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Methods to reduce sample carrier contamination for luminescence measurements

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Abstract

Equivalent doses derived from sediment measurements void of signal contamination are essential for accurate optically stimulated luminescence (OSL) dating of sediments. We identified dose-dependent luminescence signals originating from new and previously used stainless steel cups used for OSL measurements. The signal is not eliminated under typical measurement conditions for sedimentary quartz including pre-heating, infrared stimulation, and OSL stimulation at 125°C. While signals from sample carriers are typically small sources of error they may strongly contaminate sediment with low OSL sensitivities. We tested several cleaning methods on used and new stainless steel cups by measuring OSL signals after increasing irradiation doses between 0 and 100 Gy. The lowest signal from sample carriers was observed from cleaning with HF, Alconox (a detergent for silicone oil removal), and methanol. The cleaning methods that produce the lowest signal from the cups were combined to create two modified cleaning procedures that are effective in reducing the unwanted luminescence signal. Our newly modified cleaning methods are capable of reducing the luminescence signal of empty stainless steel cups to near background levels.

Introduction

Unwanted signal contributions can lead to the miscalculation of equivalent doses used for optically stimulated luminescence (OSL) ages. Recent studies have identified luminescence signals from sample carriers (Schmidt et al., 2011) and silicone oil (Vandenberghe et al., 2008) used for OSL measurements. Unlike sensitivity changes and background measurements, variable signal contamination produced by sample carriers is not accounted for in the single aliquot regenerative (SAR; Murray and Wintle, 2000) dose measurement procedure thus sample carriers pose as a source of error for sediment measurements.

Typical sample carriers used for OSL dating include stainless steel and aluminum discs and cups. Schmidt et al. (2011) observed significant dose-dependent thermal luminescence (TL) signals from clean, empty stainless steel and aluminum discs with strong TL peaks at 110 °C in ultraviolet (UV; 340 nm) and UV-blue (420 nm) detection windows. Two other peaks were observed between 150 and 300 °C in the UV range and a broad peak was observed between 150 and 300 °C in the UV-blue window with a high-temperature peak at 380 °C. Additionally, Vandenberghe (pers. comm.) identified ultra-fast decaying, thermally unstable, dose-dependent signals from stainless steel discs with a strong TL peak at 110°C with a UV (340 nm) detection window. Luminescence signals from empty sample carriers most likely arise from chemical reactions between silicone oil and sample carriers upon heating and irradiation that result in luminophores derived from defects in silica-, aluminum-, and iron-oxides (Schmidt et al., 2011). Since sample carriers are generally recycled from measurement to measurement, the luminescent film may build up with increased usage due to its impervious nature to cleaning.

Typically small sources of error have a relatively large influence on sediments with low OSL sensitivities. The goal of this study is to explore sample carriers as a possible source of contamination and devise an effective cleaning procedure in order to minimize equivalent dose distribution scatter. After determining the magnitude of contaminant signals from sample carriers, we test empty stainless steel cups under measurement conditions typically used for sedimentary quartz. Multiple cleaning methods were conducted on stainless steel cups to eliminate contaminant signals. We established modified cleaning procedures for stainless steel cups based on

the cleaning methods that produced the lowest and least variable contaminant signals. We investigate the effectiveness of our newly modified cleaning techniques by measuring equivalent doses of two Antarctic cobble surfaces with low OSL sensitivities using sample carriers cleaned prior to and after the adoption of our modified cleaning procedures.

Methods

Measurements for this study were conducted at the Radiation Dosimetry Laboratory at Oklahoma State University using a Risø TL/OSL-DA-15 Reader manufactured by Risø National Laboratory including a built-in $^{90}Sr/^{90}Y$ beta source with an internal dose rate of 100 mGy s^{-1}, and measuring OSL with blue LEDs (470 nm, 31 mW cm^{-2}), infrared stimulated luminescence (IRSL) with an IR LED array (~875 nm, 110 mW cm^{-2}), and detection in the UV band (Hoya U340, 7.5 mm, 340 nm peak; Bøtter-Jensen and Murray, 1999). A heating rate of 5°C s^{-1} in a N_2-atmosphere was used for measurement stimulation above room temperature.

Luminescence signals from sample carriers

Measurement sequence

We used stainless steel cups as sample carriers provided by Risø DTU with a diameter of 9.8 mm and thickness of 0.5 mm. Raw material for the cups is PK11NB stainless steel (Mat.No. 1.4550, DIN X6CrNiNb18-10, AISI 347) from Metal Ravne (www.metalravne.com; H. Christiansen, pers. comm.). The cups have been recycled for several years and cleaned between measurements with our standard cleaning method (termed "Ethanol" in Appendix I in the supplementary information) comprised of two 30-minute ultrasonic baths with de-ionized (DI) water and dish soap followed by individual cleaning with ethanol. We visually inspect each cup to insure all grains were removed during cleaning. All measurements on empty cups conducted in this study were not sprayed with silicone oil.

