the level of 550 mm / year.

The second type is the expositional forest-steppe that exists in a large part of the periphery of Transylvania. In this case, the level of precipitations increases from 550 mm up to 700 mm / year. The plateaus and the shaded slopes are entirely covered by nemoral oak forests and the meadow-steppe grasslands exist as tiny islets, usually on humid areas on the slopes which could not be inhabited by forests.

As soon as the precipitation level passes over 700 mm /year as it happens at the borders of the Transylvanian Basin the nemoral forests of oak, hornbeam and beech and Luvisols inhabit the whole original landscape, including the sunny steep slopes. In these conditions only the geognosic forest-steppe can develop, conditioned by the presence of massive limestone rocks and gypsum formations as is the case in the north-western part of the region (Paşcovschi and Doniţă 1967).

In the area of the expositional forest-steppe in the Transylvanian Basin there are many larger or smaller islands of Clinostagnic Phaeozems and Chernozems (named formerly “Black Clinohydromorph Soils” by the Romanian Pedologists) covered by Luvisols. These are gradually diminishing with increasing of the precipitations until they completely disappear as actual soils (Cernescu et al. 1970). They have many aspects in common with the Gley Chernozems from the nearby floodplains of the rivulets and rivers from the same area with which are actually in contiguity and which were described from the whole Central Europe as ‘Black Floodplain Soils’ or BFS by Rittweger (2000).

The typical forest-steppe landscape was certainly much widespread in Transylvania during the relatively dry and cold period of the Subboreal along with the meadow-steppe grasslands and their

associated Chernozems and Phaeozems. The shaded slopes like the ones in figure 1 – top, were almost totally covered by Chernozems and Phaeozems in almost all the Transylvanian Basin. With the increasing of the precipitation level and the onset of the Subatlantic period the nemoral forests and the Luvisols expanded on the shaded slopes and plateaus replacing the former bio-pedological structures at the periphery of Transylvania. The last areas to be inhabited by forests were on the slopes the much humid landslide ‘valleys’. Their local conditions impeded the trees to populate them - due to their high level of humidity. Consequently the dark Phaeozems and their associated meadow-steppe grasslands survived there a longer time in the conditions of the expositional forest-steppe (Fig. 1-centre). However as the level of precipitations increases further above 700 mm / year the pressure of the nemoral forests around increased a lot and the trees invaded also the landslide slope ‘valleys’. Being accumulative micro-environments, the succession of soils adjacent to this major Holocene climatic, landscape and vegetational change was preserved: the Luvisols correspondent to the actual Subatlantic conditions can be found at the top of the former Phaeozems in the areas of the landslide slope valleys (Fig.1, below).

In the drier nowadays parts of central Transylvania, where the multi-annual precipitation level is under 550mm/year the Chernozems and Phaeozems are yet the actual soils and the remained natural components of the landscape are characteristic for the forest-steppe. The C-14 dating of the dark forest-steppe soils from such areas revealed ages in between 14.000-20.000 ka BP proving that in such drier regions the forest-steppe type of landscape and its associated soils (not necessarily also the strict vegetational types) are stable structures since the upper Pleistocene (Pendea et al. 2002).

The location of the site chosen for the present study is in Sărăţii Valley to the western border of the Transylvanian Basin, near the limit with Apuseni Mountains of the Inner Carpathians, in a densely forested area from the western part of the Feleac Hills - 15, 7 kilometers west-southwest of Cluj-Napoca city. Geographical coordinates are 46°43'41.64" - 46°43'18.79" N and 23°23'52.30" - 23°24'31.19" E. The present climate of the area is a temperate sub-continental rather humid sub-mountain one with average multi-annual rainfall of 790 mm and an average temperature of 7.5 °C which encourages the development of a climatic forestry nemoral forest vegetation dominated by hornbeam (*Carpinus betulus*) and Durmast Oak (*Quercus petraea*).

