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ORIGINAL PAPER

EPR dosimetry intercomparison using smart phone touch screen glass

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Abstract This paper presents the results of an interlaboratory comparison of retrospective dosimetry using the electron paramagnetic resonance method. The test material used in this exercise was glass coming from the touch screens of smart phones that might be used as fortuitous dosimeters in a large-scale radiological incident. There were 13 participants to whom samples were dispatched, and 11 laboratories reported results. The participants received five calibration samples (0, 0.8, 2, 4, and 10 Gy) and four blindly irradiated samples (0, 0.9, 1.3, and 3.3 Gy). Participants were divided into two groups: for group A (formed by three participants), samples came from a homogeneous batch of glass and were stored in similar setting; for group B (formed by eight participants), samples came from different smart phones and stored in different settings of light and temperature. The calibration curves determined by the participants of group A had a small error and a critical level in the 0.37–0.40-Gy dose range, whereas the curves determined by the participants of group B were more scattered and led to a critical level in the 1.3–3.2-Gy dose range for six participants out of eight. Group A were able to assess the dose within 20 % for the lowest doses (<1.5 Gy) and within 5 % for the highest doses. For group B, only the highest blind dose could be evaluated in a reliable way because of the high critical values involved. The results from group A are encouraging, whereas the results from group B suggest that the influence of environmental conditions and the intervariability of samples coming from different smart phones need to be further investigated. An alongside conclusion is that the

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protocol was easily transferred to participants making a network of laboratories in case of a mass casualty event potentially feasible.

Keywords EPR dosimetry · Radiological emergency · Retrospective dosimetry · Glass

Introduction

Electron paramagnetic resonance (EPR) dosimetry of fortuitous materials has been recognized since the 1960s, but the increased concern about radiological accidents and threats in the last decade has renewed the interest of the scientific community in this topic. Among the investigated materials, glass has always been proposed as having a great potential for improvement. There have been a number of valuable studies of soda-lime glass as found in windows and wrist watches (Marrale et al. 2011; Trompier et al. 2009; Bassinet et al. 2010; Gancheva et al. 2006; Engin et al. 2006; Teixeira et al. 2005; Wu et al. 1995; Wieser and Regulla 1990; Griscom 1980). According to these works, soda-lime glass is suitable for retrospective dosimetry, because it exhibits a specific radiation-induced signal with a linear dose response (generally ascribed to an oxygen hole center) and has a detection limit estimated around 1–2 Gy (mainly limited by a partially overlapping background signal) and a 10–35 % decay at room temperature during the first 24–48 h after irradiation, after which the signal remains stable within the uncertainties. However, windows cannot provide the individual dose to the potentially exposed persons, and wrist watches are more and more frequently made with materials other than mineral glass. This is probably at least one of the reasons why the study of this material was not developed very far.

Nowadays, glass is also found in the displays of mobile phones and more generally in electronic portable devices, which represent probably the most ubiquitous personal items in a large part of the world. Such ubiquity, together with preliminary findings (Trompier et al. 2011a, b), suggests that mobile phones are the ideal item for accident individual dosimetry. Thus, liquid crystal display (LCD) glass was proposed as a material for dosimetric triage in case of mass casualties events in an EC project, MULTIBIODOSE.[1] In the first phase of the project, sixty-eight models of mobile phones were studied (Trompier et al. 2012). In this study, the types of glass found in LCD and touch screens were identified and their EPR properties evaluated, such as the presence of signals before irradiation, the line shape of radiation-induced signals, the post-irradiation signal stability, and the frequency of occurrence of each type of glass in mobile phones. In particular, one glass type showed characteristics by far more suitable to dosimetry and triage application than other types. This type of glass was found only in touch screens. For most types of smart phone models, it is commercially known as Gorilla Glass®.[2] The properties of this material were further explored (MULTIBIODOSE 2013), and a preliminary protocol based on this glass was implemented.

In November 2012, an interlaboratory exercise took place, within two EC projects, MULTIBIODOSE and RENEB,[3] at the same time on five dosimetric assays: the dicentrics; the micronucleus; the gamma-H2AX foci assays; the optically stimulated luminescence (OSL) in resistors extracted from portable electronic devices; and the EPR in touch screen glass (Kulka et al. 2012). The aim was to evaluate the provision of the implemented methods to assess correct dose categories (low exposure <1 Gy; intermediate exposure 1–2 Gy; high exposure >2 Gy). In a radiological incident, this dose characterization can be used to prioritize individuals for further clinical assessment (and, if necessary, treatment) based on their likely magnitude of exposure. Since the EPR protocol with the Gorilla Glass® (MULTIBIODOSE 2013) estimated a dose detection limit around 1 Gy, for this method, the dose categories of low and intermediate exposure were merged, and only two categories were used: low–intermediate exposure <2 Gy and high exposure >2 Gy. The OSL and the EPR tools were tested in a network of participants associated with the European Radiation Dosimetry Group (EURADOS[4]). This paper describes the EPR results of that exercise.

The EPR intercomparison was carried out in parallel in two groups of laboratories: group A received samples taken by an approximately homogenous bulk of glass fragments of several smart phones stored in the same conditions before the EPR measurements; the results from this group were expected to validate the protocol under optimal and monitored conditions. Group B received samples irradiated at different doses that came from different smart phones, and shipping and storing conditions were different among

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[1] http://www.multibiodose.eu.

[2] http://www.corninggorillaglass.com/.

[3] http://www.reneb.eu.

[4] http://www.eurados.org.

laboratories; the results from this group were expected to evaluate the method performances in a situation with multiple and not fully monitored or controlled variables, close to what could likely occur in a real accidental situation.

