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Retrospective Dosimetry after Criticality Accidents Using Low-Frequency EPR: A Study of Whole Human Teeth Irradiated in a Mixed Neutron and Gamma-Radiation Field

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In the context of accidental or intentional radiation exposures (nuclear terrorism), it is essential to separate rapidly those individuals with substantial exposures from those with exposures that do not constitute an immediate threat to health. Low-frequency electron paramagnetic resonance (EPR) spectroscopy provides the potential advantage of making accurate and sensitive measurements of absorbed radiation dose in teeth without removing the teeth from the potential victims. Up to now, most studies focused on the dose–response curves obtained for γ radiation. In radiation accidents, however, the contribution of neutrons to the total radiation dose should not be neglected. To determine how neutrons contribute to the apparent dose estimated by EPR dosimetry, extracted whole human teeth were irradiated at the SILENE reactor in a mixed neutron and γ-radiation field simulating criticality accidents. The teeth were irradiated in free air as well as in a paraffin head phantom. Lead screens were also used to eliminate to a large extent the contribution of the γ radiation to the dose received by the teeth. The EPR signals, obtained with a low-frequency (1.2 GHz) spectrometer, were compared to dosimetry measurements at the same location. The contribution of neutrons to the EPR dosimetric signal was negligible in the range of 0 to 10 Gy and was rather small (neutron/γ-ray sensitivity in the range 0–0.2) at higher doses. This indicates that the method essentially provides information on the dose received from the γ-ray component of the radiation. © 2003 by Radiation Research Society

INTRODUCTION

In the context of accidental or intentional radiation exposures (nuclear terrorism), it is essential to separate rapidly those individuals with substantial exposures from those with exposures that do not constitute an immediate threat to health (*1, 2*). The ability of electron paramagnetic resonance (EPR) to provide retrospective dosimetry is very attractive. EPR dosimetry with teeth is usually performed at X band (~10 GHz) and with enamel powder (*3*). Recent advances in low-frequency EPR (i.e. L-band spectroscopy at ~1 GHz) offer a unique potential for *in vivo* experiments (*1, 4*) without the need to remove teeth from the mouth. For that purpose, a surface coil resonator can be used to surround the top of the tooth (*1, 4*) and it has been demonstrated that around 88% of the EPR signal is then due to the enamel component of the tooth (*4*). The L-band spectrum of a whole tooth is reduced to a single composite line originating principally from radiation-generated CO_2^- (corresponding to the "dosimetric signal") and native radicals.

Up to now, most EPR dosimetry studies have concerned irradiations with photons (X or γ rays). In radiation accidents, however, the contribution of neutrons to the total radiation dose should not be neglected. In a first attempt to estimate the sensitivity of whole teeth to neutrons, an L-band EPR study was performed on whole teeth irradiated with neutrons having a wide energy spectrum, produced by a nuclear reaction in a cyclotron (mean energy around 30 MeV) (*5*). The ratio of the neutron/γ-ray (^{137}Cs) sensitivities (estimated by the slopes of the linear dose–response curves for the dosimetric CO_2^- radicals) was found to be in the range of 0.11 to 0.13. In a nuclear accident, people exposed to the radiation near the site (workers at the plant, members of the military, etc.) would receive a whole-body irradiation with neutrons having a typical spectrum in the range 10^{-8}–15 MeV. Therefore, to mimic a real nuclear criticality accident, we irradiated extracted teeth at the SILENE reactor in a mixed neutron and γ-radiation field (*6*). Teeth were irradiated in phantoms and free in air. Lead screens were also used to eliminate the contribution of γ radiation to a large extent. EPR dosimetric signals were compared to measurements carried out using conventional dosimeters. This study provides unique information on the contribution

