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ESR response to ^{60}Co-rays of ammonium tartrate pellets using Gd_2O_3 as additive

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Abstract

This work presents experimental results regarding a new ammonium tartrate blend for ESR dosimetry, with a higher sensitivity and a lower lowest detectable dose (LDD) to ^{60}Co γ-rays than the recently used pure ammonium tartrate.

The blend composed by ammonium tartrate and gadolinium-oxide (Gd_2O_3) shows a greater sensitivity (~2 times) and a smaller LDD than ammonium tartrate. The increased sensitivity was mainly attributed to the great atomic number ($Z = 64$) of gadolinium, that increases the effective atomic number of the blend; the interaction probability with photons and consequently the radical yield is therefore enhanced. Moreover ammonium tartrate with Gd_2O_3 has a linear dose response in the investigated dose range (1–50 Gy). We find this blend suitable for use in ESR dosimetry.

Keywords: ESR; Ammonium tartrate; Gd_2O_3–ammonium tartrate

1. Introduction

Since 1960s many scientists adopted the ESR technique to measure the absorbed dose due to high energy radiation. Bradshaw et al. (1962) used the amino acid L-alanine for high dose measurements, and they found an approximately linear response up to 10 kGy. The first analytical study of the alanine as dosimetric material was performed by Regulla and Deffner (1982). The advantages of these dosimeters are: cheap dosimeter material, simple and rapid dose evaluation, non-destructive analysis permitting repeated measurements and thus allowing storage for archival purposes, and wide linear dose range. Afterwards various laboratories in the world developed the dosimetric system based on alanine. This aminoacid was recognized by the IAEA (International Atomic Energy Agency; Mehta, 1999) as a routine, reference and transfer dosimeter for industrial applications in the high dose range (of the order of a few kGy). Furthermore, the alanine dosimeters are commonly adopted by several national calibration laboratories for transfer dosimetry (Nam and Regulla, 1989; Mehta and Girzikowsky, 1996, 1999; Mehta, 1999). Nette et al. (1993) showed that it is possible to measure relative absorbed doses at the 2 Gy level with a precision of 2% by means of these alanine pellets. However, the precision stated above requires alanine samples with great mass and volume; therefore, the spatial dose resolution is limited. Nevertheless, if the aim is to measure doses lower than 1 Gy or if a high spatial resolution is required, the alanine dosimeters are not suitable because they show a signal to noise (S/N) ratio too low.

Therefore, many laboratories are very interested to the development of ESR dosimeters with improved S/N-ratio at low dose levels. The way commonly followed is the research of new materials or new blends of organic and/or inorganic compounds and improvements in signal analysis (Bartolotta et al., 1999, 2001; Olsson et al., 1999).

The influence of dopants to obtain other ESR dosimeter materials more efficient are of interest (Gustafsson et al., 2005; Hassan and Ikeya, 2000). The choice of adding different atoms

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to dosimeters is justified by the interaction mechanisms of the ionizing radiation (in particular γ-photons) with the solid state matter. During the irradiation process the number of free radicals produced is strictly correlated to the number of interacting photons which depends on the photon cross section of the medium atoms. This cross section is a function of the photon energy and of the atomic number Z of the target elements. For the three main processes responsible for absorption of photons (photoelectric absorption, Compton scattering and production of electron–positron pairs) the cross section per atom increases with the atomic number Z. So, the choice of target atoms with high atomic number Z raises the number of interacting photons. Therefore, to increase the sensitivity and the efficiency of a photon detector, atoms with high atomic number should be used (Knoll, 1979).

With the aim of finding a material, sensitive enough for low dose determination, we have examined the possibility to use Gd_2O_3–ammonium tartrate as a new material, since the gadolinium atom has an high atomic number ($Z = 64$) and it has an high neutron capture cross section, that renders it useful also for ESR dosimetry in mixed fields. We have investigated the sensitivity and lowest detectable dose (LDD) of two types of solid state ESR pellets—one constituted by pure ammonium tartrate and a second one in which gadolinium-oxide (Gd_2O_3) was added. Moreover, we have realized Gd_2O_3–ammonium tartrate pellets with smaller size than ammonium tartrate ones to achieve a better spatial dose resolution.