Nine cups were initially tested to determine whether empty stainless steel cups have contaminant signals by measuring OSL at room temperature and 125°C for 100 s. Each data channel during OSL measurement represents the signal emitted in 0.4 s. Further measurements were conducted on the same cups to observe the characteristics of contaminant signals under measurement conditions used for sedimentary quartz. After irradiation of 100 Gy, signals from empty cups were measured using OSL at room temperature and 125°C for 40 s. We recorded TL curves for the cups during pre-heating to 200°C followed by measurement of OSL (125°C, 40 s). IR stimulation is commonly used in SAR measurement sequences to bleach contaminant feldspars (Wallinga, 2002; Duller, 2003; Wintle and Murray, 2006) thus we test: (1) whether the cups have signal when exposed to IR stimulation (60°C, 100 s); and (2) how the signal changes when IR stimulation (60°C, 100 s) is used prior to OSL measurement (125°C, 40s).

Measurements on the same nine cups were conducted with quartz (90-212 μm) extracted from an Antarctic beach cobble surface (sample CB10-059; Simkins et al., in review). Quartz grains (n=100-200) were adhered to the cups using silicone oil and the entire cup area was covered with a monolayer of quartz. To test whether contaminant OSL signals arise during sediment measurements, we irradiated empty cups with 100 Gy then covered the cups with bleached quartz. We bleached the quartz by pre-heating at 200°C for 60 s followed by IR stimulation at 60°C for 100 s and measurement of OSL at 125°C for 200 s. OSL signals of the irradiated cups with bleached sediment cover were measured at 125°C for 40 s following pre-heating (200°C, 10 s) and IR (60°C, 100 s) stimulation. Lastly, the cups and quartz were irradiated simultaneously with 100 Gy and OSL signals were measured following the same sequence as with bleached quartz.

Results

Stainless steel cups do not produce OSL signals in the absence of prior irradiation when measured at 125°C or room temperature (not shown) following cleaning with the standard cleaning method (Appendix I). After irradiation of 100 Gy, the decay curves have fast components with background signals established by the first second of stimulation at both room temperature and 125°C (Fig. 1a). Measurement of OSL at room temperature produces higher signals than when measured at 125°C. TL curves recorded during pre-heating (up to 200°C) show a prominent peak centered at 100°C (Fig. 1b). OSL signals from empty cups are reduced to below 500 counts for all nine cups when pre-heating (200°C for 10 s) is used (Fig. 1c). Empty cups show no signals above background when stimulated with IR with the exception of two of the nine cups with signals 20-30 counts above background (Fig. 1d). OSL signals increase following IR stimulation (Figure 1c versus 1e). Contaminant signals from empty cups are not eliminated when pre-heated (200°C, 10 s) and stimulated by IR (60°C, 100 s) prior to OSL measurement (Fig. 1e).

A TL curve recorded during pre-heating is shown for irradiated cups with bleached quartz covering the surface of the cups (Fig. 1f) as well as for irradiated cups with irradiated quartz (Fig. 1g). The OSL curves for irradiated cups with bleached quartz (Fig. 1h)

