3. Major Facilities

Portuguese Research Reactor

As is well known, the Portuguese Research Reactor (RPI) is a 1 MW swimming pool open core reactor. It is in operation since 1961, with an interruption of 3 years in the late eighties for refurbishment which included changes in the fuel enrichment from LEU to HEU. During 1999 all the LEU spent fuel was returned to the USA under an agreement which covers the return of the present fuel until 2009.

It is worth referring that a group of new operators, in training since October 1997, successfully passed their final examinations. This has implied that the new operators were performing, under supervision, essentially all the functions of the operators, including also special ones, like those concerning the return of the spent fuel.

The main users of the reactor are described in Table I. Fig. 1 indicates the reactor usage in terms of irradiation time and number of irradiations in the last decade. It is worth noting that in 1999 there was a significant increase in both the time the reactor was operating at 1 MW and the number of irradiations performed. Although the use is not yet optimal, it is worth noting that while the reactor was running at 1 MW there were, in average, two simultaneous irradiations.

Table I. Use of the RPI in 1999

User	Area	Time (%)
ITN-RPI	NAA	30.6
	Dosimetry	6.6
	BNCT Research	6.3
	Other	7.0
ITN-Chemistry	Isotope Production	21.1
	NAA	11.6
ITN-Physics	Beam Users	2.8
	Isotope Production	2.7
	Other	1.5
Univ. Coimbra-FCT	Isotope Production	0.1
CERN-LHC Division	Fast Neutron Irradiation	9.7

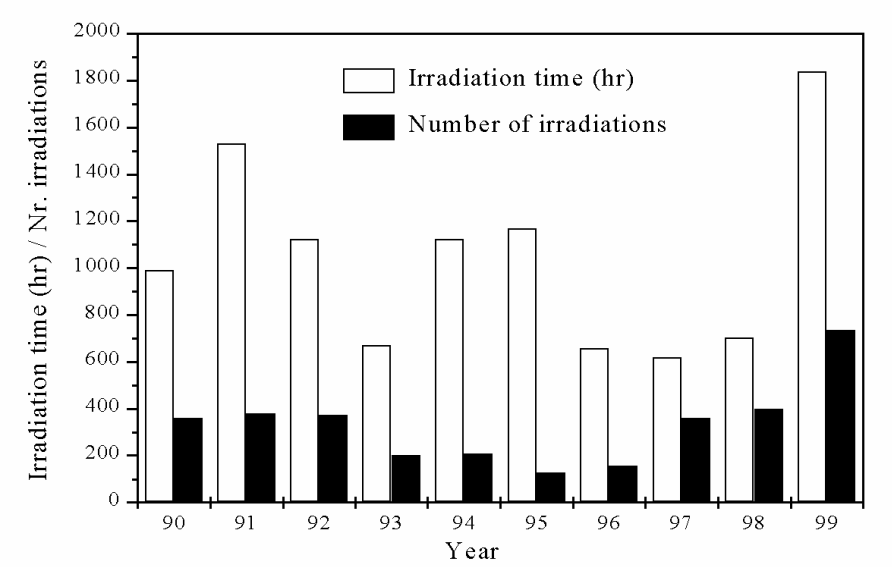


Fig.1. Irradiation time and number of irradiations in the last decade.

Of particular importance is also the admission of two new auxiliary researchers and a post-doc collaborator. The work described elsewhere shows how the new staff has been contributing to the performance of the whole group.

Radiation Technology Unit

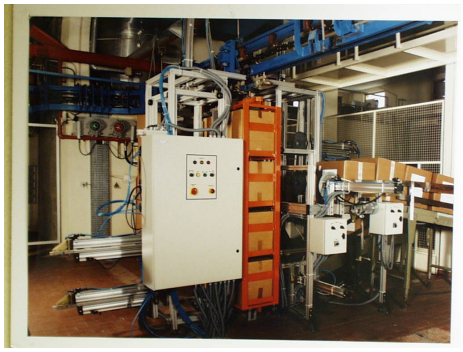
The Radiation Technology Unit (UTR) is a Cobalt-60 facility with a semi-industrial dimension, that was constructed in straight collaboration with IAEA, as a demonstration unit for the national industry. Its main application was defined for medical devices radiosterilisation.

