"ALEXANDRU IOAN CUZA" UNIVERSITY OF IAŞI
FACULTY OF PHYSICS

SUMMARY OF THE PHD THESIS

CONTRIBUTIONS TO THE STUDY OF HEAT TRANSFER PHENOMENA IN NUCLEAR CALORIMETRY

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CONTRIBUTIONS TO THE STUDY OF HEAT TRANSFER PHENOMENA IN NUCLEAR CALORIMETRY

Iași, 2013

“Alexandru Ioan Cuza” University of Iaşi
To the attention of

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We hereby inform you that on the 20th of September 2013, at 10 o’clock, in the room L1, Mrs. Cristina-Elena Ioniţă (married Mironescu) will defend, in public session, the PhD. paper **"Contributions to the study of heat transfer phenomena in nuclear calorimetry"** in view of obtaining the scientific title of Doctor in the field of Physics.

The PhD. committee will include:

Chairman: **Prof. PhD. Diana Mihaela Mardare**, “Alexandru Ioan Cuza” University of Iaşi

Scientific advisor: Prof. univ. PhD. Nicoleta Dumitraşcu, “Alexandru Ioan Cuza” University of Iaşi

Advisors: Prof. Univ. PhD. Cipriana Ştefănescu, “Gr. T. Popa” University of Medicine and Pharmacy of Iaşi

Prof. Univ. PhD. Sorin-Dan Anghel, “Babeş Bolyai” University of Cluj Napoca

Prof. Univ. PhD. Irina Radinschi, “Gh. Asachi” Technical University of Iaşi

We invite you to take part in the public session of the paper presentation.

I would like to express my gratitude and thank the scientific advisor of the paper, **Prof. univ. PhD. Nicoleta Dumitraşcu**, for her constant and highly specialised support, as well as for her advice to complete a series of measurements that seemed everlasting, to **Senior Lecturer Daniel Radu**, for the entire collaboration before the training period at the Doctoral School, as well as to **Prof. PhD. Dorina Creangă** for her useful support and competent advice, especially at the beginning of the training period.

In addition, I would also like to thank the committee for their effort in assessing this paper, as well as for their suggestions to completing the paper. Last but not least, I would like to thank to all of those who, one way or another, contributed to my scientific training and to my spiritual development and made the publication of this paper possible.

"This work was supported by the the European Social Fund in Romania, under the responsibility of the Managing Authority for the Sectoral Operational Programme for Human Resources Development 2007-2013 [grant POSDRU/CPP 107/DMI 1.5/S/78342]"and I would like to thank the entire team of the project for their financial support.

Iaşi, September 2013

The Author

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INTRODUCTION

The graphite calorimeter is one of the measuring tools for radiation doses, being used both as portable tool and as primary standard in the largest metrology laboratories of the world.

After the efficient design and creation of one of the most efficient meters of this type, namely the 3-body differential calorimeter, nuclear calorimetry benefited from special interest, being an independent branch among dosimetric methods, due to its advantages as compared to the other methods. The 3-body Domen-type graphite calorimeter quickly surpassed the other types of nuclear calorimetry as it has better performances in terms of equivalent manufacturing costs. As soon as this type of nuclear calorimeter was designed and manufactured by its inventor, the researcher Steve R Domen of NBS (National Bureau of Standards), USA, in the `70s, it was the study subject of numerous research, both experimental and theoretical, and as such this type of calorimeter is currently a reference tool in the field of radiation dose metering.

The need for studying nuclear calorimetry in general and graphite calorimetery in particular is argued as follows:

- one of the reasons refers to the practical/experimental needs, namely that the achievement of better results in the field of radiotherapy implies the administration of more accurate radiation doses to the affected tissues (usually a malignant illness) and, therefore, requests their most accurate measurement. Or, a nuclear graphite calorimeter used as primary standard offers the possibility for experimentally assessing the radiation doses absorbed with a very high precision and, in addition, allows the accurate measurement of other tools (such as, for instance, ionization chambers, that can be used in turn as auxiliary standards, etc.). Nowadays, portable graphite nuclear calorimeters have already been designed and manufactured, which led to ruling out the disadvantage of using this type of tool „on the spot" because of the sizes and weight, not only of the meter itself, but also of the auxiliary installations that serve such a tool;

- the second reason refers to the special and integral field of physics, namely Metrology (from fr. Métrologie, cf. gr. *metron* – measure and *logos* – science), regarded as a part of physics that deals with the theory and practice of accurate measurements, with establishing systems of measurement units, of measurement methods, of standards and standardization systems, including the quasi-totality legal and administrative activities related to measurements, calibrations, meters and measurement tools, as well as the monitoring of their use in economic activities.

The novelty in approaching such problems is the initiation of the preoccupations related to such a tool (a Domen-type graphite nuclear calorimeter) in our country as well, at least in terms of theory and modelling, since the practical work of this tool-type (primary standard) implies considerable financial resources and has yet to be achieved, and there is no such tool in neither laboratories in the country. Therefore, it can be modestly considered that the research work of this paper is a work of pioneering in our country and we hope that it will lead to the future development of this area of research in Romania as well.

The main purpose of the thesis is to study heat transfer phenomena that take place in a 3-body Domen-type graphite calorimeter, in order to decrease the systematic errors in measuring the absorbed radiation doses, by introducing (defining and effectively calculating) additional correction factors. Therefore, it was defined and established the correction factor caused by heat losses due to heat radiation on the absorbent's (core) surface of such a calorimeter and it was recalculated – by taking into consideration certain effects untested so far – the correction factor caused by heat losses through the connexion wires of the thermistors installed in the core. In addition, it is approached the problem of temenvironperature gradients that occur in the bodies of the calorimeter (and especially in the core) during its electrical calibration, both by the method of the analytical calculation (where possible), and by the numeric modelling of heat transfer phenomena implied by using the finite element method. This is applied both in the more simple cases, of the mono- and two-dimensional geometry (with rectangular symmetry), and in the real case, of 3D geometry.

The paper has 222 pages divided into six chapters, introduction, conclusions, bibliography and the list of the scientific papers of the PhD student. The contents of the paper is mainly represented by the original scientific discoveries of the author, achieved both independently and in collaboration and structured into eight published scientific articles, accepted for publication or sent to publishing both in specialised Romanian magazines (three), and foreign ones (five). The author considered from the beginning that the paper would be a material that could be understood, to the extent possible, only based on the data presented in the paper. This led to the need to include in the paper monographic parts in order to achieve a quasi-exhaustive presentation of the scientific framework in which research has been made, whose results are presented in the PhD paper, as well as in order to provide the scientific-information basis necessary to understand the content of the following chapters.

The information presented in the paper and the argument developed throughout the paper are supported by 90 figures and 23 tables.

In *chapter 1* it is presented the basic information related to the fundamental features and mechanisms for generating ionizing radiation (with an emphasis on the indirectly ionizing radiations), the general aspects of the interaction of indirectly ionizing radiations with the substance, the main local and systemic effects of the ionizing electromagnetic radiation over the biological systems, as well as the basic elements related to radiotherapy and to the physical, chemical and biological effects of radiotherapy.

In *chapter 2* it is presented the calorimetric method for measuring the radiation dose. Following certain basic elements related to this topic (presented in the first subchapter) it is presented information related to the use of a nuclear graphite calorimeter as primary standard, as well as the concepts of primary standards and secondary standard, the most used nuclear calorimeters are described, and the chapter ends with the presentation of the main techniques used for measuring the absorbed radiation dose, namely the irradiation and the electrical calibration of a 3-body graphite calorimeter.

In *chapter 3* it is presented one of the most often used types of nuclear calorimeters, namely the Domen-type graphite calorimeter. Therefore, the structure and the running principle of this calorimeter are presented, as well as the materials and the devices used in its construction, the heat transfer phenomena that occur in this type of calorimeter, and the essential elements related to heat measurement and control.

In *chapter 4* it is presented a new theoretical model for the 3-body Domen graphite calorimeter, called RHLC (Radiative Heat Loss Correction), which takes into consideration the „basic equations" that describe the thermal behaviour of the calorimeter. Therefore, there were introduced and defined new heat transfer coefficients (called by the author „low heat transfer coefficients"), as well as the corresponding correction factors, a new correction factor is defined and determined for the absorbed radiation dose, namely the correction factor caused by the heat losses through heat radiation.

Chapter 5 includes (the same as the other chapters) four subchapters, in which there are presented the basic elements related to heat conduction via the connection wires of the thermistors of a graphite calorimeter. There are approached theoretical models that describe heat transfer at the level of the thermistors' connection wires, which allow the recalculation of the correction factor and of the heat losses via the connection wires of the thermistors (heating and sensing) installed in the core, losses by Joule effect and the corresponding correction factor, as well as the effect of rolling the connection wires of the thermistors around the core.

Chapter 6 analyses the influence of the temperature gradients in a Domen-type graphite nuclear calorimeter, necessary for validating the comparative measuring technique of the absorbed radiation dose, namely the electrical calibration of the calorimeter. This is achieved within three theoretical models, namely: one-dimensional model, two-dimensional model with rectangular geometry and three-dimensional model, which considers both the real absorbent (core) real shape and the entire 3D geometry of the calorimeter.

The paper contains a chapter called *Conclusions*, which includes the brief presentation of the paper's entire contents, with an emphasis on the main original results obtained by the author, bibliography and the list of the published scientific papers, ongoing publication and sent to publication, as well as of the scientific papers with which the PhD student participated in some national and international scientific events.

CHAPTER 1

THE INTERACTION OF THE IONIZING ELECTROMAGNETIC RADIATION WITH THE SUBSTANCE

1.1. Fundamental features and mechanisms for the generation of ionizing radiations

1.1.1. Ionizing and non-ionizing electromagnetic radiations

From point of the view of the interaction mean of the electromagnetic radiations with the substance, they can be of two types [65, 100]:

1) *non-ionizing* electromagnetic radiations and
2) *ionizing* electromagnetic radiations.

In 1971, ICRU (International Commission on Radiation Units) advised on the use of a specific terminology that would take into account the different interaction method with the substance of the electrically charged radiations as compared to the electrically neutral ones. Therefore, all the charged particles are called *directly ionizing* radiations, and the neutral ones (among which there can be photons – quantum of the electromagnetic field) are called *indirectly ionizing* radiations.

1.1.2. Generation mechanisms of the ionizing radiations

The X radiation is also called *Röntgen radiation*, after the name of the German physicist Wilhelm Conrad Röntgen who discovered it in 1895. *X* radiations are electromagnetic radiations with a wave length lower than 10 *nm*, which means that the photon energy – quantum of the corresponding electromagnetic fields – have to be larger than $124\ eV$, but larger than $1pm$ (which corresponds to the rough *X* radiation and even to some γ radiations – the ones emitted by the disintegration products of radium).