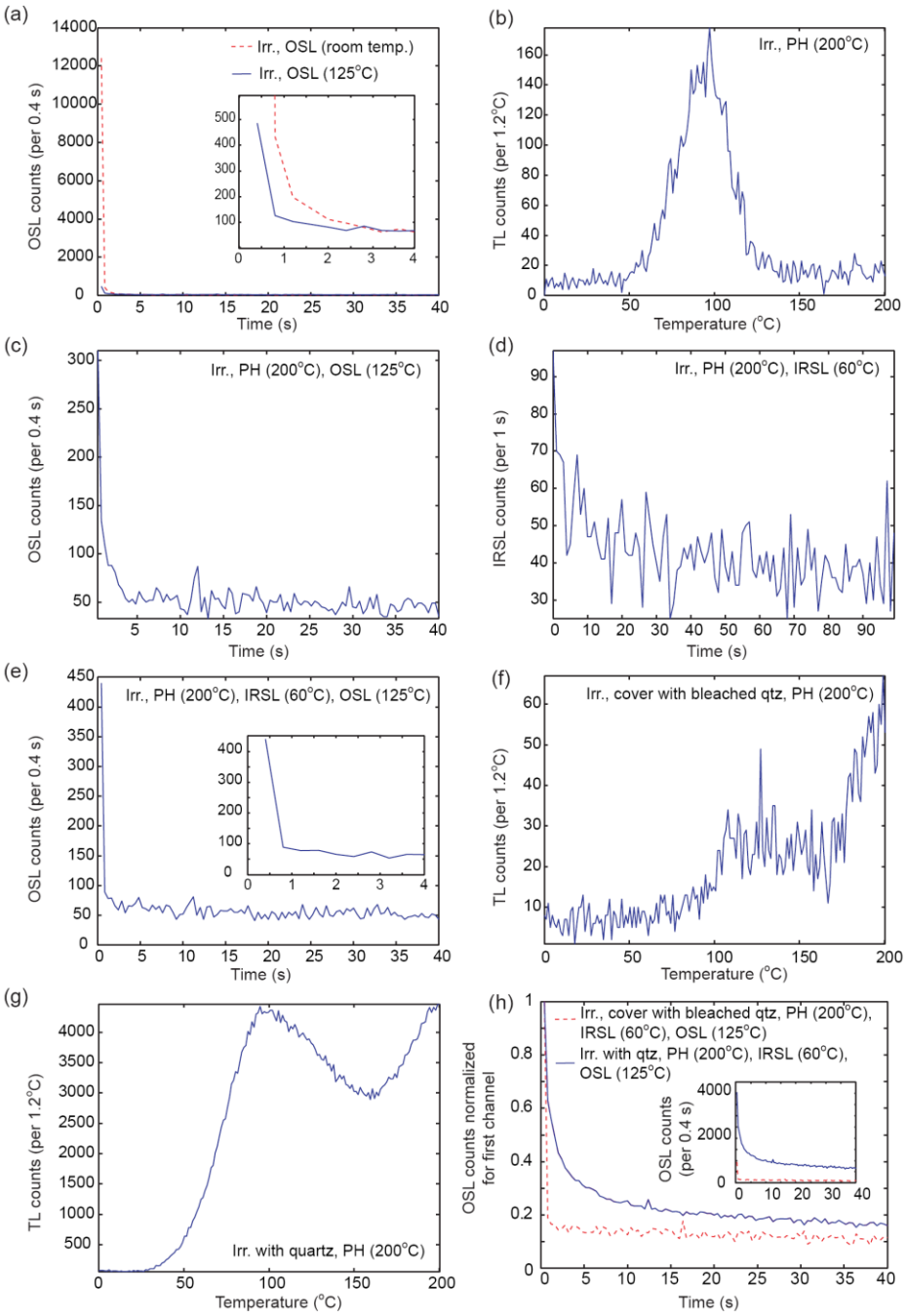

Figure 1: *Measurement of OSL signals from one representative empty cup out of the nine measured cups at (a) room temperature for 40 s (red dashed line) and 125°C for 40 s (blue line) following irradiation (irr.) of 100 Gy. (b) TL curve recorded during pre-heating (PH) to 200°C following irradiation of 100 Gy. (c) OSL curve measured at 125°C for 40 s following irradiation (100 Gy) and pre-heating (200°C, 10 s). (d) IRSL (60°C, 100 s) after irradiation (100 Gy) and pre-heating (200°C, 10 s). (e) OSL (125°C, 40 s) following irradiation (100 Gy) and pre-heat (200°C, 10 s) and IRSL (60°C, 100 s). Inset shows decay during the first 4 s of stimulation with the same y-axis as the full OSL decay curve. (f) TL curve from pre-heat (PH) (up to 200°C) using irradiated cups (100 Gy) with bleached quartz covering the cup surface. (g) TL curve recorded during pre-heat to 200°C following cup and quartz irradiation of 100 Gy. (h) Resulting OSL curves normalized for the signal in the first channel for bleached (red dashed line) and irradiated (blue line) quartz following irradiation (100 Gy), pre-heat (200°C, 10 s), and IRSL (60°C, 100 s) with an inset of the raw decay curves with the same y-axis as the normalized decay curves.*