However, for application in radiation therapy it is preferable that the material is tissue equivalent, i.e. its atomic composition and density should be as close as possible to those of soft tissue (ICRU, 1989). The Gd_2O_3–ammonium tartrate, due to the high Z of gadolinium, will absorb a different dose than soft tissue when exposed to photons, particularly at low energies (such X photons). It is, however, possible to correct for this, when the photons energy is well known.

The purpose of this work is to show how Gd_2O_3 enhances the ESR signal for ^{60}Co photons and to give an explanation for that result.

2. Materials and methods

2.1. Samples preparation

Solid state pellets for electron spin resonance (ESR) were made using a blend of ammonium tartrate (Carlo Erba, Italy) and Gd_2O_3 (Aldrich Chem. Co) in known proportions by weight.

The pellets were realized following a procedure previously optimized (Bartolotta et al., 1999) by pressing a blend of 94% of the appropriate active material (either Gd_2O_3–ammonium tartrate or pure ammonium tartrate), 5% of polyethylene (Polysciences, $MW = 700$) as binder, and 1% of magnesium stearate (Carlo Erba, Milano, Italy) as lubricant. The pellets of ammonium tartrate with Gd_2O_3 were realized by pressing a blend where the two constituent, ammonium tartrate and Gd_2O_3, were present in equal proportion in weight (47%). Table 1 reports the percentage in weight of the component present in the pellets. Each component of the blend was previously pounded in agate mortar, and afterwards sieved, to select grains of 70–125 μm, in the same size range as grains of polyethylene and magnesium stearate (about 90 μm on average). The blend was obtained by mixing with a rotating twin shell. Cylindrically shaped pellets were obtained by pressing 32 mg of each blend at 6×10^6 Pa in a stainless steel die. Finally, the pellets underwent a thermal cycle of 20 min at 130 °C and 15 min at 85 °C to improve their mechanical properties. Pellets of about 4 mm in diameter and 2.2 mm, 1.7 mm in thickness for ammonium tartrate and Gd_2O_3–ammonium tartrate, respectively, were obtained.

2.2. Irradiation

For the dose dependence studies, pellets were irradiated at room temperature with γ-photons of a ^{60}Co source (Alcyon II, General Electric, France) used for radiotherapy treatments at the Radiotherapy Department of the Oncology Hospital 'M.Ascoli' in Palermo.

Irradiations were performed at various absorbed dose values in a perspex phantom $30 \times 30 \times 30\,cm^3$, with a field size of $15 \times 15\,cm^2$, at water equivalent depth of 5 cm, and a source–detector distance of 80 cm. Fig. 1 shows the irradiation set-up. The dose rate at the effective dosimeter location was evaluated with the ENEA (Ente per le nuove tecnologie, l'energia e l'ambiente, Italy) secondary standard ionization chamber, with an overall uncertainty of 2% (95% confidence level).

The ^{60}Co irradiator IGS-3 of the Dipartimento di Ingegneria Nucleare, Università di Palermo (dose rate $\cong 2.0$ kGy/h) was used for the high dose irradiation at 3 kGy, aimed to analyse the ESR signal structure.

Table 1
Percentage in weight of the elements present in the dosimeters and in soft tissue (ICRU, 1989)

	H 1	C 6	N 7	O 8	Others	ρ (g cm^{-3})	Diameter (mm)	Thickness (mm)
Ammonium tartrate	6.98	49.15	14.30	29.53	0.04	1.2	4	2.2
Gd_2O_3–ammonium tartrate	2.93	25.08	4.86	13.33	53.80	1.3	4	1.7
Soft tissue (ICRU, 1989)	10.2	14.3	3.4	70.8	1.3	1.05		

