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The LF02 automated luminescence reader

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Abstract

In the present work the LF02 automated luminescence reader is introduced. The construction of the LF02 is part of a project aimed at creating capacities to support the activities of the luminescence dating laboratory at CEADEN in terms of carrying out sediment dating and basic research on quartz luminescence. To accomplish this task, a robust design with a basic structure capable of executing long measuring sequences was proposed. Along with the description of the reader, design concepts aimed at reducing the measurement time are analyzed in detail. To evaluate the effect of each design idea, the term reader productivity is defined. For the evaluation of the LF02 productivity, two different cases are analyzed. In the first one, the sample dose to recover is equivalent to 151 s of beta irradiation, while in the second case the time of irradiation is 833 s. The results show that the introduction of these ideas produces a significant reduction of time needed for completing a SAR sequence.

Keywords: luminescence dating, automated reader, quartz

1. Introduction

After the pioneering work by Huntley et al. (1985) proposing the optical dating of sediments and the subsequent standardization of this method through the development of the single aliquot regenerative dose (SAR) protocol (Murray & Wintle, 2000), optically stimulated luminescence (OSL) has become the major dating tool in Quaternary geology, at least for the past 100 000 years (Wintle, 2008). There is no doubt that the development and commercialization of automated luminescence readers, specifically designed for this application (Bøtter-Jensen et al., 2000; Bortolot, 2000; Richter et al., 2013), has played an important role in the dissemination of this technique. Single grain measurements based on laser stimulation (Duller et al., 1999; Bøtter-Jensen et al., 2003), new more powerful stimulation systems including violet excitation wavelengths (Jain, 2009; Lapp et al., 2015), pulsed excitation-detection systems for time resolved OSL measurements (Denby et al., 2006), EM-CCD detection systems (Kook et al., 2015) and the introduction of small X-ray irradiation sources (Thomsen et al., 2006) are some examples of the new capabilities added in recent years to these instruments.

In Cuba, the correct description and interpretation of the Quaternary geology requires the establishment of the absolute ages of rocks and sedimentary deposits. Also, the development of policies to reduce the impact of sea level rise on the Cuban territory requires the assessment of recent tectonic movement in coastal areas. In both cases, OSL dating is a valuable technique. This explains the interest in creating national capabilities to carry out sediment dating studies

Figure 1. View of the LF02 automated reader

using the OSL technique.

While most of the equipment employed in dating laboratories can also be used in other scientific laboratories, the automated OSL readers are more specific, which makes them expensive and difficult to maintain. Also, for some countries the access to this instrument is limited by export restrictions associated with the radioactive source incorporated into them. Therefore, the development of an automated reader was one of the goals of the project for the creation of the luminescence dating laboratory at CEADEN.

The present paper describes the LF02 automated luminescence reader, routinely used at the CEADEN dating laboratory. Due to the initial projection that only a single reader would be available, the reduction of the measuring time guided the conception and design stages. Therefore, along with the description of the reader, the design concepts aimed at reducing the measurement time are analyzed in detail.

2. Measuring time and reader productivity

In the present work, the measuring time is defined as the time needed to complete a sequence based on the SAR protocol (t_{SAR}). To propose solutions with a positive impact on the measuring time, a conceptual analysis of the dependence of t_{SAR} on the experimental variables becomes necessary. In general, t_{SAR} can be defined by the following equation:

$$t_{\mathrm{SAR}} = t_I + t_M \tag{1}$$

Here, t_I is the total time used for sample irradiation, while t_M represents the duration of all the processes linked to the luminescence measurement such as preheating, OSL measurement or illumination. Both, t_I and t_M, include the time spent on sample positioning.