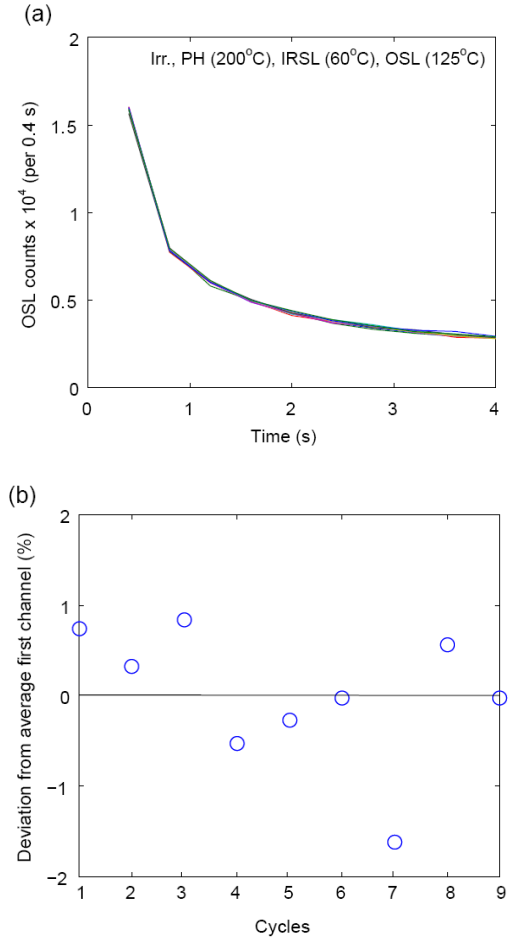

Figure 2: *(a) Decay curves for one empty stainless steel cup during the first four seconds of OSL (125°C, 40 s) for nine measurement cycles including irradiation (30 Gy), pre-heat (200°C, 10 s), and IRSL (60°C, 100 s) prior to OSL stimulation. (b) Deviation in percent from average signal in first channel for the nine measurement cycles. The line at 0 along the y-axis represents the average signal.*

shows fast decay as observed for all empty cup measurements. OSL decay curves resulting from irradiated cups with irradiated quartz (Fig. 1h) show a slower component not observed from empty cups or cups covered with bleached quartz.

Sample carrier sensitivity test

Measurement sequence

Additional measurements on one empty cup were conducted to assess whether empty cups experience sensitivity changes during measurement sequences. We used nine repeated cycles of irradiation of 30 Gy followed by pre-heat (200°C, 10 s), IR stimulation (60°C, 100 s), and OSL measurement (125°C, 40 s).

Results

OSL signals measured for the repeated cycles show less than 2 % deviation from the average OSL signal from the first channel of the nine cycles (Fig. 2). We suggest signals from stainless steel cups do not change sensitivity with repeated measurement cycles.

Cleaning procedures

Measurement sequence

Several cleaning techniques were applied to empty cups in addition to the standard cleaning method (Appendix I). Following two ultrasonic baths with dish soap and DI water, cleaning was performed with acetone, methanol, sand paper, and hydrofluoric acid (HF). In addition, Alconox (Powdered Precision Cleaner, www.alconox.com), a detergent for ultrasonic removal of silicone oils, and Rust Stain Remover (manufactured by Whink), containing approximately 3% HF, were tested as substitutes for dish soap in the ultrasonic baths. For the use of Alconox, we used an approximate mixing ratio of 10 g of Alconox per 1 liter of DI water. The ultrasonic baths with Rust Stain Remover contained 1 part Rust Stain Remover to 3 parts DI water. Each cleaning method was conducted on five randomly selected empty cups that have been previously used for sediment measurements. New (i.e. unused) cups were also tested for luminescence signals including five new cups with no cleaning treatment and five new cups cleaned with Alconox.

The higher the signal the better the resolution at which we can test the cleaning methods. For this reason, measurements of individual cleaning techniques and modified cleaning procedures were conducted with OSL stimulation at room temperature (see Fig. 1a). In order to determine the dose-dependency of the cups using various cleaning procedures, OSL signals were measured for 100 s at room temperature after irradiation of 0, 1, 10, 50, and 100 Gy. The reported signals are the sum of the first two seconds of stimulation. The slow component was removed by subtraction of the background signal (last 10 s). The cleaning methods that produced the lowest, least variable signals from the cups were combined to create modified cleaning procedures. Modified cleaning procedures were applied to the cups and tested by measuring OSL signals at room temperature for 100 s after irradiation of 100 Gy.

Results

Irrespective of the cleaning method, OSL signals were not observed for stainless steel cups that were not irradiated prior to OSL stimulation (not shown). Results from irradiated cups cleaned with various treatments are summarized in Fig. 3. Dose response curves (Appendix II) for cups cleaned with each