Panoramic views of UTR

UTR began its industrial operation in November 1988 with an activity of aprox. 334 kCi. Nowadays we are supplying irradiation services with an activity of aprox. 71 kCi.

Two operating teams and a prevention team assure the irradiation services between 9:00h to 24:00h from Monday to Friday.

In 1999 UTR stopped the operation during 3 weeks for maintenance and technical assistance. This was the beginning of a great amount of improvements in order to assure a more safety and profitable operation. These improvements were described in the project named *"Upgrade of the operating and security systems of the Radiation and Technology Unit"*.



Load/Unload/Rearrangement system

Expedition of irradiated materials

We have supplied irradiation services such as radiation sterilisation of medical devices and pharmaceuticals, and radiation decontamination of raw materials, herbs, etc, to institutions and companies. Moreover, we have given proper assistance to institutions and industry, made radiation technology transfer a reality, participated in R&D projects and promoted technical operating training.

Ion Beam Laboratory

Fig. 1

The Ion Beam Laboratory has two main equipments: The 3.1 MeV van de Graaff Accelerator and the 210 kV high fluence ion implantor.
The Accelerator utilisation in 1999 was:

Experiments –	97 %
Maintenance –	1 %
Failures –	2 %

The accelerator was upgraded in 1992 and has three beam lines (Fig. 1) with the following techniques: RBS, PIXE, NRA, Channeling, ERDA and NRB with (p, γ).

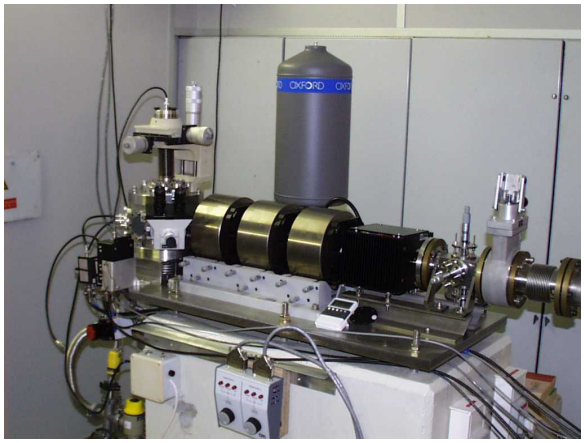


Fig. 2

In the (p, γ) beam line it was recently installed an Oxford type microprobe (Fig. 2) giving beam spots down to 1 μm. It is possible to perform PIXE, RBS, STIM (Scanning Transmission Ion Microscopy) and CSTIM (Channeled STIM) type of analysis.

The 210kV ion implantor (Fig. 3) has been mounted in 1992. It has an implantation chamber with a plate of 30 cm of diameter, allowing the implantation either of big pieces or several samples at the same time with any type of ion. It is possible to carry on the implantations from the liquid nitrogen temperature up to 600º C in a controlled way. The connection of the implantor to the RBS chamber of the van the Graaff Accelerator allows the implantation followed by the analysis without breaking the vacuum. The implantor has been used about 62 days for preparing samples for the on-going research activity. The following ion beams were produced: C^{+}, P^{+}, Co^{+}, Fe^{+}, Cr^{+}, Cr^{++}, Xe^{+++}, Hf^{++}, W^{+}, Ti^{+}, O^{+}, He^{+}, Ar^{+}.