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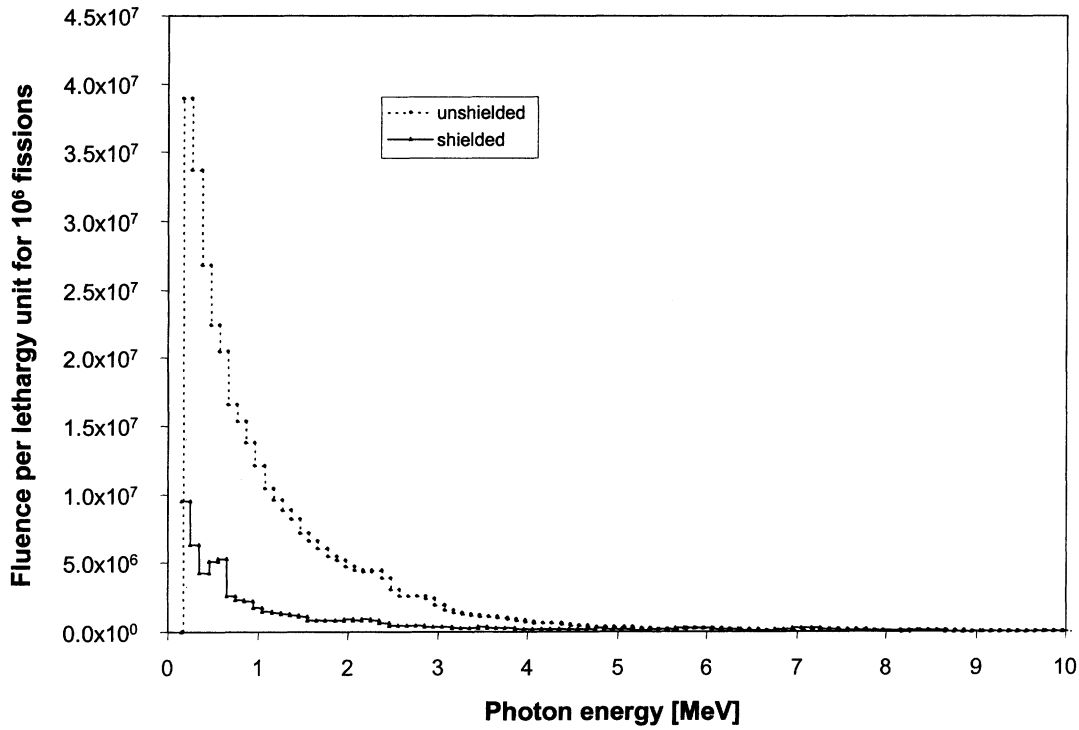

FIG. 1. Photon spectra measured 4 m from the center of the reactor for both reactor configurations.

of neutrons produced in a nuclear accident to the apparent dosimetric signal in teeth recorded with low-frequency EPR.

MATERIALS AND METHODS

Anonymized samples from existing collections were used for the study. Thirty-two teeth were selected. Twenty of them were sound permanent third molars that had been extracted for orthodontic reasons from 16–25-year-old persons in the years 1999–2001 at the UCL Dentistry School and stored in a 1% chloramine solution at room temperature. Twelve other molars were provided by IRSN. Belgian and French teeth were mixed.

Half of the teeth were irradiated at the SILENE facility on March 5, 2002 with a lead-shielded fission source and the other half on April 16, 2002 with an unshielded source. It is worth pointing out that the SILENE reactor is a solution-fueled reactor dedicated to experimental research. At the center of the reactor room, a small annular tank contains the core in which the fissile solution (71 g l^{-1} of uranyl nitrate, 93% enriched with ^{235}U) can be pumped until a supercritical level is reached in the presence of a control rod. By slowly removing the control rod, with the presence of an auxiliary neutron source, a "free evolution" mode is obtained in which the power shows damped oscillations, simulating a criticality accident involving a solution. The field created is a mixed neutron and γ-ray field. The neutron spectrum includes thermal neutrons up to high-energy neutrons (15 MeV). Several shields can be placed around the core to modify the neutron and photon spectra produced by the reactor (*6*). The two aforementioned experiments were carried out in the free evolution mode (duration ~3 min, ~2×10^{17} fissions). The teeth were located circularly at a height of 1.2 m above the floor of the room, corresponding to the mean height of the core, either free in air (called "station" positions) or inside (~2 cm deep) a paraffin cylinder 20 cm in diameter [paraffin wax from VWR, mixture of alkanes, m.p. 52–54°C, 0.90 g/cm^3 (20°C), hydrogen-rich material, close to human tissue for the interaction of neutrons]. At each position, two teeth lying close together received the same dose. The distances from the center of the core were 0.9, 1, 1.5 and 2 m, leading to a significant variation of the dose. The photon and neutron energy spectra for both reactor configurations, as measured 4 m from the core center for a steady-state reactor mode at low power, are shown in Figs. 1 and 2. The γ-ray spectra were measured with a BGO spectrometer and the neutron spectra with a ROSPEC spectrometer (*7*). The reactor mode modifies only the fluence rate of particles, not the shape of the spectra.