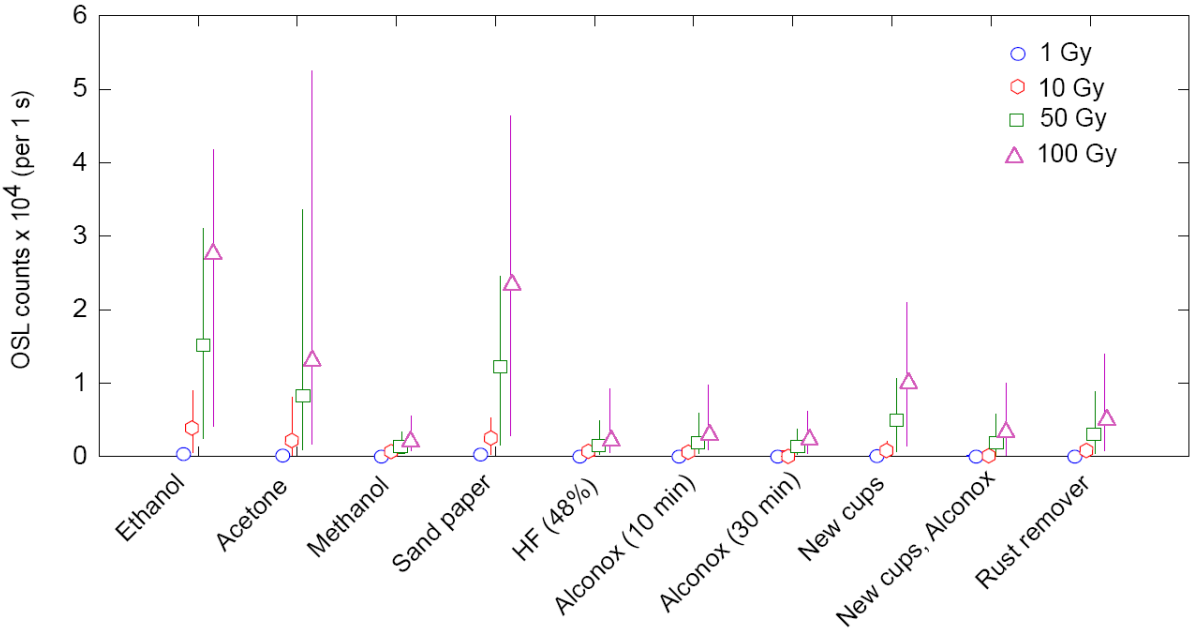

Figure 3: *OSL signals (measured at room temperature for 100 s) from five empty stainless steel cups cleaned with the cleaning methods (see Appendix I for details). The cleaning methods are listed along the x-axis. The signals include the integral from the first two channels. The error bars represent the range of signals from the five empty cups cleaned with the same method and irradiated with the same dose while the points represent the averages. OSL measurements were conducted after irradiation doses of 1, 10, 50, and 100 Gy. 1 Gy – blue circles; 10 Gy – red hexagons; 50 Gy – green squares; 100 Gy – magenta triangles.*

individual cleaning technique show, with a few exceptions, a linear trend in signal growth with increased irradiation dose. Even after 1 Gy, empty cups cleaned with ethanol, acetone, and sand paper produced luminescence signals above background (> 100 counts). After irradiation of 100 Gy, cups have OSL signals up to 53000 counts. Cleaning with ethanol (standard cleaning method), acetone, and sand paper produced the highest, most variable signals suggesting these methods are least effective in removing the luminescence signal of the cups. The use of methanol, HF (48%), Alconox, and Rust Stain Remover (3 % HF) produced the lowest and least variable OSL signals.

A combination of the most effective methods was adopted to create two modified cleaning procedures for stainless steel cups (Table 1). Method (1) consists of ultrasonic baths with Alconox, etching with 48% HF for 5 minutes, and individual scrubbing with methanol. Method (2) is similar to method (1) with the exception of using Rust Stain Remover instead of 48% HF. A total of 240 and 130 cups were cleaned with cleaning methods (1) and (2), respectively. The cleaning methods were tested on cups using irradiation of 100 Gy followed by OSL at 125°C for 100 s. The majority of cups (61%) cleaned with method (1) have OSL signals between 100-500 counts with only 2 % of the cups with signals < 100 counts. The majority of cups (89%) cleaned using method (2) have signals less than < 500 counts with a larger percentage (41%) of cups with signals below 100 counts.

