### M. Isabel Prudêncio

The Cultural Heritage and Sciences (CHS) group focus its research activity mainly on the valorisation and preservation of the archaeological and the geological patrimonies. Archaeometry, geochemistry of the earth surface and geological environment are the main areas of study. The research is largely based on the use of nuclear methods of analysis.

The analytical methods associated to the CHS group, include chemical analyses by the instrumental neutron activation analysis (INAA) - comparative method, and two absolute dating techniques - radiocarbon dating ( $^{14}$ C) and luminescence dating (TL-OSL). These methods, unique in Portugal, allow the chemical characterisation of archaeological and geological materials and the establishment of chronologies.

Geochemistry (especially of trace elements), crystal chemistry and mineralogy of geological materials (clays and rocks) are done applied to cultural assets (ceramics and monument stones, for instance). These studies also support environmental projects through the geochemical natural background knowledge, in order to evaluate anthropogenic influences, with the final goal of preserve classified natural environments.

The financed research projects are in collaboration with other national and international laboratories and universities. This group also has research activities directed to well defined end users through the ITN-IPA protocol, and also contracts/services made with other private or public institutions (ERA, EDIA, Valorsul, Robbialac, City Halls, etc).

The research activities of the CHS group can be divided in the following domains:

(1) Movable Cultural Assets (Ancient ceramics production, technology and provenance)

(2) Immovable Cultural Assets (Historical buildings and monuments);

- (3) Absolute Dating Radiocarbon Unit;
- (4) Absolute Dating Luminescence laboratory;

(5) <sup>57</sup>Fe Mössbauer spectroscopy in Geology and Archaeology;

(6) Geological Environments;

The principal achievements in each research domain during 2003 are as follows:

(1) Movable Cultural Assets - enlargement of the number of pottery Roman workshops studied in *Lusitania* (Center and South of Iberian Peninsula) in order to a better provenance and trade establishment within the roman territory based on chemical characterization; Ceramic production technology and provenance since the Pre-History to Roman times in numerous Portuguese archaeological sites, based on chemical and mineralogical characterization of pottery and potential raw materials.

(2) Immovable Cultural Assets – evaluation of the prevailing degradation state of monument stones – Évora Cathedral and Torre de Moncorvo Church, and identification of the main decay patterns, aiming at to identify and distinguish pollution sources that affect those monuments.

(3) Absolute Dating – Radiocarbon Unit. Development and application of  ${}^{14}C$  dating method units in archaeological and geological materials.

(4) Absolute Dating – Luminescence laboratory. Calibration of radioactive sources and equipment proceeded and protocols were established for different luminescence dating applications. New projects began in the frame of the ITN-IPA protocol, as well as services to the international scientific community.

(5) <sup>57</sup>Fe Mössbauer spectroscopy in Geology and Archaeology: this method (available at the Solid State group of ITN) has been applied to ancient ceramics, monument stones and geological environmental samples.

(6) Geological environments: geochemical and mineralogical studies applied to lagoon and estuarine environments were done, aiming to identify the main pollutants and thus evaluate anthropogenic inputs, in order to establish a quality control. Identification of the main factors of environmental disequilibrium, in particular in Sete Cidades (Azores), El Meleh (Tunisia), and Nador (Morocco) lagoons, in a view to its social and natural better management are ongoing FCT and European projects.

The improvement of the scientific research based on nuclear techniques of analysis and its application to the Portuguese and international cultural heritage, as well as to geological environments, is a long-term goal of the CHS group.

# **Cultural Heritage and Sciences**

# **Research Team**

### Researchers

- M.I. PRUDÊNCIO, Principal Researcher (Group Leader)
- A. M. SOARES, Auxiliary Researcher
- M. I. DIAS, Invited Auxiliary Researcher
- D. RICHTER, Invited Auxiliary Researcher\*
- K. PRZEGIETKA, Post-Doctoral

### **Collaborators**

- M.A. GOUVEIA, Collaborator

### Students

- A. JORGE, PhD Student
- M. J. TRINDADE, PhD Student
- S. VILELA, MsC student
- C. CAPITÃO-MOR, graduation student