The neutron and γ-ray tissue kerma received by each pair of teeth were estimated separately. The γ-ray dosimetry was carried out with alumina powder placed in a cylindrical plastic container, the reading being supplemented with a thermoluminescence reader (LTM FIMEL reader). Standards in terms of tissue kerma were established with a ^{60}Co reference source. This thermoluminescent material has a weak response to neutrons.[3] The neutron tissue kerma is given by diodes used as passive detectors (*8*). The dosimeters were placed on both sides of the teeth.

Three weeks after irradiation, EPR measurements were carried out at UCL using an L-band Magnettech EPR spectrometer (Berlin, Germany) equipped with a low-frequency microwave bridge operating at 1.2 GHz and an extended loop resonator (surface coil of 12.5 mm inner diameter and 2 mm thickness) designed and built by Dr. T. Walczak (Dartmouth Medical School, Hanover, NH) (*9*). A frequency counter (CUB RF minicounter, Optoelectronics) enabled the measurement of the microwave frequency. The tooth was placed on a Plexiglas cylinder against the most sensitive part (opposite to the wave guide) of the surface coil resonator surrounding the top of the crown. A reference sample of lithium phthalocyanine (LiPC, a few crystallites in a capillary tube) was regularly inserted at a fixed position inside the Plexiglas cylinder near the tooth. This LiPC line at $g = 2.0015$ with a 0.024-mT line width was used for magnetic-field calibration and intensity normalization. The spectrometer settings of EPR parameters for measurement of teeth were: 100 kHz modulation frequency, 23 mW input microwave power, 3 mT field sweep, 1024 data points, 1 min scan time, 40 scans, 0.48 s time constant, and 0.1725 mT modulation amplitude. For LiPC, the settings were: four scans, 0.01725 mT modulation amplitude, and 0.06 s time constant. Before EPR

[3] R. Medioni, G. Pescayre, F. Spurny and G. Portal, The response of some thermoluminescent materials to neutrons. Presented at the IVth International IRPA Congress, Paris, 24–30 April 1977.

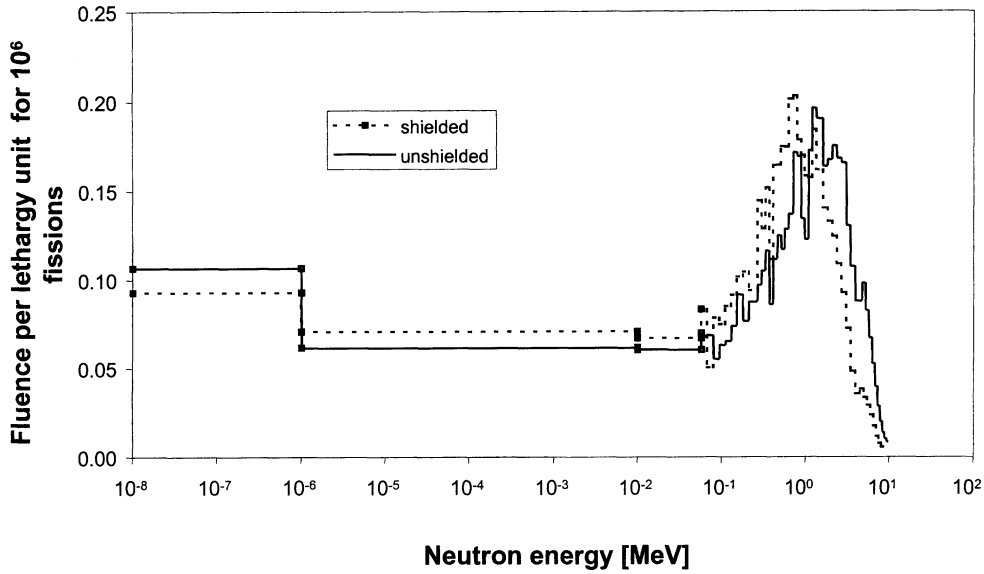

FIG. 2. Neutron spectra measured 4 m from the center of the reactor for both reactor configurations.

spectrum analysis, the cumulated spectrum of the empty resonator taken before and after the tooth spectrum was subtracted from this tooth spectrum.