Impact of contamination on equivalent doses

Measurement sequence

OSL measurements from two quartz isolates (90-212 µm; 100-200 grains) extracted from buried Antarctic cobble surfaces were measured using stainless steel cups cleaned with our standard cleaning method (Appendix I). See Simms et al. (2011) for details on sediment sample preparation. Equivalent doses were determined following the SAR measurement protocol (Murray and Wintle, 2000) using the same laboratory facilities and stimulation and detection windows mentioned in the methods section. We used a pre-heat of 200°C for 10 s, a cut-heat of 180°C for 10 s, IR stimulation at 60°C for ≤ 100 s, and OSL was measured at 125°C for 100 s. Each SAR cycle was followed by measurement of OSL at high-temperature (240°C, 10 s; Murray and Wintle, 2003). The first two channels of the OSL signal curve were used as the signal for equivalent dose calculations. The slow component of the OSL curve was removed by subtraction of the background signal (last 10 s). We consider passing aliquots to have recycling ratios < 20 %, recuperation tests with

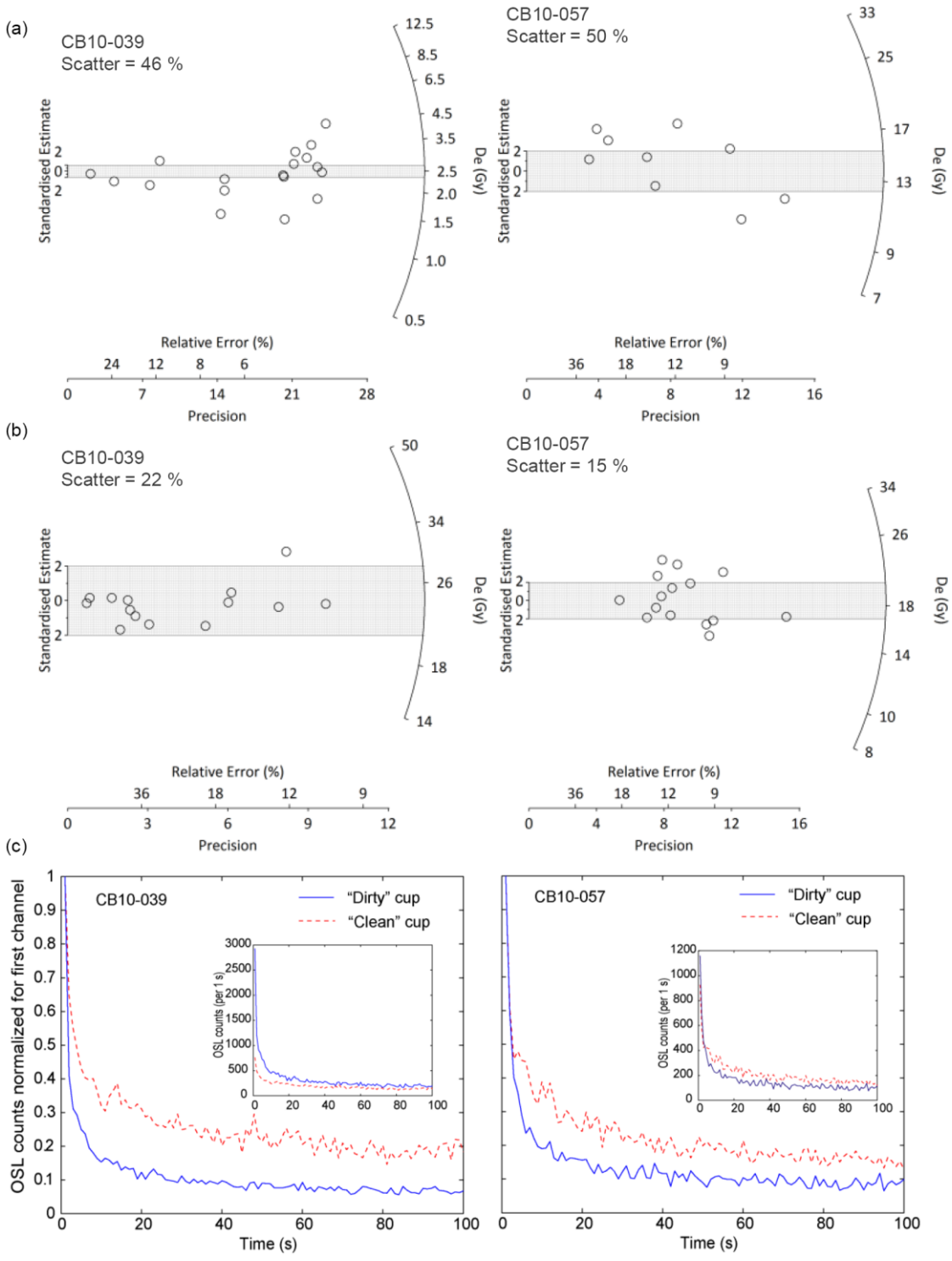

Figure 4: *(a) Radial plots showing equivalent doses distributions from two quartz samples (CB10-039 and -057) from Antarctica cobble surfaces derived from measurements made using stainless steel cups cleaned with the standard cleaning method. (b) Re-measured equivalent doses distributions from the same samples using neutral-signal cups cleaned with modified cleaning method (2). (c) OSL decay curves normalized for the signal in the first channel for samples CB10-039 and CB10-057 after irradiation of 30 and 50 Gy, respectively, using cups with contaminant signals (blue line) and neutral signals (red dashed line). The inset shows the raw OSL decay curves with the same x-axes as the normalized decay curves.*