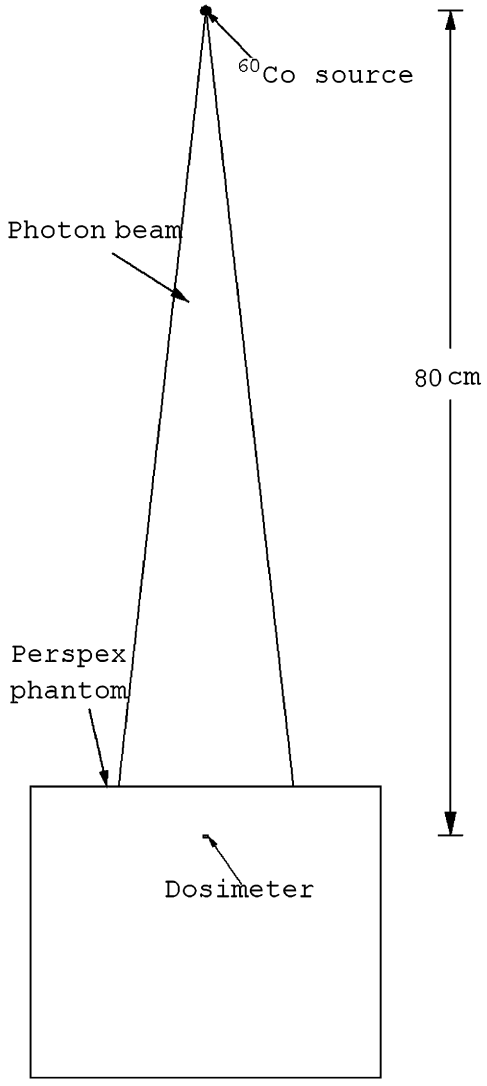

Fig. 1. Schematic representation of the irradiation set-up.

2.3. ESR measurements

The ESR spectra were recorded at room temperature with an X-band Bruker ECS106 spectrometer equipped with a rectangular cavity working in TE_{102} mode.

For the ESR measurements all dosimeters were located in quartz sample tubes.

A quartz spacer was inserted at the bottom of the sample tubes to keep the dosimeter in the location of maximum signal intensity. Fig. 2 shows the ESR spectra of a solid state ammonium tartrate dosimeter and of a Gd_2O_3–ammonium tartrate dosimeter, both irradiated at 3 kGy.

One main resolved line ($g = 2.0030 \pm 0.0005$ and 1.10 ± 0.05 mT wide) is present, due to the free radicals produced by photons in the ammonium tartrate molecule (Bartolotta et al., 2001), whose chemical form is

$H_4N^{+-}OOC–CH(OH)–CH(OH)–COO^{-+}NH_4$.

The main free radical was attributed to the break of the C–H bond in the α-position (Brustolon et al., 1996). Two other

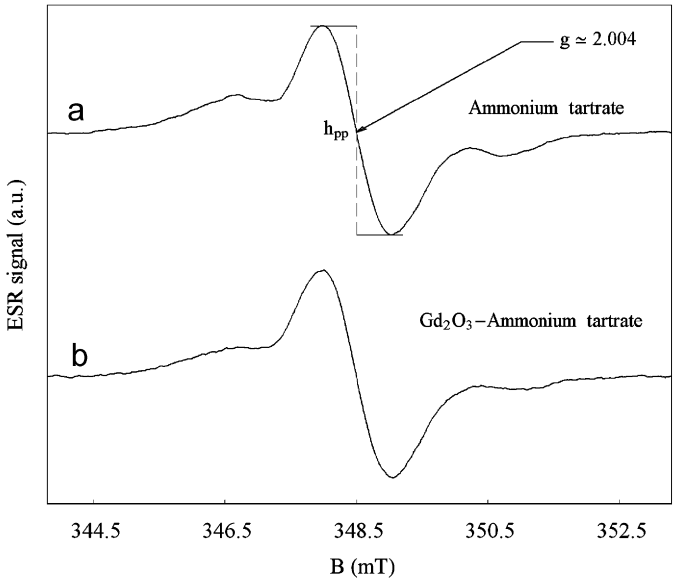

Fig. 2. ESR spectra of ammonium tartrate and ammonium tartrate with gadolinium, after Gd_2O_3-baseline elimination, pellet irradiated to 3 kGy with a ^{60}Co source.