### **Technical Personnel**

- L. FERNANDES
- A. AMARO
- R. MARQUES
- D. FRANCO
- G. CARDOSO

### Funding (€)

<b>Research Projects:</b>	74.771,72
Services:	45.507,65

Total: 120.279,40

### **Publications**

Journals:	7 and 3 in press
Proceedings:	14
Conf. Communications:	21
Other publications:	2

\* Until February

### Movable Cultural Assets Ancient Ceramics Production, Technology and Provenance The Upper Mondego Basin

M. I. Dias, M. I. Prudêncio, M. A. Gouveia<sup>\*</sup>, A. Jorge<sup>1</sup>, A. C. Valera<sup>2</sup>

### Objectives

The purpose of this archaeometric study is to emphasis on continuities or innovations in ceramic production and on raw materials resources of local populations through the third millennium BC.

### Results

(1) Fornos de Algodres Project

This project includes all the archaeometric approach of a running PhD Thesis in archaeology, which includes a Recent Pre-Historic settlement network in Central Portugal: the fortified settlements of Castro de Santiago – CAS (Chalcolithic) and of Fraga da Pena -FP (Early Bronze Age) and the settlement of Malhada – MAL (Chalcolithic/Early Bronze Age).

Chemical differences in pottery allowed the definition of several compositional groups for two of the sites, such as the FP beakers. In general the CAS sherds have a more homogeneous chemical composition. This fact reflects the probable use of different combination of raw materials in FP and MAL sites, in contrast with the exploitation of only one kind of clays in almost all of the studied ceramics of CAS. In both cases the clay materials used as paste are mainly local. In the CAS site most of the ceramics point to a granitic origin, and only a few to a sedimentary source (Fig. 1 - Cluster 4). In MAL site a larger group of ceramics also points to the use of clays from granite origin, a smaller group to dolerite vein supply and a few outliers were defined (Fig. 1 Cluster 2 and 3). In the FP site the potters must have used the weathered doleritic veins, sometimes with the addition of granitic temper (Fig. 1 - Cluster 2). In this site some, but important typological ceramics appear to be imported. What can be asked is if this dual resource exploitation option is intentional, showing a distinct technological choice, or only reflects the raw materials availability, as next FP are concentrated more dolerite veins, although some of those veins also occurs in the neighbourhood of CAS.

### Published, accepted or in press work

 M.I. Dias, A.C. Valera, M.I. Prudêncio. Pottery production technology through out the 3<sup>rd</sup> millennium B.C. on a local settlement network in Fornos de Algodres, central Portugal, 7<sup>th</sup> *European Meeting on Ancient Ceramics*, Lisboa, Portugal (in press) One important difference already noted is that in FP the attribution of especial symbolic meanings to some typological pottery groups seems to interfere in the productive phase, the case with the local bell beakers that reflect special careful production procedure. On the other hand, at CAS and MAL, even if certain recipients could have been used in different ways and with different meanings, those differentiations are not reflected in the production technological procedures.



Fig. 1 - Geochemical features of ceramics of the three sites and local clay materials.

(2) Archaeometry of Pre-historical sites from the upper Mondego basin: the Penedo da Penha and Buraco da Moura cases

A collaboration project with the University of Sheffield - Department of Archaeology and Prehistory is in course, after the successful work of a dissertation of a Masters Thesis (MSc) in 2002. This work continued as a PhD project with the same university, adding more samples, more sites of other chronologies and also field-work regarding provenance with local and regional clay materials sampling. This project is in direct correlation with the Fornos de Algodres one, being the sites excavated by the same archaeologist, having the potential of giving for Beira Alta region an important overview of ceramic technology, production and provenance from Neolithic, through Chalcolithic to the early Bronze Age.

 Jorge, P.M. Day, A.C. Valera, M.I. Dias, M.I. Prudêncio. Ceramics, style and exchange in the early Neolithic upper Mondego basin: a technological approach, 7<sup>th</sup> European Meeting on Ancient Ceramic Lisboa, Portugal (in press)

<sup>1</sup>University of Sheffield – Department of Archaeology and Prehistory, England <sup>2</sup>GAFAL archaeologist (C.M. F. Algodres), Portugal Era Arqueologia S.A. Portugal ; \* Collaborator

# Movable Cultural Assets Roman Epoch ceramics ITN-IPA Protocol

M. I. Dias, M. I. Prudêncio, M. A. Gouveia\*, A. Arruda<sup>1</sup>, J. Raposo<sup>2</sup>,

### Objectives

The ITN-IPA protocol represents a huge step in order to increment archaeometry in Portugal, especially concerning the characterisation of archaeomaterials.