The deconvolution of the composite EPR line was done with the DOSIMETRY software package developed by the GSF-National Research Centre for Environment and Health of Munich and the Institute of Metal Physics of Ekaterinburg. This software is a modification of an earlier version (*10*) used at GSF for X-band EPR dosimetry with tooth enamel. In the new version, EPR spectra can be deconvoluted by linear combinations of Gaussian functions or by simulated powder spectra that are imported from external powder spectrum simulation programs.

The native and CO_2^- signals were simulated by using the g and linewidth values obtained previously in X band [see table 1 of ref. (*11*), where two sets of CO_2^- values, orthorhombic and quasi axial, are considered]. However, with these CO_2^- values, it was not possible to reproduce the experimental L-band line width: The simulated width was either too narrow (orthorhombic case) or too wide (quasi axial case). A hypothetical CO_2^- line was therefore constructed by adding both orthorhombic and quasi-axial signals with an intensity ratio of 3:2. This hypothetical line was able to simulate the experimental line width of 0.26 mT measured in highly irradiated (875 Gy) teeth, where CO_2^- should be predominant. Actually, two types of CO_2^- radicals are known to be created in human tooth enamel: One, with an axial g tensor, formed in the bulk of the hydroxyapatite microcrystallites, and the other, with an orthorhombic g tensor, formed near the surface of the microcrystallites (*12*). In spite of the fact that the parameters of both CO_2^- components currently are not known exactly and a g strain effect could also be involved, this empirical approach was considered as a plausible solution [further details in ref. (*13*)].

The CO_2^- amplitudes were normalized to the LiPC reference sample and the mean diameter of the teeth (*1*). These amplitudes were compared to those obtained previously with 35 teeth irradiated with ^{60}Co γ rays in the range 1–100 Gy and analyzed by L-band EPR in exactly the same way as in this study (*13*).

In the 0–10-Gy γ-ray dose range, the relative standard deviation obtained by repeatedly measuring the amplitude of the same tooth may reach 20%. The same order of magnitude is observed for the variations between the teeth irradiated with the same dose. The uncertainties are due to the spectrometer and resonator (noises of different origins inherent in low signal measurement, presence of an intrinsic fluctuating resonator signal that must be subtracted from the sample signal), presence of the native signal, and variations from one tooth to another (shape of the masticatory surface, concentration of carbonate responsible for the dosimetric CO_2^- signals, radiosensitivity).

RESULTS AND DISCUSSION

Tissue Kerma in Air and in Phantom

The neutron and γ-ray tissue kerma measured with the dosimeters at each position are shown in Fig. 3. Comparing results obtained at same distance from the reactor center, one can observe that the photon tissue kerma for phantom and air with an unshielded source are very close to each other, whereas a significant increase appears in the phantom in the lead-shielded reactor configuration. In this respect, it is known that, with less than 2 cm of tissue and for a 1 MeV neutron energy beam, about 10% of the total dose in tissue is due to secondary γ rays originating from (n,γ) reactions, principally on hydrogen (*14*). From Fig. 3a, it can be noted that, for the lead-shielded configuration and a given distance, the difference between the γ-ray tissue kerma measured in air and in the phantom is around 9% of the neutron tissue kerma measured in air. The photon dose due to (n,γ) or other reactions [for example (n,p) reactions] in tissues around the teeth is measured by the alumina detectors. The neutron tissue kerma in phantom should thus be always lower than the free in air neutron tissue kerma due to the paraffin shielding effect. This was indeed observed experimentally, except for one case at 1.5 m in the unshielded configuration (see Fig. 3b). For the unshielded configuration, the differences between γ-ray tissue kerma in phantom and free in air are of the same order of magnitude as the uncertainties on the γ-ray tissue kerma.

EPR Dosimetry: Sensitivity to Gamma and Neutron Irradiation

In Fig. 4, the CO_2^- amplitudes of the teeth irradiated at SILENE are compared to the data for ^{60}Co γ rays (*13*)