In Equation 1 it is assumed that at any moment, just one process, irradiation or measurement, is taking place. In the case when irradiation and luminescence measurement are occurring simultaneously, t_{SAR} needs to be represented differently:

$$t_{\mathrm{SAR}} = t_I + (1-b) \cdot t_M \tag{2}$$

Here b is a parameter describing the time overlapping between both processes, and its value ranges from 0 (no overlapping) up to 1 (full overlapping). Equation 2, as a more general case, reveals the pathways to reduce t_{SAR}. The first approach would be the reduction of t_I or t_M; the second one would be the execution of the sequence in a way that measurement and irradiation are run simultaneously.

t_{SAR} depends on several factors such as the number of aliquots, the number of points used to construct the dose response curve, the equivalent dose of the sample and the dose rate of the beta source. Therefore, the use of t_{SAR} to evaluate the reduction of the measuring time has practical value only when the experimental parameters are fixed. Thus, it would be useful to have an indicator of the reader time performance, even when the experimental conditions are different. With such a purpose the term reader productivity (Pr) is proposed. Pr is defined by the following expression:

$$Pr = \frac{D_{\mathrm{e}} \cdot N_L \cdot N_a}{t_{\mathrm{SAR}} \cdot \dot{D}_\beta} \tag{3}$$

Here $\mathrm{D_e}$ is the sample equivalent dose (Gy) (previously

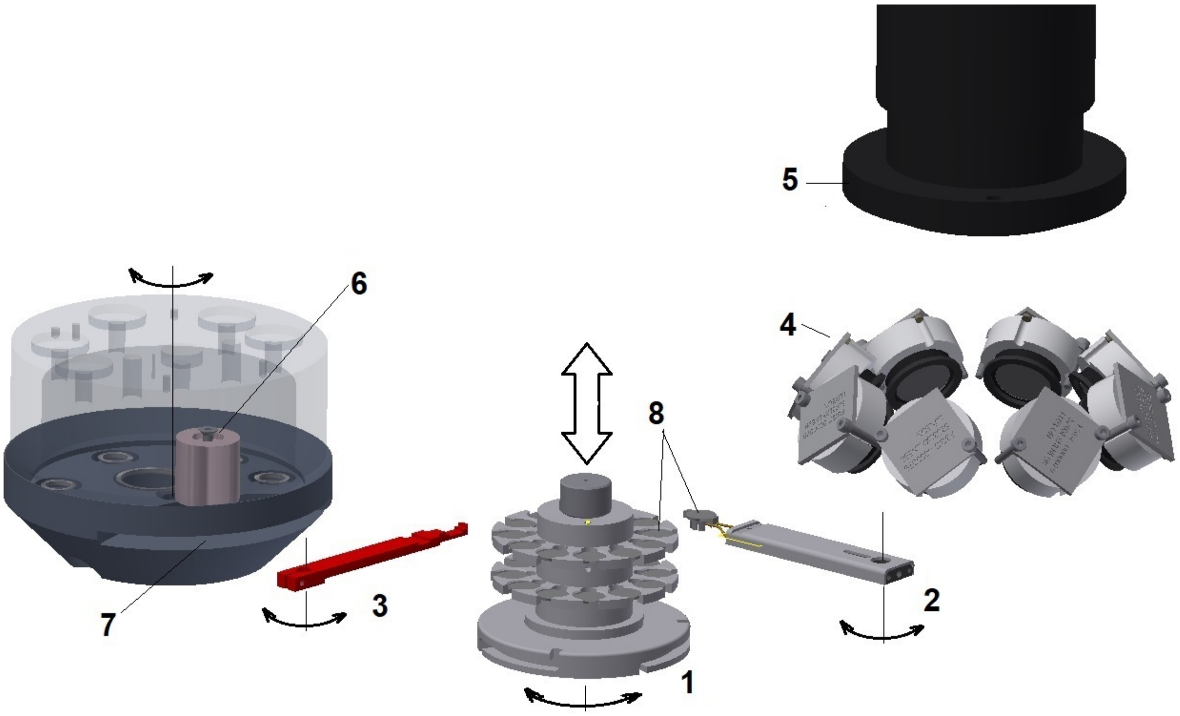

Figure 2. The schematic representation of the of the LF02 measuring chamber. The axes and arrows show the displacement of the moving elements. 1 - sample holder, 2 - measuring arm, 3 - irradiation arm, 4 - LEDs, 5 - PMT, 6 - beta source, 7 - slit for sample entry into the irradiator, 8 - sample disc.

known or measured after running the SAR sequence); N_L is the number of data types in the sequence; N_a is the number of aliquots, $\dot{D}_\beta$ is dose rate (Gy/s) of the beta source and t_{SAR} (s) is the time the reader needed to complete the SAR sequence. Even when this definition may not consider all the situations, it describes the reader time performance: the higher the value of Pr the better the reader time performance.

