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Radiological protection — Minimum criteria for electron paramagnetic resonance (EPR) spectroscopy for retrospective dosimetry of ionizing radiation —

Part 1: General principles

Radioprotection — Critères minimaux pour la spectroscopie par résonance paramagnétique électronique (RPE) pour la dosimétrie rétrospective des rayonnements ionisants —

Partie 1: Principes généraux

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 2, *Radiological protection*.

A list of all parts in the ISO 13304 series can be found on the ISO website.

This second edition cancels and replaces the first edition (ISO 13304-1:2013), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— inclusion of bibliographic references in the text;

— informative reference to ISO 13304-2 added;

— update of Bibliography.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Electron paramagnetic resonance (EPR) has become an important approach for retrospective dosimetry in any situation where dosimetric information is potentially incomplete or unknown for an individual. It is now applied widely for retrospective evaluation of doses that were delivered at considerable times in the past (e.g. EPR dosimetry is one of the methods of choice for retrospective evaluation of doses to the involved populations from the atomic weapon exposures in Japan and after the Chernobyl accident) and has received attention for use for triage after an incident in which large numbers of people have potentially been exposed to clinically significant levels of radiation[1] to [12]. Various materials may be analysed by EPR to provide information on dose[13] to [41]. Thus, EPR is a versatile tool for retrospective dosimetry, pertinent as well for acute exposures (past or recent, whole or partial body) and prolonged exposures. Doses estimated with EPR were mainly used to correlate the biological effect of ionizing radiation to received dose, to validate other techniques or methodologies, to manage casualties, or for forensic expertise for judicial authorities[42]. It uses mainly biological tissues of the person as the dosimeter and also can use materials from personal objects as well as those located in the immediate environment. EPR dosimetry is based on the fundamental properties of ionizing radiation: the generation of unpaired electron species (often but not exclusively free radicals) proportional to absorbed dose. The technique of EPR specifically and sensitively detects the amount of unpaired electrons that have sufficient stability to be observed after their generation; while the amount of the detectable unpaired electrons is usually directly proportional to the amount that was generated, these species can react, and therefore, the relationship between the intensity of the EPR signal and the radiation dose needs to be established for each type of use. The most extensive use of the technique has been with calcified tissue, especially with enamel from teeth[43] to [50]. An IAEA technical report was published on the use for tooth enamel[51]. To extend the possibility of EPR retrospective dosimetry, new materials possibly suitable for EPR dosimetry are regularly studied and associated protocols established. This document is aimed to make this technique more widely available, more easily applicable and useful for dosimetry. Specifically, this document proposes a methodological frame and recommendations to set up, validate, and apply protocols from sample collection to dose reporting. The application of this document to ex vivo human tooth enamel dosimetry is described in ISO 13304-2[52].

Radiological protection — Minimum criteria for electron paramagnetic resonance (EPR) spectroscopy for retrospective dosimetry of ionizing radiation —

Part 1: General principles

1 Scope

The primary purpose of this document is to provide minimum acceptable criteria required to establish procedure of retrospective dosimetry by electron paramagnetic resonance spectroscopy and to report the results.

The second purpose is to facilitate the comparison of measurements related to absorbed dose estimation obtained in different laboratories.

This document covers the determination of absorbed dose in the measured material. It does not cover the calculation of dose to organs or to the body. It covers measurements in both biological and inanimate samples, and specifically:

a) based on inanimate environmental materials like glass, plastics, clothing fabrics, saccharides, etc., usually made at X-band microwave frequencies (8 GHz to 12 GHz);

b) in vitro tooth enamel using concentrated enamel in a sample tube, usually employing X-band frequency, but higher frequencies are also being considered;

c) in vivo tooth dosimetry, currently using L-band (1 GHz to 2 GHz), but higher frequencies are also being considered;

d) in vitro nail dosimetry using nail clippings measured principally at X-band, but higher frequencies are also being considered;

e) in vivo nail dosimetry with the measurements made at X-band on the intact finger or toe;

f) in vitro measurements of bone, usually employing X-band frequency, but higher frequencies are also being considered.