X radiations can be of two types [84, 88, 92, 95-96, 100]:

i) *breaking radiations* (or „*Bremsstrahlung*") and
ii) *characteristic radiations*

1.1.2.1. *X* radiation generation mechanisms

i) Breaking radiations

They are produced by breaking the charged particles in the Coulombian field of the atomic cores. The production probability of this process is proportional to the kinetic energy of the electrically charged particle and to the *Z* atomic number of the target material, being inversely proportional to the particle mass.

ii) Characteristic *X* radiations

The *X* characteristic radiation is achieved when a high energy electron, which attacks a target, releases from the atom, by „collision" (in fact, through Coulombian interaction) an electron from in-depth layers. The available spot is taken by an electron from a higher layer and consequently a well-determined energy photon is emitted, which „takes over" the energy excess. There is a „vacant" spot that is occupied by another electron from a higher layer while the process continues by „falling" until the energetic balance is restored [65]. Apart from the mechanisms above, characteristic *X* radiations (of a certain radionuclide) can also be obtained by the processes of [65] *electronic captioning* and/or *internal conversion.*

1.1.2.2. γ radiation emission mechanism

Gamma radiation (γ) represents high frequency electromagnetic radiations that induce ionization processes [53, 60]. These radiations have a biological risk and are usually produced by specific disintegration (called γ disintegration) of the atomic cores (in fact, it is a deexcitation process of certain atomic cores, which are in high energy state), but they can be created via other processes as well (for instance the annihilation particle-counter particle) [65].

The γ radiation was discovered by the French physicist and chemist Paul Villard in 1900, while he was studying the radiations emitted by radium during its γ disintegration. The radiation discovered by Villard was then named by Ernest Rutherford „gamma radiation" in 1903.

The standard values of γ radiation is higher than $10\ EHz$, the corresponding wave length is lower than $10\ pm$ (less than the diameter of an atom), and the energy of the corresponding photons is higher than $100\ keV$.

One of the γ radiation sources that is most often used in medicine is the radionuclide ^{60}Co, usually prepared as capsules and under various shapes according to necessities. The disintegration diagram of this radionuclide is presented in Fig. 1 [65]. First of all, ^{60}Co disintegrates β in ^{60}Ni via the electron emission with the energy $0{,}31\ MeV$, then the nuclide ^{60}Ni in an excited state returns to the fundamental energy state and emits two successive γ quantum, with energies between $1{,}17\ MeV$ and $1{,}33\ MeV$.

This process occurs in 99.88% of the total disintegration events.

$$
\begin{aligned}
{}^{60}_{27}Co &\rightarrow {}^{60}_{28}Ni^* + e^- + \bar{\nu}_e + \gamma\ (1{,}17\ MeV) \\
{}^{60}_{28}Ni^* &\rightarrow {}^{60}_{28}Ni \qquad\qquad + \gamma\ (1{,}33\ MeV).
\end{aligned}
$$

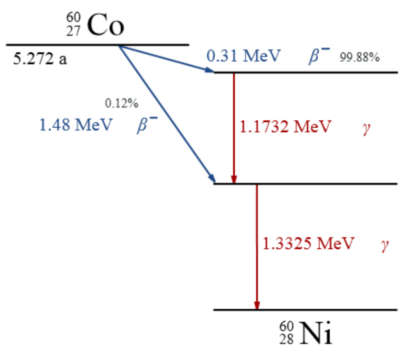

Fig. 1. The disintegration diagram of the most used γ-active radionuclide, ^{60}Co [65]

In the extended version of the paper, there are presented: the scales particular to ionizing radiations, the sources of indirectly ionizing radiation, the scales typical to the ionizing radiation sources, the detection of the ionizing radiations, the interaction of the indirectly ionizing radiations with the substance.

In the category of indirectly ionizing radiations there are also all the particles neutral from electrical point of view [65]: X and γ photons, neutrons, π^0 mesons, other neutral particles. Due to the fact that they do not have electric load, these radiations do not „feel" the presence of the Coulombian field of the cores or of the atomic electrons, and therefore they do not interact with the substance via multiple Coulombian spreading processes or Coulombian interactions with atomic cores.

In the paragraph 1.2. there are presented concepts and basic scales of the dosimetry of ionizing radiations, namely field radiometric scales, the description of the radiation field via non-stochastic scales, scales and fundamental relations in dosimetry. In addition, there are also presented scales specific to the interaction of the ionizing radiations with the substance, namely the relative biological efficiency, linear energy transfer, specific ionizing, scales specific to radioprotection and the ICRP dose limitation system.

1.3. Local and systematic effects of the ionizing electromagnetic radiation over biological systems

According to the occurrence time, biological effects are classified into [65]:

- *immediate* biological effects,
- *early* biological effects,
- *late* biological effects.

The immediate biological effects occur only after a couple of minutes or hours, the early ones after certain days, weeks or even months following irradiation, and the late ones after several years (usually as cancer) or even centuries (genetic effects, on heirs).

Another classification distinguishes between the biological effects of the ionizing radiations:

- *somatic* biological effects (they occur on irradiated persons),
- *genetic* biological effects (occur on heirs);

A classification that takes into consideration the purpose of radioprotection classifies the effects of the radiations into: *stochastic* (casual) and *non-stochastic* (necessitarian).

1.3.1. The mechanisms of producing biological effects of ionizing radiations.

In order to explain the occurrence mechanism of the biological effects of ionizing radiations, two theories were suggested, namely *the target theory* and *the free radical theory*.

The occurrence types of the biological effects, namely early (somatic) effects and late effects/occurrences.

In the extended version of the paper there are presented basic elements related to the action of ionizing radiations at molecular, cellular and tissue level:

1.4. Radiotherapy. Physical, chemical and biological effects

The first therapeutic use of ionizing radiations dates back to 1896, almost immediately after the discovery of *X* radiations. For more than one century radiotherapy continued to play an important part in treating cancers, benefiting from considerable technical progress and designed as one of the most important local and regional treatments of cancer, from which more than 50% of the patients with malignant illnesses suffer.

1.4.1. Radiotherapy –treatment method in oncology

Radiotherapy is the medical therapy that uses ionizing radiations. It is used in the treatment of various cancer types, being less involved in the pathology of non-oncologic illnesses.

1.4.2. Physical, chemical and biological effects of radiotherapy

Radiotherapy is a method that transfers a considerable amount of energy over tissues, based on which important biological lesions can occur. The purpose of radiotherapy consists of supplying a dose high enough to treat the tumour, but only up to the limit where healthy/normal organs in the nearby could be affected.

The concepts presented in this chapter are summarized below:

(i) there are presented the fundamental features and the generation mechanisms of the ionizing radiations, with an emphasis on the indirectly ionizing radiations; thus, it was shown that X radiations can be of two types: breaking („bremsstrahlung”) and characteristic (resulted via electronic captioning and internal conversion), stating in every case the way in which these radiations have been generated, as well as the traits of every particular radiation spectrum and the generation mechanism of gamma radiations is also presented;

(ii) there are presented the main scales corresponding to ionizing radiations, with an emphasis on the indirectly ionizing radiations (the quality of radiations, free road medium, the semi-attenuation thickness, the deci-attenuation thickness, the linear attenuation coefficient, the linear energy transfer);

(iii) the most important indirectly ionizing radiations are presented and classified according to two criteria: based on origin and based on the type of emitted radiation;

(iv) the scales corresponding to the sources of ionizing radiations are presented (the activity, the half-life period, volume activity, superficial activity, mass activity);

(v) there are defined: kerma (as dosimetric scaled defined for the directly ionizing radiations only), the kerma debit and the constant of the kerma debit in the air;

(vi) there are presented the main six types of detectors used for the indirectly ionizing electromagnetic radiations;

(vii) the two stages of the interaction process of the indirectly ionizing radiations with the substance are presented and there are defined: the linear (total) coefficient of attenuation, the linear attenuation coefficient, the mass attenuation coefficient, the mass (linear) coefficient for energy transfer and the mass (linear) coefficient of the energy absorption.

(viii) there are presented the concepts and basic scales of the dosimetry of ionizing radiations (the concept of field of ionizing radiations, particle flow, particle fluency, energy flow, energy fluency, energy fluency flow);

(ix) there are presented the scales and the fundamental relations in the field of dosimetry (the average absorbed energy and the absorbed dose, the specific absorbed energy, the flow of the absorbed dose, kerma, kerma flow, kerma's constituents, average ionizing energy, exposure, exposure flow, mass start/stop power);

(x) there are defined the scales specific to the interactions of the ionizing radiations with the substance (relative biological efficiency, linear energy transfer, collision stop power, specific ionizing, average specific ionizing), as well as the scales specific to radioprotection (dose equivalent, radiation's quality factor, equivalent dose, effective dose, employed effective dose, collective effective dose);

(xi) there are presented the main local and systemic effects of the non-ionizing electromagnetic radiation over the biological systems, according to the data published by „National Radiological Board", „National Protection Board" and „National Radiological Protection Board" of England [118];

(xii) there are presented the main local and systemic effects of ionizing electromagnetic radiation over the biological systems and these effects are classified according to three criteria: based on the occurrence moment (into immediate, early and late effects), based on the category of people on which these effects occur (into somatic and genetic effects) and based on a criterion specific to radioprotection (into non-stochastic and stochastic effects);

(xiii) there are presented the production mechanisms of the biological effects of ionizing radiations (theory of direct action (or target theory) and the theory of indirect action (or the free radical theory), in the latter case highlighting the vital part of the water radiolysis, taking into consideration that water is a fundamental constituent of any biological living tissue – for instance, the living and healthy tissue consists of almost 70% water, certain organs/tissues having a higher percentage of water, reaching almost 90%), as well as the two relation types between dose and effect: linear relation (dose without threshold) and sigmoid relation (dose with threshold);

(xiv) there are presented the manifestation types of the biological effects of the ionizing electromagnetic radiations on the human body (early (somatic) effects: radiodermititis, eye lesions (radio-induced cataract), gonad lesions, lesions of the human embryo, as well as late effects: radio-induced effects, chronic radiodermititis, post-radiotherapy somatic effects, genetic effects, shortening of the average life span, etc.);

(xv) there are presented the specific actions of the ionizing radiations at three major levels: molecular (at the level of the nucleic acids and on the proteins), cellular (Bergone law and Tribodeau law) and tissue level;

(xvi) it is defined the concept of radiotherapy as method for treating cancer;

(xvii) the main purpose, as well as the secondary purposes of radiotherapy are presented;

(xviii) the biological mechanism is presented (at cellular level), which ensures the efficiency of radiotherapy;

(xix) the main types of radiotherapy currently used in the specialised clinics in this type of treatment are presented:

- teleradiotherapy;
- brachytherapy;
- total body radiotherapy;
- total cutaneous radiotherapy;

(xx) the sources of ionizing radiation are listed and classified and the ones with radiotherapeutical purpose in particular;

(xxi) the main physical features of radiation used in radiotherapy are presented;

(xxii) the physical, chemical and biological effects of radiotherapy are presented.

