

Accurate calibration of a laboratory beta particle dose rate for dating purposes

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Abstract

This paper describes a novel method for calibration of the dose rate provided by a beta source used for luminescence dating. The calibration has been performed using an in-house calibration quartz that is given a dose in a Co-60 gamma ray facility. The gamma dose to the calibration quartz was calculated using Fricke dosimetry on simultaneously irradiated Fricke solution and Monte-Carlo simulations. An overall uncertainty in the dose to quartz of 0.6% (1 sigma) was achieved. The single aliquot regenerative dose (SAR) procedure was used to determine the irradiation time of the beta source needed to deliver the same dose to the sample. Results for a range of doses were combined by plotting irradiation time versus dose. A fit through the data points yielded the dose rate provided by the beta source at the sample position. This procedure resulted in an overall uncertainty in the beta dose rate of 0.9% (1 sigma). The beta dose rate was found to be in perfect agreement with an independent calibration using irradiated quartz provided by the Nordic Laboratory for Luminescence dating.

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1. Introduction

Luminescence dating techniques require the administration of an absolutely known dose to the sample. This is usually performed using a beta particle source. It is important to calibrate the dose rate at the sample position of this source as accurately and precisely as possible since the uncertainty in dose rate may be a major contribution to the overall uncertainty of the equivalent dose. Most beta-source calibrations are undertaken by comparison (using a phosphor) with a calibrated gamma ray source. The absolute calibration of this gamma source usually forms the major uncertainty in this comparison. Standard practice is to use an ionisation chamber to derive the gamma dose to the sample. This limits the overall uncertainty in the dose to the phosphor to 2–3% (Göксу et al., 1995; Armitage and Bailey, 2005). In this research we aim to reduce the uncertainty to less than 1% using Fricke dosimetry. The Fricke ferrous sulphate dosimeter is recognized to be the most suitable chemical system available for radiation dosimetry with respect to accuracy, reproducibility and linearity of response

(Fricke and Hart, 1966; ICRU, 1992). It is relatively independent of the dose rate and of radiation quality. It is the only chemical dosimetry system that is universally accepted and may be used without reference to other standards. Using a Fricke dosimeter the beta dose rate of a standard 1.48 MBq ⁹⁰Sr/⁹⁰Y source mounted on a Risø TL-DA-15B/C reader has been calibrated using quartz distributed on stainless steel discs. The results are compared with the dose rate derived using calibrated quartz from the Nordic Laboratory for Luminescence Dating.

2. Materials and methods

Our calibration procedure consists of the following steps. First, calibration quartz and Fricke solution were simultaneously irradiated in a gamma cell. Second, the dose to the Fricke solution was determined using Fricke dosimetry. Third, the gamma dose to the calibration quartz was calculated from the Fricke dose using Monte-Carlo simulations. Fourth, the single aliquot regenerative dose (SAR) procedure was used to determine the beta irradiation time, t_β , needed to deliver the same dose to the sample. Finally results for a range of doses were combined by plotting t_β versus the dose. A fit through the data points was used to determine the dose rate provided

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by the beta source at the sample position. The various components involved in this method are described in detail below.

2.1. Calibration quartz

The starting material came from a 20 kg bag of loose sand-box sand purchased from a local chain hardware store. The 180–212 μm fraction was obtained through wet sieving using a *Retsch AS 200 basic analytical sieve shaker*. This size fraction was chosen because it is a normal grain size for dating and because there is very little dependence of the laboratory beta dose rate on grain size over this range (Armitage and Bailey, 2005). The grains were then chemically treated to obtain a pure quartz sample. Treatment consisted of 40 min in 10% HCl, 60 min with 10% H_2O_2 , washing with 10% HF and subsequent treatment in 40% HF for 30 min while continuously stirring using a magnetic stir plate. The grains were then washed with 10% HCl to remove fluorides, and rinsed three times with water to remove traces of chemicals used. Finally the grains were sieved once more to avoid any grains that were badly attacked by the HF treatment. The HF treatment and subsequent washing was repeated once more because tests with infrared stimulated luminescence indicated that the sample was contaminated with feldspars or other IR sensitive minerals.

To improve the luminescence sensitivity of the quartz grains the sample was annealed at 700 °C for 1 h, cooled to room temperature and then dosed with 1000 Gy by a ^{60}Co gamma source. Next the sample was heated twice to 500 °C for 1 h. After each 500 °C heating the grains were immediately removed from the furnace to facilitate rapid cooling.