3. The LF02 automated reader

The LF02 (Fig. 1) follows the basic structure of this sort of instrument: A beta irradiation source (on the left) used for in-situ irradiation, a stimulation detection system (on the right) based on an array of LEDs, a photomultiplier working in the photon counting regime, and the automated sample positioning system holding up to 24 samples. The reader, which is controlled from a computer, has also supporting electronics with its associated software. The measuring chamber of the LF02 was designed to support vacuum. Vacuum conditions are expected to be needed for the future development of optically stimulated electron (OSE) measurement capabilities(Ankjærgaard et al., 2009).

Two mechanical arms pick up sample discs from the holder and place them in the beta irradiator or in the stimulation-detection unit, allowing that any sample can be irradiated while any other is being measured (Fig. 2). A short presentation showing the sample positioning system in action can be found at `https://www.youtube.com/watch?v=_znlq6wXB94`. The arm taking samples to the stimulation-detection unit includes a Kanthal resistive heater that reaches 500 °C with a maximum heating rate of 20 °C/s. The heating rate and temperature stabilization is realized through a tuned digital PID control system with a 2 °C maximal error.

The optical stimulation system comprises 8 ports for installing LEDs. Four of them are used for 1W blue LEDs with focusing optics ($\lambda = 470$ nm, $P_T = 200$ mW in the sample zone); two for IR LEDs with focusing optics ($\lambda = 850$ nm, $P_T = 150$ mW in the sample zone), and the other two are auxiliary ports for a wide range of applications, including another set of LEDs with different wavelength emission. Each LED unit is controlled by a current feedback stabilization system. During the design of the stimulation unit, special care was taken to produce a homogeneous illumination pattern at the sample position (Quesada et al., 2004).

For luminescence detection, the LF02 uses a 9235QB photomultiplier. The quartz OSL is filtered with 7 mm of ultraviolet glass filter UFC6 with a maximum transmittance at 360 nm, comparable to the commonly used U340 filter.

A ^{90}Sr/^{90}Y beta source, used for in-situ irradiation, provides a dose rate of 0.033 ± 0.002 Gy/s. For irradiation, the sample disc is introduced inside the irradiator shielding through a small slit (Fig. 2). Once the sample is in the irradiation position, the radioactive source, which is mounted in a horizontal rotating cylinder, is turned 180° from its parking position to the irradiation port. To irradiate highly sensitive luminescence detectors, such as Al_2O_3:C (Akselrod

Figure 3. The sample holder, hosting up to 24 discs (below), and the container.

et al., 1990), a low dose rate (0.5 µGy/s) Bremsstrahlung (X-Rays) irradiation mode was implemented (Burbidge & Duller, 2003). This method differs from the beta irradiation mode in that during the irradiation the source remains at the parking position.

The sample holder has two sections (upper and lower), each one holding up to 12 samples. The sample holder is assembled with a light-tight container (Fig. 3), to keep the samples in the dark. Once the container with the sample holder is coupled to the reader, the sample positioning system disengages the sample holder from the container and introduces it into the measuring chamber without exposing to the light. Taking advantage of this feature, the LF02 is installed in a room with no red-light illumination system.

The electronics of the LF02 is designed to resolve the simultaneous control of irradiation, sample positioning, sample heating, luminescence measurement and data transmission. The electronic core of the LF02 uses the FPGA technology. Measuring modes such as TL, CW-OSL, LM-OSL and POSL are implemented with a maximum time resolution of 500 ns per channel and 32 bits / 120 MHz counting system.