Cleaning method (1) - Alconox, HF, and methanol

Step 1: Rinse with DI water
Step 2: 30-min. ultrasonic bath (Alconox at 70°C)
Step 3: Rinse with DI water
Step 4: Etch with 48% HF for 5 minutes
Step 5: Rinse with DI water
Step 6: 30-min. ultrasonic bath (Alconox at 70°C)
Step 7: Rinse with DI water
Step 8: Scrub each cup with methanol using a cotton swab

Cleaning method (2) - Alconox, Rust Stain Remover, and methanol

Step 1: Rinse with DI water
Step 2: 1-hr ultrasonic bath (Alconox at room temp)
Step 3: Rinse with DI water
Step 4: 1-hr ultrasonic bath (Rust Stain Remover at room temp)
Step 5: Rinse with DI water
Step 6: 1-hr ultrasonic bath (Alconox at room temp)
Step 7: Rinse with DI water
Step 8: Scrub each cup with methanol using a cotton swab

Table 1: *Modified sample carrier cleaning methods*

values < 10 %, and dose recovery ratios < 25 %. The dose recovery test differs from the dose recovery test suggested by Wintle and Murray (2006) which refers to the recovery of a dose that was administered to unheated bleached aliquots by giving a dose between regenerative cycles 2 and 3 using thermal treatments as for the regenerative cycles. Statistical modeling following the common age model (Galbraith et al., 1999) yielding a weighted log paleodose was used to determine the most representative equivalent dose from dose distributions. We tested the effectiveness of our modified cleaning methods by re-measuring equivalent doses of the same sediment samples using cups without contaminant signals. The measurement sequence for re-measured samples was identical to the sequence used for equivalent doses from measurements using contaminant-signal cups.

Results

We observe large intra-sample scatter as evidenced in radial plots of equivalent dose distributions derived from quartz extracted from buried Antarctic cobble surfaces using cups cleaned with the standard cleaning method (Fig. 4a). The scatter is quantified by dividing the standard deviation of all passing aliquots by the simple average of equivalent doses and is shown as "Scatter" in the top left of the radial plots. Dose distributions of re-measured samples using cups cleaned with modified cleaning method (2) with Rust Stain Remover show the majority of aliquots plotting within two standard deviations of the equivalent dose estimate from the age model (Fig. 4b). The re-measured equivalent doses show less scatter than equivalent doses derived from measurements with cups with contaminant signals. Equivalent doses for the two samples after the adoption of our modified cleaning procedure are higher than for measurements using cups with contaminant signals (Table 2). OSL signals measured after irradiation of 30 and 50 Gy for CB10-039 and CB10-057, respectively, show faster decay curves when quartz is measured on cups with contaminant signals (Fig. 4c).

Sample	"Dirty" cups D_e (Gy)	"Clean" cups D_e (Gy)
CB10-039	2.46 ± 0.03	24 ± 1
CB10-057	13.7 ± 0.5	24.5 ± 0.5

Table 2: *Equivalent doses (D_e) of Antarctic cobble surfaces*

Discussion

Luminescence signals from sample carriers

We observe that both new and previously used empty stainless steel cups produce dose-dependent signals when measured at 125°C as well as at room temperature. Fig. 1 summarizes the characteristics of contaminant signals from stainless steel cups. Contaminant signals from empty cups are not eliminated by the thermal pre-treatment (pre-heat for 10s at 200°C) or the prior exposure to IR stimulation (60°C, 100 s). Covering the surface of the cups with bleached quartz does not mask the contaminant signals. On the contrary, despite ensuring the quartz used was bleached, OSL signals increase with cups covered with bleached quartz using the same sequence with pre-heat and IR stimulation (Fig. 1e and 1h). Sensitivity changes could potentially explain the higher signals; however, considering sensitivity changes are not observed for empty cups (Fig. 2), the higher signals cannot be attributed to sensitivity changes during measurement sequences. The fast nature observed from empty cups and cups covered with bleached quartz is not observed under typical sediment measurement conditions with irradiated quartz most likely due to the combination of slower OSL decay from the quartz with fast decay from the cups themselves (Fig. 1h). We show that contaminant signals from stainless steel cups used as sample carriers are: (1) impervious to our standard cleaning

methods, (2) not eliminated using typical measurement conditions for sedimentary quartz (e.g. thermal preheating, IR stimulation and elevated temperature OSL measurement; and (3) not masked by sediment covering the cups. These findings are in support of the work of Schmidt et al. (2011) who suggest the presence of contaminant signals from sample carriers.