CHAPTER 2

BASIC ELEMENTS IN NUCLEAR CALORIMETRY

2.1. The dosimetric method of nuclear calorimetry

Calorimetric dosimetry is, the same as in the case of the ionizing one, a slightly old measuring method, the first nuclear calorimeters being created at the beginning of the last century.

From point of view of the *interaction means* of the ionizing radiation bean, there are four main nuclear calorimeters [65], namely:

- *total absorption calorimeters* (measure the fluency of the beam);
- *dosimetric calorimeters* (measure the absorbed dose); the incident beam is partially absorbed - this is the case for Domen-type graphite calorimeter;
- *radiometric calorimeters* (measure the features of the radiation source: activity, half-value life, disintegration energies etc.) - in this case the source is inserted in the calorimeter's core;
- *calorimeters used inside nuclear reactors.*

From point of view of the *calorimetric method* used, the following nuclear calorimeters exist [65]: *isothermal calorimeters, adiabatic calorimeters, quasi-adiabatic calorimeters, stationary calorimeters.*

The main *advantages* of the calorimetric method for measuring radiation doses are [65]:

- it is an absolute method (can be calibrated independently);
- it has a wide range of measurable doses (average, large and lately, as consequence of the increase of the sensitivity of the devices used and of the improvement of the equipment corresponding to a nuclear calorimeter, even of the small doses);
- it is precise and flexible in applications.

Among the *disadvantages* of the method there can be mentioned:

- slightly elevated complexity (it needs highly accurate lab equipment and a high number of auxiliary devices, depending on the type of calorimeter);
- it has lower sensitivity as compared to other calorimetric methods, thus being difficult to apply in the field of small doses;
- it needs corrections in terms of energy loss.

The main problem that a calorimetric method has to solve is the control over the heat transfer from the thermally active body to the medium (the calorimeter's jacket), via the thermal insulator, which can never be perfect. This problem of calorimetry can be solved by identifying all the heat losses channels and applying the corresponding corrections. This can be done by defining and calculating *the correction factors.*

The basic diagram of the simplest nuclear calorimeter (the 2-body calorimeter) is presented in Fig. 2 [65]. Although there are more complex and more efficient calorimeters, in order to understand the fundamentals of nuclear calorimetry it suffices to refer to this simple mode in this subchapter.

A two-body nuclear calorimeter consists of three main parts:

1) the irradiated *sample body*, also called thermally active body or absorbent;
2) *the thermally isolating medium*, which divides the irradiated sample body from the outer cover;
3) *the outer cover* or *the jacket*, for the calorimeter's core this is the medium.

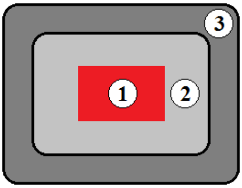

Fig. 2: The main parts of a calorimeter: 1-the thermally active body (or absorbent); 2 - the thermally isolating medium; 3 - the jacket

2.1.1. Calorimetric methods

The main problem that a calorimetric method has to solve is the control over the potential heat transfer between the thermally active body and the medium (the jacket).

The isothermal calorimeter

A control method, apparently the simplest one, is to maintain the absorbent (the thermally active body) and the jacket at constant and even temperatures (the isothermal method): $T = T_c = const.$, where T is the temperature of the thermally active body and T_c is the temperature of the jacket.

The adiabatic mean implies the control of the variation of the jacket's temperature, (T_c) so that it increases at the same time as the temperature of the thermally active body, (T) always preserving the equality $T = T_c$ (but both variable over time).

The quasi-adiabatic method is a version of the adiabatic method, developed for the purpose of controlling the residual heat losses. Within this method it is admitted the existence of a slightly low temperature difference between the jacket and the thermally active body. The temperature of the jacket, T_c, is preserved constant by means of thermostats.

The stationary method (or the heat flow method or the isothermal jacket method) implies $T_c = const.$ (by thermal control), and $T_0 < T_c$. By irradiation, the thermally active body is heated up, its temperature increases (can exceed the T_c temperature) until an equilibrium stationary state is achieved, where the power received equals the power lost to the jacket.

2.1.2. Temperature measurement and control. Electrical calibration

Temperature measurement is usually performed electrically. There can be used three categories of translators: thermocouples, metal-resistance thermometers and thermistors.

Temperature control can also be obtained electrically by using the Peltier effect. Two types of temperature control can be distinguished [65]:

i) *static control*, which implies maintaining the jacket's temperature constant, it is used in the quasi-adiabatic and the stationary methods;
ii) *the dynamic control* implies tracking the temperature of the thermally active body by the jacket's temperature (the jacket's temperature "belongs" to the temperature of the thermally active body), it is used in the adiabatic method and in the "locked" quasi-adiabatic method.

Electrical calibration

Although calorimeters allow the absolute scaling of the dosimetric sizes, this does not imply the absence of the calibration operation. For calibration there can be used calculations and/or electrical methods.

2.1.3. Thermal diffusivity

Thermal conductivity is a measure of the conduction rate of the heat. *Thermal diffusivity* α and thermal conductivity k are interconnected by the relation $\alpha = k/\rho c$, where ρ and c represent the density and the specific heat of the material. In the field of nuclear calorimetry, thermal diffusivity is usually more practical than thermal conductivity. For instance, thermal diffusivity determines the rate at which temperature gradients are diffused and dissipated in the core of a nuclear calorimeter.

2.1.4. Temperature gradients

In designing and operating calorimeters for the purpose of establishing the absorbed doses of ionizing radiations, as in the case of the other calorimeters, the effect that is difficult to anticipate and control of the temperature gradients plays a highly important part.

Since it is considered that the penetrant radiation produces even heating both in the core of a nuclear calorimeter, and in the surrounding bodies, the problem is whether during the electrical calibration of the calorimeter the same even heating of the calorimeter's body occurs.

*The heat flow theorem (*Steve R. Domen [15]*): If P is the power evenly dissipated in each of the equally caloric bodies of a two-body calorimeter (core and primary cover) so that the two bodies are included in a jacket maintained at constant temperature, then the heat change of the inner body (of the core) at any time (either heating or cooling) equals the sum of the heat changes of both bodies, when the power P is directly applied to the inner body (core) only.*

This theorem resulted from the calculation made with the purpose of controlling the behaviour of the 3-body graphite calorimeter of NBS.

The general/generalized theorem of the heat flow refers to an n -body calorimeter.

2.2. The nuclear calorimeter - primary standard tool

Nuclear calorimetry represents that part of calorimetry that is involved in the field of radiation dosimetry and especially in the dosimetry of ionizing radiation.

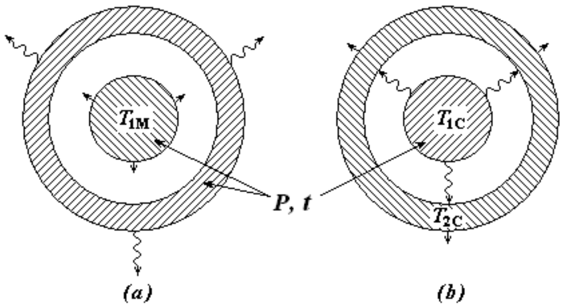

Fig. 3: Graphical representation of the heat flow theorem applied to an assembly of two bodies, for the purpose of testing the effects of temperature gradients.

Nuclear calorimetry is an integral part of Metrology and deals mainly with:

- the study of the caloric effect of ionizing radiations that interact with the substance;
- the design and practical application of equipment capable of measuring the radiation doses absorbed by various bodies/materials;
- drafting measurement methods of the absorbed radiation doses.

Since the caloric effect is a direct effect of the above-mentioned interaction and the measurement process is directly related to known physical scales, nuclear calorimetry is capable of designing and building *primary standard* measuring tools for the measurements in the field, the devices that achieve this being called *nuclear calorimeters*. In case of a primary standard, the answer to the incident radiation does not depend on various parameters of the radiation (dose rate, spectral field etc.). Moreover, the effect of the ionizing radiation whose dose must be measured on the environment used as absorbent is directly and totally measurable, so that is represents a known physical and directly measurable scale. The calibration of a primary standard is done directly, without the mediation of another measuring tool. Unlike primary tools (primary standards), measurements done with one tool */secondary standard* (for instance, ionizing chambers) depend on irradiation parameters.

2.3. Types of nuclear calorimeters

The following types of nuclear calorimeters currently exist: graphite, water, plastic - equivalent A-150, polystyrene and water, graphite and water, etc.

2.4. Techniques for measuring the radiation dose

The measurement of the radiation dose with a graphite nuclear calorimeter implies two steps, namely: (i) *irradiation* and (ii) *calibration* (usually done electrically).

In the extended version of the report there are also presented the basic elements referring to: irradiation, calibration of a 3-body graphite nuclear calorimeter, the quasi-isothermal running module of a 3-body graphite nuclear calorimeter.

The concepts presented in this chapter are summarized below:

(i) the main six dosimetric methods are listed, for each of them being indicated the sample materials used, the shield of the device used for measurements, the nature of the physical response scale, as well as the practical measurement method of the response scales;

(ii) the four main types of nuclear calorimeters are listed (isothermal, adiabatic, quasi-adiabatic and stationary);

(iii) the main advantages and disadvantages of the calorimetric method for measuring radiation doses are presented;

(iv) it is presented the central problem of any calorimetric method and there are mentioned the main methods of nuclear calorimetry, as well as the temperature measurement and control in the case of the simplest calorimeter type (the two-body calorimeter);

(v) it is highlighted the importance of the thermal diffusivity concept in comparison to the concept of thermal conductibility in the field of nuclear calorimetry;

(vi) it is presented the problem of temperature gradients, tightly connected to the two Domen's theorems (the heat flow theorem and the general heat flow theorem);

(vii) the concepts of primary standard and secondary standard are presented and it is highlighted the difference between a primary tool/standard and a secondary one;

(viii) there are presented the main types of modern nuclear calorimeters (the graphite calorimeter, the water calorimeter, the polystyrene and water calorimeter, the graphite and water calorimeter etc.)