The measuring sequences for the LF02 are generated with an application called *GenSec*. *GenSec* is a home-made application and generates a perfectly readable xml-format file with an .slf extension. The sequence file includes general information, the measuring parameters and the status of each process, indicating if the process is pending, running or done. An example of the sequence file can be found in the Supplementary Material.

To run the sequence, the defined sequence file is loaded by another application called *GenExe*, which communicates with the reader and controls the execution of the sequence. In the LF02, the input and output files have the same format. As the measuring process goes, the status of each process is updated and the results of the measurement and the time of measurement are appended to the sequence file for subsequent analysis. This allows sequence pausing, and resuming later from the first pending process.

4. Methods for t_{SAR} reduction

In this section the methods implemented in the LF02 reader to reduce t_{SAR} are explained. The methods are presented according to their impact on the time reduction starting from the one with least impact.

a) ***Increasing the optical stimulation power***. Assuming that the OSL curve is described by a single exponential decay $I(t) = I_0 e^{-\sigma(\lambda) P_{\mathrm{LED}} t}$, where $\sigma(\lambda)$ is the photoionization cross-section and P_{LED} is the intensity of the optical stimulation radiation (Kuhns et al., 2000), then the higher the value of P_{LED} the faster the decay. Presently available blue LEDs with enhanced optical power allow the construction of optical stimulation units with stimulation power at the sample position exceeding 200 mW/cm^2. Figure 4 shows the OSL curve measured in the LF02 reader with 90% of the maximal optical power of a fast component dominated quartz irradiated to 5 Gy. As it is observed, only 10 seconds are needed to measure the whole decay curve.

b) ***Merging processes during OSL measurements***. Each cycle of the SAR protocol comprises a group of processes such as preheating and OSL measurement, sequentially executed one after another in the stimulation-detection unit. When these processes are executed individually, after finishing each process the sample disc is returned back to the sample holder following a cooling down process. The merging concept implements two ideas: firstly, samples are not sent back to the sample holder until the last

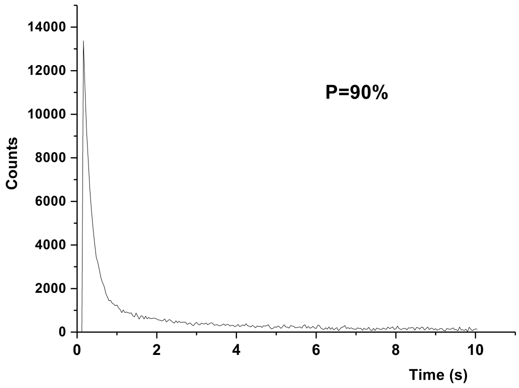

Figure 4. OSL curve for the 5 Gy reference quartz material.

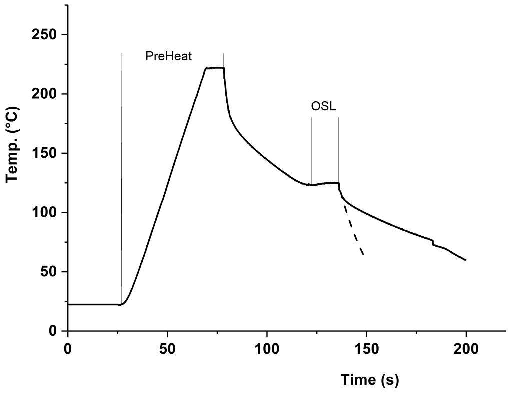

Figure 5. The continuous temperature profile of the merged preheating and OSL measurement processes. The dashed curve shows the effect of the gas jet fast cooling on the profile.

