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IRSAR – A single-aliquot regenerative-dose dating protocol applied to the infrared radiofluorescence (IR-RF) of coarse-grain K-feldspar

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Abstract: Since the infrared radiofluorescence (IR-RF) at 865 nm emitted by potassium-rich feldspars has been proven to be suitable for sediment dating, all instrumentation requirements for this new dating technique have been met. This paper describes the single-aliquot regenerative-dose protocol IRSAR (infrared radiofluorescence single-aliquot regenerative-dose) applied to coarse-grain K-feldspar. Based on state-of-the-art knowledge of the physical background and the methodology, this promising new technique can be used for the dating of clastic sediment samples in the age range up to over 250 ka.

1. Foreword

The first papers concerning the radiofluorescence of K-feldspar and its potential as a luminescence dating method used the expression “infrared radioluminescence (IR-RL)” (e.g. Trautmann et al. 1999a). In solid state physics literature, the nomenclature for luminescence phenomena refers usually to the fundamental luminescence excitation mechanisms. Whereas “radioluminescence” includes all luminescence effects induced by interaction with ionising radiation, e.g. radiophosphorescence processes like thermoluminescence and optically stimulated luminescence. The phenomenon described in the present paper arises from prompt radiative charge transitions during interaction of the samples with ionising radiation and is, irrevocably, a fluorescence process. Thus to avoid misunderstanding, we suggest the abbreviation “IR-RF” as synonym for the infrared radiofluorescence of K-feldspars

2. Introduction

2.1 Physical background

Radiofluorescence (RF) is a well known phenomenon, which arises from prompt radiative charge transitions during interaction with ionising radiation. Historically, the radiofluorescence of ZnS helped raise Rutherford (1911) to the scientific Pantheon when he used this to determine the scattering of α and β particles in a gold foil to prove the existence of the atomic structure. For radiofluorescence of ZnS the synonym “scintillator” was introduced and this kind of radiofluorescence is

still used when NaI:Tl detectors are applied to γ spectrometry tasks.

The present paper describes the radiofluorescence of coarse-grain K-feldspar, for which the experimental set-up, first described by Trautmann et al. (1998), is illustrated in figure 1.

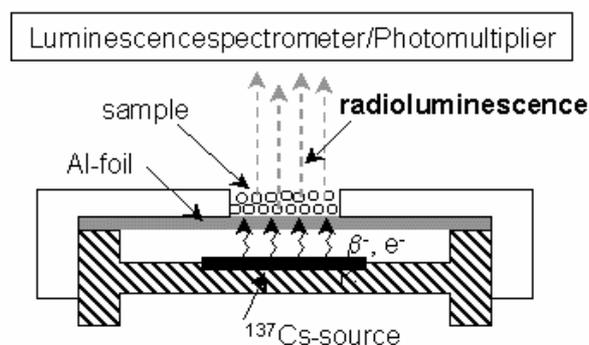


Figure 1.

Schematic of equipment for radiofluorescence measurements using a laboratory β source (after Trautmann et al., 1998)

K-feldspar coarse-grains with a grain size of 130 to 150 μm are fixed on an aluminium foil or, as reproducible mono-layers with defined thickness, on thin adhesive foil strips. The laboratory ^{137}Cs source fulfils two tasks: excitation of the fluorescence and the application of a β dose.

The simple two-term energy band model in figure 2 shows the process of prompt radiative charge trapping and recombination which arises from

interaction of K-feldspar with ionising radiation. Electrons (black dots) are excited by ionising radiation and transfer from the valence band (VB) to the conduction band (CB) (transition *a*).

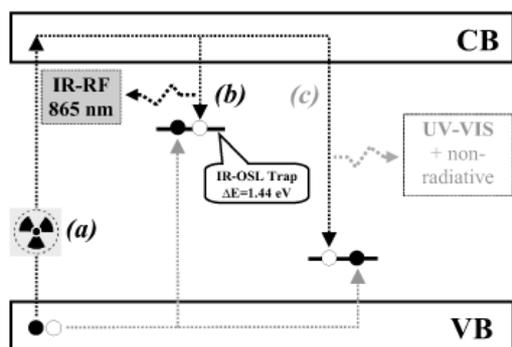


Figure 2.