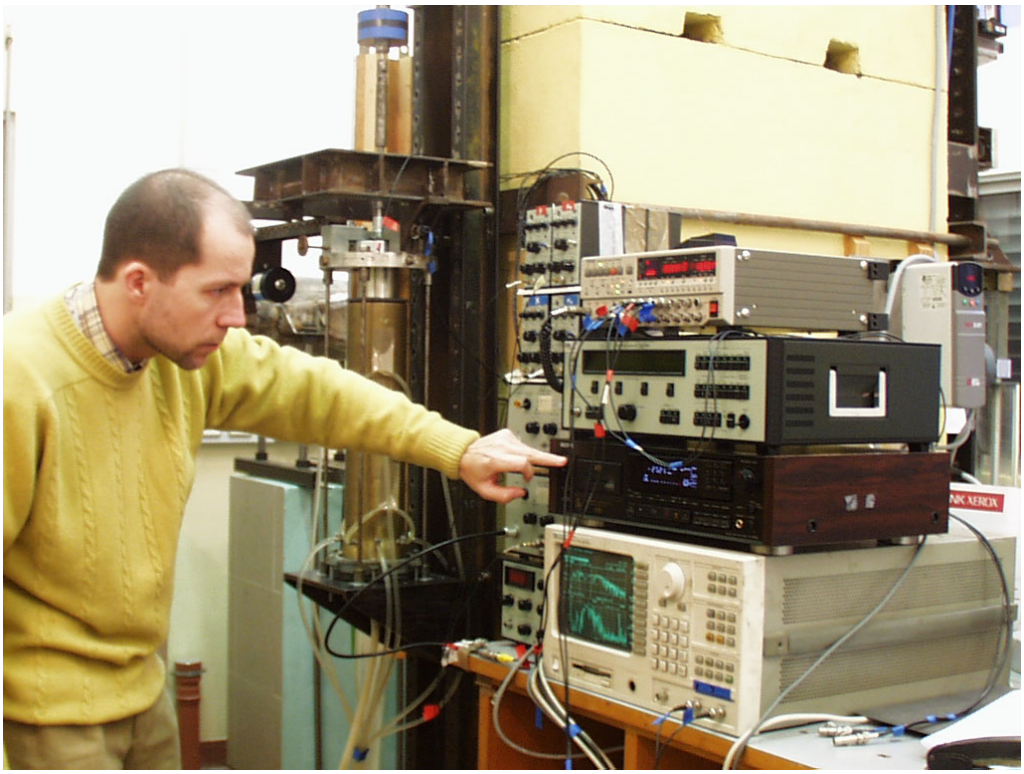
Fig. 3

Applied Dynamics Laboratory

The Applied Dynamics Laboratory (ADL) provides the experimental facilities for our group. It is well equipped for structural excitation, as well as for response measurements and analysis, including:

- signal generators, amplifiers, electro-dynamical shakers
- accelerometers, eddy-current displacement transducers, piezzo-electric force transducers
- precision microphones
- conditioning amplifiers
- sonometer and vibrometer
- stroboscope
- digital data recorder
- spectrum analyser
- PC-based acquisition system
- controller for rotating machinery
- wind-tunnel

The Applied Dynamics Laboratory is equipped for finite element computations, applied to static and dynamic analysis of complex structures. Such computations are performed using CASTEM 2000, a general purpose computer code developed by our partners from CEA (France). More advanced analysis are performed with specific computational tools, developed at *ADL*. The test rigs are designed at *ADL* and developed at ITN workshop facilities.



EPA – Small Angle Neutron Scattering Spectrometer

F.G. Carvalho, F.M.A. Margaça, A.N. Falcão, J.Neves, A.Saraiva, J.S. Sousa and J.F. Salgado

Small Angle Neutron Scattering, SANS, provides one of the best-suited tools for the characterisation of materials microstructure in the range 1-100 nm. It provides information on the shape, size and volume fraction of heterogeneities in the bulk, at the colloidal scale. Nowadays, there is interest in producing homogeneous structures or extremely fine-scale second phases in quite different materials. The characterisation of such materials at this spatial scale is thus particularly important so that the demand for SANS instruments is increasingly high, world-wide.

EPA, the SANS facility designed for installation at RPI is a medium resolution SANS instrument, with relatively good detector count rate, for a range of Q-values of 0.01 $Å^{-1}$ – 0.5 $Å^{-1}$. These characteristics of EPA enable the application of SANS to different fields of research such as Physics, Biology, Metallurgy, Polymer Science and Geology. The expected instrument's performance, in spite the modest RPI flux and the absence of a cold source, follows from the previous extensive work carried out at ITN on instrument design optimisation (Margaça *et al*., Nucl. Instrum. Methods, **A274**, (1989) 606; J. Appl. Cryst. **24** (1991) 994 and Falcão *et al*., J. Appl. Cryst. **27** (1994) 330). The EPA general layout is shown in Fig.1.

