doi.org/10.15392/2319-0612.2024.2568
2024, 12(4) | 01-17 | e2568
Submitted: 2024-07-19
Accepted: 2024-10-23

Optically stimulated luminescence in quartz pellets under blue and green illumination

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Abstract: In this study, pellets fabricated from powdered rose quartz are investigated for their potential as ionizing radiation detectors, taking advantage of the abundant availability of quartz in the Earth's crust. For this purpose, optically stimulated luminescence (OSL) technique, using blue and green light stimulation, was used. The pellets were irradiated with a ^{90}Sr/^{90}Y beta source to doses from 80 to 550 mGy (dose rate = 0.08Gy/s). Analysis of the OSL decay curves showed a good fit with 3 exponential components (fast, medium, and slow). A linear dose-response relationship was observed within this interval of dose for both light stimulations. The repeatability of the OSL signal was found to be 3% for blue light and 2% for green light. Additionally, the fading experiment indicated that, after 1 hour, 41% and 35% of the OSL signal remains for blue and green stimulation, respectively. The results obtained highlight the promising usefulness of rose quartz-based pellets as effective detectors of ionizing radiation.

Keywords: rose quartz, blue-OSL, green-OSL, dosimetry.

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Submitted: 2024-07-19
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Luminescência opticamente estimulada em pastilhas de quartzo utilizando iluminação azul e verde

Resumo: Neste estudo, foi investigado o potencial de pastilhas fabricadas a partir de pó de quartzo rosa como detectores de radiação ionizante, considerando a abundante disponibilidade de quartzo na crosta terrestre. Para isso, foi utilizada a técnica de luminescência opticamente estimulada (OSL), utilizando estimulação de luz azul e verde. As pastilhas foram irradiadas por uma fonte beta de 90Sr/90Y em um intervalo de doses de 80 a 550 mGy (taxa de dose = 0,08Gy/s). A análise das curvas de decaimento OSL mostrou um bom ajuste para 3 componentes exponenciais (rápido, médio e lento). Foi observado uma dose-resposta linear dentro deste intervalo de dose para ambas as estimulações luminosas. A repetibilidade do sinal OSL foi de 3% para luz azul e 2% para luz verde. Além disso, a medida de desvanecimento indicou que, após 1 hora, 41% e 35% do sinal OSL permaneceram para a estimulação azul e verde, respectivamente. Os resultados obtidos destacam a utilidade promissora das pastilhas à base de quartzo rosa como detectores eficazes de radiação ionizante.

Palavras-chave: quartzo rosa, azul-OSL, verde-OSL, dosimetria.

1. INTRODUCTION

Quartz exhibits a chemical composition consisting silicon dioxide (SiO_2), and is one of the most important silica polymorphs in natural geological formations, generally occurring in igneous, metamorphic and sedimentary rocks [1,2]. The α-quartz represents the crystalline structure commonly observed in nature [3,4]. In this configuration, the structure is characterized by a hexagonal crystalline system, with its unit cell formed of three silicon atoms and six oxygen atoms. The bonds between Si^{4+} and O^{2-} generate a tetrahedral polyhedron, with silicon positioned at the center and oxygen at the vertices of the structure [3]. In terms of global reserves of large crystals, Brazil has the highest occurrence, accounting for approximately 95% of production, followed by Madagascar, Namibia, China, South Africa, Canada and Venezuela [5].

Throughout the process of crystallization, various physical, chemical, and thermal mechanisms can determine the concentration and characteristics of defects and impurities present in quartz [6]. Defects in the quartz structure can lead to luminescence and coloration phenomena [4,7,8]. Among the impurities that frequently replace Si^{4+} in the quartz structure are $Al^{3+,}$ Ga^{3+}, Fe^{3+}, B^{3+}, Ge^{4+}, Ti^{4+}, and P^{5+} [9]. While the precise cause of rose quartz coloration remains somewhat indeterminate, study have suggested the inclusion of dumortierite [4].

To exhibit luminescence, the sample must possess specific defects within its crystalline lattice [10]. Quartz, a mineral found globally, is formed under diverse geological conditions, which results in a wide range of defect types and concentrations depending on its origin [11]. This variability is a challenge for the use of quartz in TL and OSL dosimetry, as different samples can exhibit distinct luminescence responses due to variations in the nature and abundance of these defects. A chemical analysis of the sample should be performed to correctly interpret the outcomes.