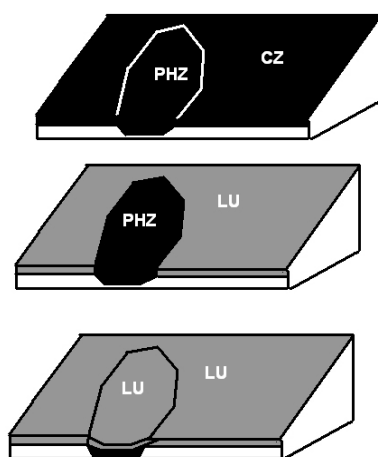


Figure 1. Scheme of soil cover on a long shaded (north exposed) slightly inclined slope from the typical forest-steppe (up), expositional forest-steppe (middle) and full nemoral zone (below) in the Transylvanian Basin. In the typical forest-steppe the slope surface is occupied by Chernozems (CZ) and the large derasional or landslide much humid 'slope valleys' by Phaeozems (PHZ). In the peripheral nemoral zone (expositional forest-steppe) the forest invaded the well drained parts of the slope where Luvisols appear (LU) while the mesic/mesohygrophile meadow-steppes on Clinostagnic Phaeozems survived as isolated patches on the much humid derasional or landslide slope valleys. When the annual precipitation level increases over 700 mm the nemoral forest occupies even the steep sunny slopes and also these humid slope valleys from the shaded slopes where the Luvisols replace the Phaeozems. However the very deep and very dark humus rich mollic horizon survives here below the albic horizons of the actual Luvisols due to the accumulative nature of the micro-environment in such 'slope valleys'. Such a situation can be seen on the left slope of Sărății Valley between Luna de Sus and Stolna in the western part of the Feleac Hills where the site analyzed here is located.

The site was discovered in 1996 by Al.S. Bădăraș and F. Pendea and it was appreciated as one of the best locations in Transylvania for studying the Holocene paleosols. Unfortunately, no profound studies were conducted here at the time and no protective measures were implemented for this site with exceptionally well preserved paleo-pedological structures.

During the year of 2008, while the site was not supervised in any way, the work to the nearby passing „Transylvania Motorway” almost totally damaged it: tons of red clays excavated from the vicinal hills were deposited on the left slope of the Sărății Valley exactly in the areas where there are two deep ravines deeply cutting two landslide slope valleys where the paleosol structures investigated in the present studies were located. Consequently we were forced to take the samples in September 2008

from various sheltered paces in the Sărății Valley where we found them yet preserved (Figure 2).

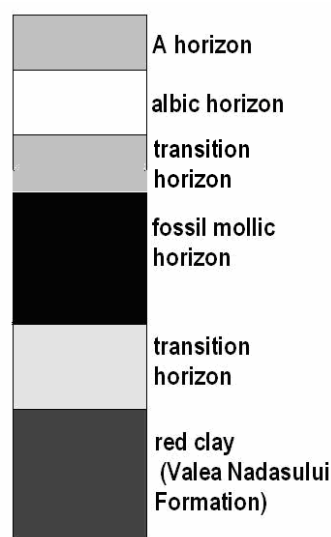


Figure 2. The paleo-pedological structure from Sărății area (placed in a landslide slope small valley exposed to north) from up to bottom: the A horizon with a weak humus accumulation of the nowadays Luvisol (7cm); the albic horizon of the actual Luvisol (41cm) color 7,5YR 7/5, no structure, sandy-loam texture; transition horizon (28cm); the fossil mollic horizon of the former Phaeozem (59cm) very dark color 5YR 2/1, massive structure, clayey texture, no calcium carbonates present; transition horizon (35cm); the red clays of the Valea Nadasului Formation of a mid-Eocene age. The most important level for tracing the Subboreal-Subatlantic climate change is the transition between the albic horizon and the fossil mollic horizon below.

### 3. OPTICALLY STIMULATED LUMINESCENCE DATING

#### 3.1. Principles

Luminescence dating is based on the property of certain minerals to store the energy of nuclear radiation. A low level of nuclear radiation is omnipresent in nature, and the longer the minerals are exposed, the more energy they store. By stimulation with heat or light, the minerals can release part of this energy by emitting a small amount of light, which is called luminescence. The intensity of this luminescence signal is a function of the total accumulated radiation dose, and hence also to the total acquisition time. There are two variants of luminescence dating, namely thermoluminescence (TL) and optically stimulated luminescence (OSL) dating, depending on whether the luminescence is stimulated by heat or light, respectively. OSL dating is also simply referred to as optical dating. In both cases the latent dating information is carried in the

form of trapped electrons. These electrons are produced by the interaction of the nuclear radiations with the atoms of the mineral and then may get trapped in certain defects of the crystalline structure. The number of trapped electrons is a measure for the total dose – the paleodose (the total amount of energy absorbed from the ionizing radiation) the mineral has received over a certain period of time. If also the rate by which the mineral has been absorbing the dose – the annual dose, is determined, this period (age) can be obtained.