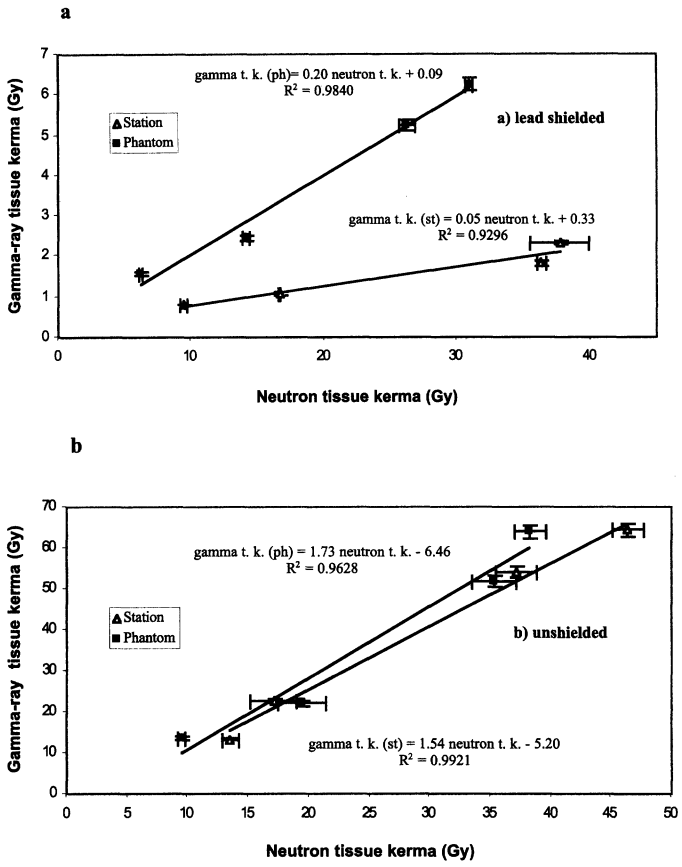

FIG. 3. Photon tissue kerma compared to neutron tissue kerma (t.k.) shown with standard deviations for the different distances from the reactor center (0.9, 1, 1.5 and 2 m) and configurations. Panel a: lead-shielded reactor; panel b: for the unshielded reactor. ph, Phantom; st, station.

obtained previously by using the γ-ray air kerma. Although different symbols are used for the data obtained at SILENE free in air and in the paraffin phantom, the comparison is made with all the SILENE data and by completely neglecting the contribution of neutrons. For γ-ray air kerma up to around 10 Gy (see inset in Fig. 4), both response curves overlap, indicating a negligible contribution from neutrons. At higher doses, the SILENE data exceed the data for ^{60}Co γ rays, but the uncertainties are also higher [the slope for the ^{60}Co data is 4.51 ± 0.09 (± SD), whereas for the SILENE data it is 5.97 ± 0.74]. This 32% increase in the slope is reduced to 26% when only the "station" free-air data are considered in the regression. The phantom data exhibit a 10% increase in the slope compared to the "station" data. This paraffin/air effect should be more readily observable in X band (work in progress at IRSN). The difference between the ^{60}Co γ-ray and the SILENE data must be related to the neutron contribution and the different calibration and geometry conditions used at GSF (*13*) and IRSN. Dentine, through its weight and organic matter and water content, can be suggested to play a definite role during neutron irradiation, contrary to the case of photon irradiation (work in progress at IRSN). At GSF, groups of five molars were irradiated inside a Plexiglas phantom box placed 100 cm from the cobalt source (*13*). Also, the air kerma rate is converted to absorbed dose rate in hydroxyapatite using a factor of 0.975 (*13*). At IRSN, the conversion factor soft tissue kerma/air kerma is 1.102, corresponding to photons of 1.25 MeV (^{60}Co γ rays).

The detection limit in this study can be estimated to be around 1.5 Gy from the dose yielding a signal-to-noise ratio