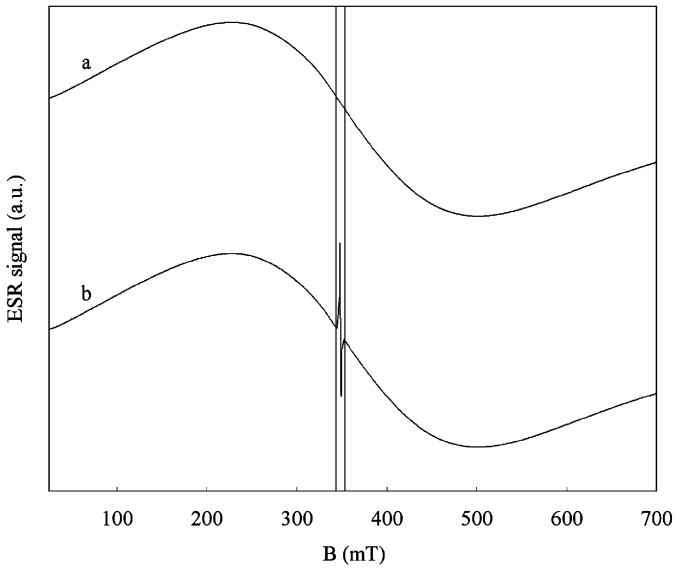

Fig. 3. The ESR spectrum of a solid state Gd_2O_3–ammonium tartrate pellet: (a) unirradiated and (b) irradiated at dose of 3 kGy with the ^{60}Co source.

structures appear, symmetrically spaced at about 1.8 mT from the central line; their characteristics are under investigation, but they are not relevant for dose evaluation, which is the aim of the present work. The peak-to-peak signal height h_{pp} of the central line was measured and used as dosimetric parameter (ASTM, 1995).

The dosimeters realized with Gd_2O_3–ammonium tartrate (unlike the dosimeters with pure ammonium tartrate) show also a very large ESR signal (∼ 270 mT) due to Gd_2O_3, centered in the $g \sim 2$ region field, as shown in Fig. 3. The contribution of the Gd_2O_3 signal in the magnetic field range swept for dosimetric purposes (343.5–353.5 mT) is linear (Fig. 3a) and

the ESR spectrum of ammonium tartrate is inside this range (Fig. 3b). To eliminate this background signal a linear baseline was subtracted to the spectrum of each Gd_2O_3–ammonium tartrate dosimeter. The h_{pp} for these dosimeters was measured after this baseline correction. The ESR spectrum, shown in Fig. 2, of a solid state ammonium tartrate with a gadolinium dosimeter irradiated at 3 kGy, was obtained after the baseline elimination.

2.4. Recording parameters

The ESR recording parameters were appropriately chosen to obtain the highest signal to noise ratio (S/N), even though some signal distortion was introduced due to power saturation and to large modulation amplitude.

A detailed analysis of the ESR signal intensity h_{pp} as a function of the recording parameters was carried out both on Gd_2O_3–ammonium tartrate and on ammonium tartrate dosimeters. In Fig. 4 the h_{pp} values of the two dosimeter types irradiated at 100 Gy as a function of the square root of the microwave power are shown (modulation amplitude 0.85 mT). For each blend these values were normalized to their maximum. For both dosimeter types the signal intensity increases linearly with the square root of the microwave power up to about 1 mW; it reaches its maximum at about 5 mW and decreases afterwards. The selected microwave power value was therefore for both blends 1.59 mW, that does not introduce excessive signal distortion.

For the same dosimeters, the dependence of h_{pp} on the amplitude of the 50 kHz modulating magnetic field, between 0.01 and 2.5 mT, is shown in Fig. 5 (microwave power 1 mW). At low modulation amplitude values (below $\sim$ 0.8 mT) the ESR signal intensity increases linearly; it reaches its maximum value at about 2 mT and decreases afterwards. The trends of the signal intensities of the two compounds as a function of modulation amplitude are similar. To choose the optimal modulation amplitude we have tried to maximize the S/N ratio avoiding to excessively distort the line shape. The chosen modulation amplitude was therefore 0.96 mT.