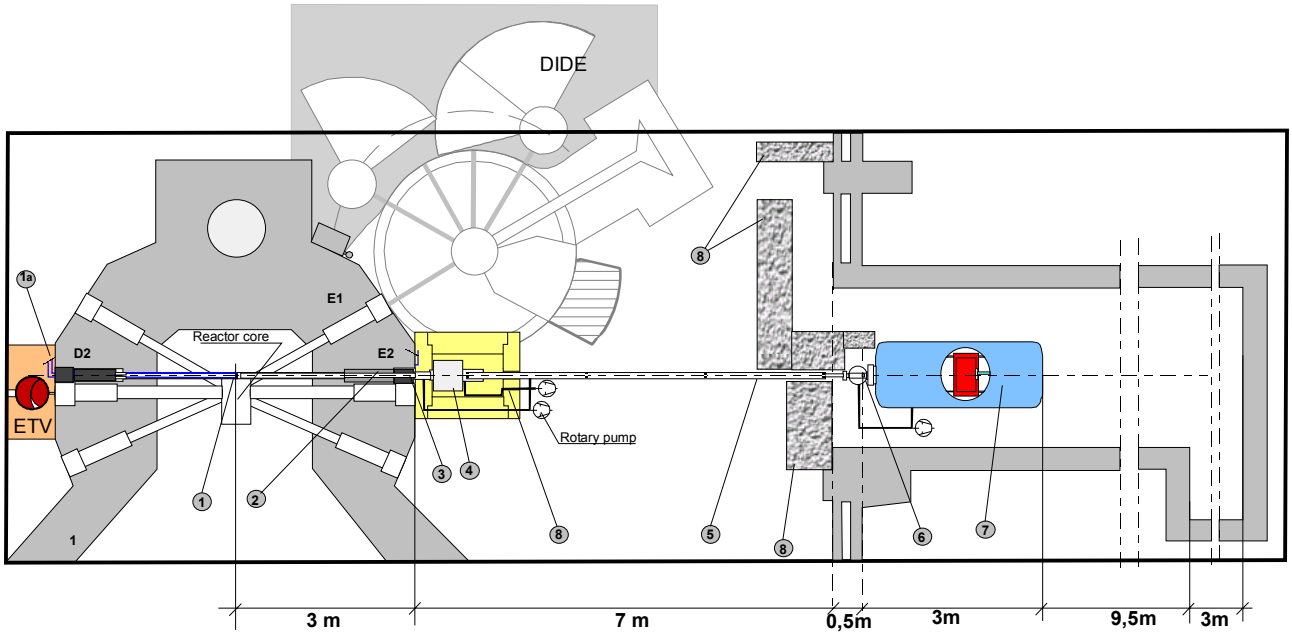


Fig.1: General layout of EPA.1-neutron scatterer; 1a-ends of the water circuit; 2-in-pile collimator assembly; 3-beam shutter; 4-mechanical velocity selector; 5-out-of-pile collimator; 6-sample chamber; 7-position sensitive detector; 8-shielding walls. DIDE is the nearby 2-axis diffractometer facility and ETV a time-of-flight diffractometer.

During 1999 the detector carriage was built and successfully tested mechanically inside the detector chamber with the PSD neutron area detector in position. The whole hardware of EPA is ready for installation. Permission is still awaited to start the installation work. Command and control electronics and data handling protocols have been essentially completed during the year and are ready to be tested *in situ*.

Further work involves the installation of the in-pile and out-of-pile components of the instrument and the commissioning of EPA. This can be accomplished during 2000 unless unforeseen difficulties are raised by the reactor operation as has ocurred in the past.
Research activities planned in the organic-inorganic hybrid materials (ormosils) project will immediately benefit from the availability of the EPA instrument whose operation should begin as soon as possible.

DIDE – Two-Axis Neutron Diffractometer

A. N. Falcão, F. M. A. Margaça, J. F. Salgado and F. G. Carvalho

A two – axis diffractometer (DIDE) is being installed at ITN to investigate powder samples using the thermal neutron beam of the Portuguese Research Reactor (RPI). The instrument favours intensity over resolution, but it was designed to be flexible, allowing set-ups that provide accessibility to various *q*-ranges and experimental resolutions, making it useful in the investigation of magnetic and crystallographic structures. Led by the present demand from on-going research at our Laboratory, the initial set-up of the instrument is suited for the investigation of magnetic structures.