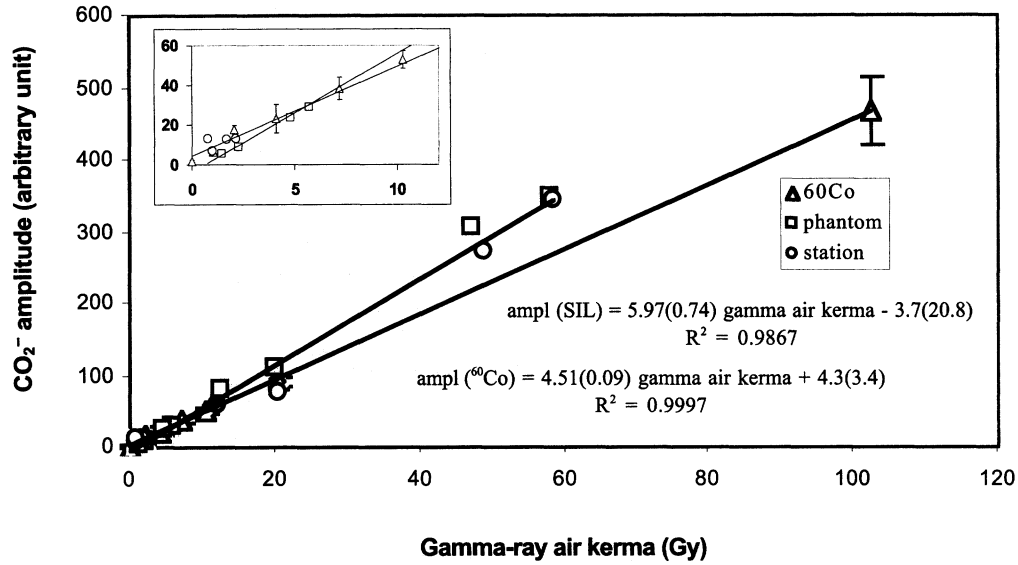

FIG. 4. The CO_2^- amplitudes of the teeth irradiated at SILENE (□ for the paraffin phantom data and ○ for the free-air "station" data) as a function of the photon air kerma compared to the data for ^{60}Co γ rays (△). Error bars for the data for ^{60}Co γ rays (cumulative spectra of five teeth per dose) are standard deviations obtained from three EPR measurements performed 2 weeks, 2 months and 4 months after irradiation. The SILENE data were obtained from the cumulative spectra of two teeth analyzed 3 weeks after irradiation. In the equations, the standard deviations for the slope and intercept are shown in parentheses.

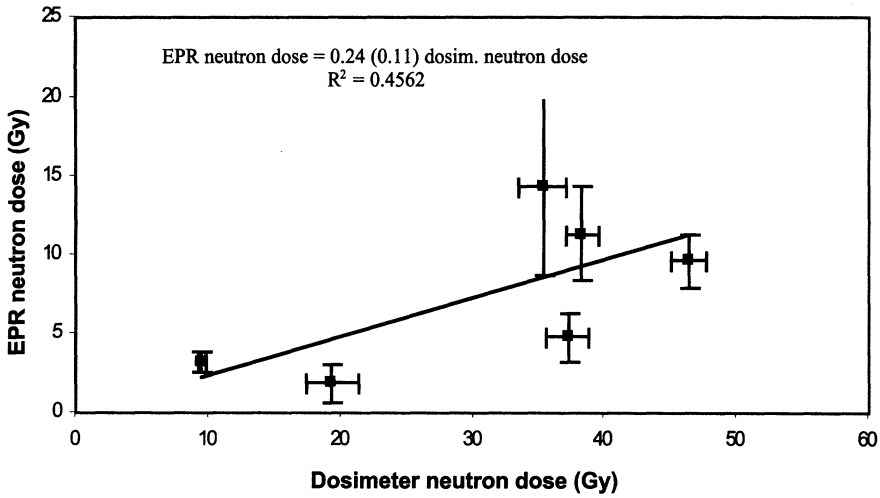

FIG. 5. EPR tooth neutron doses ($D_{60Co} - D_\gamma$) relative to measured neutron doses. EPR error bars are standard deviations obtained with two teeth per dose.

of two. The intercept of the regression line divided by the slope yields the dose corresponding to unexposed samples. The detection limit is also sometimes defined as two standard deviations of this dose, yielding 1.4 Gy for the ^{60}Co data, 1.6 Gy for the SILENE phantom data, and 3.1 Gy for the "station" data. It should be noted, however, that the data for ^{60}Co γ rays were obtained from the cumulated spectra of five teeth per dose measured three times, whereas the SILENE data were obtained from the cumulative spectra of only two teeth per dose, measured only once.

Considering the aforementioned uncertainties in the range of 0 to 10 Gy (the range of dose which can be considered as relevant for the triage of people irradiated in nuclear accidents), it is clear that the contribution of neutrons to the apparent dosimetric signal is negligible.