Some characteristics of quartz allow it to be used to evaluate doses of ionizing radiation; luminescence is one of these features. In dosimetry, it can be used for archaeological and paleontological dating, nuclear accident dosimetry, and other applications [11]. Ionizing radiation releases charges in the mineral, and a fraction of these charges become trapped in suitable traps for long time, giving rise to luminescence after some stimulus. The dosimetric characteristics of quartz can be measured using techniques such as thermoluminescence (TL) and optically stimulated luminescence (OSL) [12]. Thermoluminescence manifests when a material, post-irradiation, emits light upon heating [13]. Analogously, Optically Stimulated Luminescence (OSL) occurs when a previously irradiated material emits light upon stimulation by light [7]. Typically, the OSL signal obtained under light stimulation at constant power (*continuous wave* - CW-OSL) is observed as a decay curve, *i.e.*, luminescence gradually diminishes over time, reflecting the finite number of trapped charges [14]. The use of the OSL technique offers several advantages over TL. Firstly, it can be carried out at room temperature, which avoids the reduction of luminescence efficiency [15]. Additionally, this technique allows for the repeated measurement of the OSL signal in a sample, providing enhanced flexibility and reliability in dosimetric analyses [16].

Currently, the most prevalent OSL detectors are synthetic, carbon-doped aluminum oxide (Al_2O_3:C) and beryllium oxide (BeO)[16]. However, natural quartz (SiO_2) has been receiving attention in luminescence dosimetry because quartz presents a cost-effective alternative to synthetic materials due to its abundance [17–22]. This way, this research promotes a deeper understanding of the physical processes associated with the OSL signal of rose quartz, using mathematical deconvolution of the curves, and evaluating the OSL signal using different wavelengths of stimulus. In fact, the study of CW-OSL at different stimulation wavelengths is important to comprehensively evaluate quartz potential as a radiation detector.

2. MATERIALS AND METHODS

2.1. Pellets

In this work, a natural sample of rose quartz originated from Minas Gerais, Brazil, (Fig. 1a) was used. The sample was pulverized resulting in particle sizes below 75 µm (Fig. 1b). The sample was heated at 500 ºC for 1h to remove any residual signal. The selected powder was mixed with Durabond 950 binder, known for its resistance to high temperatures, rigidity, resistance to thermal shock, and oxidizing atmospheres, among other properties, without showing luminescence. For pellet fabrication, a ratio of 0.310g of rose quartz powder was combined with 0.090g of binder and divided into five pallets with $8.16 \times 10^{-2} \pm 6.89 \times 10^{-3}$ g of mass and thickness 2.00 ± 0.5 mm. The mixture was subsequently compressed using a 3-ton hydraulic press (model SchwingSiwa NID 15T/130) to form the pellets. The pellets were dried at 100 ºC for 1 hour in an oven with circulating air (FABBE, Brazil, mod. 119).

Figure 1: (a) natural crystal and (b) pellet samples.

2.2. X-ray fluorescence

The sample was pulverized employing particle sizes below 75 µm. The elements present in the sample were determined by X-ray fluorescence spectrometry using a Malvern Panalytical XRF spectrometer (model Zetium) in standardless calibration STD-1.

2.3. Optically Stimulated Luminescence

Continuous wave optically stimulated luminescence (CW-OSL) measurements were performed employing the commercial Risø reader (model DA-20). Blue light (470nm, 90mW/cm^2) and green light (525nm, 42mW/cm^2) emitting diodes were utilized for stimulation. Samples were irradiated using a $^{90}Sr/^{90}Y$ beta source (dose rate 0.08 Gy/s) in a dose range of 80-550 mGy (the minimum achievable dose is obtained with 1 s irradiation). Luminescence signal was detected using an ET PDM9107-CP-TTL photomultiplier tube through a pair of Hoya U-340 filters with a combined thickness of 7.5 mm. These filters offer an effective transmission band of 250–390 nm full width at half maximum (FWHM). The OSL curves were separated into components for both stimulation LEDs.