The beam extracted from the reactor has a cross-section with a vertical dimension as large as possible and the instrument is equipped with a vertically focusing monochromator. The beam extracted from the reactor is filtered out of the higher order wavelength contamination and is collimated towards the focusing monochromator, placed at a fixed position, 1.7 m away from the reactor wall. The beam divergence can be varied by interchangeable Soller collimators (15', 20' and 30'). The monochromator holder allows the mounting of two different sets of crystals. At start only one set of HOPG (002) crystals with 30' mosaic spread is available. Monochromator take-off angles in the ranges 25º-65º; 85º-105º can be chosen. The sample to monochromator distance can be varied from 1.4 m to 2 m. The divergence of the beam striking the sample can also be defined by interchangeable Soller collimators. The detector is a BF3 filled multi-wire detector covering an angular range of 80º with an angular resolution of 0.1º. Beam profile at sample position was accessed by means of Monte-Carlo simulation. The calculated value of the 2.5 Å neutron flux at sample position obtained with the PG(002) monochromator in open beam geometry is 6.8×10^5 n cm^{-2} s^{-1}. Calculated of experimental resolutions of Bragg peaks recorded at the detector are represented in inset c of Fig 1 as a function of scattering angle. Also in the same figure experimental points obtained with an existing time of flight diffractometer ETV are represented for the sake of comparison.

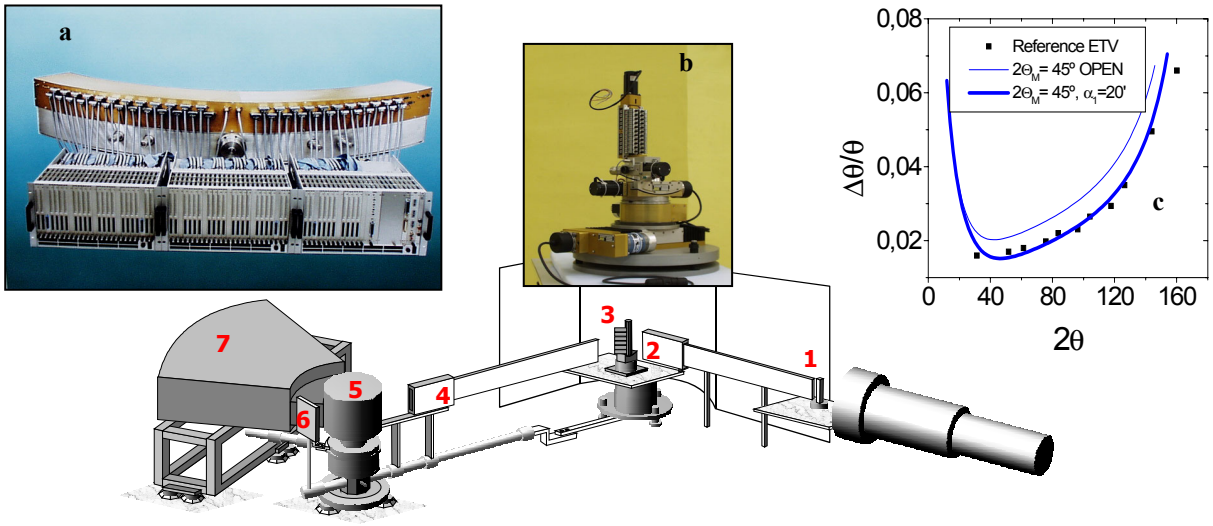


Fig. 1 - Schematic view of the beam-line of the two-axis diffractometer DIDE. **1** – high order filter; **2** – collimator; **3** – focusing monochromator; **4** – collimator; **5** – sample chamber; **6** – beam-stop; **7** – detector. The insets show pictures of the multidetector (**a**) and the focusing monochromator (**b**), and calculated resolutions of Bragg peaks recorded at the detector as a function of scattering angle (**c**).

The project has been supported by ITN funds. The overall reference budget for the installation of the diffractometer (excluding the multidetector that was offered by the French CEA) was 37 MPTE. A total of 25.7 MPTE has been allocated and spent (21.1 MPTE in 1998, and 4.6 MPTE in 1999). Almost all the components necessary for the minimal installation required for the instrument to go into test phase have been constructed. Still lacking is the remote control electronics, which is being developed in collaboration with the LLB (Saclay) and should be ready by February 2000.