(ix) it was presented the standard reference material in nuclear calorimetry (and not only), adopted by ICRU (International Commission on Radiation Units and Measurements) in 1969, as well as the materials with absorption properties of the radiations related to it and which are most often used;

(x) there are presented the main difficulties in building a nuclear calorimeter, namely the heat defect (especially on water calorimeters) and temperature gradients (especially on graphite calorimeters);

(xi) there are presented the main three running modules of a 3-body graphite nuclear calorimeter (quasi-adiabatic module, heat compensation module and quasi-isothermal module);

(xii) there are presented the main measuring techniques (irradiation and calibration) in nuclear calorimetry.

CHAPTER 3

THE DOMEN-TYPE GRAPHITE DIFFERENTIAL CALORIMETER

3.1. Structure and running principle

The 3-body graphite calorimeter has been designed and manufactured for the first time by Steve R. Domen, at the beginning of the '70s and it can be considered that he set the grounds of a new era of graphite-based nuclear calorimetry. S.R. Domen was the first to introduce a third body, the secondary shield, apart from the core and the primary shield, which represented the calorimeter until then (2-body), and thus it was created a new calorimeter with better properties in terms of heat loss to the environment. The general diagram of the graphite calorimeter belonging to NBS (very similar to the ENEA-IMRI one) is presented under Fig.4.

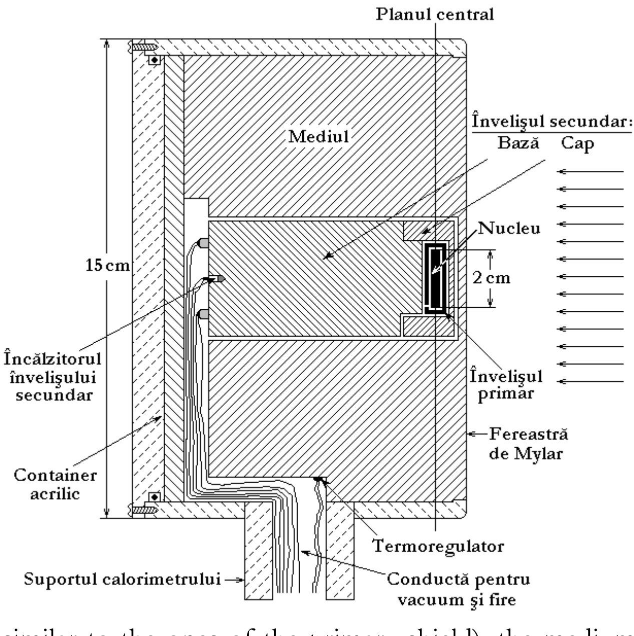

Fig. 4. Section via the NBS graphite calorimeter (and/or ENEA-INMRI), side view[15, 76]

The main constructive elements of a 3-body Domen - type graphite calorimeter are: the core, the primary shield (consisting in turn of two major elements, namely: the basis of the primary shield and the cap (or the cover) of the primary shield), the secondary shield (consisting in turn of two major elements, similar to the ones of the primary shield), the medium, the heating elements of the calorimeter's body (usually microthermistors), the medium's thermoregulator (a heating device - most often consisting of a special thermistor, whose purpose is to preserve the air temperature as constant as possible), the window (the frontal part) of the calorimeter, the radiation inlet window, the container/jacket (contains the first four main constructive elements: the core, the primary shield, the secondary shield and the medium), the wiring pipe and the vacuum pipe and the calorimeter's support.

In order to ensure the lowest heat transfer between the main elements of the calorimeter, the spaces between them are vacuumed, the usual pressure being Pa ($10^{-8}\,atm$).

The principle of the calorimetric dosimetry (in general terms, not only in the case of Domen-type calorimeters) seems simple (at first sight). The energy absorbed by the irradiated substance is transformed into its internal energy and can be identified either in the increase of the temperature of the irradiated sample body (the case of the Domen-type calorimeter) or in the change of its aggregation state (by melting or evaporation).

In the actual applications there must also be considered phenomena that appear as consequence of the action of the ionizing radiation: endothermic chemical reactions, the change of the crystal network of the irradiated sample body, heat losses (heat transfer towards the environment).

The sensitivity of the method is slightly low: 50 of cGy absorbed lead to a change in temperature of the irradiated substance by almost $\Delta T = 10^{-4}\,^{\circ}C$. In other words, the thermal effect is rather low, and the method can be easily applied in the field of average and large doses. However, as consequence of the technological progress recorded during the last decades, the calorimetric method can currently be successfully applied even in the field of small doses.

3.2. Materials and devices used to build the calorimeter

The usual materials used to build a 3-body Domen-type graphite calorimeter are the following: graphite (the primary material, all the four constructive elements that are essential to the calorimeter are built out of it, namely: the core, the primary shield, the secondary shield and the medium), Mylar and the aluminium Mylar (it is used for the calorimeter's window, as electrically isolating element upon the installation of the thermistors, namely for sheathing the various surfaces of the calorimeter's body, in order to reduce heat losses by reflection), KAPTON (it is used to make a special film that will cover the inlet window of the ionizing radiation in the calorimeter), PERSPEX (poly(methyl) methacrylate is an acrylic thermoplastic material, transparent, also called “acrylic glass” used to make the calorimeter’s jacket, its support etc.), polystyrene (it is used to manufacture supports - isolating from thermal point of view - of the core), epoxydic resin (used as glue - which must both a good electrical insulator, used to solder the alloy wires $Pt - Ir\ (10\ \%\ Ir)$ of the thermistor on the enamelled copper wires), the enamelled copper wires (they are used on all electrical circuits inside the calorimeter), ceramics (used to install the secondary shield), asbestos (it is used as thermal insulator of the medium).

The main *devices* used to build the 3-body Domen-type graphite calorimeter are the thermo-elements that allow the control of the temperature on all the four major constructive elements of the calorimeter. In general, the core and the primary shield are heated up by using the thermistors (of various sizes adapted to the sizes of these bodies) and the secondary shield and the medium are heated up by using highly-accurate resistors. Sometimes however, the secondary shield is also heated by thermistors.

3.2.1. The definition of the δ and τ constants of a thermistor

- *The dissipation constant* of the thermistor, δ, is defined as the relation, at a given air temperature, T_0, of a certain variation of the dissipated power in the thermistor and the variation of the thermistor's body temperature variation caused by this power variation, T. The size δ is not a proper constant, but it varies by T_0, and $(T - T_0)$ as well.
- *The time thermal constant* of the thermistor, $\tau = C/\delta$ (where C is the caloric power of the thermistor) equals the time necessary for the thermistor to reach 63.2 % of the temperature difference when it is subject to a temperature change, at the null dissipated power[1]. In the extended version of the report, under the paragraph

3.2.2. The experimental determination of the constants δ and τ of a thermistor

are presented as measurement methods for the values of the two constants of a thermistor, both in static air, and in graphite. For a "VECO ultra-small bead 41A14" thermistor, the following values were obtained:

$$\delta_{sa} = 0.0902\ mW/^{\circ}C \pm 0.35\% ; \quad \delta_g = 0.8651\ mW/^{\circ}C \pm 0.35\%$$

$$\tau_{sa} = 0.4989\ s \pm 0.32\% ; \quad \tau_g = 0.0048\ s \pm 0.32\% .$$

For this purpose, the circuits presented under Fig.5 were used:

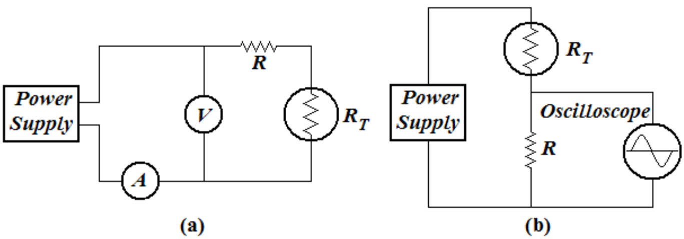

Fig. 5: Testing circuits (a) for the measurement of dissipation constants and (b) for the measurement of time thermal constants with low and very low values[93]

3.3. Heat transfer phenomena in the Domen-type graphite calorimeter

The design of the graphite calorimeters depends on the prior knowledge of the heat transfer mechanisms between the various bodies of the calorimeter, on the relations between the heat exchange and the corresponding temperature variations, relations implied by these mechanisms, as well as on the measurement and control

[1] Usually the null (negligible) dissipated power condition is given by the inequality $R(T)/R_0(T) > 0.999$.

methods of the temperature. As it is widely known, there are three different heat transfer phenomena, namely: *convection* (free or natural), *conduction* and *thermal radiation*. In the extended version of the paper there are presented: the natural (free) convection, the thermal conduction and the thermal radiation. In addition, under the paragraph **Heat transfer phenomena inside a 3-body Domen-type graphite calorimeter**, there are presented (only in the extended version of the report) the three heat transfer phenomena, as well as their relative importance within a Domen-type nuclear calorimeter.

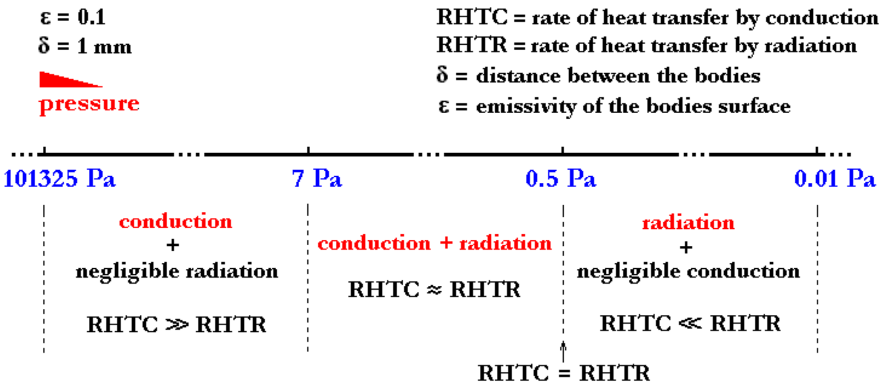

Fig. 6: Diagram of the connection between the air pressure value and the dominant phenomenon via which the heat transfer takes place

3.4. Temperature measurement and control

Temperature measurement is usually done electrically. *Temperature control* can be done either electrically or by using the reverse thermo electrical effect.

Electrical calibration

Although calorimeters allow the absolute measurement of the dosimetric scales, the calibration operations are still necessary. For the calibration stage there are used particular calculations, as well as electrical methods. Calculations are based on the fundamental equation of calorimetry, which must be applied for each type of calorimeter and imply the accurate knowledge of other constants of the tool: the caloric capacities of the calorimeter's body, the heat transfer coefficients between the calorimeter's bodies etc. Electrical calibration is a completely independent method.