3. RESULTS AND DISCUSSIONS

The results of the chemical analysis by XRF (Table 1) are presented in percentage (%). Al_2O_3, Fe_2O_3, and Co_3O_4 were identified as the major components in the sample. Generally [4], the most common cations involved in the Si^{4+} substitution process in the quartz crystal structure are Al^{3+}, Ga^{3+}, Fe^{3+}, Ge^{4+}, Ti^{4+}, and P^{5+}.

Table 1: XRF results of rose quartz sample.

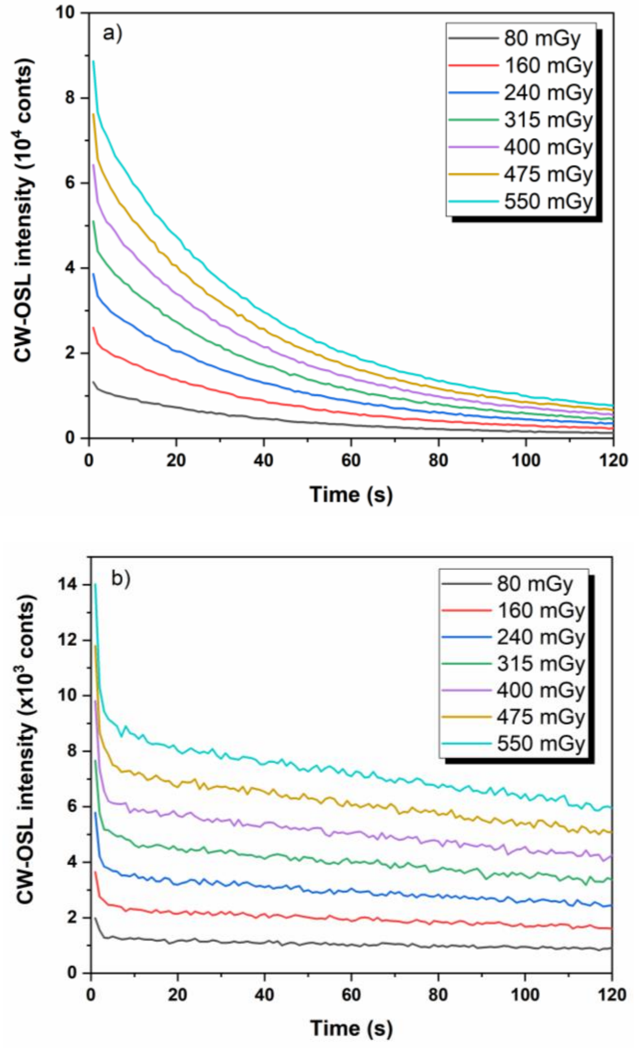
Elements	SiO_2	Co_3O_4	Al_2O_3	CaO	Fe_2O_3	Cl	P_2O_5	NiO	ZnO	ZrO
%	99.6	0.16	0.06	0.03	0.02	0.02	0.01	<0.01	<0.01	<0.01

According to the literature [11], OSL in quartz occurs due to defects related to the $[AlO_4]^-$ and [X/M+]+ centers. The $[AlO_4]^-$ defect is a substitutional center that forms when Si^{4+} is replaced by Al^{3+} in the quartz crystal lattice. To maintain charge neutrality, the $[AlO_4]^-$ center must associate with positive defects, such as (i) alkali ions (M^+), (ii) hydrogen ions (H+), or (iii) a hole (h). The $[X/M^+]^+$ defect is an interstitial ionic center, where impurity X is stabilized by an interstitial alkali ion (M^+). In this case, the X atom can be Ti, that binds to alkali ions (M^+) to enhance the stability.

The OSL characteristics of rose quartz were investigated for blue and green stimulation light, and the results are shown in Fig.2. It is possible to observe that the OSL intensity for blue light was much larger than for green light. Therefore, with the blue stimulus a higher rate of de-trapping is being caused in relation to the green stimulus, and this phenomenon may be connected to the higher power of the blue LED setup and the higher energy of the blue light, compared with the green stimulation. Accordingly, the OSL background signal with blue light is larger than that with green light.