On the basis of these results, the following set of standard ESR recording parameters was chosen:

- field set, 348.5 mT,
- field sweep, 10 mT,
- microwave power, 1.59 mW,
- modulation amplitude, 0.96 mT,
- time constant, 655 ms,
- number of cumulated scans, 3.

Since h_{pp} depends on the orientation of each pellet inside the resonating cavity (Kojima et al., 1995), all samples were read out at four orientations and the h_{pp} were corrected by the fractional weight content of ammonium tartrate in the sample. The mean value h_r of the signals of the overall twelve readouts of three dosimeters, irradiated at the same value of dose, was used for dose measurement. This procedure decreases the uncertainties associated with ESR measurements. The sample standard deviation of the signal were always within ±3%.

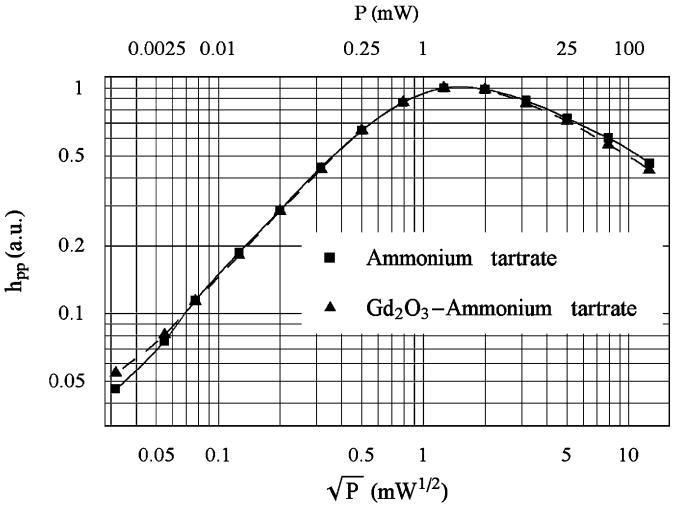

Fig. 4. ESR signal amplitude (h_{pp}), normalized to its maximum value, as a function of microwave power square root.

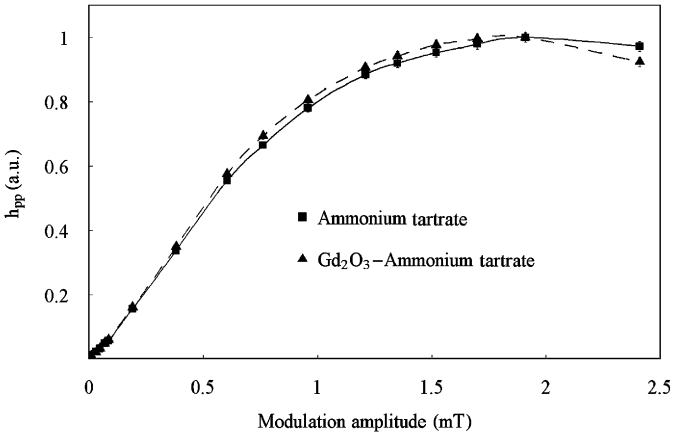

Fig. 5. Effect of magnetic field modulation on the peak-to-peak height (h_{pp}) of ammonium tartrate and Gd_2O_3–ammonium tartrate signals.

3. Results and discussion

The ESR signal intensity of ammonium tartrate and ammonium tartrate with Gd_2O_3 pellets was studied as a function of dose by irradiating groups of three dosimeters with the ^{60}Co source at absorbed doses in water, D_γ, in the 1–50 Gy range. Since the irradiation center is an oncological hospital, the availability for dosimeter irradiation was reduced and we could irradiate the dosimeters of ammonium tartrate at the doses 3, 4, 5, 10, 50 Gy and the dosimeters of ammonium tartrate with gadolinium at the doses 2, 3, 4, 5 and 33 Gy. Fig. 6 reports the values of h_r as a function of dose in water for the two compounds. The error bars corresponds to ± one standard deviation.