The notions presented in this chapter are summarized below:

(i) the main 11 constructive elements of a 3-body Domen-type graphite calorimeter are listed;

(ii) the practical electrical calibration method for a 3-body Domen-type graphite calorimeter is presented;

(iii) there are mentioned the materials and the devices used to build a 3-body Domen-type graphite calorimeter;

(iv) there are presented the main physical properties of the materials and devices used to build the 3-body Domen-type graphite calorimeter from ENEA-INMRI (Italy);

(v) it is presented the practical, experimental method used for identifying the two main constants of a microthermistor, namely the dissipation constant and the time thermal constant, both in static air and in graphite;

(vi) there are briefly presented the three heat transfer phenomena (natural convection, conduction and thermal radiation) and consequently, based on the analysis on the corresponding particular conditions of a 3-body Domen-type graphite calorimeter, there are presented the heat transfer phenomena that can take place more efficiently in such a calorimeter (namely the heat radiation from the surface of the calorimeter's body and the conduction through the connection wires of the thermistors, epoxydic resin, Mylar film, etc).

CHAPTER 4

THE MODEL WITH LOSSES CAUSED BY HEAT RADIATION OF THE DOMEN-TYPE GRAPHITE CALORIMETER

4.1. Correction factors of the absorbed radiation dose

In order to provide the most accurate value of the absorbed radiation dose, measured by means of a calorimeter, there are defined the correction factors of the absorbed radiation dose, corresponding to the real dose of the identified absorbed radiation.

In general, an F_c correction factor of the absorbed radiation dose is defined as the relation between the value of the measured dose by taking or not into consideration that correction

$$F_c = \frac{D_{corr}}{D},$$

so that the value of the measured dose by taking into consideration the corresponding correction D_{corr} „ considered the real value of the absorbed radiation dose, $D_r \equiv D_{corr}$, will be given by the relation

$$D_r = F_c D.$$

Examples of correction factors:

- the correction factor due to the vacuumed spaces between the calorimeter's body;
- the correction factor due to the difference between the intensity of the ghost and the calorimeter's intensity;
- the correction factor due to the air decrement;
- the correction factor due to the gradient of the dose absorbed in the core;
- the correction factor due to the Kaptopn film located in front of the calorimeter;
- the correction factor due to the protection board of the calorimeter's front;
- the correction factor due to the distance between the radiation source and the defined standard reference point;
- the correction factor due to the difference of heat transfer between the electrical calibration and the irradiation;
- the correction factor due to the measuring depth, etc.

4.2. The RHLC model („**R**adiative **H**eat **L**oss **C**orrection")

The simplest theoretical model of a 3-body Domen-type graphite calorimeter is presented „in extenso" in the reference paper of Steve R. Domen [18].

The RHLC model is based on the following system of differential equations (in which the author introduces the terms marked with red):

$$\begin{cases} C_1\dot{T}_1 = P_1 - K_1(T_1 - T_2) - e_1\left[(T_M + T_1)^4 - (T_M + T_2)^4\right], \\ C_2\dot{T}_2 = P_2 + K_1(T_1 - T_2) - K_2(T_2 - T_3) + e_1\left[(T_M + T_1)^4 - (T_M + T_2)^4\right] - \\ \qquad e_2\left[(T_M + T_2)^4 - (T_M + T_3)^4\right], \\ C_3\dot{T}_3 = P_3 + K_2(T_2 - T_3) - K_3 T_3 + e_2\left[(T_M + T_2)^4 - (T_M + T_3)^4\right] - e_3\left[(T_M + T_3)^4 - T_M^4\right], \end{cases}$$

where $T_M = T_0 + T_{medium}(^\circ C) = 273{,}15 + 25 = 298{,}15\ K$ is the constant temperature of the medium in Kelvin and

$$\begin{cases} e_1 \equiv e_{c-j} = \sigma A_c F_{c-j} = \sigma A_c \Big/ \left[\varepsilon_g^{-1} + \left(A_c / A_j^{int}\right)\left(\varepsilon_{My}^{-1} - 1\right)\right], \\ e_2 \equiv e_{j-sh} = \sigma A_j^{ext} F_{j-sh} = \sigma A_j^{ext} \Big/ \left[\varepsilon_g^{-1} + \left(A_j^{ext} / A_{sh}^{int,\,My}\right)\left(\varepsilon_{My}^{-1} - 1\right)\right], \\ e_3 \equiv e_{sh-m} = \sigma A_{sh}^{ext,\,My} F_{sh-m} = \sigma A_{sh}^{ext,\,My} \Big/ \left[\varepsilon_{My}^{-1} + \left(A_{sh}^{ext,\,My} / A_m^{int,\,My}\right)\left(\varepsilon_{My}^{-1} - 1\right)\right]. \end{cases}$$

For the particular geometry of the 3-body graphite calorimeter of ENEA-INMRI, Rome, Italy, by considering the matrix elements, $e_i,\ i = 1,\ 2,\ 3$ the following values were identified:

$$e_1 = 0{,}232 \times 10^{-11}\ W \cdot K^{-4},\ e_2 = 0{,}344 \times 10^{-11}\ W \cdot K^{-4},\ e_3 = 1{,}157 \times 10^{-11}\ W \cdot K^{-4}.$$

The meaning of the above-mentioned scales is presented in the extended version of the report.

4.3. The "reduced" heat transfer coefficients

Based on the system of differential equations in the previous subchapter, new heat transfer coefficients can be defined, $K_i^*,\ (i = 1,2,3)$ as well as new correction theoretical/calculated factors, due to heat losses, F_C^* and F_{C+J}^*, by the relations

$$K_1^* \overset{def.}{=} \frac{P_1}{T_1(\infty) - T_2(\infty)} - \frac{e_1\left\{[T_M + T_1(\infty)]^4 - [T_M + T_2(\infty)]^4\right\}}{T_1(\infty) - T_2(\infty)} \overset{not.}{=} K_1 - K_1^{rad} < K_1,$$

$$K_2^* \overset{def.}{=} K_2 - \frac{e_2\left\{[T_M + T_2(\infty)]^4 - [T_M + T_3(\infty)]^4\right\}}{T_2(\infty) - T_3(\infty)} \overset{not.}{=} K_2 - K_2^{rad} < K_2,$$

$$K_3^* \overset{def.}{=} K_3 - e_3\left\{[T_M + T_3(\infty)]^4 - T_M^4\right\} \Big/ T_3(\infty) \overset{not.}{=} K_3 - K_3^{rad} < K_3.$$

and

$$F_C^* \equiv \frac{K_1^* \int_0^{t_h} (T_1 - T_2)\,dt}{C_1\ T_1(t_h)} = F_C - \frac{e_1 \int_0^{t_h} \left[(T_M + T_1)^4 - (T_M + T_2)^4\right] dt}{C_1\ T_1(t_h)} \overset{not.}{=} F_C - F_C^{rad} < F_C$$

and

$$F^{*}_{C+J} \equiv \frac{K^{*}_{2} \int_{0}^{t_h} (T_2 - T_3)\, dt}{C_1 \left[T_1(t_h) + T_2(t_h) \right]} = F_{C+J} - \frac{e_2 \int_{0}^{t_h} \left[(T_M + T_2)^4 - (T_M + T_3)^4 \right] dt}{C_1 \left[T_1(t_h) + T_2(t_h) \right]} \overset{not.}{=}$$

$$F_{C+J} - F^{rad}_{C+J} < F_{C+J} < F_C ,$$

which highlight the possibility of decreasing the uncertainty of the correction caused by heat losses (proportionally with F_C and F_{C+J} factors).

4.4. The correction factor of the absorbed radiation dose caused by heat radiation losses

For the 3-body graphite calorimeter with the features listed under Table 4.1 in the extended version of the report, the numerical solution of the system of differential equations in subchapter 4.2 is graphically represented in Fig.7.

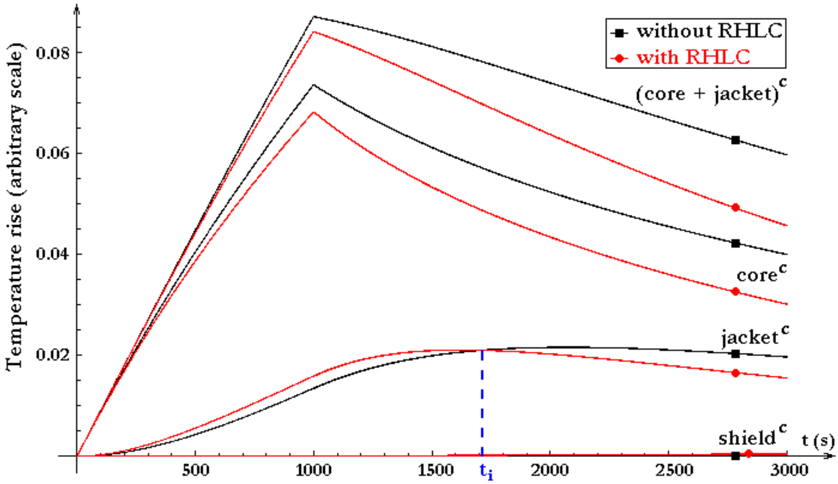

Fig. 7. Variation in temperature increase of the three bodies on the inside of the graphite calorimeter, with RHLC (red curves) and without RHLC (black curves), for $t_h = 1000\ s$ heating

The overall energy lost through radiation by the surface of the core, $W_C^{rad}\big|_{t\in[0,\ t_h]}$,during the heating, $t \in [0,\ t_h]$ is given by

$$W_C^{rad}\Big|_{t\in[0,\ t_h]} = \sigma A_c \left[1/\varepsilon_g + \left(A_c / A_j^{int} \right) \left(\varepsilon_{My}^{-1} - 1 \right) \right]^{-1} \int_0^{t_h} \left\{ [T_M + T_1(t)]^4 - [T_M + T_2(t)]^4 \right\} dt .$$

The correction factor of the absorbed radiation dose, caused by heat losses through heat radiation on the surface of the core is defined as follows

$$k_{SB} \equiv D_g / D_g^{*} ,$$

where D_g is the dose whose value is affected by the heat losses of the core through heat radiation, and D_g^* is the dose whose value is not affected by these losses.