It is important to study the sensitivity of the sample to ionization radiation, to understand better the behavior of dose-response curve. Therefore, the luminescence signal of rose quartz was investigated in the range of 80 to 550 mGy. The results showed that the OSL decay curve shape is independent of the dose, in the range used in this study. Additionally, it was also observed that the decay times, as expected, are different for the different wavelengths, with the OSL signal under blue light exhibiting significant decay up to 80s of illumination for all doses, whereas under green light, this decay decreased after 100s. Furthermore, a correlation between the dose and the signal OSL was noted, indicating that a higher dose corresponds to a higher OSL signal.

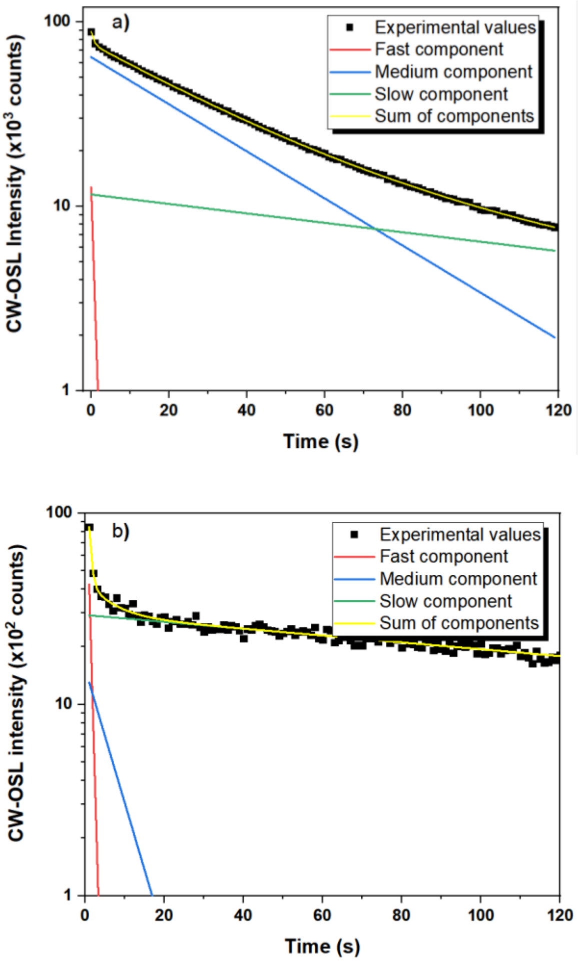
Figure 2: OSL decay for rose quartz pellets using (a) blue and (b) green light stimulation.



The quartz CW-OSL curve is characterized as the sum of multiple exponentials [14,23]. According to the literature [24–26], it is known that the OSL decay curve of quartz can be deconvolved into two or more distinct components. Fig.3 illustrates the fitting of the CW-OSL decay curve following a beta dose of 550 mGy, using blue and green light stimulation. This observation highlights that the quartz sample is best characterized by three components, regardless of the wavelength of stimulus (blue and green). For the mathematical analysis, the following equation was used:

$$I_{cw}(t) \approx \frac{dn}{dt} = \sum_{i} n_i \alpha_i \, exp^{(-\alpha i t)} \qquad (1)$$

Figure 3: Deconvolution of the CW-OSL curve of rose quartz previously irradiated with beta dose of 550 mGy for the (a) blue and (b) green light stimulation. The dots represent the experimental results, and the yellow line is the sum of all components.



Based on the deconvolution analysis in Fig. 3, the following classification for both wavelengths can be established: the red line represents the fast component, the green line corresponds to the medium component, and the blue line corresponds to the slow component. For better classification, it was used the relationship $\tau = 1/\alpha$, as illustrated in Table 2.

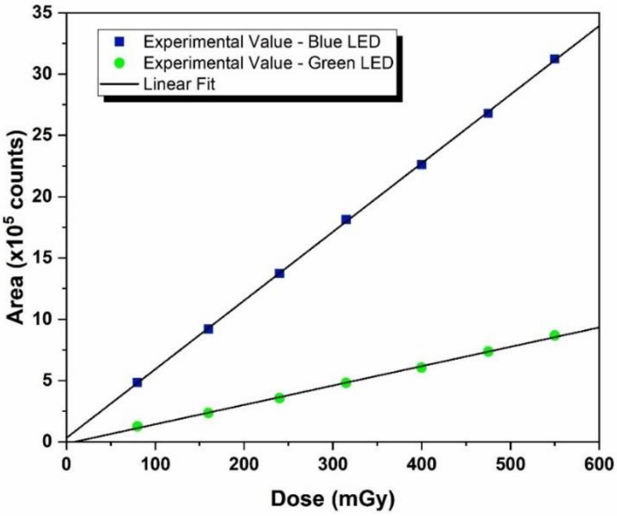
Table 2: Integral of CW-OSL components for blue and green LED.