Materials and methods

Participants recruitment and instructions

Thirteen institutes from nine European countries and one US institute participated in the interlaboratory comparison. The participants were split into two groups: group A (participants 4, 11, and 13) and group B (participants 1, 2, 3, 5, 6, 7, 9, and 10). Participants 2, 4, and 5 were MULTIBIODOSE partners (Helmholtz Zentrum Muenchen, IRSN, and ISS, respectively), and they were the organizers of this exercise.

During a two-day preparatory meeting, organized at IRSN, the participants were trained on the use of the protocol, i.e., sample preparation, measurement, signal evaluation, and uncertainty assessment. This training was considered necessary, because most of the participants did not have any previous expertise on EPR dosimetry with glass.

The participants received the samples that they were asked to measure at their own laboratories with the aim to determine the calibration curve and to assess the blind doses. Participants were asked to report the signal amplitude, the calibration curve parameters, the blind doses, the EPR spectra, and details on storing conditions. Participants 8 and 12 received the samples, but they did not report results because of difficulties in handling the spectra and of serious failures of their instruments, respectively.

Sample preparation

The EPR measurements were taken with smart phone touch screens made of Gorilla Glass®. Twenty-two smart phones of the same model were purchased from the same supplier, and the glasses were disassembled from the smart phones.

The main difference between the two groups was in the sample selection: for group A, an approximately homogeneous batch of mixed samples was used, whereas for group B, individual samples coming from different mobile phones were used at each dose. Specifically, for the samples of group A, glasses from five smart phones were crushed in pieces of the size of approximately 1–3 mm, mixed together, and then separated again in nine aliquots of approximately 600 mg, out of which four aliquots were irradiated at calibration doses and three at blind doses (as described in the next section). The two remaining aliquots were not irradiated. Every aliquot (irradiated and non-irradiated) of 600 mg was divided into three fractions of about 200 mg. The three participants received a whole set of calibration samples, blind dose samples, and two non-irradiated samples. One of the two non-irradiated samples was blind, i.e., the participant did not know that it had not been irradiated. All samples were stored in the same conditions at one laboratory for the first week and then shipped to the participant laboratories. For the samples of group B, touch screens from nine smart phones were used separately for every dose. Seven touch screens were irradiated (as described in the next section), and two were not irradiated. In this case, the glass samples from different smart phones were not mixed and every calibration and blind sample was coming from a different smart phone. Each of the nine touch screens was crushed and divided into ten aliquots of about 200 mg, which were distributed among the participants.

Sample irradiation

Calibration samples were irradiated with 0.8, 2, 4, and 10 Gy, and one sample was kept non-irradiated as a control. The blind doses were as follows: 0, 0.9, 1.3, and 3.3 Gy. The irradiation was performed at IRSN with a ^{60}Co radiation source calibrated in terms of air kerma. The dose rate in air kerma was 2 Gy/min. A 4-mm PMMA plate was used to insure electronic equilibrium. The dimension of the beam was 10 × 10 cm.

EPR measurements

No detailed measurement protocol was distributed. The participants were left free to choose the EPR acquisition parameters within of a suggested range of modulation amplitude (between 0.1 and 0.3 mT) and microwave power (defined for different microwave cavities). The only requirement was to replicate three times the EPR measurement of each sample. Participants used about 100 mg for every single measurement: an aliquot of 100 mg was taken from the total 200 mg amount of glass for every dose, measured, and then mixed again, and a different aliquot was taken for the following measurement.

Sample storing

For group A, all samples were stored in the same conditions for one week after irradiation at IRSN and then distributed to participants.

For group B, samples were distributed to participants on the irradiation day and stored in the participating laboratories in different environmental conditions. No special recommendations were given to participants regarding sample storing, except of keeping the samples exposed to light for at least 5 days in order to speed up the decay of unstable EPR signal components. This requisite was based on preliminary findings where the presence of a spectrum component

sensitive to light was observed (MULTIBIODOSE 2013). This component was stable up to at least one year when the sample was stored in dark, whereas it was canceled within 5 days after irradiation if the sample was exposed in light. Although the fading mechanism and the origin of such component have not been elucidated, the participants agreed to expose their samples to light for at least 5 days. Participants were asked to report details of the storing conditions. Temperature was monitored in every laboratory and limited in the 19–23 °C range. Light exposure conditions (artificial or natural light) varied notably among participants: artificial light (participants 1 and 2); natural cloudy/shadow light (3, 4, 7, 9, 11, and 13); direct bright sunlight (10); and a combination of all (5, 6). An analysis of the relation between storing conditions (illumination and duration) and results was tempted, but the poor statistics did not allow evidencing any influence. The number of days between irradiation and measurements varied within 5–70 days after exposure to ionizing radiation.

Signal evaluation

A dedicated spectrum analysis software was provided to participants to evaluate the EPR-dose-dependent signal. The software "EPR dosimetry" (Koshta et al. 2000), which was designed for the isolation of the EPR-radiation-induced signal in tooth enamel EPR spectrum, had been adapted to the EPR spectrum of Gorilla Glass® within the framework of the MULTIBIODOSE project. EPR dosimetry is based on the best fit of the experimental spectrum with a set of Gaussian lines, spectrum-simulated lines, and experimental reference spectra. The spectrum equation is as follows:

$$F(H) = \sum (A_i f_i(H)) \quad (1)$$

where A_i and $f_i(H)$ are the amplitude and the shape of signal i at field value H, respectively.