In 2003 CHS group has eleven roman epoch ceramic characterization running projects within the ITN-IPA protocol. For all of them analytical results were obtained, and for some important research objectives were achieved. We will enhance the following:

A – "The "Alcáçova de Santarém" during Roman Epoch". Archaeologist: A. M. Arruda

B – "Characterization of roman ceramics from *Sellium* (Tomar) ". Archaeologist: S. Ponte

C – "Roman ceramics from the Tagus estuary: production and consumption centers". Archaeologists: J. Raposo

### Results

A – "Alcáçova de Santarém", an archaeological site located in central Portugal, on a plateau by the Tagus river, has evidences of occupation since the VIII<sup>th</sup> century B.C.. In Roman times, *Scallabis* (nowadays Santarém) was an important roman city of *Lusitania*. The results obtained showed that the imported products derived from at least 4 amphorae production regions and related foodstuff. The comparison with well-known amphorae production centers point to the importation of: (1) fish products from Tagus estuary production sites such as "Quinta do Rouxinol" in *Lusitania*; (2) wine, oil as well as fish products from Guadalquivir basin (*Baetica*); (3) wine from Italy; and (4) oil from Cartago.

### Published, accepted or in press work

- M.I. Prudêncio, M.I. Dias, J. Raposo, M.A. Gouveia, C. Fabião, A. Guerra, J. Bugalhão, A.L. Duarte, Sabrosa, Chemical characterisation of amphorae from the Tagus and Sado estuaries production centres (Portugal). *Ceramic in the Society. Proceedings of the* 6<sup>th</sup> International Meeting on Ancient Ceramics, Fribourg, Switzerland (2003), (S. Di Pierro, V. Serneels, M. Maggetti, eds.), 245-253.
- M.I. Dias, M.I. Prudêncio, J. Raposo, C. Fabião, A. Guerra, J. Bugalhão, A. Sabrosa, A.L. Duarte, M.A. Gouveia, Amphorae from the Lower Tagus (Portugal)

B – The archaeological excavations done in *Sellium* (Tomar, Portugal) revealed the existence of a large number of roman *amphorae* of various types and chronologies, and *doliae*. The results obtained showed the existence of amphorae and *doliae* imported from known production centers, a few outliers (unknown origin) and one group that may correspond to a local production. Importation is also possible from wine and oil *amphorae* from the South of the Iberian Peninsula (*Baetica*) during the I<sup>st</sup> and II<sup>nd</sup> centuries; import of fish products and wine *amphorae* from Tagus basin production centers since the I<sup>st</sup> cent till the Vth century; and from Peniche since the I<sup>st</sup> century till Augusto/Tiberio times.

C - The archaeological work carried out at the Tagus estuary - Roman sites of P. Cacos (PC), Q. Rouxinol (QR) and R. Correeiros (CR) - allowed to understand the crucial importance of this lower course of the river to the economy at the time. At QR two kilns and possible remains of a third one were found. CR, in Lisbon, was occupied by several fish-salting industries. Results obtained allows to clear differentiating amphorae production within the two studied Tagus basin sites, presenting PC the higher contents of Na, Co, As and especially U, and QR the lower amounts of U, and the higher of Fe. A patent relationship between QR and the majority of CR ceramics is possible to establish, even a more chemical heterogeneity exists in the latter, as expected.