Cleaning methods

Previously used and new cups have contaminant signals when cleaned with multiple techniques; however, signals were not observed without prior irradiation. The signals observed following various cleaning methods (Fig. 3) suggests the contaminant signal from stainless steel cups is variable and dose dependent. Considering sensitivity changes were not observed for empty cups, we attribute the growth in signal magnitude with increasing irradiation dose to the dose-dependency of the contaminant signals.

Signals produced by new stainless steel cups most likely result from silicone oil used in manufacturing of the cups. Luminescence signals from previously used cups most likely arise from reactions that take place upon heating and irradiation of previously used cups resulting in luminophores derived from defects in silica-, aluminum-, and iron-oxides (Schmidt et al., 2011). We find that our standard cleaning method does not sufficiently remove OSL signals from new and previously used cups. However, we identify the most effective cleaning methods for stainless steel cups including methanol, Alconox, HF (48 %), and Rust Stain Remover (3 % HF).

Modified cleaning procedures (Table 1) minimize luminescence signals produced by stainless steel cups. Both cleaning procedures use HF in different concentrations. HF is well known for its ability to dissolve iron- and silica- oxides and is commonly used to remove impurities from stainless steel. Of the two cleaning methods, method (2) is more effective at reducing the signal to less than 100 counts or otherwise close to background signals (Fig. 3). Additionally, although HF is present in Rust Stain Remover, cleaning method (2) eliminates the use of strong concentrations of HF.

We advise that following the first use of either modified cleaning procedure, cups should be tested for luminescence signals following irradiation of 100 Gy. Cups that produce signals above 100 counts should be discarded or re-cleaned and re-tested prior to use for equivalent dose measurement of dim quartz. However, for brighter quartz, contaminant signals from stainless steel cups > 100 counts may be acceptable. After cups are initially cleaned and used in sample measurements, we conduct routine tests on the cups after each cleaning treatment by selecting five clean cups at random to verify that the cups have signals less than 100 counts.

Impact of contamination on equivalent doses

Luminescence signals from stainless steel cups contaminate OSL measurements of sedimentary quartz (Figs. 1 & 4). Considering signals from cups are dose-dependent, natural OSL signals from sediment for dating purposes are not contaminated by sample carriers; however, the dose response curve results from a combination of signals from sediments and stainless steel cups. The resulting higher equivalent doses of sediment samples (Table 2) using cups with no signal suggest that the dose-dependent luminescence signals from stainless steel cups previously dominated the dose response curve without contributing to the natural signal from dated samples. When using cups with contaminant signals, we suggest sediment natural signals appear relatively small compared to regenerated dose responses thus equivalent dose estimates underestimate true OSL ages. Our modified cleaning methods allow for more robust sample measurements and equivalent dose estimates for Antarctic cobble surfaces with low OSL sensitivities.

Conclusions

We report variable, dose-dependent luminescence signals from new and previously used empty stainless steel cups used as sample carriers for OSL measurements. Contaminant signals are reduced but not eliminated using pre-heat (200°C, 10 s) and IR (60°C, 100 s) stimulation. Signals generated by the cups are not masked by sediment cover. The remaining contaminate signal from stainless steel cups introduce a source of error for OSL measurements of sedimentary quartz. Our standard cleaning method is not effective in removing the luminescence signal from stainless steel cups. Two modified cleaning methods combine the use of Alconox, HF (48% and 3%), and methanol in order to reduce unwanted signals from new and used stainless steel cups. After cleaning cups using our modified cleaning methods, much of the dose distribution scatter initially observed for Antarctic cobble surfaces is resolved. The application of these cleaning methods to stainless steel cups used as sample carriers reduces signal contamination of low OSL sensitivity quartz where typically small sources of error can produce relatively large measurement uncertainty. Our modified cleaning methods may potentially be useful for other applications of luminescence methods to produce more robust measurements void of unwanted contamination from sample carriers.

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Supplementary information for this article is available at the Ancient TL web site (www.aber.ac.uk/ancient-tl).

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Reviewer

D.A.G. Vandenberghe

Editor's comment

This is the latest in a series of papers in Ancient TL dealing with unwanted signals during luminescence measurements, and they highlight the need for care. The practise of checking sample holders prior to measurement is a good one. Although the formation of phosphors by chemical reactions with the substrate of the sample carrier is a concern, in my experience the most common problem that colleagues find is that they have not completely removed all the quartz and/or feldspar from their carriers, and that it is this which may provide a problem.