In this work, the $f_i(H)$ functions were based on experimental spectra measured with a non-irradiated sample and measured with a 10-Gy exposed sample at least 5 days after irradiation; these were used as model spectra by the participants. In this paper, they will be called "reference spectra." The participants could either create their own model spectra measuring the non-irradiated and the 10-Gy samples or use a set of model spectra provided by participant 2 and acquired before the start of the intercomparison. Some participants tested both sets of "reference spectra," but no influence was evidenced on the estimated doses. The reference spectra and the experimental spectra were taken with the same acquisition parameters and in the same external conditions, except that a larger number of spectrum scans for the reference spectra were used in order to improve signal-to-noise ratio.

Data analysis

Critical levels (CL) and detection limits (DL) were calculated from the unweighted calibration curves (Zorn et al. 1997; Wieser et al. 2008; Fattibene et al. 2011 and references therein); DL and CL refer to two different concepts. The critical level allows the measured signal to be distinguished from the background noise, and it is inherent to the instrument. In other words, if the result of a measurement is higher than the critical level, then the measurement is detecting a physical effect, with a given probability α of being false positive. The detection limit specifies the minimum (true) value of the measurand (here the EPR signal intensity), which can be detected with a given probability β of error using the measuring method in question. In other words, the detection limit allows a decision to be made whether the method under question is suitable for a given purpose of the measurement.

Statistical tests were carried out to identify outliers, leverage points, and influential points of the calibration curves. These are described in "Appendix 1." The results of the blind dose test were analyzed in two steps: firstly, the evaluation of the ability to assess the dose; secondly, a check on the ability of the method to assign the dose category in case of a triage.

Results

Calibration curves

All participants determined the dose–response curve using the calibration samples irradiated at IRSN. The calibration curves of group A and group B laboratories are shown separately in Fig. 1. The data points are the mean value of three replicated measurements, while the error bars represent one standard deviation (SD) of the three measurements; the SD values were in the 1–15 % range (within the symbols in the figures). All curves were normalized to the 10-Gy data point. The parameters of the linear fit and the related statistics are shown in Table 1. The linearity of the curves was demonstrated statistically at 5 % significance level for both groups [A: Pearson's r (0.998) F test (prob $> F = 0$); B: F test (prob $> F$ in the 0.007–4.6 $\times$ 10^{-10} range)]. The residuals (not shown) followed a normal distribution for all laboratories (according to Chen–Shapiro test). The distribution shape of the residuals suggested that variance was constant throughout the dose range. The deviation of the experimental points from the linear fits within each laboratory, and the interlaboratory variation of the EPR measurements at each dose were much larger in group B than in group A. The largest interlaboratory variation of the EPR measurements was observed at the 0.8-Gy calibration dose.

CL and DL values of group A calibration curves were very similar for all laboratories and were in the 0.37–0.40- and 0.74–0.79-Gy dose range, respectively. These values are appropriate for detection of doses below 1 Gy. Both CL and DL values were significantly higher for group B than for group A, and in most cases, they were not appropriate for dose estimation below 1 Gy. Laboratories 5 and 10 reported the lowest slope values and the highest detection limits.

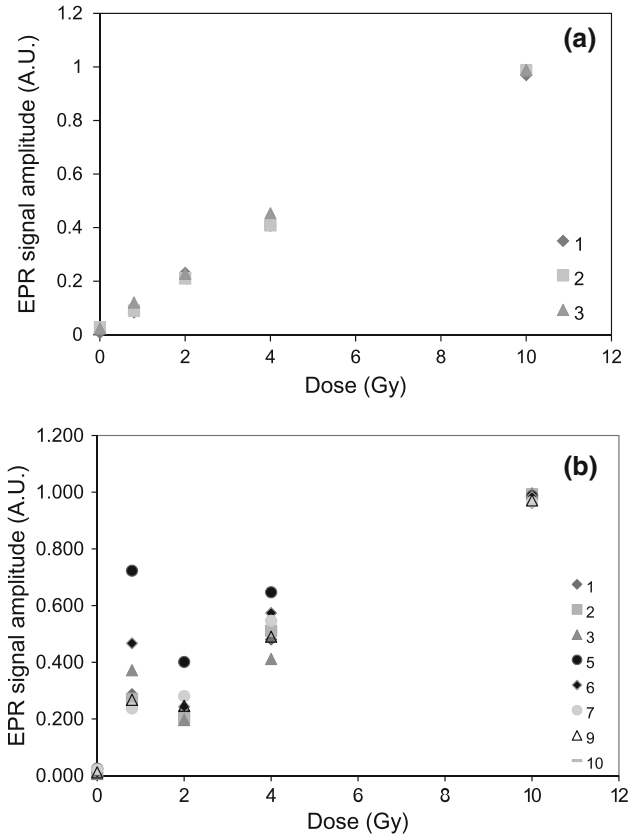

Fig. 1 Calibration curves normalized to the amplitude of the 10-Gy dose sample, for participants of group A (*top*) and group B (*bottom*)

Identification of outliers and influential data points in the calibration curves

During the analysis, all participants identified the calibration sample at 0.8 Gy as having an unusual line shape and higher intensity. For this reason, some participants provided alongside the results obtained without the 0.8-Gy calibration point. This remark from the participants stimulated us to investigate whether the 0.8-Gy dose data point was an influential point in the calibration curves. For this purpose, statistical tests were carried out to identify outliers, leverage points, and influential points of the calibration curves. These tests are described in "Appendix 1." The results of these tests did not suggest that the 0.8-Gy data point was an influential point for any participant.

Determination of blind doses

Figure 2 shows the dose obtained by all participants from the mean value of three replicated measurements of the blind dose samples and Table 2 reports the associated uncertainties. The empty cells of the table are related to data that were not reported by the participants.

