Instead of introduction

In front of you is the first issue of the Newsletter devoted to promotion of the application of various nuclear and other analytical techniques for characterization of the Cultural Heritage Objects (CHO) alongside with radiation based treatment for conservation and consolidation purposes. Since 2005, under the patronage of the IAEA, several regional TC projects regarding this subject were successfully realized and in few days, RER0034 project entitled „Enhancing the Characterization, Preservation and Protection of Cultural Heritage Artefacts“ is coming to the end. Fortunately, there is a strong intention and goodwill among researchers involved to continue activities in this field in the future and to continue promotion of the application of the scientific methods in characterization and treatment of CHO. That is way, although this newsletter is one of the achievements of the current RER0034 project, it will not be ended with it and we hope that it will last as long as the interest of the researchers exists. We hope that here you will find basic information regarding different projects relevant to this subject, funded by IAEA or some other institutions and organizations, interested case studies and results, new publications and events, success stories etc. The idea of networking professionals from natural, technical and social sciences will certainly be easier realized with this Newsletter and dissemination of the results will be more successful. So be invited to make contributions to next issues since your own ideas and information you want to share, will be a foundation of the success of this Newsletter. We also hope that this will be just starting point for all next actions in disseminations of your results which surely will be useful for both communities. The realization of web portal devoted to this subject should made easier exchange of the information between professionals in this field and distribution of this Newsletter to all interested parties.

So this is just a first step...

Velibor Andric, VINCA Institute of Nuclear Sciences, Serbia
Contributions from participating countries

Ion Beam Analysis Methods in Advanced Historical Textiles Research: Technical Study of Metal Threads from Romanian Historical Textiles (15th–18th century)

Research on gold threads from Romanian medieval textiles by using IBA analysis techniques (PIXE and RBS) started in 2011 when our scientists group project proposal (IBATEX) was accepted for beam time access at ATOMKI-HAS (Debrecen, Hungary) within the EU-CHARISMA FIXLAB programme and continued in 2012 with IBATEX 2 in collaboration with the ATOMKI-HAS researchers.

Study was focused on technological characteristics of the metal threads in order to identify the specific constituent elements, especially the trace elements, their concentration and structure.

Main objective was to demonstrate the necessity of integrating the advanced modern IBA methods with the classical analytical techniques (XRF, SEM-EDX) for an in-depth applied research that can bring new developments and rich accurate information on processes governing historical metal threads technology of production, their origin and provenance.

PIXE and RBS methods proved to be ideal by their features for this kind of research, information resulted on metal composition, the elements distribution and concentration and the compositional depth profiles being used to determine the technique of production, gilding technology, to clarify and complete the existing information concerning their provenance, to identify if there are any qualitative differences between them, and to indicate, if possible, their area of spreading.

Textiles from which the samples were prelevated were velvet brocades and religious embroideries worked in the traditional Byzantine technique and dating between the 15th and the 18th centuries, some of them produced locally (Byzantine Romanian Embroidery School from Putna Monastery) and some imported (Western and Ottoman Empire workshops) as appeared in the Romanian historical literature. Samples were metal wires and strips wrapped around a silk core or without a core fiber and cross-sections of gilded wires. Gilded silver wires were embedded in synthetic resin, cross-sectioned and finely polished for obtaining smooth thin uncontaminated sections.

Optical microscopy in reflected, polarized and UV fluorescence light at different magnifications was also used for preliminary examination of metal threads.

Fig.1 Microscopic images (reflected light, 100x) of J2 (metal wire wrapped around a red silk core) and K2 samples (metal strip wound around a yellow silk core), their cross-section images.

PIXE measurements were performed under 3 MeV and intensity of 100 – 500pA on 23 cross-sections and 50 samples taken from 18 medieval textiles. Also, PIXE maps showing elemental distributions were collected for each sample. RBS was carried out on 36 samples using an alpha particles beam with energy of 2MeV and intensity of a few hundred pA. Analyses were performed in vacuum and the size of the beam was of a few microns. The raw data have been analyzed by the deconvolution of PIXE spectra with the GUPIX code.

Fig.2 PIXE maps showing some of constituent elements distribution.
The major and minor constituent elements (Ag, Au and Cu), also trace elements like Ca, Pb, Fe, Hg, Zn, Ni, Bi, Co, Cr or Mn were possible to be detected by PIXE, and important clues on the metal threads provenance, also on manufacturing techniques used were revealed.