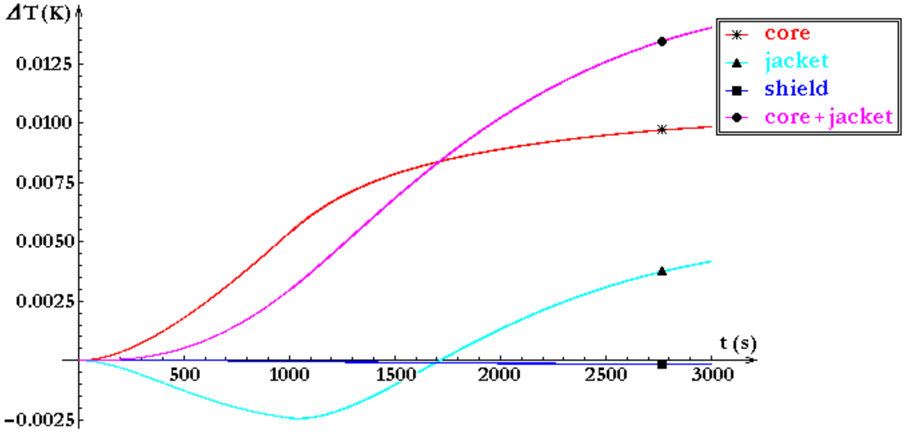

Fig. 8. The $\Delta T_i = \Delta T_i(t)$, $(i = \overline{1,4})$ dependencies for an overall time of $3000\ s$ ($1000\ s$ plus $2000\ s$ the heating thermal relaxation)

$$k_{SB}^t = \frac{1+\left(F_{C+J}^c\right)^*}{1+\left(F_C^r\right)^*}\frac{1+\left(F_C^r\right)^*+\left(F_C^r\right)^{rad}}{1+\left(F_{C+J}^c\right)^*+\left(F_{C+J}^c\right)^{rad}} = \frac{C_1\left[T_1^c(t_h^c)+T_2^c(t_h^c)\right]+\left(K_2^c\right)^*\int_0^{t_h^c}\left(T_2^c-T_3^c\right)dt}{C_1T_1^r(t_h^r)+\left(K_1^r\right)^*\int_0^{t_h^r}\left(T_1^r-T_2^r\right)dt}$$

$$\times\frac{C_1T_1^r(t_h^r)+\left(K_1^r\right)^*\int_0^{t_h^r}\left(T_1^r-T_2^r\right)dt+e_1\int_0^{t_h^r}\left[\left(T_M+T_1^r\right)^4-\left(T_M+T_2^r\right)^4\right]dt}{C_1\left[T_1^c(t_h^c)+T_2^c(t_h^c)\right]+\left(K_2^c\right)^*\int_0^{t_h^c}\left(T_2^c-T_3^c\right)dt+e_2\int_0^{t_h^c}\left[\left(T_M+T_2^c\right)^4-\left(T_M+T_3^c\right)^4\right]dt}.$$

The actual calculations give the following theoretical value for the correction factor k_{SB}: $k_{SB}^t = 0{,}999623 \cong 0{,}9996$. If k_{SB} is directly determined from the quantity of the heat lost through heat radiation on the surface of the core, during the heating process ($t_h^c = 100\ s$), when the calorimeter runs as electrical calibration, the $k_{SB} = 0{,}999648 \cong 0{,}9996$ value results, which is very close to the theoretical one (the difference between the two values is only $0{,}0025\ \%$).

To sum up, if in order to describe the running of a 3-body graphite calorimeter it is adopted the model based on the suggested system of differential equations, which considers the heat losses through heat radiation on the surface of the core, then the radiation dose absorbed in the graphite must be corrected via the factor $k_{SB} \cong 0{,}9996$ (or, in percentage, $0{,}04\ \%$).

CHAPTER 5

THERMAL CONDUCTION THROUGH THE CONNECTION WIRES OF THE THERMISTORS IN A GRAPHITE CALORIMETER

5.1. The theoretical model

The purpose was to consider: *(A)* the analysis of the heat losses through the connection wires of the heating thermistors and *(B)* the analysis of the heat losses through the connection wires of the sensing thermistors. The heating thermistors belong to an active electrical circuit (in which energy is dissipated), while the sensing thermistors belong to an inactive circuit[1].

All the processes are analysed in the time interval $t \in [0,\ t_h]$, where t_h is the heating time of the calorimeter's core. Working hypotheses:

- the spatial-temporal dependence of the temperature in a connection wire of the thermistors is $T = T(x,\ t)$, where x is the spatial coordinate, measured along the wire, and t is the temporal coordinate (see Fig.9).
- the thickness of the enamel layer of the wires is small enough $(5 \times 10^{-6}\, m)$ as to be neglected, therefore it can be considered that $R_w \cong R_m$; the caloric capacity of the enamel layer of the wires can also be neglected;
- the Joule effect cannot be taken into consideration; it will be analysed separately and the correction factor corresponding to these losses will also be defined and determined;
- first of all, an immediate increase in temperature will be considered (at the constant value T_{hot}) at the hot end of the thermistors' connection wires;
- the material scales (resistivity, density, specific heat, thermal conductivity, etc.) of the wires do not depend on the temperature;
- except for the single "point" at the hot end of the wires, the temperature field is uniform at the initial moment;
- the two ends of all the wires are plane-parallel and isothermal surface (transversal scales (in section) of the wires (the section area is almost $5 \times 10^{-10}\, m^2$) are small; therefore, the radial variation of the temperature can be neglected, so, from mathematical point of view the problem can be addressed on one scale only);
- the Peltier-Seebeck effect (which appears on the contact region between the two different materials of the wires - an alloy of *Pt-Ir* and copper), as well as the Thomson effect are neglected.

---

[1] The circuit of the sensing thermistors works in „zero electrical power dissipation" module.

The mathematical model that takes into consideration all the hypotheses above, as well as the combined effects of the losses of heat via conduction and thermal radiation are based on the following equation with partial derivatives [85]:

$$\gamma\ \partial\theta(x,t)/\partial t = \partial^2\theta(x,t)/\partial x^2 - \beta^2\theta(x,t) \qquad (\#)$$

where $\theta(x,t) = T(x,t) - T_{cold}$ is the temperature increase (K) of the wire above the constant temperature (T_{cold}) of the cold wire, $\alpha = k\rho^{-1}c^{-1}\left(\equiv \gamma^{-1}\right)$ is the thermal diffusivity of the metal used to manufacture the wire (see Table 5.1 of the extended version of the report), and $\beta^2 = 8\ \sigma\ \varepsilon_w\ T_{cold}^{\,3}\ R_w^{-1}\ k^{-1}$.

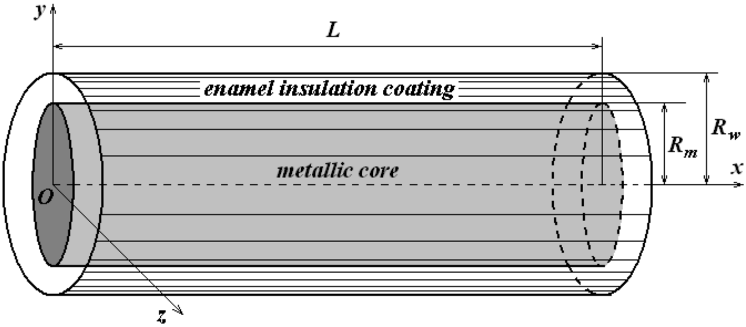

Fig. 9. The spatial orientation of the coordinate axes attached to a connection wire of the thermistors: R_m is the radius of the metallic core of the wire, and R_w is the overall radius of the wire.

The cold end of the wires is outside the calorimeter and therefore it is considered that its temperature matches the laboratory's, $T_e = 298{,}15\ K$. The analytical solution of the equation (#) leads to the solution:

$$T(x,t) = T_{cold} + \left(T_{hot} - T_{cold}\right)\left\{\frac{\sinh\left[\beta\left(L-x\right)\right]}{\sinh\left(\beta L\right)} - \frac{2}{L}\sum_{n=1}^{\infty}\left[\frac{n\pi / L}{\beta^2 + \left(n\pi / L\right)^2}\right]e^{-\omega_n^2 t}\sin\frac{n\pi x}{L}\right\}.$$

The overall energy E_{cond}^{tot}, transferred through the thermal conduction phenomenon in each wire, during the entire heating process, is given by the relation

$$E_{cond}^{tot,h} = \int_0^{t_h}\left[dE(x,t)\right]_{cond}^{x=0} = -\pi k R_w^2 \int_0^{t_h}\left[d_t T(x,t)/dx\right]_{x=0} dt\ ,$$

and the overall energy, E_{rad}^{tot}, lost through thermal radiation via a sole wire within the same time interval, is

$$E_{rad}^{tot,h} = \int_0^{t_h} dt \int_0^{L} d\left[d_x E\left(x,t\right)/dt\right]_{rad} = 2\pi R_w \varepsilon_w \sigma \int_0^{t_h} dt \int_0^{L}\left\{\left[T(x,t)\right]^4 - T_{cold}^4\right\}dx\ ,$$

where L is the wire's length.

An approach more similar to reality refers to:

1) ruling out the hypothesis according to which the temperature increases immediately at the hot end of the thermistors' connection wires;

2) taking into consideration the explicit temporal dependencies of the temperatures at the end of the thermistors' connection wires (both for the heating thermistor and for the sensing thermistors);
3) taking into consideration the actual structure of the thermistors' connection wires; each wire consists of a smaller part, of length L_{Pt-Ir}^{eff}, directly connected to the thermistors' bodies and consists of an alloy of $Pt-Ir$ $(10\%\ Ir)$, and a greater length $L_{C-ext} = L_{C-J} + L_{J-S} + L_{S-E}$, which consists of enamelled copper.

5.2. The calculation of the heat losses through the thermistors' connection wires

5.2.1. Preliminary results

Based on the relations presented in the extended version of the report, at the beginning of this subchapter, and on the numerical values of the scales that take part in it (presented in the tables 5.1 and 5.2 of the same extended version) there can be numerically assessed the heat losses, both in the connection wires of the heating thermistor, and in those of the sensing thermistors. The overall energy supplied to the core during the entire heating process is: $W_1 = \int_0^{t_h} P_1\, dt = 5{,}88\ mJ$. Obviously, a small part of it is lost via the connection wires of the thermistors, and this requires the introduction of a corresponding correction factor for the absorbed dose. As usual, this factor is given by the relation between the absorbed dose when these losses are taken into consideration and the absorbed dose without taking into consideration the corresponding losses: $k_w \equiv D_g / D_g^*$. In the hypotheses considered until present time, the calculations lead to the value $k_w^{core \to Pt-Ir} = (5880\mu J - 8{,}42\mu J)/5880\mu J \cong 0{,}9986$

The correction factor corresponding to the model with immediate temperature increase at the end of the wires is $k_w^{inst} = 0{,}9985$, and the one corresponding to the model that starts from the premises that the wires are entirely made of copper, it is $k_{w,\,Cu}^{inst} = 0{,}9918$.