process of the group is executed; and secondly, the sample is heated with a continuous temperature cycle, and only when the last process in the group is executed, the sample is cooled down to the safe temperature. With these ideas, the time used in sample positioning and in cooling down the sample is reduced. Figure 5 shows the temperature profile of a merged pre-heating and OSL measurement. After linear pre-heating to 220 °C, and keeping that temperature for 10 s, the sample is not returned to the sample holder; the system waits for the natural decrease of sample temperature to 125 °C and following a temperature stabilization, the OSL curve is measured for 10 s. Then the sample is cooled down to a safe temperature (60 °C) after which the sample is sent back to the holder. The merging of the OSL measuring processes is defined during the sequence edition. After defining individual processes, the processes to be merged are selected and the command "MERGE" is applied.

c) ***Fast cooling***. Before the sample is returned to the sample holder the system waits until the sample cools down to a safe temperature of 60 °C. If the cooling process is based on the natural heat exchange, depending on the sample temperature it may take 1 – 2 minutes before the safe temperature is reached. To reduce this time, in the LF02 a gas jet nozzle located beneath the heater is used to rapidly cool down the sample at the end of the heating profile. With this system, the cooling down process from 125 °C is reduced from 1 minute to less than 20 seconds (Fig. 5).

d) ***Two-queues scheme***. In the LF02 reader, a sequence can be executed using three different schemes. The first one is the "first sample first" (FSF) scheme, in which all the processes of the first sample are executed first; and then all the processes of the second sample, and so on. The second scheme is the "first process first" (FPF) scheme, in which the "first process" of all the samples is executed first and then the "second process" of all the samples and so on. The third scheme, which is based on the possibility of independently moving samples to irradiation or to the measuring unit, is called "two-queues" (2Q). In the 2Q scheme both types of processes, irradiation and measurement, are run simultaneously. As soon as the system detects that either the irradiation source or the measuring system is available, it looks for a sample, whose first pending process matches the type of the available port and executes it. Using this scheme, due to the time overlapping of the irradiation and measuring processes, a reduction of the overall measuring time is produced.

e) ***External irradiation***. As it was explained above, the sample container of the LF02 allows the transport of the sample discs without exposing them to light. In addition to this, the small sizes and the thin walls of this aluminum container make possible the simultaneous irradiation of all the samples by irradiating the container in a gamma irradiation facility, avoiding sample disc manipulation. For old samples requiring long times of beta irradiation the external irradiation is a convenient method to reduce the irradiation time when all the samples are expected to receive the same dose. The external irradiation is defined during the sequence edition as an independent process. When the LF02 finds an external irradiation process the sequence advances until all the samples are waiting for the external irradiation. After this point the reader automatically sends the sample holder to the container and advises the operator of an awaiting external irradiation. The external irradiation can reduce the overall irradiation time of a single dose point from several hours to some minutes. Due to the relatively high dose rate of gamma irradiation facilities, the external irradiation is not suitable for young samples.

5. Materials and methods

A previously sensitized quartz material with diameters between 180 and 250 µm and showing a predominant fast component in the OSL signal was used to prepare two samples. The first sample is an internal reference material gamma irradiated in a ^{60}Co secondary calibration facility to 5.0 ± 0.1 Gy. The other sample was irradiated in a gamma irradiation facility at CEADEN for 300 s. The dose received by this sample was measured in the LF02 reader using a standard SAR protocol resulting in a dose of 27.5 ± 1.6 Gy. For these two samples a 6 mm diameter spot of quartz grains was mounted on a stainless steel discs.

The reference conditions for the SAR protocol were: Preheat temperature, 220 °C at 5 °C/s for 10 s; cut heat 200 °C at 5 °C/s for 5 s; optical power = 25%; OSL measuring time 40 s and test dose of 30 s. The preheat temperature was determined from a preheat plateau test. When using increased optical power (90 %), the conditions are the same except the OSL curve was measured during 10 s. Using the reference conditions, the beta dose rate was found to be