By 15th and 16th century, most of the silver was extracted from lead and copper ores by cupellation, so Pb presence could be explained by the silver refining process used in those times. According to Biringuccio (“The Pirotechnia”), a fine cupelled silver was employed by medieval smiths to make the so called “spun silver or gold” which is the flattened strip wound around a silk yarn used in embroideries. Presence of Hg may indicate a gold amalgamation technique employed for gilding, but also, a mercury amalgamation gold refining process that have been used in the 14th century in the Middle East, and only later on, by the middle of the 15th century, in both Europe and the Middle East.

Calcium showed a homogeneous distribution and its presence resulted to be related to the silk core threads. Other trace elements of interest for provenance of historical metal threads textiles were Bi, Fe and Zn.

RBS results brought new information on the gold very thin layer structure, and clarify the fact that in some cases the gold is separate from the silver bulk by an interface layer that resulted by atomic diffusion of silver into the gold layer.

Considering that the manufacture of the selected embroideries is very precise in time, the results obtained could contribute as a reference and could constitute an important database on historical metal threads textiles to further studies. Since metal wrapped yarns are easily transportable and an easy medium of trade it is possible that the Au/Ag ratios, or the ratios between major and minor elements (Cu, Pb, etc.) or the trace elements (Hg, Bi, Fe etc.) to may indicate the manufacturing sites for the metal thread yarns. A tentative European assignment could be made for the mixed metal and a Middle Eastern assignment for the specimens containing Au or Ag in the absence of the other.

This study has been made possible due to the financial support by the Transnational Access to Research Infrastructures activity in 7th framework programme of the EU (CHARISMA Grant Agreement n. 228330) which is gratefully acknowledged and thanks to technical help and support of the ATOMKI-HAS researchers.

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Recent Studies on the Application of Nuclear Methods for the Construction of a Temporality Approach on Neolithic and Archaeological Contexts of Perdigões Archaeological site, south Portugal

The Archaeological Problem

The Perdigões site is one of the largest known Portuguese Chalcolithic settlements, occupied during the late 4th - 3rd millennium B.C in the Reguengos de Monsaraz region, in the South of Portugal. This circular shaped settlement spreads over an area of 16 ha, and is delimited by two concentric ditches (Fig. 1).

The interpretive approaches that have been made are consciousness of its structural complexity; topographic and landscape structure that has been excavated, allowed the development of interpretative discourses of the spatial organization of the site, also by means of astronomical and cosmological aspects (Valera, 2008). The ongoing research is attentive to the fact that we are dealing with a reality that was dynamic, diverse, and complex, obtained from a register that provides fossilized palimpsest as a static, homogeneous, partial and still poorly known (Valera et. al., 2007).

The burial remains are diversified and mainly constituted by pottery, lithic artefacts, limestone and bone idols, pecten shells, bone combs, adorn artefacts, etc... (Fig. 2). On a global perspective one can say that the group of pottery artefacts includes all the typical morphologies of the Late Neolithic and Chalcolithic of the South West of the Iberian Peninsula and that there are differences between the funerary and the domestic recipients. According to the main goals of the archaeological project some of the answers can only be reached by an archaeometric approach, specially the ones concerning provenance and production technologies of the funerary ritual pottery, when compared with the domestic function ceramic, as well as, a diachronic approach, from Neolithic to Chalcolithic, in relation to ceramic production strategy.
The Used Methods

The chemical analysis of both ceramics and clay raw materials was done by means of instrumental neutron activation analysis (INAA). The mineralogical composition was obtained by X-ray diffraction (XRD).

The Relevant Results

Chalcolithic pottery from Perdigões was previously studied (Dias et al, 2005; 2007), together with clay materials representative of regional geological contexts, and the resource to more diversified raw materials was identified for the production of funerary rituals ceramics. More recently also Neolithic pottery was studied. We also may infer that, as predictable, most of the ceramics, independently of the studied chronology, points to the resource to local clays available close to the site, and have a certain chemical homogeneity, with the above mentioned exception of funerary pottery, especially from tomb 1, which points to a spread in the clays resources (Fig. 3) (Dias et al., 2012). Thus reflecting different origins, perhaps due to the use of Perdigões necropolis by peripheric, but related, communities. In fact, by combining a greater chemical heterogeneity and a more distant geological origin then "common" ones, these ceramics used in funerary rituals may be originated from peripheral known contexts and which are located much close to that of geological origin of the used clays.