The participants of group A were able to assess correctly the three doses within 1 SD, except for two doses where the agreement was within 2 and 3 SDs. The participants of group B, at a first glance, were able to distinguish between the lower and the higher irradiated samples, but not between the low and the intermediate dose samples. Highest reported doses were in the 2.9–4.5-Gy dose range and were on average 2.1 ± 0.5 Gy higher than the lowest

Table 1 Linear fit parameters of the calibration curves and related statistics, critical values, and detection limits

Group	Participant	Y-intercept (A.U.)		Slope (A.U./Gy)		X-intercept (Gy)	Statistics		Critical value (Gy)	Detection limit (Gy)
		Value	SE	Value	SE		Adj. R^2	Pearson's r		
A	4	0.018	0.008	0.096	0.002	0.187	0.998	0.996	0.40	0.79
A	11	0.029	0.007	0.097	0.002	0.299	0.998	0.997	0.37	0.74
A	13	0.040	0.008	0.096	0.002	0.417	0.998	0.996	0.40	0.79
B	1	0.098	0.028	0.088	0.006	1.122	0.944	0.974	1.57	3.10
B	2	0.087	0.028	0.092	0.006	0.946	0.950	0.976	1.49	2.94
B	3	0.101	0.041	0.088	0.008	1.153	0.885	0.945	2.33	4.61
B	5	0.322	0.079	0.070	0.016	4.609	0.560	0.769	5.60	11.91
B	6	0.176	0.054	0.083	0.011	2.123	0.802	0.903	3.20	6.38
B	7	0.125	0.026	0.087	0.005	1.440	0.958	0.981	1.35	2.67
B	9	0.099	0.027	0.089	0.005	1.114	0.950	0.977	1.48	2.92
B	10	0.190	0.053	0.079	0.012	2.403	0.756	0.880	4.16	8.46

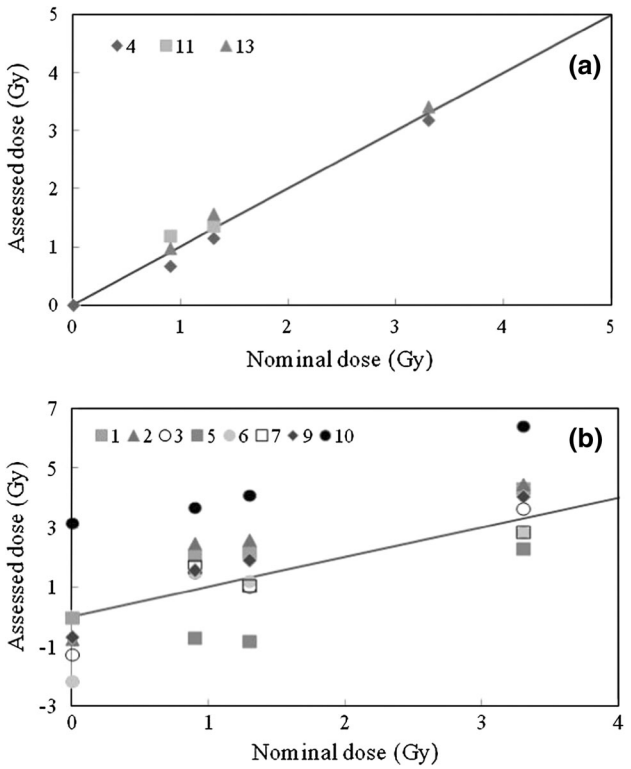

Fig. 2 Measured dose per participant of group A (*top*) and B (*bottom*)

doses for all participants, except for participants 5 and 10 (for the latter, a thorough discussion is carried out below). Some participants provided a dose significantly lower (i.e., exceeding the associated uncertainty) for the 1.3-Gy irradiated sample than for the 0.9-Gy irradiated sample (participants 6 and 7). The non-irradiated samples were identified (significantly) as irradiated by one participant (participant 10).

However, interpretation of results provided by group B participants needs a deeper reading. Comparison between Tables 1 and 2 suggests that for five participants (3, 5, 6, 7, and 10), most of the values reported for the low–intermediate doses were below the CL. Even the lowest CL of group B, which was obtained by participant 7, was 1.35 Gy, i.e., higher than both the lowest and the intermediate blind doses. At such doses, no participant could conclude whether these samples had been irradiated or not. Excluding participants 5, 6, and 10, the highest blind dose was always higher than the CL, and therefore, the participants were able to identify the highest blind sample as irradiated. However, only for four participants (1, 2, 7, and 9), the highest dose was also higher than the DL.

The results of participant 10 were significantly higher than those of the other participants, and the assessed dose was more than three standard deviations higher than the nominal dose, but the Grubbs test could not identify these values as outliers; moreover, they turned out to fall within the range [Q1–1.5 × IQR: Q3 + 1.5 × IQR] where Q1, Q3, and IQR are the first quartile, the third quartile, and the interquartile range of the distribution, respectively. Only two replicate measurements of the non-irradiated sample of participant 10 were identified as outliers according to the aforementioned tests. The relevant overestimation and the large SD of the assessed doses call for a further investigation of the results obtained by this participant.

Discussion and conclusions

The EPR results of the present intercomparison evidenced a diversity in the method performances according to whether the samples were coming from a homogeneous batch of glass (as those distributed to group A) or from different smart phones (as those of group B). The variability of the EPR response per unit dose and of the EPR spectrum line shape of samples of group B, and the consequent large fit error of the calibration curve were especially critical for the determination of the DL. The standard error of the calibration curve slope was, on average, a factor of 5 higher in group B than in group A, which corresponds to a detection limit that was fivefold higher in group B than in group A. The calibration-based detection limit was 0.75 Gy for the three participants of group A, but approximately in the 2.7–3.1-Gy dose range for four participants of group B and (sometimes significantly) higher than 4 Gy for the other four participants.