The dose response of both dosimeter types could be well fitted with the straight line equation

$$h_r = a + bD_\gamma \quad (1)$$

as shown by the correlation coefficients reported in Table 2; the other results obtained through this analysis are also reported in Table 2.

The LDD was evaluated as the dose value that produces in the irradiated pellets an ESR signal equal to the mean value of the zero dose signal in unirradiated pellets plus three standard deviations (Bartolotta et al., 1993). Therefore, we measured the signal intensity of six unirradiated pellets for each blend in the magnetic field range where we expect to find the ESR signal of the free radicals produced after irradiation (between 347.7 and 349.2 mT). From the measurements of the background signal and the calculation of its standard deviation, we obtain the LDD through an inversion of Eq. (1).

Our results (Fig. 6, Table 2) show that the sensitivity (the slope of the calibration straight line function) of the dosimeters realized with Gd_2O_3–ammonium tartrate is greater (about twice) than the sensitivity of ammonium tartrate ones. This sensitivity enhancement could be due to the relatively high atomic number ($Z = 64$) of gadolinium that involves an increase of the photon interaction probability, which is proportional to the photon cross section of the dosimeter atoms. For the three main processes responsible for absorption of γ-rays (photoelectric absorption, Compton scattering and production of electron–positron pairs) the cross section per atom increases with the atomic number Z of the target atoms. In particular, the cross sections for photoelectric absorption and Compton scattering increase with atomic number because the number of electrons (which might interact with photon) increases with Z; the cross section for production of electron–positron pairs increases with the atomic number because the intensity of the electric field of the nucleus (wherein the pair production occurs) increases in intensity with Z (Kaplan, 1963). The ^{60}Co γ-rays have a mean energy of 1.25 MeV. Hence, the predominant interaction is the Compton scattering and the production of electron–positron pairs is less relevant. The photoelectric absorption is the least probable effect.

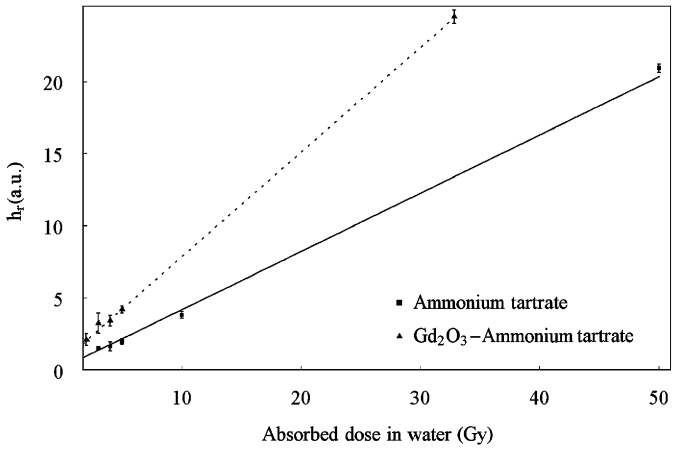

Fig. 6. Response curves of ammonium tartrate and Gd_2O_3–ammonium tartrate in the range 1–50 Gy. The fitting curves are linear functions.

In pair production process created positrons and electrons would break the C–H bond in ammonium tartrate with increasing scattered electrons. On the other hand, the interaction between photons and electrons results mainly in Compton scattering with the scattered electrons and the photons with the lost energy. The cascade of the electrons induced by the scattered electrons produces radicals by breaking of the bond.

The addition of Gd_2O_3 implies an increase of the number of the secondary electrons produced by the radiation in the dosimeter, producing extra free radicals. Hence, increase of the production of radicals would be due to the increase of atomic number. So, the choice of target atoms with high atomic number Z, such as the gadolinium, raises the number of photons that release their energy inside the dosimeter and, consequently, the ESR signal. The sensitivity of the dosimeter increases and the detection efficiency is enhanced with the addition of the gadolinium.