The paleodose is a combined dose, resulting from exposure to  $\alpha$ ,  $\beta$  and  $\gamma$  radiation, it cannot be measured directly. Therefore, it is determined as an equivalent dose ( $D_e$ ), i.e. the amount of artificial dose delivered by irradiation in the laboratory that induces a luminescence signal identical to the natural one in the sample. The annual dose is derived by calculation based on the estimation of environmental nuclide concentrations.

The moment that is dated with luminescence techniques is a zeroing event during which all the effects on the mineral of its exposure to radiation are removed. For sediments, the zeroing event, called bleaching, is the exposure of the sediment grains to sunlight during transportation, prior to deposition. Once the zeroing agent is removed, for instance when sedimentary minerals are deposited and shielded from sunlight by other grains falling on top of them, the luminescence signal starts to build up again.

One of the underlying principles of optically stimulated luminescence (OSL) dating is that sediments collected for dating have remained undisturbed since their burial. Bioturbation is a common phenomenon in soils, thus hampering the use of luminescence methods for direct dating of these formations.  $D_e$  replicates from undisturbed and fully bleached sediments are unskewed, showing low overdispersion. Bioturbated sediments, however, may show highly skewed multi-model  $D_e$  distributions with higher overdispersion values (Bateman et al 2007). True burial ages may be derived only through the application of statistical analysis and usually using single grain OSL measurements which are not straightforward (Roberts 2000), thus OSL ages obtained on soil horizons are not very often encountered in literature.

### 3.2. Sample collection and preparation

For luminescence dating, the soil samples were taken from an outcrop located in the mentioned site by using 30 cm steel tubes of 7 cm diameter which were hammered into the mid parts of the albic

and mollic horizons described above. Tubes were sealed with black plastic bags and only the inner part (that has not been exposed to light) of the material had been used for luminescence analysis, the outer ends being used for gamma spectrometric investigations.

For annual dose (gamma spectrometric) investigations) samples have been pulverized, packed in a cylindrical geometry and stored for one month in order for radioactive equilibrium to be reached in uranium series.

For equivalent dose (luminescence) estimation, 63-90  $\mu\text{m}$  quartz grains were extracted from two samples using conventional procedures (Zimmerman, 1971; Lang et al., 1996; Frechen et al., 1996) under subdued red-light conditions (HCl (10%) and  $\text{H}_2\text{O}_2$  (30%) treatment, wet sieving, heavy liquid (sodium metatungstate  $\text{Na}_6[\text{H}_2\text{W}_{12}\text{O}_{40}] \cdot x\text{H}_2\text{O}$  density (2.62-2.75  $\text{g/cm}^3$ ) separation (Mejdahl, 1985) and HF (40%) attack.

### 3.3. Equivalent dose measurements

Luminescence analysis were performed on a sample of Albic Luvisol –Laboratory code: 3A (LU), and a sample of Clinostagnic Phaeozem – Laboratory code: 4B (PHZ).

All measurements have been performed on an automated TL OSL Risø reader model DA-20 (for details reference is made to Thomsen et al., 2006) using blue light stimulation  $470 \pm 20$  nm (36  $\text{mW/cm}^2$ ) and detecting the quartz OSL emission at 380 nm through a 7.5 mm thick Hoya U-340 filter. Irradiations have been carried out automatically in the reader by a radioactive beta ( $^{90}\text{Sr}/^{90}\text{Y}$ ) 1.48 GBq (40 mCi) source delivering 0.17 Gy/s in quartz.