When looking at the results of the assessed blind doses, those by group A were very satisfactory. Participants of this group were able to identify correctly all blind doses (0.9, 1.3, 3.3, and 0 Gy) within 95 % confidence interval. The agreement between the measured and the nominal doses was within 20 % in the dose range lower than 1.5 Gy and within 5 % for the dose of 3.3 Gy, for all the participants of this group. The results of group B were less satisfactory. As a consequence of the high DL values, no participant of group B was able to assign the dose correctly for doses around 1 Gy, and only four participants out of eight assigned correctly the highest dose. All participants of group B, except for one, were able to identify correctly the non-irradiated samples.

It should be pointed out that, in principle, the calibration curve that was used to assess the unknown dose should have been built only with the calibration doses higher than the detection limit. This was the case for the calibration curves of all participants of group A. For group B, however, four participants calibration doses lower than 2 Gy and the other four participants calibration doses lower than 4 Gy should have been excluded from the linear fit. Note that, based on preliminary findings (MULTIBIODOSE 2013), such high

Table 2 Mean values of the doses measured by every participant of groups A and B ± the standard deviation of the three replicated measurements. The figure in brackets is the uncertainty associated to the calibration curve (1 sigma). All figures are expressed in Gy

Actual dose (Gy)	Participant ID												
	A							B					
	4	11	13	1	2	3	5	6	7	9	10		
0.9	0.7 ± 0.2 (0.2)	1.2 ± 0.7 (0.2)	1.0 ± 0.1 (0.2)	2.1 ± 0.2 (0.8)	2.5 ± 0.0 (0.7)	1.5 ± 0.4 (1.1)	−0.7 ± 0.6 (2.8)	1.5 ± 0.1 (1.6)	1.7 ± 0.1 (0.6)	1.6 ± 0.2 (0.7)	3.7 ± 0.5 (2.0)		
1.3	1.2 ± 0.1 (0.2)	1.4 ± 0.1 (0.2)	1.6 ± 0.1 (0.2)	2.2 ± 0.2 (0.8)	2.6 ± 0.1 (0.7)	1.0 ± 0.3 (1.1)	−0.8 ± 0.2 (2.8)	1.2 ± 0.1 (1.6)	1.1 ± 0.2 (0.6)	1.9 ± 0.2 (0.7)	4.1 ± 0.5 (2.0)		
3.3	3.2 ± 0.2 (0.2)		3.4 ± 0.1 (0.2)	4.3 ± 0.1 (0.8)	4.5 ± 0.0 (0.7)	3.6 ± 0.5 (1.1)	2.5 ± 0.4 (2.8)	2.9 ± 0.1 (1.5)	2.9 ± 0.1 (0.6)	4.1 ± 0.1 (0.7)	6.4 ± 0.3 (2.0)		
0.0	−0.3 ± 0.1 (0.2)			0.4 ± 0.0 (0.8)	−0.7 ± 0.0 (0.7)	−1.2 ± 0.6 (1.2)	−3.2 ± 0.4 (2.7)	−2.1 ± 0.1 (1.6)		−0.7 ± 0.0 (0.7)	3.2 ± 0.6 (2.1)		

DL values were completely unexpected. When it was realized that unexpected factors had occurred which led to high DL values in some laboratories, the design of the intercomparison should have been modified and the exercise repeated. This will certainly be an objective of future work, but, at this stage, the authors believe that the results obtained so far are nevertheless worth to be analyzed and published. In fact, the results of the intercomparison, on the one hand (group A), confirmed the hypothesis that the glass found in touch screens is intrinsically suitable for dosimetric triage, with a DL lower than 1 Gy and a high potential for interlaboratory transfer. On the other hand, the results from group B are also interesting, because they allow to evidence possible pitfalls of the method that could turn out to be troublesome in a real situation.

Possible explanations for the unexpected results from group B are given here, together with suggestions for further investigation:

- The reason for the variability of the background line shape and of the radiation-induced signal increase per dose unit of glass, when coming from different smart phones, cannot be explained by different quality and composition of the original glass making the touch screens. In fact, the EPR signals of the Gorilla Glass® were studied in several glasses coming from different smart phone models and brands in a previous study (Trompier et al. 2012), and variations like the ones observed in the present work were not reported.
- Inexperience of participants might be one cause of the high variability of results, but it does not apply to all participants of group B, because participants 2 and 5 were two of the organizers of this exercise and had the highest experience. As shown in "Appendix 2," participant 5 performed reasonably well when they measured samples that had been stored fortuitously in the absence of light.
- A more sound explanation could be a different sensitivity of the glass to environmental parameters, which were variable among laboratories and not always monitored. Exposure to light and, or, temperature or a combination of the two might have an impact on the EPR spectrum. For instance, the worse performing participants (5, 6, and 10) exposed their samples to direct bright sunlight. In the present study, it was not possible to draw any conclusion on the possible influence of sample storing and preparation conditions on the EPR spectrum, but these should be certainly studied in the future;
- The effect of the intersample variability on the calibration curve uncertainty could be partially bypassed using several samples, coming from different smart phones, per calibration dose. This would average

out the individual variations and would lead to a reduction in the fit error of the calibration curves and then of the DL.

- Finally, the EPR acquisition parameters, i.e., modulation amplitude and microwave power, were not optimized for every spectrometer. It is expected that acquisition parameter optimization can impact on the quality of EPR signal identification and then on the spectrum deconvolution.