Component	τ of blue stimulus (s)	τ of green stimulus (s)
Fast	0.68	0.61
Medium	34.00	6.21
Slow	169.64	242.68

In the case of the green stimulus, the results showed that the signal is predominantly influenced by the slow component, although the medium and fast components contribute with a small percentage to the initial signal. For the blue stimulus, both the slow and medium components significantly contribute to the overall OSL signal measured up to 120 s. Additionally, it was observed that the sum of the components (yellow curve) closely reproduces the experimental values, indicating a good fit for both light wavelengths. The deconvolution yielded a correlation coefficient of $R^2 = 0.99$ for blue light and $R^2 = 0.98$ for green light.

To examine the CW-OSL dose response, the area under the CW-OSL curves was calculated, and presented in Fig. 4, as a function of dose. The rose quartz pellets exhibited a linear dose response, with a coefficient of determination of $R^2 = 0.99$ for both stimuli. The graphs were adjusted without removing the background because it is not a significant part of the signal. This result was highly satisfactory, as a coefficient of variation closer to 1 indicates minimal variation between the OSL signal and the radiation dose. In dosimetry applications, a material exhibiting a linear dose response is desirable, as it obviates the necessity for non-linearity calibration [27]. This property of quartz makes it suitable for use as a dosimeter [11,16,28].

Figure 4: Dose response of CW-OSL using (a) blue and (b) green LEDs.



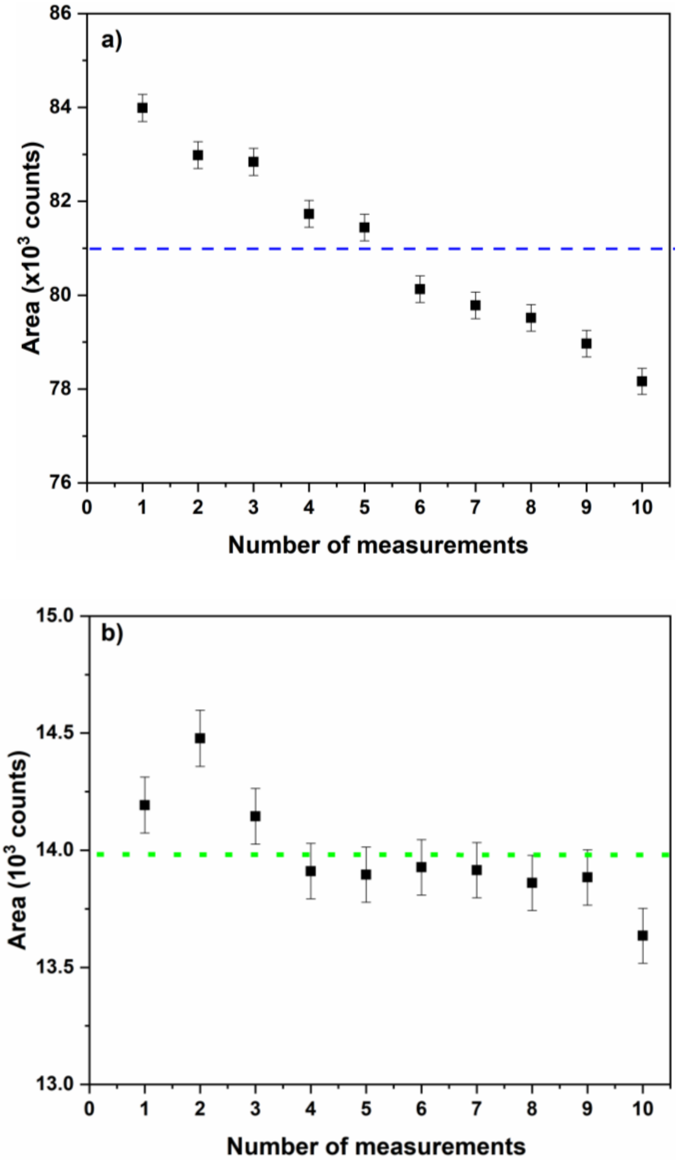
An important parameter is the measurement of the lowest detected dose (LDD) of the sample. To determine this, Equation 1 was used [29,30]:

$$LDD = (\bar{B} + (3\sigma_b))f_b \tag{2}$$

where $\bar{B}$ represents the OSL background signal of the non-irradiated samples, σ_b is the standard deviation of the measurements from the non-irradiated samples, and f_b is the calibration factor, obtained as the inverse of the slope of the total dose-response curve. The calculated value in this work was 1 mGy.