For the biological samples, the in vitro measurements are carried out in samples after their removal from the person or animal and under laboratory conditions, whereas the measurements in vivo are carried out without sample removal and may take place under field conditions.

NOTE The dose referred to in this document is the absorbed dose of ionizing radiation in the measured materials.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
retrospective dosimetry (including early or emergency response)
dosimetry, usually at the level of the individual, carried out after an exposure using methods other than the conventional radiation dosimeters

3.2
electron paramagnetic resonance
EPR
electron spin resonance
ESR
magnetic resonance technique which is similar to nuclear magnetic resonance (NMR) but based on the net spin of unpaired electrons, such as free radicals and electron defects centers in matrices

Note 1 to entry: The terms EPR and ESR are essentially equivalent and are widely used. The term electron magnetic resonance (EMR) also sometimes is used because it is analogous to nuclear magnetic resonance (NMR).

3.3
radical/paramagnetic centre
species with unpaired electron(s)

Note 1 to entry: Paired electrons have the same quantum state except for opposite spins; unpaired electrons do not have a "partner" with the opposite spin. When the unpaired spin is on a molecule, it is usually termed a radical; when the unpaired electron is in a matrix, it often is termed a paramagnetic centre.

3.4
in vivo measurement
measurement carried out within the living system, such as measurements of *paramagnetic centres* (3.4) in teeth within the mouth

3.5
in vitro measurement
measurement carried out on materials outside the organism

Note 1 to entry: The term ex vivo also has been used in the literature for sample measured in vitro but irradiated within the organism.

3.6
quality assurance
planned and systematic actions necessary to provide adequate confidence that a process, measurement, or service satisfies given requirements for quality

3.7
quality control
planned and systematic actions intended to verify that systems and components conform with predetermined requirements

4 Confidentiality and ethical considerations

All individual identifying information of persons who provided samples should not be attached to the information on the samples and kept only in a secured place. The corresponding samples should be identified by codes with indication only of parameters that are useful for scientific purposes and for making decisions. Data linking the code to the person can be kept if they are done so in a secure manner, with access limited to the persons in charge of the data.

Where appropriate, permission for obtaining and measuring the samples should be obtained under the rules of the jurisdiction where the samples are obtained.

5 Laboratory safety requirements

5.1 Magnetic field

With conventional EPR spectrometers, the magnetic field (for EPR signals with g-factor near 2,0, typically 350 mT for X-band and 1 200 mT for Q-band) is restricted to the region between the pole caps of the magnets, and therefore, there is no associated health risk (can affect watches or credit cards if brought very close to the pole gap).

Due to the open nature of some in vivo EPR spectrometers, the magnetic field (for EPR signals with g-factor near 2,0, 40 mT for L-band) combined with large gaps between the poles has the potential to project the 0,5 mT line beyond the confines of the room. This line needs to be determined and appropriate shielding placed for areas that exceed this limit and that are accessed by the general public. The establishment of the 0,5 mT limit is based on concerns about potential effects on pacemakers, which could pose a significant hazard from the magnetic fields that are employed with open in vivo EPR spectrometers. The conventional limit is 0,5 mT (which is very conservative) and surveys should be made to confirm that this field is not exceeded where a person with a pacemaker could be positioned[53].

Effects of modulation fields on tissues or tooth restorations are not a significant hazard.

5.2 Electromagnetic frequency

5.2.1 in vitro measurement

The configurations used for in vitro measurements have no hazard for exposure of operators, as the spectrometer usually fully constrains the microwave to the sample with no significant amount distributed outside of the resonator.

5.2.2 in vivo measurement

Measurements in vivo have the potential hazard of local heating of tissue. The operative safety limit is that established for NMR in terms of permissible rates of energy absorption. In practice, this is a potential hazard only at high incident microwave power levels — typically >1 W, which is at least a factor of 3 greater than that in existing instruments.