Procedure designation	**Procedure description**	t_{SAR} **(hours)**	**Time reduction (%)**	***Pr***	**D$_e$(Gy)**
A0	5 Gy sample, P_{LED}=20%, no merging, no fast cooling, FPF mode	23	-	0.26	5.0 ± 0.2
A1	5 Gy sample, P_{LED} =20%, no process merging, fast cooling, FPF mode	16	31	0.37	5.1 ± 0.1
A2	5 Gy sample, P_{LED} =90%, process merging, fast cooling, FPF mode	12.5	45	0.48	5.1 ± 0.2
A2-2Q	5 Gy sample, P_{LED} =90%, process merging, fast cooling, 2Q mode	10	56	0.60	4.8 ± 0.2
A2-2Q-HD	27.5 Gy sample, P_{LED} =90%, process merging, fast cooling, 2Q mode	16.3	-	2.24	27.5 ± 1.6
A2-2Q-EXT	27.5 Gy sample, P_{LED} =90%, process merging, fast cooling, 2Q mode, external irradiation	10	38	3.66	26 ± 2.4

Table 1. Comparison of the time needed to complete a SAR sequence (t_{SAR}) and reader productivity (*Pr*) using different procedures. The time reduction is calculated using procedures A0 and A2-2Q-HD as reference for the 5 Gy and 27.5 Gy samples respectively. The equivalent dose (D$_e$) obtained for each procedure is also presented.

0.033 ± 0.001 Gy/s. At this dose rate, the 5 and 27.5 Gy doses are produced after 151 and 833 s of irradiation respectively.

A ^{60}Co gamma irradiation facility was used for external irradiation. With the purpose of homogenizing the dose, during the external irradiation the container is mounted in a spinning system. To achieve irradiation uniformity better than 4% only the upper section of the sample holder was used (12 samples). Considering the beta dose rate as a reference value, the dose rate of the irradiation facility at the time of this experiment was 0.090 ± .006 Gy/s (324 ± 22 Gy/h), which is 2.7 times higher than the beta source dose rate. The overall transient dose appearing during the introduction and extraction of the container was 1.02 ± 0.05 Gy.

The value of t_{SAR} is defined as the time for completing a sequence of SAR protocol on 12 aliquots. Beside of the initial dose measurement (L_0) , the OSL response for three dose points (L_1, $L_{2,}$ L_3) was used for the construction of the dose response curve. Two additional measurements were used for recuperation (L_4) and recycling (L_5) tests. A constant test dose of 30 seconds irradiation was given after the measurement of each dose point; and the corresponding OSL response (T_0 trough T_5) was measured. For the 5-Gy sample, the dose points were 100, 150 and 250 s of beta irradiation; for the 27.5 Gy sample, the dose points were 700, 850 and 1000 s of beta irradiation and when the gamma irradiation facility was used, the irradiation times were 270, 300 and 400 s. In the last case additional 20 minutes per irradiation point were employed for sample transferring and preparative works.

For both samples a linear regression was used to construct the dose response curve. For the 5 Gy sample the regression coefficient was better than 0.99; for the 27.5 Gy sample the regression coefficient was lower, 0.98, due to non-linear behavior of the dose response curve in that region. The criteria for aliquot acceptance were 1.00 ± 0.05 for the recycling ratio and 0.00 ± 0.05 for the recuperation. Among the 72 aliquots used in this study only 2 were rejected.

6. Results

Six different procedures were used to evaluate the proposed methods of time reduction. The procedures use different combinations of the methods proposed above. Table 1 shows the designation and the description of each procedure. The procedure denoted as A0 uses the reference conditions for the SAR protocol described in the previous section and it is executed with FPF scheme. The A1 procedure uses the same conditions as the A0 but the fast cooling method is added at the end of each heating profile. The A2 procedure combines increased stimulation power (90%), merging of processes during OSL measurement and fast cooling. Both A1 and A2 procedures are executed with FPF scheme. The A2-2Q procedure uses the same conditions as A2 but instead of using FPF scheme uses the 2Q scheme of sequence execution. All these procedures were tested with the 5 Gy sample.