If we add to this the fact that, in both excavated tombs, only secondary deposits have been identified, the possibility gets some relief of the distant communities use Perdigões in the management of their burial practices, which would reinforce the idea that the aggregator power of this site.

Bibliography


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Obsidian Studies Using Nuclear Techniques in Hungary

Introduction

Obsidian is a success story in lithic provenance studies. The beauty, rarity and adaptability of the material for the purpose of making stone tools made it popular and widely known both in prehistory, folklore and archaeological / anthropological special studies.

Looking at the palaeogeographical maps of the Neogene period, however, (Hámor 2001) we can see that during large part of the Neogene when the actual image of the current Carpathian Basin was formed, the territory was covered with large water bodies, remains of the Tethys Ocean of the Mesozoic, turning gradually into a huge with brackish water called Paratethys.

Apparently, the formation of obsidian requires special conditions; however, these conditions are typically met in young volcanic regions of the tectonically active zones like the Circum-Pacific region or the Mediterranean islands. Collecting information on obsidian worldwide, H. Pollmann (1999) compiled distribution map for geological obsidian sources all over the World. As his work was mainly directed at collecting bibliographical information, he did not, and could not make a critical assessment of his sources – this task should be handled by the experts of the individual regions themselves. Such a critical review was accomplished by the author in 2004 on the occasion of the 34th International Symposium on Archaeometry, Zaragoza, Spain (Biró 2004, 2006) for the territory of Hungary and the Carpathian Basin.

Summing up fast, in the so-called Carpathian Region, we have to consider 3 main obsidian sources, termed in archaeometrical practice Carpathian 1 (Slovakian obsidian), Carpathian 2 (Hungarian obsidian) and Carpathian 3 (obsidian in the NW parts of the Ukraine).

Obsidian is a special kind of rock and gemstone in many ways. Though it looks like a mineral on the strength of its homogeneity, it is a volcanic rock with generally very high silica ($\text{SiO}_2$) content. Obsidian is formed from rhyolitic lava by quenching, i.e., the very fast, practically instantaneous cooling and solidification of the magma. These circumstances can be most easily met at volcanic islands surrounded by large water bodies like sea or ocean, occasionally lakes and ice sheet. The result is a solidified rock with no apparent mineral phases. The glass will, by the advance of geological times, crystallize starting from the surface and turn into felsitic volcanic rock with growing number of crystallites and, later, crystals of zeolite and feldspar.

Depending on the composition of the magma, however, the glass can be fairly stable over millions of years.

How can we speak of obsidian volcanism in the centre of Europe, the most continental part of the continent?

Fig. 1 Depot of giant obsidian blade cores from the collection of the Hungarian National Museum. Photo by J. Kardos

Fig. 2 Carpathian 1, 2, and 3 obsidians
Contributions from participating countries

Petroarchaeology and archaometry of obsidian

In the meantime, obsidian became a favourite subject for provenance studies on international scale. Following the first communications on the so-called “Mediterranean” obsidian regions (term applied in rather wide sense in various works of C. Renfrew and his collaborators), more and more techniques were successfully applied on sourcing obsidian starting with Optical Emission Spectroscopy (OES) (Cann & Renfrew 1964), later on using Instrumental Neutron Activation Analysis (INAA) (Gordus et al. 1968) and Fission Track Dating (FTD) (Bigazzi & Bonadonna 1973). The first successful geochemical characterisation of the Central European (Hungarian and Slovakian) obsidians was realised by INAA (O. Williams and colleagues, Warren & al. 1977, Williams & al. 1984.). Anglo-Saxon archaeometrical research introduced the name “Carpathian obsidian” for these sources with the ease of visitors from far away and grouped known sources accordingly (C1, C2).

Soon after the successful characterisation by INAA, the source-characteristic geochemical differences were also detected by Energy Dispersive Electron Spectroscopy (EDS) and X-ray Fluorescence (XRF) techniques (Biró et al. 1986, 1988) and further sub-groups could be ascertained.