- 3. production center: review of five years of archaeological and archaeometric research, 7<sup>th</sup> *European Meeting on Ancient Ceramics*, Lisboa, Portugal (in press)
- M.I. Prudêncio, M.I. Dias, A.M. Arruda. Importation of food products in "Alcáçova de Santarém" during Roman epoch, 7<sup>th</sup> European Meeting on Ancient Ceramics, Lisboa, Portugal (in press)
- M.I. Prudêncio, M.I. Dias, A.M. Arruda. Amphorae in Sellium (Portugal) from I<sup>st</sup> century to V<sup>th</sup> century – importation and regional production, 7<sup>th</sup> European Meeting on Ancient Ceramics, Lisboa, Portugal (in press)

<sup>1</sup>University of Lisbon; <sup>2</sup>Ecomuseu Municipal do Seixal; \*Collaborator

### Movable Cultural Assets Pre-historic ceramics ITN-IPA Protocol

M. I. Dias, M. I. Prudêncio, M. A. Gouveia\*, A. C. Valera<sup>1</sup>, M. Lago<sup>2</sup>

### **Objectives**

CHS group has three running projects within the ITN-IPA protocol, of pre-historic ceramic characterization. Perdigões archaeological site is one of the most important Chalcolithic settlements in South of Portugal, due to its size, spatial organization and archaeological material record (ceramics, lithic artefacts, limestone and bone idols, pecten shells, bone combs, adorn artefacts, metallurgy, human and animal bones).

Ceramics of this site comprise the typical morphologies of Late Neolithic and Chalcolithic of the SW of Iberia. Previous work highlights some differences between domestic and funerary pottery, presenting the last a higher chemical heterogeneity.

More samples were studied of ceramics and also clay materials derived by weathering from schists, greywackes, diorites, gabbros, volcanites, dolerite veins, schists with metabasites and also Tertiary clays, representative of regional geological contexts, aiming to establish potential raw materials for Perdigões pottery, so one can ascertain their provenance as well as the existence of diversity in ceramic productions according to different purposes/uses.

#### Results

A multivariate statistical approach distinguishes one main group, which includes around 50 % of the analyzed samples, and a second one about 30% of the samples, including ceramics of both domestic (in majority) and funerary context. The third group (20 %) is mainly constituted with funerary ceramics of tomb 1 (Fig. 1 and 2).

A regional origin for most of the ceramics is probable, with a spread of resources in the funerary pottery. The third group is the only one, which point to the use of weathered schists. On the other hand quartzodiorite derived clays were probably the most used raw material, as well as diorites and associated gabbros, and in some cases also Tertiary clays.

Considering one of the main questions (the possibility of a specific production of pottery recipients used in

### Published, accepted or in press work

1. M.I. Dias, M.I. Prudêncio, A.C. Valera, M. Lago & M.A. Gouveia, Composition, technology and functional features of Chalcolithic pottery from Perdigões, (Portugal). Reguengos de Monsaraz Proceedings of the 33rd International Symposium on Archaeometry, Amsterdam, The Netherlands (in press)

funerary rituals), it seems that diversified raw materials were used in pottery of funerary rituals. This could reflect different origins, perhaps due to the use of Perdigões necropolis by peripheric, but dependent, communities.



Fig. 1 - Cluster analysis of Perdigões ceramics.



Fig. 2 - K-means clustering method applied to Perdigões ceramics.

 M.I. Dias, A.C. Valera, M. Lago, M.I. Prudêncio,. Provenance and production technology of pottery from Perdigões, Reguengos de Monsaraz, 7<sup>th</sup> European Meeting on Ancient Ceramics, Lisboa, Portugal. (in press)

<sup>&</sup>lt;sup>1</sup>Era Arqueologia S.A. Portugal

<sup>&</sup>lt;sup>2</sup> Era Arqueologia S.A. Portugal

<sup>\*</sup>Collaborator

## Movable Cultural Assets Alqueva Dam Impact - Era Arqueologia Projects

M. I. Dias, M. I. Prudêncio, M. A. Gouveia\*, A. C. Valera<sup>1</sup>, J. Albergaria<sup>1</sup>, S. Brazuna<sup>1</sup>

### **Objectives**

The construction of the Alqueva dam is a project evolving large impact studies in several areas, with specialists of the most varied subjects. Considering that a huge area will be submersing, a program of archaeological rescue was design and Era Arqueologia S.A. has executed several excavations in sites of various chronologies.



Fig. 1 – Alqueva Dam

Archaeometric study has been done in several sites within three archaeological running projects.