Nevertheless, participants of both groups A and B were able to discriminate between irradiated and non-irradiated samples and, for group A participants, in addition between high and low doses.

A final outcome of this exercise was that no major problems were identified by the participants in applying the protocol. The possible minor trouble that required solving was the operational systems compatibility of the dedicated software. The absence of problems is especially meaningful if one considers that the participants were not experienced on the protocol and that they had received only a relatively short training course of 2 days. The protocol can be considered easily transferable to a potential network of laboratories in case of a mass casualty event.

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Appendix 1

A data point that is graphically far from the relationship between x and y described by the other points generally deserves to be further investigated. In the case of the present exercise, the 0.8-Gy calibration data point was far from the linear pattern of the other data points for most participants (see Fig. 1, lower panel) and required further investigation. For this reason, although keeping in mind the weak statistical power of the sample, specific statistic tests were carried out to identify outliers, leverage points, and influential points of the calibration curves (Draper and Smith 1998; Chatterjee and Hadi 1986):

a. Outliers are data points that appear to deviate markedly from other members of the sample in which it occurs, as defined by Grubbs (1979). In this exercise, outliers were identified by the Grubbs test (Grubbs 1979) and by the deleted studentized residuals (Draper and Smith 1998). One individual measurement (out of the three replicates of participants 3, 6, and 9) for the dose 0.8 Gy exceeded slightly the Grubbs test value (assumed as 2).

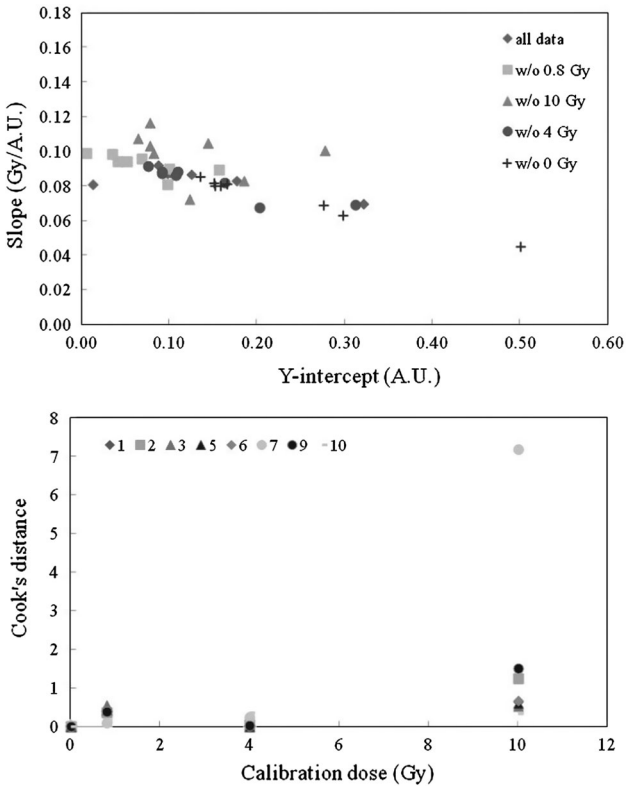

Fig. 3 *Top* estimated slopes versus estimated Y-intercepts obtained by removing one calibration data point at a time. *Bottom* Cook's distance test

b. Leverage points are the points of the independent variable (in the present analysis, the calibration dose) having the potential to dominate a regression analysis, but not necessarily to influence it. These were identified according to the Sokal test. The 0.8-Gy calibration dose points resulted not to cause large changes in the linear fit parameter estimates when they were deleted. The only dose point with a statistically high leverage value was the 10-Gy dose point.

c. Influential points are data points that greatly affect the parameters of the regression line and therefore deserve further investigation. They are typically outliers weighted for their leverage value. The influential points of the calibration curves were identified by the Cook distance test (Cook and Dennis 1979). This parameter was evaluated for the calibration curve of every participant, and it was repeated after removing one calibration data point at a time point from all the calibration curves. In Fig. 3 (top), the resulting estimated slopes versus estimated intercepts obtained