Installation has been delayed both by bureaucratic reasons and by the lack of manpower. In 1999 it was possible to uncouple the installation of the in-pile and out-of-pile parts. The installation of the in-pile part was concluded and that of the out-of-pile part started. In the course of 2000, the installation will have to completed, and a furnace and a cryostat will have to be purchased.

ETV – Time-of-Flight Diffractometer

A. N. Falcão, F. M. A. Margaça, J. F. Salgado and F. G. Carvalho

Time-of-flight is a general experimental method of finding the energy of a neutron by measuring the time it takes to travel between two points. When a white beam of thermal neutrons interacts with a powder crystalline sample without energy transfer, the time-of-flight histogram of the neutrons reaching the detector can be easily related to the microscopic structure of the sample.

ETV is a time-of-flight diffractometer. The white neutron beam extracted from the reactor is pulsed by a converging slit chopper and collimated towards the sample. The scattered neutrons are collected in two detector banks making 2θ angles of 90º and 45º. Typical values of the chopper-to-sample and sample-to-detector (2θ=90º) flight paths are 154 cm and 120 cm respectively. The energy window observed, hence the range of interplanar spacings d_{hkl}, depends on the rotation speed of the chopper. Overall the range 1Å to 8Å can be accessed.

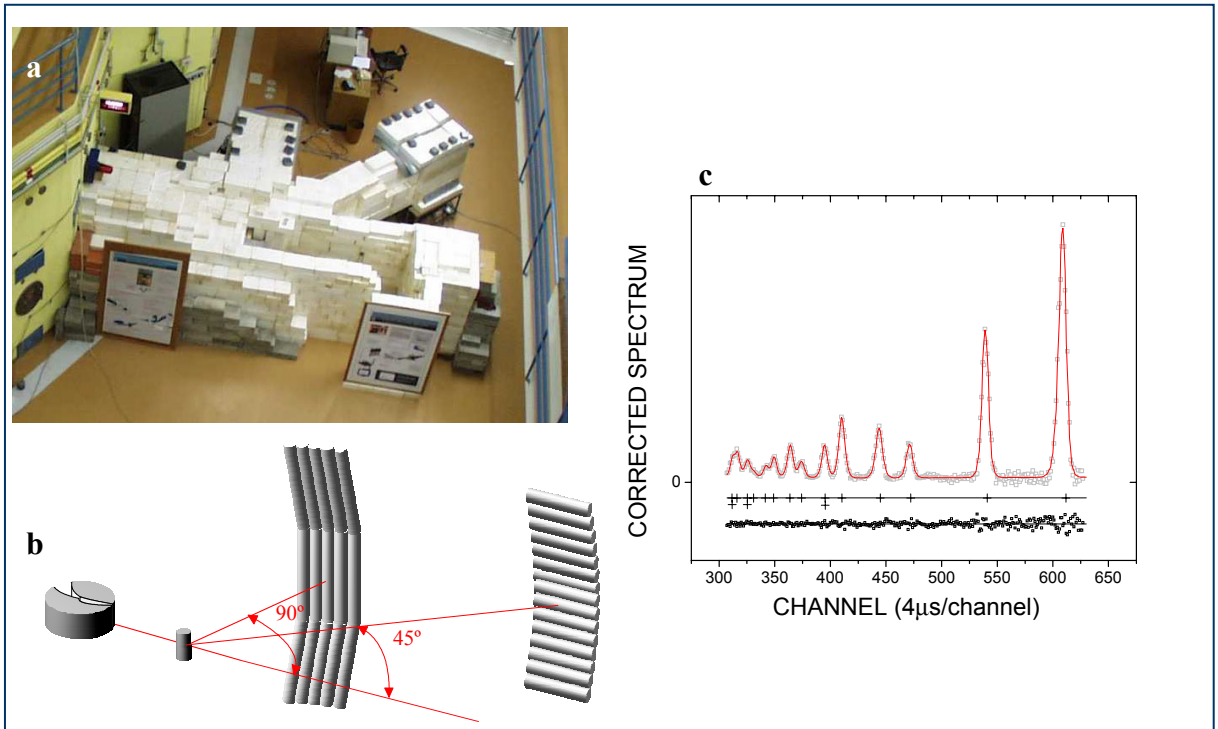


Fig. 1 *– Time-of-flight diffractometer ETV.* ***a*** *– view of the instrument installed at the Portuguese research reactor RPI;* ***b*** *– schematic view;* ***c*** *– corrected spectrum of a powder Si sample.*