In the left bank of Guadiana river four sites were studied: (1) recent prehistory site of Moinho de Valadares (MV), a settlement occupied from late Neolithic to early Bronze Age; (2) Monte do Tosco (MT), an enclosure Chalcolithic site, reoccupied during the early Bronze Age; (3) Monte da Pata 1 (MP) and Castelo das Juntas (CJ), two late Iron Age sites.



Fig. 2 – Moinho de Valadares and Monte do Tosco sites

In the right margin, in Monsaraz, the site of Xerez de Baixo 13 has also been studied with a chronology of the  $1^{st}$  century a.C. which may extend to the  $2^{nd}$  century a.C.

### Published, accepted or in press work

- M.I. Dias, M.I. Prudêncio, F. Rocha, J. Albergaria, A. Jorge, A.C. Ramos, S. Melro, Barris ibéricos no SW peninsular. Resultados preliminares de um estudo arqueométrico. O Arqueólogo Português (in press).
- M.I. Dias, J. Albergaria, A. Jorge, A.C. Ramos, S. Melro, M.I. Prudêncio, F. Rocha. Ceramic productions of Late Iron Age archaeological sites of the left bank of Guadiana river, Portugal, 7<sup>th</sup> European Meeting on Ancient Ceramics, Lisboa, Portugal (in press).

### Results

Ceramics from MV site present two main types of chemical composition, independent of typology, and a third one regarding building materials. One group is mainly associated to Neolithic-Calcolithic ceramics. For MT ceramics is more evident the differentiation according to the chronology. Comparing both sites, it is interesting to enhance geochemical similarity between Chalcolithic ceramics, related with quartzodiorites and diorites and gabbros derived clays. Also a chemical correspondence can be established between MT bell beakers, MT bronze ceramics and MV building materials with schists and tertiary clays. For both sites most of ceramics were fired in temperatures near 800°C, and in some cases traces of mineral phases transformations indicate higher temperatures.

Obtained results of MP and CJ point to a possible use of local clay deposits (mainly the schist ones) for most of the ceramics, as well as for the building materials. In some cases the use of weathered material derived from quartzodiorites is also probable. In the case of CJ, it is possible to define two main groups based on chemical data, none of which with any specific typological correlation. In MP, ceramics appear to have a more homogeneous chemical composition, although two outliers may be defined. For the majority of the cases, mineralogical phases of transformation indicating very high temperatures during the firing process were not detected (should not overcome 800°C).

Xerez de Baixo results point to a homogeneous chemical composition for most of ceramics, independently of typology, and confirm a local production using local clay materials, mainly quartzdiorites and diorites and associated gabbros. No mineralogical phases transformation indicating very high temperatures during the firing process, which shouldn't have past 800°C, were detected.

- 3. M.I. Dias, S. Gomes, S. Brazuna, M.I. Prudêncio, F. Rocha. Ceramic productions of Xerez de Baixo in the right margin of Guadiana river, Portugal, 7<sup>th</sup> European Meeting on Ancient Ceramics, Lisboa, Portugal (in press).
- 4. M.I. Dias, A.C. Valera, M.I. Prudêncio, F. Rocha. Ceramic productions during the 3<sup>rd</sup> early 2<sup>nd</sup> millennium B.C. in thr left bank of the Guadiana river, Portugal, 7<sup>th</sup> European Meeting on Ancient Ceramics, Lisboa, Portugal (in press)

<sup>&</sup>lt;sup>1</sup>Era Arqueologia S.A. Portugal; \* Collaborator

### Immovable Cultural Assets Historical Buildings and Monuments

M. I. Prudêncio, M. A. Gouveia \*, C. A. Alves<sup>1</sup>

### **Objectives**

Several geogenic and anthropogenic pollution can contribute to the alteration/contamination and degradation of historical buildings and monuments. In order to establish well-defined and scientifically based conservation strategies it is necessary to identify and to distinguish the pollution sources that affect the monuments. The evaluation of the prevailing degradation state of the stones, the identification of decay patterns and the understanding of the geochemical cycles of the pollutants will contribute to understand the susceptibility of different igneous stones under different pollution settings. This research has been supported by FCT.

#### **Results**

A detailed characterization of the stone decay aspects, and the study of pathologies in the Church of Torre de Moncorvo (Fig. 1) and Braga monuments, was done with sampling increase.