Among the C2 (Hungarian) obsidians two variants could be differentiated clearly; C2T type (Tolcsva environs) and C2E (Mád-Erdőbénye environs). The three types can be separated with a trained eye macroscopically as well. C1 is clear / transparent, with shiny glassy surface, black in blocks; C2T (Tolcsva type) is opaque, even in relatively thin slice, black or sometimes reddish (mahogany obsidian); C2E is also opaque but dark graphite grey, often striped. All of them are known from archaeological context, on regional and even long distance range, too, but C1 is far the most popular version.

Geochemical characterisation of Carpathian obsidians, differentiating the main groups and all of them from other potential concurrent European sources was successful using a number of techniques. Apart from neutron activation analysis which is still the most accepted routine method, measuring the main components and some trace elements by EDS and XRF, as well as ion beam analytical techniques proved equally successful and are more available for us and less destructive.
Contributions from participating countries

Very important contribution to the characterisation of Carpathian obsidians is the determination of the age of formation, i.e., geological age (G. Bigazzi in Biró et al. 2000b). It can help in cases when the geochemical composition is undistinguishable like certain C2 types interacting, on the basis of main composition, with one of the Sardinian sources and C1 type, interacting with the highest quality Central Anatolian obsidians. The relatively old age of Carpathian obsidians would clearly identify them in problematic cases; however, no suspect interacting piece was located so far, though we cannot exclude the possibility.

Recent Hungarian contribution to obsidian studies

Following the afore mentioned distribution studies the author started to collect data relevant to obsidian distribution from literature as well as personal survey of the archaeological collections (Biró 1981, 1984). Effort was made to implement objective, instrumental methods for analysis.

Unfortunately, most of the methods were destructive at least to some degree and while the historical information obviously justified the damage in debitage relatively close to the source areas, for more distant and unique pieces it was necessary to adopt effective but non-destructive methodology. Our efforts fortunately met with technical possibilities using Proton Induced X-ray / Gamma Emission Spectroscopy (PIXE-PIGE) techniques (Elekes et al. 2000, Biró et al. 2000c, Rózsa et al. 2000) and Prompt Gamma Activation Analysis (PGAA) (Kasztovszky & Biró 2004, 2006, Kasztovszky et al. 2008), all are non-destructive multi-element techniques for geochemical characterisation. Initial application of the latter method for quantitative determination of bulk elemental composition turned out to be adequate and sensitive enough for obsidian characterisation in view of comparative material from all known sources of Europe.

Reference samples were obtained by personal trips to sources as well as kind donations and exchange of samples by means of the comparative raw material collection of the Hungarian National Museum (Lithotheca, Biró & Dobosi 1990, Biró et al. 2000a). Regular field surveys supported our basic source database, including special new elements as the extremely rare red obsidian in our region (Biró et al. 2005) and confirmation and characterisation of the long-debated obsidian sources in Ukraine (Rosania et al. 2008).

Archaeological interpretation of obsidian distribution data was evaluated from chronological and quantitative aspects (Dobosi 2011 for Palaeolithic distribution and routing; Biró 1998 for Neolithic stages and Biró 2009 for an essay on overall assessment of Prehistoric obsidian use in Central Europe).

Fig.4 Archaeological distribution map of Carpathian obsidians

Extending the scope: the prehistoric border-lines

Distribution data were collected and surveyed several times. (Biró 2004, 2006). Seemingly we have to count on three basic sources lying in the adjacent regions of South-East Slovakia, North-East Hungary and Western Ukraine. From the Hungarian provenance data we could see a basic dominance and popularity of Slovakian (C1) obsidian, a regional role of Hungarian (C2T, C2E) obsidians. According to what we know so far, the Ukrainian (C3) obsidian was used only locally.

Knowing the archaeological distribution data it became more and more pressing to focus on the limits of Carpathian obsidian distribution and possible interaction zones with obsidian coming from other parts of the Mediterranean region, i.e. the Western Mediterranean (Italy) and the Eastern Mediterranean (Greece and Anatolia).
Instrumental obsidian characterisation data already justified the presence of Carpathian obsidian in the sphere of both: i.e., Grotta Tartaruga (Williams et al. 1984) and Mandalo (Kilikoglou et al. 1996). To know the limits more exactly, we could organise the investigation of essential number of archaeological obsidian finds from most of the neighbouring countries (Croatia, Serbia, Romania, Ukraine and Poland) with the help of international collaboration projects and we are trying to cover more.