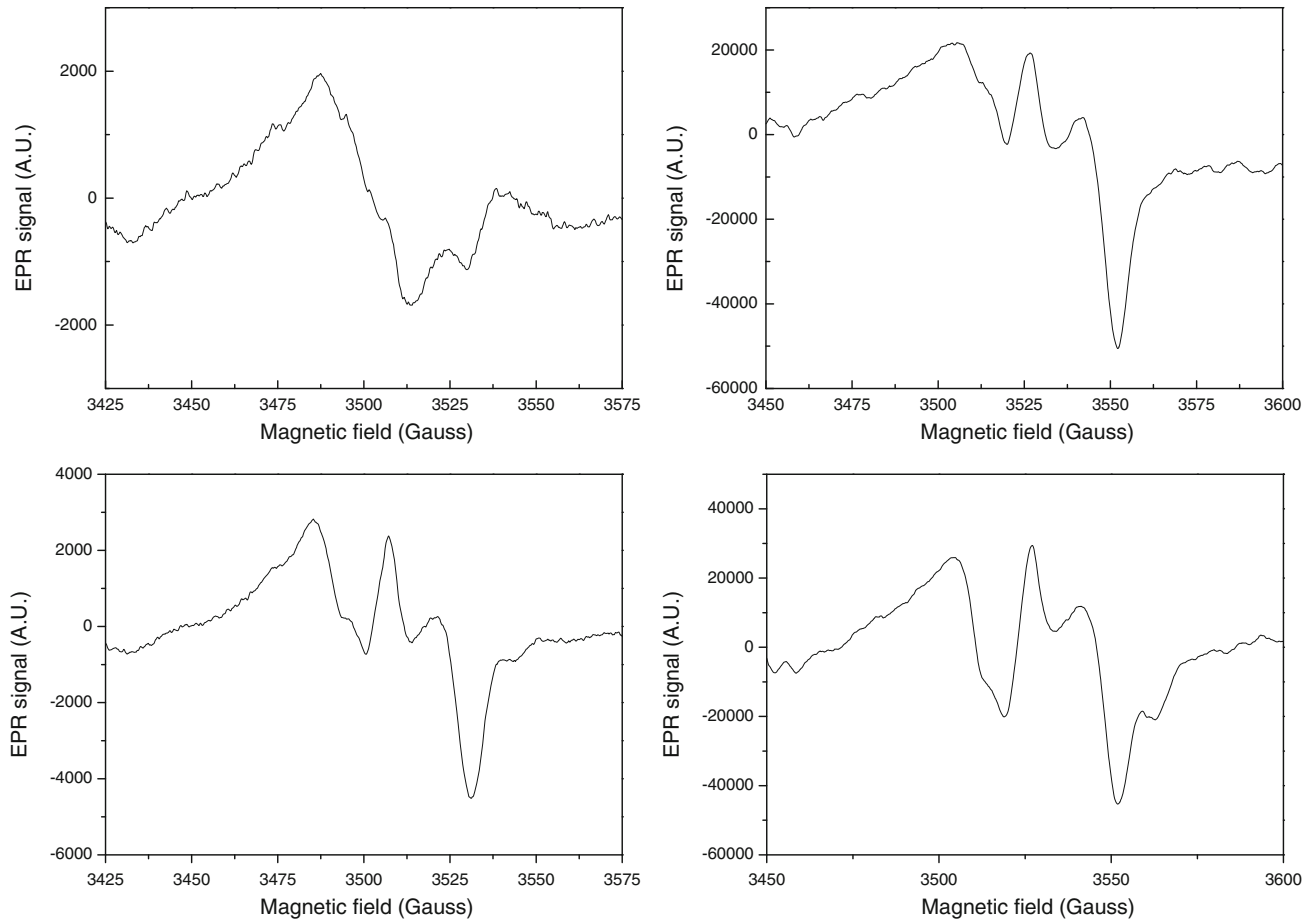

Fig. 4 EPR spectra of the samples irradiated at 0.8 Gy (*top*) and 10 Gy (*bottom*) for participants 13 (*left*) and 5 (*right*)

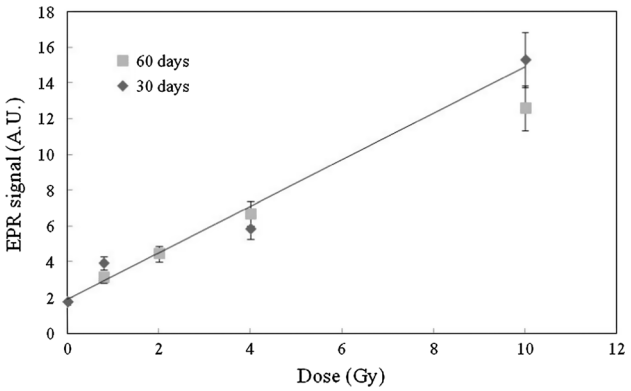

Fig. 5 Calibration curve obtained by participant 5 with samples of glass taken from the same batch used for group B, but stored under controlled conditions and in condition of absence of light (as far as possible)

by removing one calibration data point at a time is plotted. The estimated coefficients are all bunched together regardless of the removed data points, except for the calibration curve of participant 5 when the points 0.8, 4, and 10 Gy were eliminated, and for the curves of participants 5, 6, and 10 when the 0-Gy calibration point was eliminated. The Cook distance (Fig. 3, bottom) indicated that only the 10-Gy point was influential for some participant (2, 7, and 9). This was an expected result given the fact that the 10-Gy calibration dose is an extreme point of the calibration curve and obviously a high leverage point. Although this test is controversial and there are different opinions on the threshold to be chosen for the Cook distance, it is possible to state that no influential points were found.

Although no outliers were identified, it is out of doubt that a problem existed in some participants' measurements in the signal line shape at 0.8 Gy, which appeared different from the signal observed by the other participants. Figure 4 shows the comparison of the spectrum for 0.8 and 10 Gy for participants 5 and 13. Whereas the signal at 10 Gy appeared similar in shape and intensity (although within an expected intersample variability), the signal for the 0.8-Gy irradiated sample was evidently different in line shape and intensity. Excluding the 0.8-Gy calibration data point from

the calibration curve of some participants, the statistics indeed improved significantly. For instance, for participant 5, the CL and DL would drop to 2.3 and 4.6 Gy, respectively, and Pearson's r would increase to 0.953. Participant 10 also showed a signal line shape different from others at all doses, and this is the reason why it was not possible to identify any influential point for this participant. Reasons for this will have to be further investigated.

Appendix 2

After distributing the samples to participants of group B, the remaining part of the samples was stored in the laboratory of participant 5, in a cabinet, i.e., as far as possible in the absence of light. This turned out to be a lucky fortuity. When it became clear that the participants of group B were measuring large fluctuations in the calibration samples, those remaining fragments were measured by participant 5. The measurements were carried out 30 days after irradiation and repeated after further 30 days. The calibration curves are shown in Fig. 5. The fit error in the calibration curve appeared to be significantly smaller than that of Table 1. The estimated values for the blind doses were as follows: 0.69 ± 0.375, 1.16 ± 0.62, 3.46 ± 0.35, -1.2 ± 0.358, for the 0.9, 1.3, 3.3, and 0 Gy doses, respectively, showing satisfactory agreement between the actual and the measured doses for the blind test. Although these data should be taken with prudence because they were measured one and 2 months after irradiation, they are an indication that inexperience may be only partly responsible for the bad performance of participants of group B. Albeit very well experienced, participant 5 performed differently between the intercomparison and when using samples which had not been exposed to light.