Also field and laboratory work has proceeded in the Alentejo region, particularly in granites of Évora region (quarries and outcrops), which able a comparison study with Sé de Évora church stones.

A collaboration project with the University of Minho -Department of Earth Sciences, including a Masters Thesis (MsC), is in course concerning the effects of different pollution sources in the decay of igneous stone monuments of the north of Portugal. The chemical characterization of soils of the surrounding area of selected monuments, as well as a detailed study of the coatings of the monument stones, is being done.

Important results have already been achieved, namely those concerning secondary deposits of the stones, of different compositions, which contribute to the degradation of the monument stones. In figure 2 it is presented one type of these kinds of deposits and its chemical composition.

### Published, accepted or in press work

1. M.I. Prudêncio, Aplicação do método de análise por activação em estudos de degradação e conservação de monumentos megalíticos



Fig. 1. Main façade of the Torre de Moncorvo Church.



Fig. 2 - SEM - EDS analyses of secondary deposits of monument stones.

construídos em rochas graníticas, *Arqueologia, Arte e Radioactividade* (2003), ITN ed., 13-15.

<sup>&</sup>lt;sup>1</sup>Univ. Minho; \* Collaborator

# **Absolute Dating - Radiocarbon Unit**

M.I. Prudêncio, A.M. Soares, R. Marques

### **Objectives**

This dating method supports the scientific community (archaeological and geological) in solving chronological problems, as well as other entities which need this kind of information, like city halls and water companies.

Several projects are running with the archaeological community, financed by the Portuguese Institute of Archaeology (ITN-IPA protocol).

This laboratory has also services running with the geological community, contributing to date geological samples and events.

Considering that this dating method is only available at ITN in Portugal (Fig. 1), is of major importance to continue with the very good performance of the laboratory, increasing projects and services, and also continue to approach scientific community and students, within the "Ciência Viva" program and numerous visits from schools and universities.

### Results

During this year about one hundred samples of different types were processed, such as human and animal skeletal bones, coal, mud, wood, peat, shells (in majority) and water.

This dating technique able dates various kinds of materials from several ages. The obtained results vary from  $34040 \pm 2100$  BP,  $6060 \pm 70$  BP,  $1740 \pm 70$  BP to Modern Age.

Eight new projects were approved within the ITN-IPA protocol and started during this year:

### Published, accepted or in press work

- A.M.M. Soares , A duna de Magoito revisitada. *Revista Portuguesa de Arqueologia* 6(1) (2003) 83-100.
- A.M.M.; Soares, P. Barros, P. Queiroz, J.M.A. Dias, L. Rocha, W. van Leewaarden, Lagoonar Deposits Eastward of Quarteira (Algarve) – Preliminary Results, *Thalassas*.19(2b) (2003) 180-181.

- Meghalitic study in Central Alentejo and the Megalithic necropolis of the TÊRA-PAVIA hill.

- TEMPOAR II- Territories and mobility in the "Alto-Ribatejo".

- ESTRELA – The medieval occupation of the NW side of the Serra da Estrela".

-The fortified site of Cárcoda in the Proto-history of central Portugal.

- Natives and Phoenicians in Almaraz.

- The fortified site of Castro de Palheiros. Study and divulgation of one archaeological site occupied in the Chalcolithic and Iron Age .

- The dolmen of Carniçosas (Figueira da Foz). Excavation, valorization and dinamization.

- The Necropolis of São Bartolomeu (Monsaraz).

This laboratory also supported research work for graduation and PhD thesis, in collaboration with other ITN group, and dated samples as services for private and public entities and institutions.



Fig. 1 – Vacuum line for benzene synthesis at  ${}^{14}C$  laboratory.

- A.M.M. Soares, A.C. Sousa– Aeolianites of the Coastal Region of Lisbon – a contribution to their dating, *Thalassas*. 19(2b) (2003) 182-183.
- A.M.M. Soares, A datação por radiocarbono de conchas marinhas – novos dados, Arqueologia, Arte e Radioactividade (2003) ITN ed., 11-12
- 5. A.M.M. Soares, A.C. Sousa,- Cronologia Absoluta. In "São Julião". *Cadernos de Arqueologia de Mafra, 2* (in press).