Simple two-term energy band model which shows the fundamental physical idea behind IR-RF dating, especially the radiative electron transition from the conduction band (CB) to the IR-RF (IR-OSL) trap emitting characteristic fluorescence light at 865 nm.

Electrons thus transferred to the conduction band only remain a few nanoseconds in this delocalised state and immediately are forced to recombine with holes and electrons in the valence band (direct CB→VB transition not shown in the figure). These are attracted to hole traps in the band gap, resulting from the presence of several intrinsic or extrinsic defects, and this leads to recombination at metastable traps or recombination centres (transition *b* and *c*) emitting characteristic fluorescence light. In contrast to other materials, only the types of K-feldspar classified as microcline, orthoclase (and amazonite) show radiofluorescence in the infrared at 865 nm (IR-RF), which is interpreted as radiative electron transition from the conduction band to the trap (transition *b*). In all other feldspars, the transitions from the conduction band to the dosimetric traps are non-radiative and the energies of the electrons are dissipated by phonon processes somewhere in the crystal lattice. Because of the similarity of the IR-OSL excitation energy of 1.44 eV (855 nm) and the wavelength of the IR-RF at 865 nm (1.43 eV), Trautmann et al. (1999a,b) concluded that the IR-RF trap and the IR-OSL trap originate from the same crystalline defect. The other special feature of the IR-RF process is the completely different behaviour of the dose response curve. The IR-RF signal decreases exponentially with given dose and its saturation level is reached after about 1300 Gy dose. This can also be explained using the model in figure 2: The intensity of the IR-RF intensity depends on the electron

density in the IR-RF (IR-OSL) trap. The higher the density of trapped electrons, the lower is the probability that other electrons can undergo the transition into this trap; this results in a decrease of the IR-RF intensity. Furthermore, the stability of the dosimetric signal does only depend directly on the thermal behaviour of the IR-RF (IR-OSL) trap. This transition also takes place in the radioactive environment of the K-feldspar crystals in sediments. Therefore, the IR-RF (IR-OSL) trap is filled continuously during burial. The processes in nature and in the laboratory are absolutely comparable, apart from different dose rates. The “radiative” filling of traps continues in the laboratory. Thus measurement of the IR-RF is the first opportunity in luminescence dating to monitor the electron density in a trapping state and to use it as primary dating information.

The electron density in the trap can be altered by sunlight with wavelengths less than 550 nm within about one hour (Trautmann et al. 1999a,b) and by a laboratory solar simulator within a few minutes (Krbetschek et al. 2000). This fulfils the bleaching requirements for sediment dating.

The radiofluorescence signal is not restricted to the infrared. Emissions in the UV-VIS can also be excited (transition *c*) and they are understood to be radiative recombinations into recombination centres during interaction with ionising radiation. With dose, their intensities mostly increase and can be explained in the same way as the decreasing IR-RF intensity: the more traps that are filled with electrons, the higher is the probability for electron recombinations into recombination centres.

Transitions shown in grey for holes (black circles) are needed for charge neutrality in irradiated crystals. During laboratory irradiation, holes will continuously move from the valence band to UV-VIS recombination centre(s) as well as to the IR-RF (IR-OSL) trap. This explains why the IR-RF saturation level shows a constant residual IR-RF signal. More detailed information about the physical background and modeling approaches of this special luminescence effect are given in the literature review in section 2.2 below.