With the aim of correlating the dosimeter sensitivity with the dosimeter composition, we computed the mean atomic number, Z_m, for each blend through the following formula:

$$Z_m = \sum_{i=1}^{n} w_i Z_i, \quad (2)$$

where w_i is the fraction in weight of the element i contained in the dosimeter and Z_i is its atomic number. In Table 3 we reported the results normalized to the value obtained for ammonium tartrate dosimeters. From this table it is evident that the relative mean atomic number is much greater than the sensitivity ratio. Therefore, we have evaluated, for each dosimeter type, the effective atomic number Z_{eff} using the procedure suggested in literature for 1.25 MeV photons (Shivaramu and

Table 2
Results (a and b parameters ± S.D. and correlation coefficient) of the fitting procedure for the two typologies of samples

Compound	Parameter		Correlation coefficient	LDD (Gy)
	a (a.u.)	b (sensitivity) (a.u.)		
Ammonium tartrate	$(17 \pm 3)\ 10^{-2}$	$(403 \pm 5)\ 10^{-3}$	0.9923	2.9
Gd_2O_3–ammonium tartrate	$(62 \pm 7)\ 10^{-2}$	$(724 \pm 9)\ 10^{-3}$	0.9989	1.1

In the last column the lowest detectable dose (LDD) is reported.

Table 3
Comparison of the experimental sensitivity ($S \pm$ S.D.) of the two compounds (with respect to the ammonium tartrate sensitivity) with the mean atomic number (Z_m) and effective atomic number (Z_{eff})

Compound	Z_m	$Z_m/Z_{m(tartrate)}$	Z_{eff}	$Z_{eff}/Z_{eff(tartrate)}$	$S/S_{tartrate}$
Ammonium tartrate	6.78	1.00	4.01	1.00	1.00
Gd_2O_3–ammonium tartrate	30.10	4.44	6.37	1.59	(1.80 ± 0.03)

Ramprasath, 2000). The results are shown in Table 3, where the experimental relative sensitivities of the dosimeters are compared with their relative effective atomic numbers. Our results show that the relative effective atomic number is much closer to the sensitivity ratio than the mean atomic number. So, the introduction of the gadolinium atom have increased the effective atomic number and, consequently, the number of interacting photons which can produce free radicals in the ammonium tartrate.

Therefore, dose in water being equal, the ESR response h_r of the ammonium tartrate dosimeters with gadolinium is about a factor 2 greater than that one of the dosimeters without gadolinium.

4. Conclusion

We analyzed in the present paper the ^{60}Co photon response of two different types of ESR pellets, realized with ammonium tartrate and with a Gd_2O_3–ammonium tartrate blend. Through a study of the ESR signal we obtained the parameters which allow dosimetric measurements by the read-out of the peak-to-peak amplitude, h_{pp}, and we chose the optimal microwave power and modulation amplitude values. Through an analysis of the dose response we observed that the sensitivity for the ammonium tartrate with Gd_2O_3 is about two times greater than pure ammonium tartrate. This sensitivity improvement can be correlated with an increase of the effective atomic number Z_{eff} of the pellets after the addition of the gadolinium ($Z = 64$). Another consequence of the addition of Gd_2O_3 is the reduction of the LDD ($\sim$ 1 Gy) which is below the values of simple ammonium tartrate ($\sim$ 3 Gy) used in this work. Moreover, the thickness of Gd_2O_3–ammonium tartrate pellets is smaller (0.7 times) than ammonium tartrate ones. This allows to achieve a better spatial dose resolution. On the other hand, the Gd_2O_3 ammonium tartrate pellets tested in this work are indeed not ideal for clinical applications because of the high Gd content. If the energy of the photons is well known, it is possible to correct for the difference in mass energy absorption properties. In conclusion, the ammonium tartrate with Gd_2O_3 shows radiometric features that designate it as a good candidate for low dose measurements. The improvement of the response and the use as dosimeter in radiation therapy of these promising Gd–ammonium tartrate pellets are linked to the study, in progress, of the ESR signal at different Gd_2O_3 concentrations and to a study of their photon energy dependence. Low concentration of the Gd_2O_3 added is on request to obtain a dosimeter closer to tissue equivalence. Other studies with different sensitive materials and additive are in progress, taking into account relative concentration and dosimeter thickness.

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