### **Absolute Dating - Luminescence Laboratory**

M.I. Prudêncio, D. Richter, K. Przegietka

### **Objectives**

The optimization of the luminescence dating laboratory using various techniques and materials is a general objective. The promotion of this dating technique in national and international communities was done and a significant feedback occurred.

The calibration using various techniques and materials was a major goal of the good performance of this dating laboratory. Several protocols were established regarding the three main luminescence dating applications of TL, B-OSL, IRSL, applied to the four main materials of heated flint/quartzite, coarse grain quartz sediment, polymineral fine grain sediment and pottery/bricks.

Thus the development and application of the technique for dating various kinds of samples was also a major objective.

### Results

The calibration results were found to be good to excellent, and the laboratory is now perfectly running.

Quite a few important dates were obtained within the running projects, namely OSL results for timing of glaciations in the Pyrenees (Andorra) from glaciofluvial deposits, and TL-dating from flint samples, which shows signs of heating, from Schoningen Palaeolithic site (Germany) and Hummal Palaeolithic site.

Regarding luminescence dating of small samples of heated silex, new ways in dating archaeological sites were found by employing the orange-red emission and a single aliquot regeneration (SAR) protocol.

In Fig. 1 the variation of the TL sensitivity of the orange-red emission due to heating is shown. No significant change is observed, which allows the use of a very simple SAR-protocol, consisting of 2

### Published, accepted or in press work

- D. Richter, A. Zink, K. Przegietka, G.O. Cardoso, M.A. Gouveia, M.I. Prudêncio, Source calibrations and blind test results from the new Luminescence Dating Laboratory at the Instituto Tecnológico e Nuclear, Sacavém, Portugal, Ancient TL 21 (2003) 1-7.
- 2. E. Trinkaus, A.E. Marks, J.P. Brugal, S.E. Bailey, W.J. Rink, D. Richter, Later Middle Pleistocene Human Remains from the Almonda Karstic System, Torres Novas, Portugal.

regeneration points, which are set just above and below the intensity of the natural signal. Table 1 shows the results obtained with the new SAR orangered protocol and standard procedures on the same samples.



Fig1. Recycling of a single aliquot for each sample, normalized to the first cycle

SAMPLE	UV-BLUE	ORANGE-RED
	(GY)	(GY)
GK9	$\textbf{11.7} \pm 0.4$	$\textbf{10.9} \pm 0.4$
AUB168	<b>57.8</b> ± 1.7	<b>68.0</b> ± 1.6
JA324	<b>56.2</b> ± 3.1	<b>65.4</b> ± 1.4

Table 1: Comparison of equivalent dose (Gy) determinations using standard (UV-blue) and the new SAR (orange-red) protocol.

During 2003, two projects were approved within the ITN-IPA protocol and selection and preparation of samples are ongoing.

- (1) Territories and mobility in Ribatejo;
- (2) Paleoalmonda.

Journal of Human Evolution **45** (2003) 219-226.

 W.J. Rink, D. Richter, H.P. Schwarcz, A.E. Marks, K. Monigal, D. Kaufman, Age of the Middle Palaeolithic Site of Rosh Ein Mor, Central Negev, Israel: Implications for the Age Range of the Early Levantine Mousterian of the Levantine Corridor. *Journal of Archaeological Science* **30** (2003) 195-204.

## <sup>57</sup>Fe Mössbauer spectroscopy in Geology and Archaeology

J. C. Waerenborgh<sup>1</sup>, A. P. Jesus<sup>2</sup>, A. Mateus<sup>2</sup>, J. Figueiras<sup>2</sup>

### **Objectives**

Identification Fe-containing of phases and determination of the oxidation state and distribution of Fe in different crystallographic sites. This investigation is applied to the establishment of metallogenetic models for ore-forming systems and in the study of the mineralchemistry behaviour of Fe in weathering processes or in the alteration mechanisms of rocks used as building materials and in clay firing.

### Results

Iron is the most abundant element on the Earth as a whole, making up to 30% of its total mass. It constitutes over 80% of the core and is also the 4th most abundant element of the crust. Natural solid materials such as soils, sediments, rocks, etc., typically contain Fe in a concentration that is sufficiently high to allow the observation of good-quality Mössbauer spectra.