2.2 Literature review

Trautmann et al. (1998) reported pioneering investigations of spectral and dosimetric radiofluorescence (RF) properties of alkali feldspars, Trautmann et al. (1999a) realised the potential of the dating application of the infrared radiofluorescence (IR-RF) that only occurs in potassium feldspars, such as microcline and orthoclase. The IR-RF was explained as the radiative transition of electrons from the conduction band via the excited state to the ground state of the 1.44 eV IR-OSL trap, emitting

characteristic luminescence at 865 nm. This was inferred from the strict single exponential decay of the IR-RF dose curves for all K-feldspars. Trautmann et al. (1999a,b) and Trautmann (2000) presented dating results, which mostly agreed very well with other dating results. Krbetschek et al. (2000) summarised methodological aspects of the IR-RF technique and Krbetschek and Trautmann (2000) investigated the dosimetric RF behaviour of other materials such as quartz and halite. Trautmann et al. (2000) presented a spectral study of single feldspar grains and pointed out the potential of IR-RF single grain dosimetry. The challenge of a very precise beta source calibration for the IR-RF technique has been solved by the introduction of a radiofluorescence method using $\text{Al}_2\text{O}_3:\text{C}$ crystals as probe dosimeters (Erfurt et al. 2000; Erfurt et al. 2001). Together with an automated multi-spectral radioluminescence (RL) reading system for geochronometry and dosimetry, Erfurt et al. (2002) precisely described the calibration protocol using the RF at 415 nm of $\text{Al}_2\text{O}_3:\text{C}$ dosimeters. They also reported on dating results which correspond very well with independent ages. Furthermore, the physical background and the methodology of the IR-RF dating technique were investigated by Erfurt and Krbetschek (2002) and, among other things, they found, that the IR-RF dose curves can be modelled more precisely using a stretched single exponential decay. They also suggested the use of a combination of additive dose and regeneration dose curves (slide technique) for the estimation of the palaeodose. Schilles and Habermann (2000) and Schilles (2002) reported on an experimental device for the measurement of IR-RF of K-feldspar and physical features of this emission.

In this paper we present state-of-the-art knowledge of the methodology of the IR-RF dating technique, resulting in a standard single-aliquot regenerative-dose protocol, called IRSAR.

3. Measurement conditions

3.1 IR-RF instrument

All measurements were carried out using the automated multi-spectral radioluminescence (RL) instrument described by Erfurt et al. (2002).

3.2 Sample preparation and geological settings

All samples were prepared using standard procedures (Aitken 1998). Geological settings and detailed sample preparation descriptions of the samples (GOS4, HURL1, SHO, GRÖ8 and OOK1) can be found elsewhere (Preusser 1999; Erfurt et al. 2002; Trautmann et al. 1999b). From the IR-RF measurements already carried out, it was clear, that samples that were additionally treated by flotation to extract the whole feldspar fraction before the K-

feldspars were separated by density, showed much higher IR-RF intensities. Therefore we used the flotation technique to extract quartz from feldspar as described by Miallier et al. (1983)

3.3 Physical and methodological requirements of IR-RF dosimetry for sediment dating

This new IR-RF dating technique is characterised by the following methodological features:

- Extracted K-feldspars which underwent flotation, prior to being separated by density, showed stronger IR-RF intensities; this may indicate enhanced enrichment of K-feldspars (Erfurt et al. 2002)
- Due to the continuous excitation by ionising radiation and the simultaneous observations fluorescence, the method is a single-aliquot technique with only one regenerative dose cycle compared to the commonly used SAR protocols applied to quartz and feldspar (Murray and Wintle 2000; Wallinga et al. 2000)
- The RF spectra of K-feldspars show, an interfering RF peak at about 710 nm; this has to be considered when filters for the isolation of the IR-RF peak are chosen (figure 3)
- The IR-RF dose curves follow a stretched single exponential dose curve function (Erfurt et al. 2002) with non-significant fit residual (see figures 4 and 5)
- After bleaching by light with solar emission characteristics, the samples exhibit strong phosphorescence (figure 6)
- Since the IR-RF is measured continuously during interaction with ionising radiation, all transitions of charges in the material are characterised by a dynamic (transient) equilibrium which compares to processes taking place in the radioactive environment of the sediments (Erfurt and Krbetschek 2002)
- Compared with secondarily excited radiophosphorescence techniques, such as TL and OSL, IR-RF the dosimetric informations based primarily on excited electronic transitions and the electron density in the IR-RF (IR-OSL) trap is monitored (Erfurt and Krbetschek 2002)
- The method does not need thermal pre-treatment of samples and the IR-RF shows no change in the dose curve response during regeneration cycles (figure 4)
- The mean saturation dose is about 1200 to 1500 Gy. However, due to the small dynamic range of the IR-RF process and the decrease of the sensitivity with dose, the dose curves are resolvable up to about 600 to 800 Gy (Erfurt et al. 2002)