Several tests have been used to check the purity of quartz extracts in this work. In most studies, a luminescence signal above background in response to IR stimulation is attributed to feldspars, as at room temperature the fast component of quartz OSL is not stimulated by IR (Aitken, 1998), whereas a large variety of feldspars respond to IR excitation by emitting in the UV (Krbetschek and Rieser 1995, Krbetschek et al., 1997). The samples have been analyzed based on this criterion and yielded negligible IR signal compared to signal collected under blue stimulation. The “IR depletion ratio” test (Duller, 2003), has been applied to every aliquot analyzed throughout this work, and no aliquots had to be rejected.

Linearly modulated-OSL measurements (Bulur et al 2001) indicated that the luminescence signal of interest is dominated by a fast component (Fig. 3), and thus amendable to be analysed through

the single aliquot regeneration protocol (Murray and Wintle 2003, Wintle and Murray 2006)

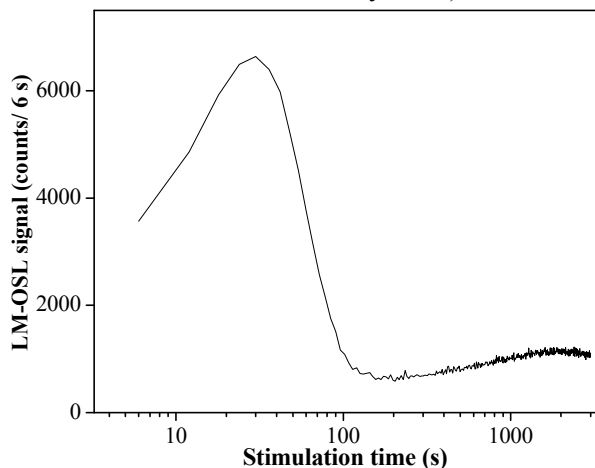


Figure 3. LM-OSL signal of quartz extracted from sample 4B (PHZ). The signal was recorded at 125 °C, the stimulation power being linearly increased from 0 to 40 mW/cm<sup>2</sup> in 3000s.

Continuous wave blue-light stimulated luminescence signals (40 s at 125 °C) have been used for dating, integrated from the first 0.32 s of stimulation. The background correction has been performed subtracting the signal collected between 2.5 and 3.8 s of stimulation.

The main assumption underlying the single aliquot regeneration protocol (SAR protocol) is that sensitivity changes occurring throughout a measurement cycle can be corrected for using the luminescence responses to a constant test dose and thus only one aliquot can be analytically used for determining an equivalent dose. The measurement procedure starts with the measurement of the natural OSL signal after the aliquot has been preheated ( $D_{i=1}=0$  Gy). A test dose is then given, followed by another preheat and the measurement of the OSL response to the test dose. The test-dose preheat (cutheat) is meant to empty the shallow traps. This measurement cycle is then repeated after a regenerative dose is given, and this as many times as desired. Various regenerative doses can be used throughout the experiment but the test dose is kept constant. By dividing the regenerated OSL signals by the corresponding test dose signals, a growth curve is obtained, which is corrected for sensitivity changes (example shown in figure 4).

Intrinsic rigor tests (as recommended by Murray and Wintle 2003, Wintle and Murray 2006) performed indicated that the quartz samples analysed behaved well in the measurement protocol. The recycling ratios close to unity show that we can successfully correct for sensitivity change throughout the measurement cycles. The low

recuperation values (< 1%) indicated that there is no significant unwanted charge transfer into the dosimetric trap. Dose recovery tests demonstrate that we can also successfully correct for sensitivity changes occurring during the first preheat. Furthermore, the measured to given dose ratio as well as the equivalent dose is independent of preheat temperature from 220 °C to 280 °C, both in the case of low (160 °C) and higher cutheat treatments (220 °C- figure 5). This is further evidence that our measurement protocol is robust (sensitivity changes are taken into account, the sampled signal is thermally stable, and any unwanted thermal transfer is irrelevant).