In 2003 Mössbauer spectroscopy has identified the magnetic Fe oxide cumulates in the Beja layered

gabbro complex as being maghemites. This complex has been the subject of a detailed investigation by the Geology Dept. of the Fac. Sciences of the Univ. of Lisbon

The grain size of the Fe oxides and hydroxides in soils and sediments around waste disposal sites were estimated by Mössbauer spectroscopy. These results were obtained in the frame of a joint research project which aims at understanding the dispersion of pollutants around waste disposal sites located in climatic and geological settings that are common in Portugal.

In the frame of a joint research project with the Technical University of Lisbon an investigation of the heat induced colour modifications in monument limestones was initiated. This study will allow the estimation of the highest temperature range at which the stones of the cathedral of Lisbon were submitted during an 18th century urban fire.



Mössbauer spectrum of natural magnetite. The inner doublet is due to an impurity phase.

### Published, accepted or in press work

1. P. Jesus, A. Mateus, J.C. Waerenborgh, J. Figueiras, L. Cerqueira, V. Oliveira "Hypogene titanian, vanadian maghemite in reworked oxide

cumulates in the Beja layered gabbro complex" *Can. Miner.* (accepted)

<sup>&</sup>lt;sup>1</sup> Solid State Group of ITN

<sup>&</sup>lt;sup>2</sup> Dept. Geology of Fac. Sciences Univ. Lisbon, P-1749-016 Lisboa, Portugal.

### **Geological environments**

M. I. Prudêncio, M.I. Dias, J. C. Waerenborgh<sup>1</sup>, M.A. Gouveia\*

### **Objectives**

Geochemistry, crystal chemistry and mineralogy studies are applied in environmental and geological projects, with a view of the preservation of classified natural environments and valorisation of natural resources.

The characterization of the natural background is essential for the evaluation of the anthropogenic input in the geological environment. In this way, an important objective of the CHS group is the study of the geochemistry of the earth surface, particularly the behaviour of trace and major elements in secondary natural systems, and the alterations due to pollution actions.

Among the CHS running projects, concerning the quality control and identification of pollutants of geological environments, the following projects are in course:

#### Lagoon environment

A. - Non-point Pollution in the Hydrosphere Due to Agricultural Practices: Source Characterization and Pollutants Mobility in the Vadose Zone

B. - COLASU – Sustainability of Mediterranean coastal lagoon ecosystems under semi-arid climate.

#### Estuarine environment

C. – Continuous monitoring of trace elements in the vicinity of the Municipal Solid Waste Processing Plant (CTRSU).

### Results

Lagoons with different origin are studied: the Sete Cidades lagoon (Fig. 1) corresponding to a volcano crater in S. Miguel Island Azores (FCT project) and two coastal lagoons in the north of Africa -Nador in Morocco and El Meleh in Tunisia (EC project). The chemical characterisation lagoon sediments and soils around the lagoons was done by INAA.

The winter sampling campaign has been done by ITN team in El Meleh, Tunisia (fig. 2) in the lagoon and surrounding region, in order to collect soils, sediments and parent rocks.

### Published, accepted or in press work

 M.I. Dias, M. Suarez Barrios, S. Prates, Las bentonitas de Benavila (Portugal). Caracterización mineralógica e propriedades, Geogaceta 35 (2003) 99-102.

<sup>1</sup> Solid State Group of ITN; \* Collaborator



Fig. 1. Sete Cidades lagoon, S. Miguel Island, Azores.



Fig. 2 – El Meleh winter 2003 sampling campaign.

Regarding the estuarine environment, the study of the evolution of trace elements concentration around the Municipal Solid Waste Processing Plant (Tagus estuary) a continuous monitoring program running since 1998 and financed by Valorsul, was concluded in 2003.

Chemical analyses by INAA of soils, vegetation, water, sediments, and chimney filters of the incinerator were undertaken, so a more accurate evaluation of the pollutants contribution of this central was established.

 B.J.H. Vieira, P.M. Soares, M.I. Prudêncio, M.C. Freitas, A.F. Rodrigues, Caracterização química (terras raras e outros elementos) de solos das Ilhas de Santa Maria e Terceira (Açores, Portugal), Geociências (in press).