References

Bassinet C, Trompier F, Clairand I (2010) Radiation accident dosimetry on glass by TL and EPR spectrometry. Radiat Prot Dosim 98(2):400–405

Chatterjee S, Hadi SA (1986) Influential observations, high leverage points, and outliers in linear regression. Stat Sci 1(3):379–393

Cook R, Dennis (1979) Influential observations in linear regression". J Am Stat Assoc 74(365):169–174

Draper NR, Smith H (1998) Applied regression analysis, 3rd edn. Wiley, New York

Engin B, Aydas C, Demirtas H (2006) ESR dosimetric proprieties of window glass. Nucl Instr Meth B 243:149–155

Fattibene P, Wieser A, Adolfsson E, Benevides LA, Brai M, Callens F, Chumak V, Ciesielski B, Della Monaca S, Emerich K, Gustafsson H, Hirai Y, Hoshi M, Israelsson A, Ivannikov A, Ivanov D, Kaminska J, Ke W, Lund E, Marrale M, Martens L, Miyazawa C, Nakamura N, Panzer W, Pivovarov S, Reyes RA, Rodzi M, Romanyukha AA, Rukhin A, Sholom S, Skvortsov V, Stepanenko V, Tarpan MA, Thierens H, Toyoda S, Trompier F, Verdi E, Zhumadilov K (2011) The 4th international comparison on EPR dosimetry with tooth enamel: Part 1: report on the results. Radiat Meas 46(9):765–771

Gancheva V, Yordanov ND, Karakirova Y (2006) EPR investigation of the gamma radiation response of different types of glasses. Spectr Acta A 63:875–878

Griscom DL (1980) Electron spin resonance in glasses. J Non-Cryst Solids 40:211–272

Grubbs FE (1979) Procedures for detecting outlying observations. Army Statistics Manual DARCOM-P706-103, Chapter 3. U.S. Army Research and Development Center, Aberdeen Proving Ground, MD 21005

Koshta AA, Wieser A, Ignatiev EA, Bayankin S, Romanyukha AA, Degteva MO (2000) New computer procedure for routine EPR-dosimetry on tooth enamel. Description and verification. Appl Radiat lsot 52:1287–1290

Kulka U, Ainsbury L, Atkinson M, Barquinero JF, Barrios L, Beinke C, Bognar G, Cucu A, Darroudi F, Fattibene P, Gil O, Gregoire E, Hadjidekova V, Haghdoost S, Herranz R, Jaworska A, Lindholm C, Mkacher R, Mörtl S, Montoro A, Moquet J, Moreno M, Ogbazghi A, Oestreicher U, Palitti F, Pantelias G, Popescu I, Prieto MJ, Romm H, Rothkamm K, Sabatier L, Sommer S, Terzoudi G, Testa A, Thierens H, Trompier F, Turai I, Vandersickel V, Vaz P, Voisin P, Vral A, Ugletveit F, Woda C, Wojcik A (2012) Realising the European network of biodosimetry (RENEB). Radiat Prot Dosim 151(4):621–625

Marrale M, Longo A, D'oca MC, Bartolotta A, Brai M (2011) Watch glasses exposed to 6 MeV photons and 10 MeV electrons analysed by means of ESR technique: a preliminary study. Radiat Meas 46(9):822

MULTIBIODOSE (2013) Project final report. http://www.multibiodose.eu/News/MBD%20final%20publishable%20summary.pdf

Teixeira MI, Ferraz GM, Caldas LVE (2005) EPR dosimetry using commercial glasses for high gamma doses. Appl Radiat Isot 62:365–370

Trompier F, Bassinet C, Wieser A, De Angelis A, Viscomi D, Fattibene P (2009) Radiation-induced signals analysed by EPR spectrometry applied to fortuitous dosimetry. Ann Ist Super Sanità 45(3):287–296

Trompier F, Bassinet C, Della Monaca S, Romanyukha A, Reyes R, Clairand I (2011a) Overview of physical and biophysical techniques for accident dosimetry. Radiat Prot Dosim 144(1–4):571–574

Trompier F, Della Monaca S, Fattibene P, Clairand I (2011b) EPR dosimetry of glass substrate of mobile phone LCDs. Radiat Meas 46(9):827–831

Trompier F, Fattibene P, Woda C, Bassinet C, Bortolin E, De Angelis C, Della Monaca S, Viscomi D, Wieser A (2012) Retrospective dose assessment in a radiation mass casualty by EPR and OSL in mobile phones. In: The proceedings of the 13th IRPA International Congress, 13–18 May 2012, Glasgow, UK, 2012: P- 02.30

Wieser A, Regulla DF (1990) Ultra high level dosimetry by ESR spectroscopy of crystalline quartz and fused silicate. Radiat Prot Dosim 34:291–294

Wieser A, Fattibene P, Shishkina EA, Ivanov DV, De Coste V, Guettler A, Onori S (2008) Assessment of performance parameters for EPR dosimetry with tooth enamel. Radiat Meas 43(2–6):731–736

Wu K, Sun CP, Shi Y (1995) Dosimetric properties of watch glass: a potential practical ESR dosimeter for nuclear accidents. Radiat Prot Dosim 5:223–225

Zorn ME, Gibbons RD, Sonzogni WC (1997) Weighted least-squares approach to calculating limits of detection and quantification by modeling variability as a function of concentration. Anal Chem 69:3069–3075