2.2. Gamma irradiation facility

A ^{60}Co Gammacell 200 irradiator (AECL, 1970) was used for gamma irradiation of the quartz and Fricke solution. The radioactive source in this facility comprises 24 rods (active dimensions of each rod $\varnothing 0.8\text{ cm} \times 12.9\text{ cm}$, total activity of all rods 7.0 ± 0.35 (1σ) TBq on May 26, 2005) positioned in annular symmetry (pitch diameter 13.97 cm) around the irradiation chamber in the centre of a lead shielding. The cylindrical irradiation chamber has a height of 14 cm and a diameter of 9 cm. The radioactive rods around the irradiation chamber produce a uniform field in the centre of the chamber of 0.091 Gy s^{-1} (dose rate to water on May 26, 2005).

2.3. Sample holder for gamma ray irradiation

The sand is contained in a fused quartz walled cylindrical vial (inner diameter 8.0 mm and wall thickness 1.0 mm). The vial is placed in a cylindrical container made of carbon-filled polyethylene (see Fig. 1) which is placed in the centre of the irradiation chamber. The wall thickness of the polyethylene container (5.5 mm) is enough to provide charged particle equilibrium within the sample and Fricke solution. The polyethylene material has been chosen in order to minimize wall effects and reduce the dependence on calculated corrections

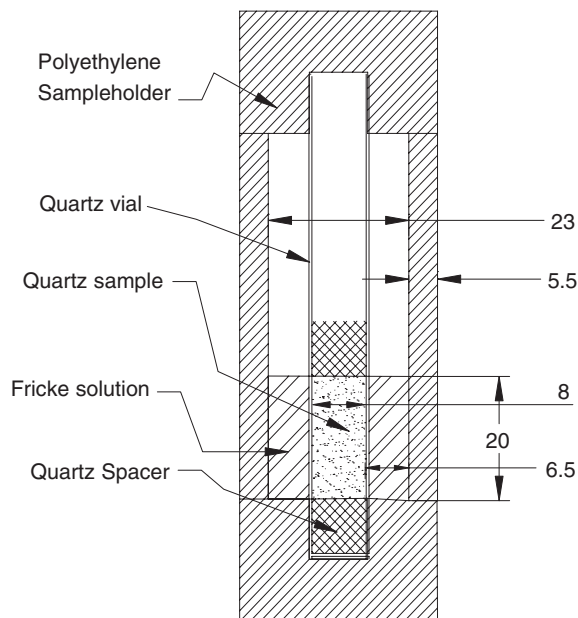


Fig. 1. Cross section of the cylindrical sample holder used to give the quartz sample a precise dose. The quartz sample (1.08 cm^3) is contained in a fused quartz vial which in turn is contained in a polyethylene holder filled with a Fricke solution (6.74 ml) up to the same height (20 mm) as the quartz sample. The sample holder is placed in the centre of a cylindrical irradiation chamber surrounded by ^{60}Co rods producing an uniform radiation field over both the Fricke solution and the quartz sample. Dimensions in mm.

(Ma et al., 1993). The polyethylene contains 2.5% carbon making the container light tight. The container is filled with an aqueous solution (6.74 ml) of ferrous ions (Fricke solution) up to the same height as the calibration quartz (see Fig. 1). This volume allows four independent measurements of the optical density (see next section). Beneath and on top of the sand sample two solid spacers (fused quartz, density 2.2 g/cm^3) are placed. The density of the sand sample itself is 1.40 g/cm^3 .

2.4. Fricke dosimetry

The dose to quartz has been derived from the dose to the Fricke solution surrounding the quartz sample. The Fricke (ferrous sulphate) dosimeter is an aqueous solution of ferrous ions (0.001 M FeSO_4 , 0.001 M NaCl and $0.8\text{ N H}_2\text{SO}_4$). Ionizing radiation converts ferrous ions, Fe^{2+} , into ferric ions, Fe^{3+} , with a radiation yield proportional to the absorbed dose. The increase in the concentration of Fe^{3+} , compared to the concentration in the unirradiated controls read on the same day, is calculated from the increase in the optical density (OD) at 303 nm measured with a spectrophotometer (Perkin Elmer Lambda 40). The absorbed dose to the Fricke solution, D_F , is

$$D_F = \frac{\Delta\text{OD}}{\varepsilon G \rho l}, \quad (1)$$

where ΔOD is the increase in OD at 303 nm, ε is the molar extinction coefficient of Fe^{3+} at 303 nm, G is the radiation chemical yield of Fe^{3+} , ρ is the density of the Fricke solution and l the length of the light path of the photometer cell.

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