- The IR-RF trap shows very high thermal stability. There is no significant change in the dating results if the samples are heated up temperatures ranging from 250 to 350°C (figure 7)

3.3.1 Spectral aspects, filter choice

Krbetschek et al. (2000) mentioned IR-RF detection problems that may result from the interference of a neighbouring RF emission band at 710 nm. For a better understanding of filter selection, we have used a Gaussian distribution function in order to simulate different interference ratios for the RF Gauss peaks, separated by different filters (resulting spectra in figure 3). To simulate the “worst case” scenario, we used the mean Gaussian fit data of saturated K-feldspars (about 1500 Gy) with a very strong emission at 710 nm.

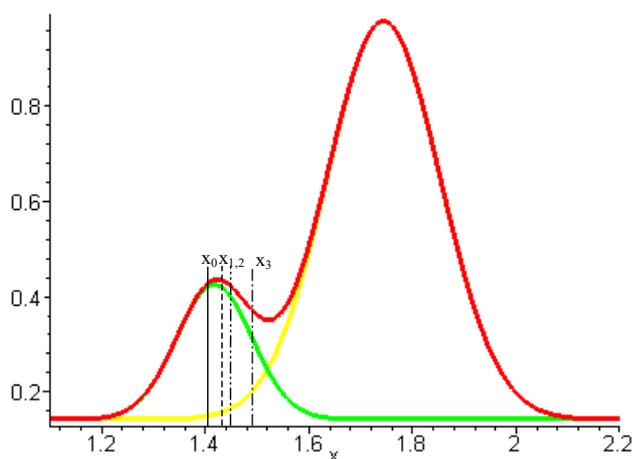


Figure 3.

Simulated saturated spectrum generated by mean values of Gaussian distributions from an number of RF spectra of K-feldspars in the energy range from 1.2..2.0 eV. The vertical lines indicate the integration intervals ($x_0..x_1 =$ Andover 200FC35-25/8650, $x_0..x_2 =$ Schott RG850, $x_0..x_3 =$ Schott RG830)

An interference filter (Andover 200FC35-25/8650) with a bandwidth of 18.7 nm FWHM (after Erfurt et al. (2002)) and the characteristics of the Schott RG850 and Schott RG830 filters (suggested as the detection window by Schilles (2002)) have been used as the integration regions. This Gaussian distribution of the spectrum was integrated (fig. 1) at 50 % transmission for all filters (see figure 3: $x_0..x_3$ as integration intervals for the simulation of different filter transmissions). The results of this simulation were corrected for the changing detector sensitivities in this wavelength interval. The computed percentage of the simulated signal overlapping are for the

interference filter (Andover 200FC35-25/8650) 5.2%, for the Schott RG850 6.4% and for the Schott RG830 12.1%. Typical values for signal interference (e.g. sample Bur10, described by Krbetschek et al., 2000) were estimated using this spectral simulation. The ratios were not more than 0.9% for the Andover 200FC35-25/8650, 1.0% for the RG850 and 1.8% for the RG830. In contrast to Schilles (2002), we do not recommend using filters which transmit below 840 nm. Optimal results can be reached using an interference filter centred around the IR-RF emission at 865 nm, such as the previously mentioned Andover 200FC35-25/8650 or with much higher transmittance (but also much more expensive), filters of the Chroma Technology HQ series (HQ865/20M). However, since the IR-RF signals are the strongest RF emissions in microcline and orthoclase, the typically low transmission percentages of interference filters (of only about 40 to 50%) still leads to sufficient PMT counting statistics.