The most recent summary was presented on the 39th International Symposium on Archaeometry, Leuven (May 2012, Kasztovszky et al. 2012). Accordingly, we can see interaction zones of the Carpathian (mainly C1) obsidian towards the SW and SE borders of the distribution zone; interaction with Lipari obsidians was observed on the territory of former Yugoslavia (currently: Croatia and Bosnia). Towards the SE, the only Thessalian piece (Mandalo) raises the possibility of an interaction zone. Towards the North (NW, NE) we can expect no interactions; here, the question is the extent of obsidian transport and the authenticity of the pieces. The furthermost pieces reported in this direction are from Zealand, Denmark (approx. 1400 km ACF from the sources); we had no occasion to study these pieces as yet.

**Conclusion**

Obsidian was an attractive and much desired commodity in prehistoric times and it is a popular and successful subject for the research of prehistoric societies. Non-destructive methods and representative comparative sample collection help to make our work more efficient.

There are, though, some important things to keep in mind:

1. we still do not know the exact location of the most important primary C1 sources (material of Cejkov and Kasov workshops, Kasov and Nyírlugos cores, Bánesz 1967, Bánesz 1991, Kaminska & Duda 1985)

2. obsidian distribution and obsidian trade is only one element of a most versatile and colourful prehistoric network of connections and should be interpreted accordingly among “sourceable” and “non-sourceable” goods.

**References**

**Acknowledgement**

The invaluable support of the following grants and projects is acknowledged here: OTKA T- 025086, OTKA K-62874, OTKA K-100385, TéT Hungarian-Croatian bilateral project, CHARISMA EU FP7 projects.

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Meetings and Events in 2013.

Training course on Dating CH Artefacts, Zagreb, Croatia, May 2013

Regional Training Course on Dating of Cultural Heritage Artefacts using Nuclear Analytical Techniques was held in Zagreb, Croatia, from 20 to 24 May, 2013. The course was organized within the IAEA project RER/0/034 entitled „Enhancing the Characterization, Preservation and Protection of Cultural Heritage Artefacts“. One objective of the this RER project is to improve the characterisation, preservation, protection and authentication of cultural heritage (CH) artefacts by effective utilisation of nuclear analytical techniques (NATs) and radiation technology, and absolute dating by applying nuclear techniques is essential for the proper characterization of CH objects.

The course was organized jointly by the IAEA and the Croatian counterpart institutions: Ruđer Bošković Institute (RBI, through its three laboratories: Laboratory for Ion Beam Interactions, Laboratory for Measurements of Low-level Radioactivity, and Radiation Chemistry and Dosimetry Laboratory), Croatian Conservation Institute (CCI) and the Academy of Fine Arts of the University of Zagreb, and Ines Krajcar Bronić from RBI was the Course Director. There were 32 participants selected from 17 countries: Albania, Azerbaijan, Bosnia and Herzegovina, Bulgaria, Hungary, Lithuania, The Former Yug. Republic of Macedonia, Montenegro, Poland, Portugal, Romania, Serbia, Slovenia, Slovakia, Turkey, Ukraine and Croatia.

The main part of the course was devoted to the absolute dating methods most often used in dating of CH artefacts: radiocarbon ($^{14}$C) dating, luminescence (TL and OSL) dating, and dendrochronology. Lectures on radiocarbon dating included the basis of the method and its restrictions, sample preparation techniques with special emphasis on the preparation for the Accelerator Mass Spectrometry (AMS) measurement, measurements techniques, data evaluation, presentation, calibration, and interpretation, as well as numerous archaeological case studies. Since radiocarbon can be used also as a natural tracer, a lecture on its application in environmental and geochronological research and climatology and oceanology was also given. The lectures on radiocarbon dating were prepared by the RBI staff (Nada Horvatinić, Bogomil Obelić, Jadranka Barošić, Ines Krajcar Bronić). Thermoluminescence (TL) and Optically-stimulated luminescence (OSL) as methods of dating ceramics and bricks were presented both through the physics lying under the methods and numerous examples that helped explaining what time periods were actually determined. Dendrochronology is the most precise method for dating wood and at the
Meetings and Events in 2013.

same time related to radiocarbon dating through calibration of radiocarbon ages to obtain calendar ages. These lectures were given by Marco Martini from University of Milano Bicocca, Milano, Italy. The lectures were supplemented by the relevant demonstrations in the analytical laboratories: in the radiocarbon and dosimetry laboratories, and at the radiation treatment and accelerator facilities.