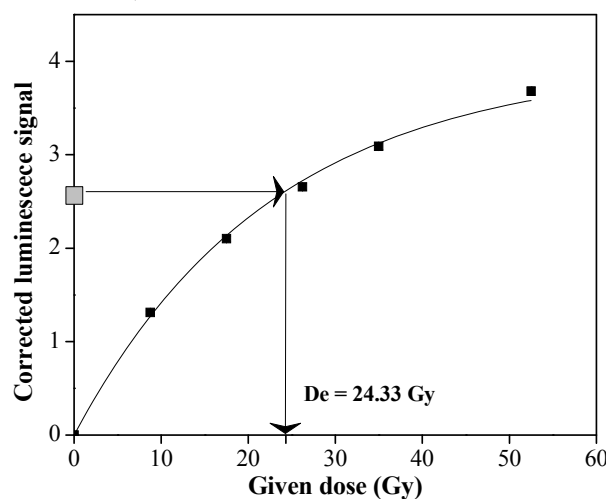


Figure 4. Typical dose response growth curve of quartz extracted from sample 4B (PHZ). The equivalent dose is obtained by interpolating the natural luminescence response on this growth pattern. For the represented aliquot an average equivalent dose of 24.3 Gy was obtained.

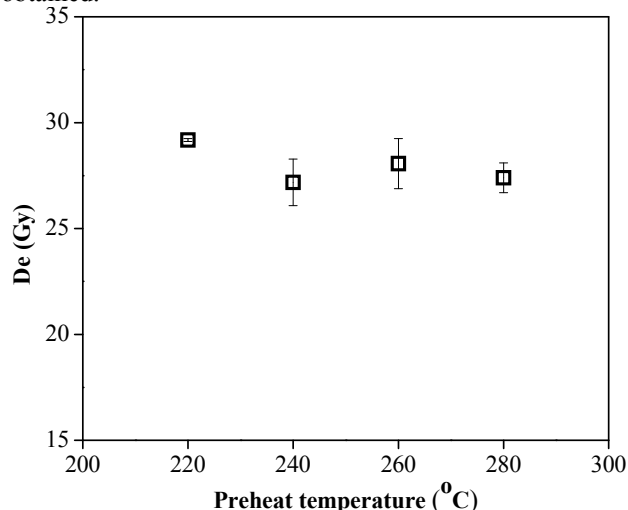


Figure 5. Preheat plateau for sample 4B (PHZ). The thermal treatments consisted in a 10 s preheat at variable temperatures, in combination to a cutheat of 220 °C

Equivalent doses were determined for both samples performing 30 replicate measurements. An average value of 28.3 Gy was obtained for sample 4B clinostagnic phaeozem while for sample 3A albic luvisol we have obtained an average value of 12.6 Gy.

### 3.4. Annual dose measurements

In order for ages to be calculated the annual dose rate was computed based on natural radionuclide concentrations. The specific activities of radionuclides of interest have been determined through high resolution gamma spectrometry using an ORTEC hiperpure germanium detector having the following characteristics: active volume of 181 cm<sup>3</sup>, 0.878 keV FWHM at 5.9KeV, 1.92 keV FWHM and

34.2 % relative efficiency at 1332.5 keV, calibrated in efficiency using IAEA standards. Radionuclides in the uranium series were assumed to be in secular equilibrium. The cosmic dose rate was estimated based on Prescott and Hutton (1994). Relevant dosimetric information is presented in table 1.

Table 1. Relevant dosimetric information for luminescence age determination of the analysed samples. Quoted errors are only of statistical nature.

SAMPLE	U-238 (Bq/kg)	Th-232 (Bq/kg)	K-40 (Bq/kg)	Total dose Rate (Gy/ka)
4B (PHZ)	33 ± 1	47 ± 2	604± 18	2.9 ± 0.1
3A (LUV)	29 ± 1	36 ± 1	564± 16	2.6 ± 0.1