The emission at 920 nm in K-feldspar (Erfurt and Krbetschek 2002) does not have an influence on the dose characteristics of the the 865 nm peak; this is because of the very low sensitivity of the used Hamamatsu PMT R943-02 in this wavelength region (Erfurt et al. 2002).

3.3.2 Dose curve fitting

The experimental data and the luminescence model suggested by Trautmann (2000) give a single exponential dose curve function which described the IR-RF data. The IR-RF was measured using RF spectra together with a Gaussian distribution of the spectra. Only a maximum of about 30 dose points were used for the single exponential fit function. Using the automated RL instrument (Erfurt et al. 2002), commonly about 200 and up to 800 dose points are measured depending on the absorbed dose. Such a high data density results in a very precise dose curve record. It has been observed by Erfurt and Krbetschek (2002) that the IR-RF dose characteristic does not exactly follow a strict single exponential function. Therefore they suggested, that a stretched exponential function (equation 1) can describe a behaviour which is often encountered in disordered condensed-matter systems (e.g. by Pavesi and Ceschini (1993)).

$$(1) \quad \Phi(D) = \Phi_0 - \Delta\Phi(1 - \exp(-\lambda D))^\beta$$

(Φ_0 ..initial IR-RF flux, $\Delta\Phi$..dose dependent change of the IR-RF flux, λ ..exponential parameter, D ..dose, β ..dispersive factor)

They suggested that the dispersion of charge carrier transition or release rates and trap energies in

multiple trapping-detrapping mechanisms can be expressed in the dispersive factor β in equation 1. Erfurt and Krbetschek adapted such a function for the IR-RF dose curve description and discussed its influences on the model introduced by Trautmann (2000).

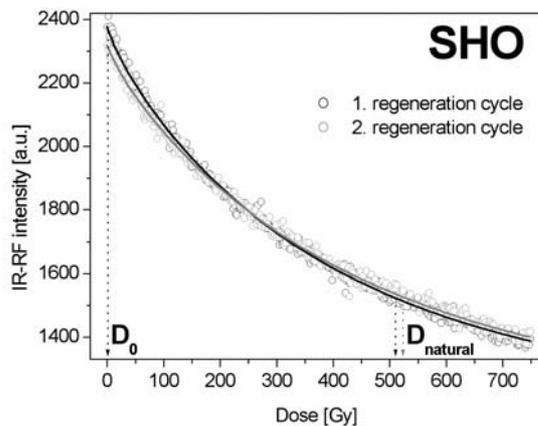


Figure 4. Dose curves of regeneration cycles of sample SHO. One can see the very good fitting of a stretched exponential function to the IR-RF data. Also slight changes in the luminescence response after a second regeneration cycle resulting in only a very small change of the dose determination are visible.

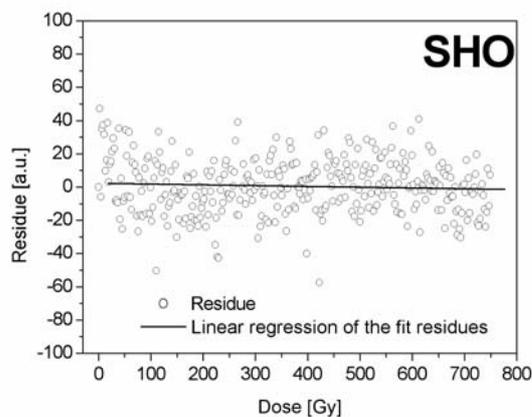


Figure 5. Fit residual of the IR-RF dose curve fit of sample SHO (figure 4) using equation 1. The residual were fitted using a linear regression function. One can see the precision of the parameter determination using this stretched single exponential fit function. Together with the high number of measured dose points (a few hundreds) the IR-RF data show good agreement of experimental observation and theoretical considerations (Trautmann 2000, Erfurt and Krbetschek 2002).