Another critical aspect in dosimetry is the repeatability and reproducibility of the luminescent signal [27]. Because of this, the repeatability of blue and green light was investigated. In these measurements, 10 irradiations and 10 consecutive readings were performed for the same sample, irradiated each time to a dose of 550 mGy. The results are shown in Fig. 5.

Figure 5: Repeatability of a rose quartz pallets using (a) blue and (b) green LEDs. The dotted line represents the average of the values obtained with 10 CW-OSL measurements.



To verify the repeatability of the sample, the coefficient of variation (C.V.) was used, which accesses the variability of a set of data in percentages. The C.V. is the ratio between the standard deviation and the arithmetic mean of the collected data and can be defined using equation 2. The CW-OSL results for blue and green light yielded a C.V. of ~3% and ~2%, respectively. Although the C.V. results are less than 5%, it is possible to notice that there is a tendency of a decreasing signal as the pellet is reused, that is more pronounced for the blue light stimulus.

$$\mathrm{C.V.} = \frac{\sigma}{m}\ 100\% \qquad (2)$$

Another important characteristic of a luminescent material is the ability to show the same signal that would be got immediately after irradiation, if the irradiated sample is stored for an extended period [27]. The fading of the CW-OSL signal for both stimuli was investigated by irradiating the samples to a dose of 550 mGy. For this purpose, three measurements were performed: one immediately after irradiation, a second one after a pause of 1800s after irradiation, and the third one after a pause of 3600s after irradiation. The results revealed that after 30 minutes, 60% and 56% of the total CW-OSL signal persisted for the blue and green stimuli, respectively. After 1 hour, these values remained at 41% and 35% of the total CW-OSL immediate signal for blue and green LEDs, respectively.

4. CONCLUSIONS

This study investigated the optically stimulated luminescence (OSL) of rose quartz pellets using blue and green stimuli. The pellets were produced in a proportion of 78% quartz and 22% Durabond 950 binder. The results indicated that the most intense signal was obtained with blue light, which is consistent with the expected outcome, given that blue light has a shorter wavelength and higher photon energy. This characteristic results in enhanced efficiency in stimulating the luminescent centers within the rose quartz sample, thereby optimizing its use as an OSL dosimeter. The OSL decay curve of the rose quartz sample demonstrated a satisfactory fit with three components (fast, medium and slow), with R^2 value of 0.99 for blue LEDs and 0.98 for green LEDs. Furthermore, the dose-response relationship exhibited linearity in the investigated dose range (80 – 550 mGy) for both stimuli. The repeatability analysis indicated that the OSL signal from the pellets presented C.V. of 3% and 2% using blue and green light stimulation, respectively, although a decrease in signal was observed with the sample reuse. Additionally, fading results showed that after a one-hour pause, the retained signal was at 41% and 35% of the total *continuous-wave* OSL signal for blue and green LEDs, respectively. Our future research will focus on developing strategies to mitigate signal fading and further enhance the stability of the luminescent response

5. FUNDING

I. A. Ferreira thanks to FAPESP (#2021/05042-0) and (#2022/14925-5). M.C.S. Nunes thanks to FAPESP (#2021/12758-1), (#2022/14516-8) and (#2023/18315-0). E.M. Yoshimura is grateful to FAPESP (#2018/05982–0) and CNPq (#311657/2021–4). N. M. Trindade is grateful to FAPESP (#2018/05982-0, #2019/05915-3, #2024/03006-4) and CNPq (#409338/2021-4); M. Chithambo is indebted to Rhodes University and the National Research Foundation of South Africa.

6. CONFLICT OF INTEREST

All authors declare that they have no conflicts of interest.

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