Looking to results obtained at γ-ray doses higher than 10 Gy, and using the dose–response curve obtained previously with 35 teeth irradiated with ^{60}Co γ rays for converting EPR amplitude into ^{60}Co γ-ray equivalent dose (D_{60Co}), it appears that this EPR dose is higher than the γ-ray dose (D_γ) measured at SILENE. The difference between D_{60Co} and D_γ yields the neutron contribution to the total dose, according to $D_{60Co} = D_\gamma + xD_n$, where D_n is the measured neutron dose and x is the ratio of the neutron and γ-ray sensitivities (*15*). This procedure is strictly valid only if the photon energies are higher than 300 keV where the dose response of tooth enamel is independent of photon energy (*16*). It can be used as a good approximation in our case because the photon component of the SILENE photon spectra under 300 keV in terms of dose is small (<7%). Figure 5 shows the linear response obtained for EPR and dosimeter neutron doses with the data for six (out of eight) bare reactor high doses yielding a slope x of 0.24 $\pm$ 0.11 and a poor correlation coefficient. This value can be considered as a maximum one. For γ-ray doses below 10 Gy, since the contribution of the neutrons does not have any visible effect on the amplitude of the EPR signal, the experimental error (2σ) can be used to estimate the maximum value of the neutron sensitivity. In this case, a mean value of x of 0.1 is found.

A precise value for the contribution of neutrons cannot be assigned using the present data and uncertainties. We can only suggest a possible contribution in the range of sensitivity between 0 and 0.2. It is likely that a more accurate value could be estimated using EPR spectrometers operating at higher frequency, but this method would be usable only for extracted teeth and not *in vivo*. The range of sensitivity observed appears to be consistent with previous studies dealing with the dose response of human teeth to neutrons. Bochvar *et al.* (*17*) reported a theoretical estimate of 0.03 for the relative sensitivity of hydroxyapatite to fast neutrons of 1 keV–1 MeV compared with γ rays (^{60}Co, ^{137}Cs). For whole teeth irradiated with neutrons with a wide energy spectrum that were produced by a nuclear reaction in a cyclotron (mean energy around 30 MeV) (*5*), we found a ratio for the neutron/γ-ray (^{137}Cs) sensitivity in the range 0.11 to 0.13. Using an accelerator-based neutron source with a mean neutron energy of 280 keV and X-band EPR analysis of the irradiated enamel, experimental values of the ratio of the neutron/γ-ray (^{137}Cs) sensitivities ranging from 0.08 to 0.12 were obtained by Khan *et al.* (*18*). A higher value of 0.33 $\pm$ 0.08 relative to ^{60}Co was found by Fattibene *et al.* (*15*) with 2.8 MeV neutrons after X-band EPR analysis complemented by Monte Carlo calculation. The different values obtained should be correlated to the neutron energies and experimental configurations used in the different works.

Finally, as already stated in recent publications (*5, 15, 18*), even if the neutron contribution is not negligible in the case of high exposure, the limiting factor remains that EPR spectra of γ- and neutron-irradiated teeth are apparently identical. There is thus no measurable way in teeth to discern between the two radiations.

In the absence of appropriate personal dosimeters, be-

cause the tooth EPR spectra are essentially sensitive to the γ-ray component of the exposure, other materials belonging to the irradiated person (body, clothing) should be investigating as potential neutron + γ-radiation EPR detectors. For example, fingernails (*19*) and buttons (*20*) with their light nuclei should be more sensitive to neutrons than teeth and might be candidates depending on the complexity of their EPR spectra and the thermal stability of their radicals. We also suggest that the EPR method should be used in conjunction with other methods that can assess the neutron dose such as the classical neutron activation analysis in spite of the relatively short half-lives involved. For example, in the case of neutron overexposure, ^{32}S isotopes from hair can be activated and produce, through (n,p) reactions, ^{32}P if the neutron energy is higher than 2.2 MeV. Since the occurrence of a neutron accident is very often known quickly, the 14-day half-life of ^{32}P could be considered as acceptable. In this respect, the extreme stability of the CO_2^- radicals in teeth is an undeniable advantage.

CONCLUSIONS

The L-band EPR dosimetric CO_2^- component amplitudes were related to the neutron and γ-ray doses obtained with dosimeters placed on both sides of the molars and compared to the EPR amplitudes obtained previously with ^{60}Co γ rays. The contribution of the neutrons to the global dose is not detectable up to around 10 Gy. Thus, at the present stage of L-band spectrometry, this method essentially provides information on the dose absorbed by whole teeth from the γ-ray component of the radiation. L-band EPR spectroscopy technique can be decisive for rapid and first non-invasive dose estimation after a nuclear accident, especially when the irradiated persons are not wearing dosimeters. The γ-ray dose can be estimated with around 20% uncertainty. This is in good agreement with the IAEA recommendations that request in the first 24 h after a nuclear accident a dose estimation with a 50% uncertainty (*21*). The neutron component of the radiation should be estimated by other materials or methods.

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