Applying equation 1 to the data of figure 4, the dose curve of sample SHO can be described yielding residuals close to zero (figure 5) (more examples in Erfurt et al. (2002) and Erfurt and Krbetschek (2002)).

3.3.3 Effects after bleaching I: phosphorescence

Trautmann et al. (1999a,b) pointed out the spectral bleaching behaviour of the IR-RF signal and Krbetschek et al. (2000) discussed the implication for dating methodology. If the samples are bleached using the Osram HTI lamp (which is part of the RL instrument described by Erfurt et al. (2002)) K-feldspar show very strong phosphorescence at 865 nm. Figure 6 illustrates this effect, observed for a saturated sediment sample OOK1 (about 1500 Gy given dose). The decay of the phosphorescence is exponential. The phosphorescence was measured immediately after the bleaching light was shut off from the samples. Because of an 18 seconds time delay between shutter closing and the first phosphorescence measurement, we extrapolated the exponential decay function to the initial phosphorescence as shown in figure 6.

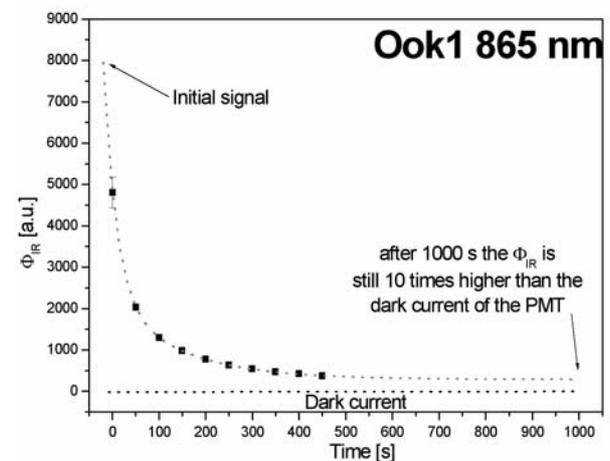


Figure 6. Typical phosphorescence kinetics at 865 nm measured directly on sample OOK1 after bleaching for 30 minutes. The decay follows an exponential function and complete depletion of the signal is measured only after a few thousands seconds. This may interfere with the IR-RF signal, if regeneration is started immediately after bleaching.

The phosphorescence in figure 6 was measured for only 450 seconds. On extrapolation of the phosphorescence characteristics, one can see that the intensity is not completely decayed. Therefore, it is important to wait about one hour after bleaching, before the regeneration of the palaeodose can be

started, because of a possible superposition of the phosphorescence after bleaching and the radiofluorescence.

3.3.4 Effects after bleaching II: changes in the IR-RF dose curve response

After a second regenerative dosing, the reconstructed dose curves show no significant differences from that of the first cycle. Figure 4 presents a typical example of a sediment sample (SHO). A change in the dose curve response is commonly expressed in terms of sensitivity changes.

As one can see in figure 4, such effects may occur to a small extent but do not change the dose reconstruction (using the IRSAR protocol) adversely. If a palaeodose would be estimated using these two regenerated dose curves applying the IRSAR protocol (see section 4), a deviation of only 3 % occurs ($D_{\text{black}}=508.9$ Gy and $D_{\text{grey}}=525.9$ Gy).