In addition to these main topics, the lectures included also radiation treatment of CH objects, and especially the Croatian experience in treatment of objects damaged during the war. This topic was presented by Irina Pucić, Tanja Jurkin, Branka Katušin Ražem and Dušan Ražem. The complementary NATs that give additional information on the structure and/or composition of the material and that can be used for indirect dating of objects based on already published information on features distributed along the time scale, included X-ray fluorescence technique (presented by Vladan Desnica, Academy of Fine Arts), Raman spectroscopy (presented by Vlasta Mohaček-Grošev, RBI) and various accelerator-based techniques (PIXE, PIGE, ERDA, etc.) presented by Stjepko Fazinić (RBI).

One afternoon was devoted to the visit to the CCI, where Mario Braun presented the Institute itself, Iskra Karniš-Vidović presented the process of restoration of the Croatian Apoxiomenos including radiocarbon dating of the organic remains discovered in the statue, Lea Čataj presented several archaeological investigations in which radiocarbon dating was successfully used, and Domagoj Mudronja showed the new Science Laboratory of the CCI.

As usually, the IAEA sponsored the Hospitality event on Monday evening, where all the participants, lecturers and auxiliary staff had a chance to talk to each other in a friendly atmosphere. On Tuesday evening the whole course was invited by the City Museum of Zagreb to the exhibition “In the service of archaeology”. The authors of the exhibition, Zoran Mašić and Aleksandra Bugar, summarized various sciences used in modern archaeology for interdisciplinary investigations. The participants had also a chance to go through the permanent exhibition of the Museum.

At the beginning of the course all the participants were divided into 6 groups named “Wood and charcoal”, “Ceramics, pottery, bricks”, “Bones, teeth, antler”, “Metals and alloys, glass”, “Paper, parchment” and “Textile, leather, canvas”. The assignment of each group was to follow which of the presented techniques is appropriate for their material and what can be inferred from these analyses, and to present their observation in a form of a project assuming that they obtained an artefact made of the given material for the analysis. The presentations of the participants, given on the closing day, showed that the participants followed the lectures and learned what was presented during the course, and also showed a great creativity in the presentations of their “projects”. Participants and lecturers also actively participated in the Round-table discussion on Wednesday afternoon. The lecturers on radiocarbon and TL dating once more stressed the importance of keeping in mind what is being dated: radiocarbon dating gives the time elapsed since the death of the organism (e.g., cutting the wood) and not the date of the use of the material (wood). Similarly, TL dating gives the time elapsed since the last firing of the pottery. Concerning radiocarbon dating, the discussion on sampling, sample storage, sample transport and treatment was useful to all the participants, especially when the sampling for AMS method (small samples, < 1 mg) is concerned.

Prof. Martini pointed out that pottery samples from archaeological excavations for TL dating should not be taken away without prior consultations with the TLD experts because various environmental conditions have to be determined to obtain realistic TL age. Both the lecturers and the participants agreed that all the presented methods are simple “at first approximation”, but when we go more closely into the details, the things become more complex, but for the proper interpretation of the obtained results (ages) one has to take into account also the possible pitfalls of the methods. It was also pointed out that for complete investigation of an artefact multi-proxy studies are necessary since each of the applied techniques can give a contribution to the final characterization of the object. A few additional remarks on presentation of radiocarbon results and use of calibration software were given. Participants were free to ask questions, and one of them was “What was the strangest sample that you have dated in the Radiocarbon laboratory?”. The answer was human hair and an Egyptian mummy. Some of the participants proposed to start an intercomparison study on radiocarbon dating, but it was pointed out that this year or latest in 2014, a new international radiocarbon intercomparison exercise will be organized by the University of Glasgow. Dinara Abbasova proposed to have some joint application to either FP7 or IAEA Coordinated Research Project, and willingness for joining was expressed.

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