5.3 Biohazards from samples

Biological samples measured in vitro should be handled in conformance to the rules of the jurisdiction for routine practice for handling biological samples.

Measurements of teeth in vivo should follow the routines practiced for ordinary dentistry in regard to potential contamination from subjects to operators or other subjects.

6 Collection/selection and identification of samples

All samples should be collected in as uniform manner as possible and the circumstances of the collection noted, although this may not always be able to be controlled by the measuring laboratory. If prior coordination between the collecting and the measuring laboratories is possible, requirements about the sample collection, selection (of donors, location, or materials) and storage (sample holder, integrity of the sample and of the container, temperature, light, UV) should be given. If information about samples is available, keep record of them (this information can be about the location of the sample, origin or history of the sample, information about donor, etc.). All samples should have a unique identifying code associated with them.

7 Transportation and storage of samples

If sample collection is made in a place other than the measuring laboratory, then samples should be transported and stored under specified environmental conditions. These conditions should be coordinated between the collecting and the measuring laboratories. Conditions of transportation and storage of the sample may affect the integrity of the sample and also modify the quantity of paramagnetic centres or the nature of the paramagnetic centres in the samples. Environmental parameters such as light and other types of radiations (UV, X-rays, gamma), temperature, humidity, oxygen, sample conditionings in water or disinfectant solution, for example, contamination (e.g. dust), may significantly affect the nature and quantity of paramagnetic centres in the samples. Therefore, specific attention should be paid as to the conditions of transportation and storage to avoid or limit as much as possible the influence of environmental parameters on the samples. Details for transport and storage of tooth samples for ex vivo measurements are provided in ISO 13304-2[52].

If possible, the influence of these parameters on the radiation-induced signal line shape and intensity should be investigated to establish the optimum conditions for transportation or storage and to avoid unnecessary precautions. When samples are known to be sensitive to one or several environmental conditions or the influence of these parameters or samples is not known, it is highly recommended that precautions are taken so as to avoid conditions that could affect the samples.

Transportation conditions, including dates, ways of transportation, and mode of control of transportation conditions, should be recorded. Appropriate sample packaging should always be used to prevent sample physical damage.

Procedures to avoid X-ray exposure of the sample during airport controls should be implemented. The dose at the X-ray hand luggage control is of the order of the microgray, so it can be considered negligible for some applications. If not, when the sample is transported in hand luggage, then authorization for X-ray exemption should be obtained in advance in order to avoid hindrance at the airport security controls. X-ray dose to the hold luggage can be higher. For shipping, appropriate labelling (including a note that the package contains radiation-sensitive dosimeters and, therefore, should not be irradiated) should be used. When this is not possible, unirradiated identical control samples or dosimeters should be placed in the package.

After the samples are received, they should be stored under stable conditions and the temperature and humidity should be monitored and recorded. Exposure to light should always be avoided.

8 Preparation of samples

Sample preparation should be performed according to an established and explicit protocol. Details for creation of a protocol for ex vivo measurements of tooth samples are provided in ISO 13304-2[52].

For in vitro and ex vivo measurements, sample preparation is usually needed to accomplish several goals, including: achieving a sample size that fits in the measurement tube; reducing anisotropy; ensuring disinfection; eliminating paramagnetic impurities from the sample; drying the sample; and stabilizing the EPR signals.

When required, preparation of the sample can be done by grinding, crushing, cutting, drilling, or other mechanical treatments. During these operations, sample overheating should be avoided by using water irrigation or other cooling systems. Metal contamination of the sample can be avoided by using hard alloy tools.

Water irrigation of nails can influence the RIS and should be applied with care.

As needed, sterilization, cleaning, deproteination, and/or delipidation are performed using chemical agents. Thermal treatment (annealing, freezing) can be used to accelerate or slow down recombination of the radicals. Samples with significant amounts of moisture can be dried before the EPR measurements to improve signal-to-noise ratio.

The setup of a protocol for sample preparation shall ensure no disturbing effect of the protocol on the EPR signals (lineshape and intensity) used for dose estimation, and no generation of additional EPR