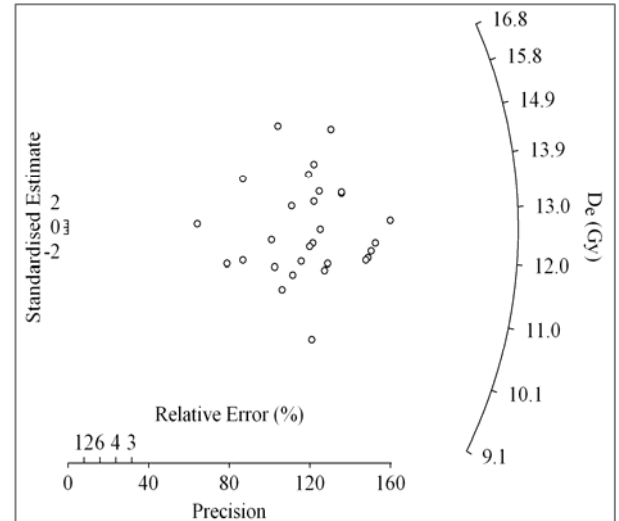
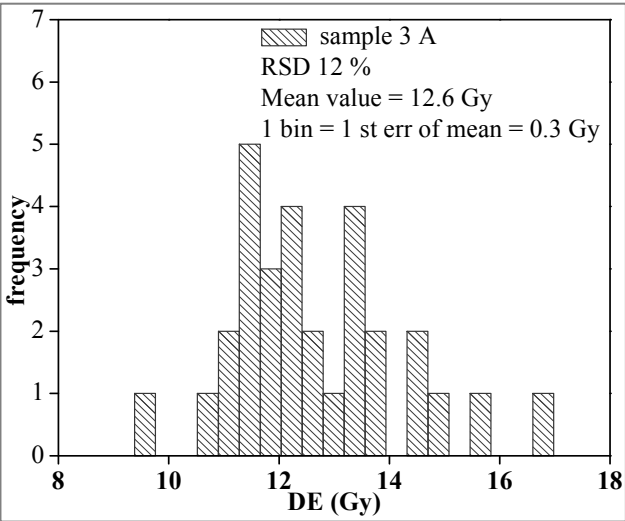
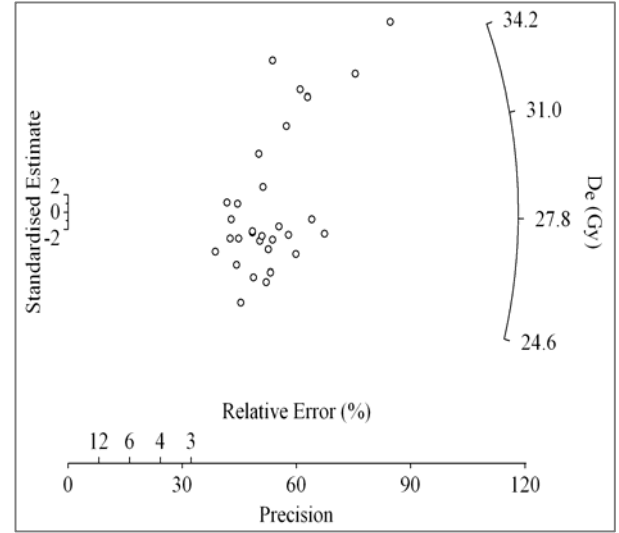
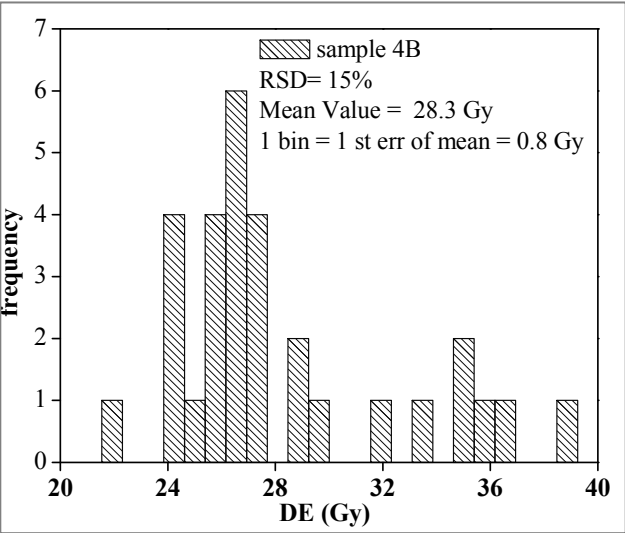


Figure 6. Equivalent dose distributions obtained on 30 aliquots of quartz extracted from sample 4B (chernozem) and sample 3A luvisol. Over dispersion can be noticed.