3.3.5 Saturation dose, signal stability and upper dating limit

Trautmann et al. (1999b) concluded from spectral measurements, that the IR-RF saturation dose lies at about 800 Gy. Measuring the IR-RF with higher dose resolution (number of measured dose points) using the automated RL instrument (Erfurt et al. 2002), a saturation dose of about 1200..1500 Gy was estimated (see figure 4). On the other hand, the technical dating limit depends mainly on the dynamics ($d\Phi/dD$) of the IR-RF signals at high given doses. Based on state-of-the-art knowledge, it can be assessed that the IR-RF dose curves are resolvable up to 650 Gy (Erfurt et al. 2002) and this results in an (mean) upper dating limit of around 250 to 280 ka, assuming a sediment dose rate of about 2.5 mGy a^{-1} . Thus, the high saturation dose together with the thermal stability of the IR-RF signal has potential to increase the upper age limit of luminescence dating. Figure 7 shows the thermal stability of the IR-RF signal after pulse annealing of sample SHO to 100°C , 175°C , 250°C and 350°C . The equivalent dose of sample SHO at room temperature is ≈ 600 Gy. The reconstructed dose after a preheat only slightly changes, giving values at $100^\circ\text{C} \approx 595$ Gy, $175^\circ\text{C} \approx 580$ Gy and $250^\circ\text{C} \approx 565$ Gy. After annealing up to 350°C , the signal is altered and the reconstructed dose equals to ≈ 515 Gy. This test implies, that the IR-RF signal seems to be thermally stable up to about 250°C .

4. The dating protocol IRSAR (infrared radiofluorescence single-aliquot regeneration)

Summarising all aforementioned methodological aspects, we describe in the following the IRSAR

(infrared radiofluorescence single-aliquot regeneration) dating protocol.

The IRSAR protocol applied to the IR-RF on K-feldspar:

1. Separation of K-feldspars using standard procedures combined with the flotation technique to separate quartz from feldspars
2. Measuring of the IR-RF intensity at natural dose D_N
3. Bleaching of samples under solar conditions for 30 minutes without changing the geometry
4. Waiting for at least one hour until the phosphorescence excited by the bleaching light is decays completely
5. Regeneration of the IR-RF signal to the natural dose level with preferably a high number of dose points
6. Fitting data to a stretched single exponential function (equation 1)
7. Using fit parameters (Φ_0 , $\Delta\Phi$, λ and β) and the IR-RF signal ($\Phi(D)$) at the natural dose level to calculate the palaeodose D

After an investigation by Erfurt et al. (2002), the application of the protocol is also shown in figure 8. The first step is the measurement of Φ_{natural} connected to the palaeodose D_e .

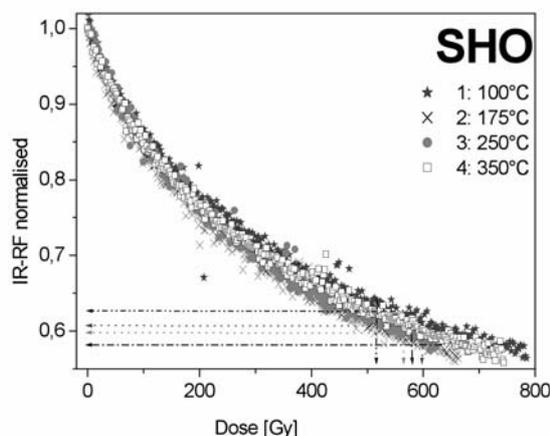


Figure 7. Comparison of IR-RF dose curves after preheat of 100°C (grey stars), 175°C (black crosses), 250°C (grey circles) and 350°C (open black squares). Comparing the determined equivalent dose for the unheated sample of 598 Gy, the reconstructed doses of the preheated samples at 100°C (595 Gy), 175°C (580 Gy) and 250°C (565 Gy) only changes slightly. This indicates the high thermal stability of the IR-RF process.

During bleaching for 30 minutes, the IR-RF increases up to the maximum IR-RF signal Φ_{bleached} . After bleaching the IR-RF signal Φ_{natural} related to the palaeodose D_e is regenerated, by measuring 200 dose points. The applied laboratory dose then equals the equivalent dose, absorbed during burial after the last light exposure of the sediment.