5.2.2. Some additional corrections

These additional corrections refer to the thermal transfer that can take place through the substance used to anchor the thermistors (epoxydic resin) and through the Mylar film, which electrically isolates the *Pt - Ir* wires from the graphite body of the core. Indeed, the thermal conductivities of these materials have values that cannot be neglected, namely $k_g \cong 0{,}43\ W \cdot m^{-1} \cdot K^{-1}$ [27] and $k_{Mylar} \cong 0{,}15\ W \cdot m^{-1} \cdot K^{-1}$ [103], and have to be taken into consideration in order to establish the effect of the heat transfer through these materials. The considerations related to this topic will not be presented here (as they can be found in the paper

[82]), but only the final results will be presented. Therefore, taking into consideration the effect caused by the multiple reflections suffered through thermal radiation (emitted by wires) via the "combined factor perspective-emissivity", whose expression is $F_{v-e} = \varepsilon_g \left\{1-\varepsilon_{My}\left[1+\left(R_C/R_J\right)^2\right]^{-1}\right\}\left[1-\left(1-\varepsilon_g\right)\left(1-\varepsilon_{My}\right)\right]^{-1} = 0,96758,$, for the correction factor due to heat losses through wires it can be obtained the value

$$k_w^{core} = (W_1 - W_{loss}^{Cu,\,net})/W_1 = (5880\ \mu J - 0,74\ \mu J)/5880\ \mu J \cong 0,9999$$

5.3. Losses by Joule effect and the corresponding correction factor

The calculation of the overall heat losses of the core of a graphite calorimeter via the Joule effect that occurs in the thermistors' connection wires leads to the value

$$W_{loss}^{Joule,\,net} = 2t_h \rho_{Cu}^e I_h^2 (L_{C-ext} - p_h F_{e-v} L_{C-J})/\pi (R_w^{Cu})^2 = 5,857\ \mu J$$

where I_h is the amperage of the direct current passing through the connection wires of the heating thermistors, the meaning of all the other scales that appear in this expression being listed in the extended version of the report. The corresponding correction factor is

$$k_w^{Joule} = (W_1 - W_{loss}^{Joule,\,net})/W_1 = 5880\ \mu J - 5,857\ \mu J/5880\ \mu J \cong 0,9990$$

and leads to the following value of the overall correction factor due to losses through the connection wires of the thermistors during the electrical calibration of a graphite calorimeter $k_w = k_w^{core} \cdot k_w^{Joule} \cong 0,9989$.

5.4. The effect caused by rolling the connection wires of the thermistors around the core

By taking into consideration this effect, the correction factor k_w of the measured radiation dose caused by heat losses through the thermistors' connection wires increases from 0.9989 to 0.9999, which is unanimously accepted as the maximum possible "contribution" of this type of losses [44]. Thus, the considerations of this paragraph leave out the difference between the experimental The working hypotheses of the model and the extreme conditions for the corresponding equation $\frac{1}{r}\frac{\partial}{\partial r}\left[r\frac{\partial T(r,z)}{\partial r}\right]+\frac{\partial^2 T(r,z)}{\partial z^2}=0$, are presented in the extended version of the report. The conclusion: rolling the connection wires of the thermistors around the core results in total disposal of the losses caused by conduction from these wires (thus $k_w^{core}=1$), and thus only the losses by Joule effect remain. The recalculation of the latter, in view of the suggested mode, leads to the value $W_{net\ loss}^{Joule} = 0.11\ \mu J$, which means that the new value of the correction factor k_w^{Joule} is $k_w^{Joule} = (W_1 - W_{net\ loss}^{Joule})/W_1 = 0.9999$; thus, the resulting value of the overall correction factor caused by losses through the connection wires of the thermistors during the electrical calibration of a 3-body Domen-type graphite calorimeter is [44]:

$$k_w = k_w^{core} k_w^{Joule} = k_w^{Joule} = 0.9999 .$$

CHAPTER 6

MODELLING TEMPERATURE GRADIENTS FROM A GRAPHITE NUCLEAR CALORIMETER

6.1. The temperature gradients of a Domen-type graphite calorimeter

One of the most often used methods for calibrating a graphite calorimeter, used as primary standard in measuring the absorbed radiation doses, which is operated in quasi-adiabatic module or together with the compensation of the heat losses, is the *comparative method* [15, 17]. The main idea of this method is to compare the answer of the temperature sensor of the calorimeter at a very well established quantity of energy/heat, with the same radiation response. This calibration type is usually achieved by the simultaneous dissipation of a certain quantity of electricity which is dissipated in the heating thermistor(s), installed both in the core (which is used as radiation absorbent), and in the primary shield of the calorimeter [83]. Since this calibration method is comparative, it is very important that the core of the calorimeter be identically heated in the two cases: *(i)* when it is irradiated with radiation beam and *(ii)* when the necessary electricity quantity is dissipated in it via the heating thermistors. This condition is not accurately met because the core and the constituents near the core are evenly heated when the calorimeter is irradiated with a slightly higher energy beam (in this case the temperature gradients in the core are negligible) while in the case of the electrical heating (during the calibration) the same bodies can be heated unevenly and the temperature gradients can no longer be neglected.

In general, there are two major causes that lead to the occurrence of temperature gradients during the electrical calibration of the calorimeter.

i) the uneven electrical heating (via micro-thermistors) of the calorimeter's bodies;

ii) the existence of thermal barriers between thermistors and the fabric of the calorimeter's bodies (in this case, graphite).

6.2. The one-dimensional model

Since the change of φ temperature, in the case of electrical calibration of the calorimeter, is determined mainly by the thermal conduction phenomenon, it will be determined as solution of the one-dimensional equation of the heat, $\partial\varphi/\partial t = \alpha\ \Delta\varphi$, where the diffusivity is given by the relation $\alpha = k/\rho c$. Here k represents the thermal conductivity, ρ is the mass density and c is the specific heat of the core's fabric (graphite). The final temperature change of the core can be determined from the energy conservation law, namely $\varphi_\infty = Q/mc$, where Q is the heat/energy supplied to the core, m the mass of the core, and c is the specific heat of the graphite.

Under one-dimensional aspect, the heat equation can be explained as follows

$$\partial\varphi(x,\ t)/\partial t=\alpha\ \partial^2\varphi(x,\ t)/\partial x^2\ . \qquad (*)$$

The initial and borderline conditions for this equation, which model a situation similar to an adiabatic calibration, are the following: (i) initially, the temperature is overall the same (in the entire "volume" of the core); (ii) an end of the core is thermally isolated; (iii) at the other end of the core a constant thermal flow is applied on a time interval t_h; (iv) the final temperature is φ_∞ everywhere.

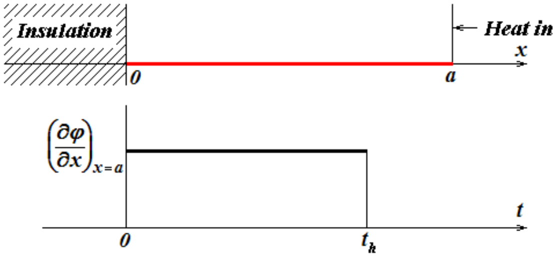

Fig. 10: Graphic representation of the extreme conditions for the one-dimensional model

Let it be the time necessary to reduce the maximum of $\varphi(x,\ t)$ to $0{,}1\ \%$ of φ_∞. The numerical evaluation for $a=20\ mm$, $t_h=60\ s$ and $\varphi_\infty=1\ mK$ gives for the time t_r the value $(t_r)_{0{,}1\ \%}\ =\ 1{,}3687\ s$, where it was considered the solution of the equation (*): $\varphi(x,\ t)=(\varphi_\infty/t_h)\Big\{t-(t-t_h)\theta(t-t_h)+2\sum_{n=1}^{\infty}\Big[\Big(1-e^{-\beta n^2 t}\Big)-\Big(1-e^{-\beta n^2(t-t_h)}\Big)\theta(t-t_h)\Big]\Big((-1)^n/\beta n^2\Big)\cos(n\pi x/a)\Big\}$. If $1\ll\beta t<\beta t_h$ (in this case $\beta\cong 2{,}041\ s^{-1}$), then the temperature of the entire core will increase at the same pace, $\varphi(x,\ t)$ reaches a quasi-constant aspect in the spatial variable x and increases quasi-linearly in the temporal variable t. If this status is reached when the heat supply is disconnected (which is mathematically expressed by the inequality $1\ll\beta t_h$) then a higher limit value can be assessed for the „relaxation" time $t_{r,\max}$, with the help of the lowest terms of the Fourier series, for instance the time measured after the cessation of the core's heat supply, in order to reach $0{,}1\ \%$ of φ_∞ is at most $t_{r,\max}=\beta^{-1}\ln\left(a^2/3\cdot 10^{-3}\alpha t_h\right)\cong 1{,}6\ s$

6.3. The two-dimensional model with rectangular geometry

Under two-dimensional aspect, the heat equation can be expressed as

$$\partial\phi(x,y,\ t)/\partial t=\alpha\left(\partial^2\phi(x,y,\ t)/\partial x^2+\partial^2\phi(x,y,\ t)/\partial y^2\right). \qquad (**)$$

The extreme conditions (initial and borderline) for the equation above, which models a situation similar to an electrical calibration, are the following: *(i)* at the initial moment the temperature field in the core is even, the temperature increase $\phi(x,y,t)$ can be considered null, without reducing the generality of the problem; *(ii)* the border of the square (for simplicity reasons a rectangular symmetry will be considered) that models the core (See Fig.11) is perfectly thermally isolated, except for a small part, with a width of 2ε, located near the centre of one of the sides and centred on this centre, via which the core is heated by a constant heat flow of t_h seconds; (*iii*) the initial temperature has the balance value ϕ_∞.