For the 27.5 Gy sample the reference procedure is the A2-

2Q-HD. This procedure uses the same time reduction methods used in the A2-2Q procedure, and is denoted differently to mark the measurement of a sample with higher equivalent dose. The A2-2Q-EXT procedure uses the same conditions as A2-2Q-HD, with the difference that the dose points of L1, L2, L3 and L5 are given externally while test doses are given with the beta source. The results are presented in Table 1.

When comparing the procedures used in the 5-Gy sample, it is observed that the application of each method produces a reduction of the 23 hours needed for completing the reference procedure A0. In this sequence, the total irradiation time is 3.2 hours; which indicates that most of the time is dedicated to luminescence measurement.

The fast cooling introduced in the A1 procedure drastically reduces t_{SAR}. The reason of this significant time reduction is due to the reduction of time needed to cool down to the safe temperature, especially after preheating to 220 °C. The incorporation of other methods in A2 and A2-2Q procedures, allows decreasing the measuring time to less than half the time used in the reference method A0. When using *Pr* to analyze reader time performance, an increment of its value is observed as new methods of time reduction are added

In the case of the A2-2Q-HD procedure, the time needed to deliver a beta dose point is several times longer than the time spent in executing the group of processes to measure a single OSL signal; therefore, an almost complete time overlapping should be expected. For this procedure, the value of t_{SAR} (16.3 hours) is close to the total irradiation time (13.8 hours); indicating that the luminescence measuring process occurs behind the irradiation. Although the total irradiation time of the A2-2Q-HD procedure is 4.3 times greater than the one of A2-2Q, the corresponding ratio for t_{SAR} is less than 2. This is explained because in the low dose case, the time overlap is very low and very often the sequence gets blocked waiting for a luminescence measurement process to be finished. Finally, the application of the external irradiation method reduces the measuring time by nearly 40%, however this time reduction is principally due to the reduction of the irradiation time.

The evaluation of *Pr* for all procedures is also shown in Table 1. It can be observed that, as new methods of time reduction are applied, the reader productivity increases. A significant increment is found after the application of the fast cooling method. Another important increment is observed from the A2-2Q procedure to the A2-2Q-HD; confirming the previous conclusion that the 2Q scheme is more effective when beta irradiation times are longer than the typical 3-4 minutes duration of the luminescence measuring process (see Fig. 5). As expected, the higher *Pr* is obtained for the A2-2Q-EXT procedure in which all the proposed methods are combined.

The methods used here to reduce t_{SAR} change the conditions under which the equivalent dose is normally measured: higher photon counting rates, continuous heating profile, fast sample cooling, etc. Therefore it is important to check that these modifications do not produce significant variations in the measured equivalent dose. The results of this study are presented in Table 1. In the case of the 5-Gy sample, the results show that there is good agreement between the procedures and that all the procedures give a result close to the expected dose.

From the point of view of measuring conditions, there is almost no difference between A2-2Q and A2-2Q-HD procedures, except a higher photon counting rate during the OSL measurement in the A2-2Q-HD procedure. Nevertheless, for the 27.5 Gy sample used in this procedure, the photon counting rate is quite below the maximal counting rate of the detection system. Therefore, the accuracy of the 27.5 Gy dose obtained with the A1-2Q-HD method can be inferred from the correctness of the A1-2Q method, previously established. The comparison of the results given by A1-2Q-HD and A1-2Q-EXT methods shows good agreement, noticing a higher dispersion for the A1-2Q-EXT method; mainly motivated by the operator intervention during the gamma irradiation process.

7. Conclusions

The LF02 automated luminescence reader, routinely used at the CEADEN dating laboratory, has been described. Five methods proposed to reduce the time spent on equivalent dose measurements using the SAR protocol were evaluated. Three of these methods are related to the reduction of the luminescence measuring time, one related to the reduction of the irradiation time and the other oriented to produce time overlapping of irradiation and measurement. The application of these methods allowed a time reduction to less than half of the time without the implementation of the methods. The proposed *Pr* parameter proved useful for evaluating the reader productivity under different conditions. Based on the increment of *Pr*, the application of all these methods improved the reader time performance.

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Reviewer

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