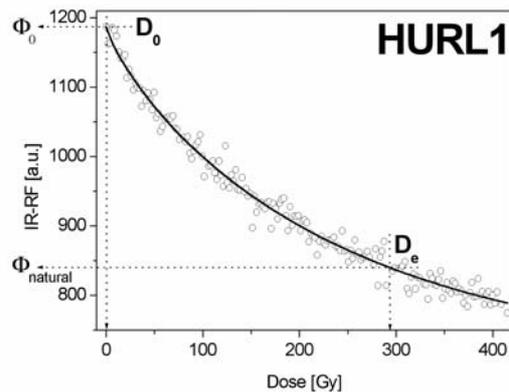


Figure 8. IR-RF dating example applying the IRSAR protocol to sample HURL1 (Erfurt et al. 2002). The dose curve was measured by reading 200 dose points.

We propose to make a standard sample preparation, together with additional flotation before feldspar separation by density to enrich K-feldspars in the sample. Furthermore one should guarantee to have a packed mono layer of sample grains for dosimetric reasons. Therefore we suggest to use sample plates as described by Erfurt et al. (2002).

The palaeodose can also be estimated graphically. However it is more precise to calculate the palaeodose using equation 1.

A comparison of ages determined by the IRSAR protocol and ages estimated using IR-OSL, U/Th and ^{14}C is presented in figure 9 for sample GOS4, GRÖ8 and HURL1. This again underlines the good agreement of IRSAR data to other dating techniques.

8. Discussion and summary

We have presented a single-aliquot regenerative-dose protocol applied to the infrared radiofluorescence (IRSAR) of coarse-grain K-feldspar. It is a robust and precise method to determine the equivalent dose connected to the last light exposure of clastic Quaternary sediments with the possibility to observe the primary dating information in terms of the electron density in the IR-RF (IR-OSL) trap. The physical and methodological

characteristics show that this new promising technique does not need special treatment of the samples (e.g. for sensitivity changes), compared to other luminescence methods (SAR on quartz and feldspar). Furthermore, the IR-RF can be measured using a very simple experimental set-up. Based on state-of-the-art knowledge of the physical background, we assume a reliable dating range from about 10 ka over up to over 250 ka. With regard to the saturation dose levels as well as the thermal stability of the IR-RF signal, there is some potential to increase the upper dating limit.

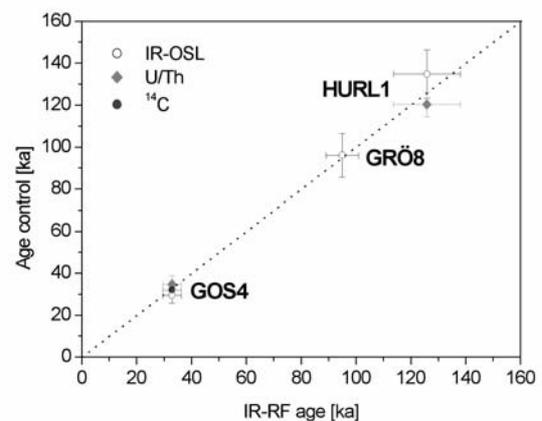


Figure 9. Comparison of IR-RF ages with IR-OSL, ^{14}C and U/Th ages (data after Trautmann et al. (1999) and Erfurt et al. (2002)) of samples GOS4, GRÖ8 and HURL1

However, much more work is needed to investigate the IR-RF more precisely, especially its behaviour at higher dose levels as well as on effects which may affect the stability of the transient equilibrium of the fluorescence occurring during interaction of the minerals with ionising radiation.

This paper should encourage the luminescence dating community to apply and to investigate this new IR-RF dating technique.

Acknowledgements

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Purchase of IR-RF instrumentation

If there are questions about the purchase of IR-RF instrumentation, please ask the corresponding author.

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Rewiever

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