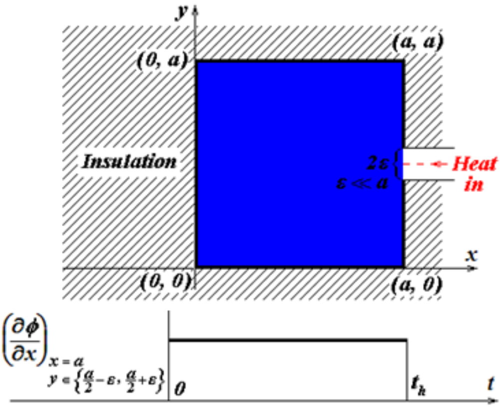

Fig. 11: Graphic illustration of the borderline conditions for the equation (**)

The solution of the equation (**) is given by the relation (***), and the numerical evaluations based on this solution lead to the following values: $\phi_{\max} \cong 1{,}1343\ \phi_\infty$ (obtained for $x = a,\ t = t_h$ and $y = a/2$) and $(t_r)_{0{,}1\ \%} = 1{,}368738\ s$. To sum up, the time (measured after the disconnection of the core's heat supply) necessary to reach, for instance, the value $0{,}1\ \%$ of ϕ_∞ is lower than 2 seconds.

$$\begin{aligned}\phi(x,y,t) = \frac{\phi_\infty}{t_h}\Bigg\{ & t-(t-t_h)\theta(t-t_h)+2\sum_{n=1}^{\infty}\left[\left(1-e^{-\beta n^2 t}\right)-\left(1-e^{-\beta n^2 (t-t_h)}\right)\theta(t-t_h)\right]\times \\ & \frac{(-1)^n}{\beta n^2}\cos\frac{n\pi x}{a}+2\sum_{m=1}^{\infty}\left[\left(\left[\left(1-e^{-\beta m^2 t}\right)-\left(1-e^{-\beta m^2 (t-t_h)}\right)\theta(t-t_h)\right]\frac{1}{\beta m^2}+\right.\right. \\ & 2\sum_{r=1}^{\infty}\frac{(-1)^r}{\beta\left(m^2+r^2\right)}\cos\frac{r\pi x}{a}\left[\left(1-e^{-\beta\left(m^2+r^2\right)t}\right)-\left(1-e^{-\beta\left(m^2+r^2\right)(t-t_h)}\right)\theta(t-t_h)\right]\Bigg)\times \\ & \left.\cos\frac{m\pi}{2}\cos\frac{m\pi y}{a}\right]\Bigg\}\ . \end{aligned} \tag{***}$$

6.4. The three-dimensional model

As it was indicated in the previous paragraphs concerning one- and two-dimensional models, the change/increase in temperature made by an uneven heating (more precisely „point-like”) of a graphite calorimeter's core during its electrical calibration is not very different from the even heating specific to irradiation after almost two seconds since the cessation of the core's electricity supply.

This result was obtained by means of theoretical models based on ordinary differential equations, by more research teams, among which there can be remembered: DuSautoy [22], Ioniță *et al.* [39, 41] etc. At the same time (in order to check whether the temperature change caused by an uneven heating of the core of a graphite calorimeter during its electrical calibration is significantly different from the even heating ensured by irradiation) a series of investigations have been conducted (such as Duane [21] and McEwen and Duane [61]), based on the finite element method for modelling the entire calorimeter and not only its ore. In addition, some of the modelling of these authors also refer to the more general, three-dimensional case.

In the extended version of the paper there are presented detailed aspects related to the theoretical/analytical modelling of the problem, the heat transfer mechanism between thermistor and graphite, the numerical modelling of the problem and the analysis of the temperature gradients.

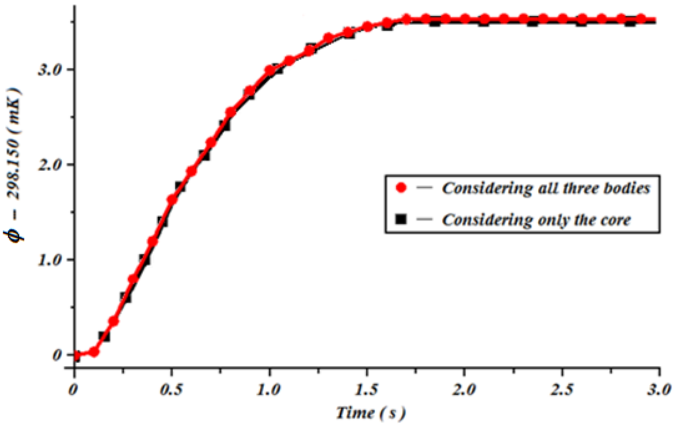

Fig. 12: The increase in temperature of the core based on time, measured from the beginning of the core's heating, in two cases: *(i)* the 3D numerical analysis of the entire calorimeter (considering all three interior bodies of the calorimeter) – the red curve (the dotted curve) and *(ii)* the 3D numerical analysis of the nucleus only – the black curve (the square curve)

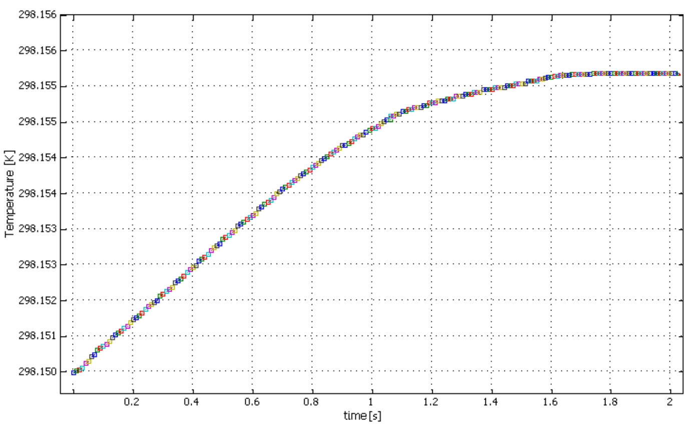

Fig. 13: Temporal dependency of the core's temperature in the location points of the sensing thermistor s(P_1 and/or P_2) for the first heating $\Delta t = 2\ s$. This is the temperature „measured" by the sensing thermistors.

As it can be noticed in Fig.13, after more than two seconds following the disconnection of the core's heat supply, the temperature gradients have been completely „dissipated'. In addition, in Fig.12 there can be noticed that for this type of calorimeters, due to the specific geometrical symmetry, the 3D modelling of the entire calorimeter (with all three bodies) does not bring significant changes to the one that only considers the core.

CONCLUSIONS

1. It is suggested a new theoretical model for explaining the electrical calibration of a 3-body Domen-type graphite calorimeter, called Radiative Heat Loss Model (RHLC – „*R*adiative *H*eat *L*oss *C*orrection”);
2. New heat transfer coefficients are defined (called by the authors „low heat transfer coefficients” K_i^*, $(i=\overline{1,3})$), as well as the corresponding correction calculated/theoretical factors $(F_C^*$ şi $F_{C+J}^*)$;
3. The differential equation system that underlies the new model is numerically solved, thus establishing the temporal variation of the calorimeter’s bodies’ temperature during its electrical calibration;
4. It is introduced (defined and effectively established) a new correction factor for the absorbed radiation dose, called „correction factor due to heat losses through heat radiation” (noted k_{SB});
5. The k_{SB} correction factor is calculated by using two methods (by using the induced theoretical formula, as well as directly, from the general definition relation), thus obtaining a very good correspondence between the two values (difference of 0.0025% for a correction value of 0.04%)
6. The spatial and temporal distribution of the temperature is established in a connection wire of the thermistors, both in transitory regime and in stationary regime for both types of thermistors – heating and sensing;
7. It is suggested and analysed a new theoretical model for calculating the heat losses through the connection wires of the thermistors, which takes into consideration the time variation of the wires’ hot end temperature, as well as the structure of the two materials (alloys of $Pt-Ir$ $(10\%\ Ir)$ and copper) of the wires;
8. Additional corrections are brought to the suggested model by taking into consideration the conductibility other than zero, both of the adhesive used for fastening/installing the thermistors and of the Mylar film used as electrical isolator between the thermistors’ connection wires and the graphite body of the calorimeter’s core;
9. It is identified and established a new factor that contributes to mechanism of the heat loss through the thermistors’ connection wires, called by the authors „the sight-emissivity combined factor” and the correction factor (k_w^{core}) is calculated for the absorbed radiation dose, due to the heat losses through the thermistors’ connection wires, due to conduction and thermal radiation;
10. $k_w = k_w^{core} \cdot k_w^{Joule}$, pentru pierderile totale de căldură prin fire; It is established the correction factor due to heat losses by Joule effect in the thermistors’

connection wires, k_w^{Joule}, as well as the total/resulting correction factor, $k_w = k_w^{core} \cdot k_w^{Joule}$,for the total heat losses through wires;

11. It is studied the effect of rolling the thermistors' connection wires over the values of the correction factor due to the heat losses through these wires, thus obtaining a higher value of this factor, only if this effect was neglected; therefore, rolling the thermistors' connection wires (and especially those corresponding to the heating thermistor) around the calorimeter's core seem to be a highly indicated procedure in order to obtain the most accurate measurement results.

    There are presented the causes that lead to the occurrence of temperature gradients, as well as practical solutions used throughout the history of nuclear calorimetry in order to reduce the scale and thus the effect of these gradients.
12. It is established the superior limit value of the „relaxation" time $t_{r,\max}$, defined as the time measured after the cessation of heat supply to the core, so that the change/increase in its temperature, φ/ϕ reaches $0{,}1\ \%$ of the final equilibrium value, $\varphi_\infty/\phi_\infty$, in three different cases corresponding to: the one-dimensional model, two-dimensional model (in a rectangular geometry) and the three-dimensional model (which takes into account theaccurate geometry of the core). In all three cases it was found that this time does not exceed two seconds. There are estimated the maximum and minimum values that can be reached by temperature gradients in the core of a Domen-type graphite nuclear calorimeter;
13. It is analysed in detail the heat transfer mechanism between a thermistor and its surrounding medium (graphite in this case) for the purpose of finding the most pertinent answers to the two questions below:

- Is the temperature „measured" by the two sensing thermistors equal at any time with the average temperature of the core? If not, how much is the difference?
- How great is the error that occurs when the there are calculated the heat losses through thermal radiation on the surface of the core by taking into consideration the average value of the temperature on the surface of the core instead of the actual local spatial and temporal distribution of the temperature?

14. It is obtained the important result according to which, in the case of the geometry specific to a Domen-type graphite nuclear calorimeter, 2D modelling is sufficient (therefore a 3D model is not necessary) to analyse the effect of the temperature gradients that occur in the calorimeter's bodies (and especially in the absorbent/core) over the results obtained during the electrical calibration of such a calorimeter.

Under the section *Personal papers and publications* there are listed, in three different lists, the published scientific papers (5, out of which 4 are quoted ISI and 2 were published during the PhD training), accepted for publication (1 quoted ISI and published „online" during the PhD training), sent for publication (2) and the papers presented at national and international conferences and workshops (5).

The paper is completed by an alphabetical *bibliography* that contains 118 titles (the extended/full version).

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