Table 2. Average, respectively maximum and minimum equivalent doses derived from a selected number of aliquots denoted by n. Uncertainties in equivalent doses quoted represent only random errors. For the age calculation a total moisture content of  $20 \pm 5\%$  was assumed, based on Singhvi 2001, and a factor of  $0.94 \pm 0.45$  was adopted for correcting the attenuation of beta radiations in 63-90 $\mu\text{m}$  quartz grain (Aitken, 1985). Systematic uncertainties of 5% concerning the calibration of the gamma spectrometer, respectively 3% for the calibration of the radioactive source. The last columns presents the ages and the corresponding overall uncertainties calculated using the system of Aitken (1976).

<i>Sample</i>	<i>De average (Gy)</i>	<i>De minimum (Gy)</i>	<i>De maximum (Gy)</i>	<i>Average AGE (ka)</i>	<i>Minimum Age (ka)</i>	<i>Maximum Age (ka)</i>
4B (PHZ)	$28.8 \pm 0.8$ (n=30)	$24.0 \pm 0.8$ (n=6)	$35.1 \pm 0.8$ (n=7)	$10.9 \pm 1.4$	$9.2 \pm 1.2$	$13.4 \pm 1.8$
3A (LUV)	$12.6 \pm 0.3$ (n=30)	$11.8 \pm 0.2$ (n=20)	$14.4 \pm 0.3$ (n=10)	$4.3 \pm 0.6$	$4.0 \pm 0.5$	$4.9 \pm 0.5$

### 3.5. OSL ages and discussions

The equivalent dose distributions are presented as histograms and radial plots respectively in figure 6.

Radial plots allow visualizing and comparing several estimates with different precisions. The plot was originally developed by Galbraith (1988, 1990) for fission track dating and was subsequently also introduced in luminescence dating. In a radial plot, equivalent doses  $De_i$  are plotted in an (x, y) system where the x-axis represents the precision or the relative standard error (%) ( $x_i = 1/\sigma(De_i)$ ) of the individual measurements. The y-axis plots  $y_i = (De_i - De_0)/\sigma(De_i)$ . The central point of the plot ( $De_0$ ) is the average value. Thus, measurements on any straight line radiating from the origin have the same equivalent dose but may have been measured with different precisions. Equivalent doses that are statistically consistent within 2 expected standard error of the mean will fall between a band extending  $\pm 2s$  units around a common radial line. Overdispersion can be observed in the data, as most values do not fall in this band. This is not an experimental artifact or a result of quartz OSL signal intrinsic characteristics, as it was demonstrated that our measurement protocol is robust, and the precision for individual equivalent doses obtained is always better than 3%. The scatter might be caused by the low resolution that is achieved when sampling soils for luminescence dating (7 cm in our case). In order to find the best estimate for the true value of the burial dose measurements should be repeated using smaller amount of grains and by applying statistical analysis of the dataset. However, minimum and maximum ages based on a selected limited number of analysed aliquots can be calculated and are presented in table 2.

## 4. CONCLUSION

Quartz grains extracted from fossil soil covers in the Transylvanian Basin forest steppe zone displayed satisfactory optically stimulated

luminescence characteristics: bright signals dominated by a fast component and a general good behavior in the single aliquot regeneration protocol in terms of dose recovery, recycling and recuperation.

The overdispersion observed in the  $De$  distributions needs further investigations, however, the minimum, respectively maximum optical ages obtained can be regarded as at least partial evidence that the dark soil cover ( $9.2 \pm 1.2$  ka -  $13.4 \pm 1.8$  ka) was a stable feature in the Subboreal and their replacement with Luvisols ( $4.0 \pm 0.5$  ka -  $4.9 \pm 0.5$  ka) reflects a climatic transition from a drier climate (Subboreal) that has favored the extension of the Boreal forests and steppic grasslands to a wetter milder one (the Subatlantic) that favored the expansion of the nemoral and subtropical forests in the Holocene. Thus, these paleo-pedological structures from Transylvania consisting of a fossil mollic horizon of a Phaeozem covered by an albic horizon of an actual Luvisol can be regarded as a paleoclimatic marker.

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