The instrument is essentially used for training and educational purposes in general. In the course of 1999, it was used to perform the experimental work included in introductory lectures to neutron scattering (collaboration with the Physics Department of the Faculty of Sciences of the Lisbon University), and to introduce neutron diffraction techniques to students from Instituto Superior Técnico, Technical University of Lisbon, and from the Physics Department of Aveiro University.

As soon as the two-axis diffractometer DIDE is commissioned, the set-up of the time-of-flight diffractometer will be changed. The 2θ=45º detector bank will be removed and the sample environment improved to include sample rotation and temperature variation. Also, the electronics will be renewed.

High Temperature Materials Laboratory, MA^{3}T

A. D. Sequeira e J. B. Manteigas

Owing to the limited space available at the Physics Department it was no longer possible to accommodate new laboratories that were urgently needed. To improve this situation, a small extension of the Physics Department of ITN was constructed. This way it was possible to install a new infrastructure, the High Temperature Materials Laboratory, MA^{3}T and also accommodate the new machine shop of the department and a new laboratory to manipulate radioactive samples. The last two will be operational in 2000 and will not be referred to hereafter.

The construction started in August 1998 and was finished one year later in the Summer of 1999. The funding originated from two different sources. The main part was institutional for the construction and in part for the furnishing of the laboratories (work benches and office room furniture); the laboratory equipments and part of the furniture was funded by a PRAXIS project.

Main facilities

The main facilities of this new infrastructure are an X-ray laboratory, a metallographic laboratory, a laboratory for thermal treatments, a control room, a technical annex and two office rooms.

The X-ray laboratory has a powerful X-ray source (a rotating anode with 18 kW) that can accommodate a second diffractometer. All the control of the instruments is done remotely from the control room next to the laboratory.

The sample preparation and metallographic observation of metallic and ceramic samples is made at the metallographic laboratory. Currently the laboratory has a limited set of metallographic equipment but has already a chemical *hotte,* for the electrochemical surface preparation of samples, and optical microscopes (magnification range 3x -1000x). The laboratory for thermal treatments has already a high temperature furnace with a maximum temperature of 1800ºC.

In the technical annex are situated the chillers for cooling of the rotating anode X-ray generator and air conditioning, the water pumps and the deposits of inertia, the air compressed system and a 30 kW UPS to support the X-ray generator.

The completion of the supply of the laboratories with new equipment for sample preparation, namely polishers, grinders and mounting press for hot mounting of samples will depend on future funding from research projects.

Fig. 1. The front view of the new Laboratory on a sunny Sunday afternoon.

High-Temperature Double Crystal X-ray Diffractometer, Hotbird

A. D. Sequeira, N. Franco, J. Neves, A. Santos, F.M. Margaça, N. Pinhão and M. R da Silva[1]

With the support of the FCT, through a PRAXIS project, a high temperature X-ray diffractometer was projected and constructed at the physics Department of ITN. The diffractometer is a multipurpose instrument with applications ranging from the phase identification and determination of residual stresses on polycrystalline alloys to two-dimensional mapping of Bragg peaks in single crystalline materials. This instrument can perform diffraction experiments from room temperature up to 1400 ºC allowing *in situ* studies. These high temperature experiments, in particular on single crystalline samples, are very useful and also very rare.

The construction and installation of the *Hotbird*, a *high resolution X-ray diffractometer*, was finalized being now in operation. The success of the construction of the diffractometer resulted from a huge effort from a varied team of experts in different technical fields. Emphasis should be given to three particular aspects: the project of mechanical components, that were all constructed at the ITN machine shop; the project of electronic components and control boards; and finally the development of the software for remote control, data acquisition and data analysis.

With the Hotbird the team intends to study high temperature phenomena in superalloys, ceramics and semiconductors. The Hotbird is particularly suited to perform determination of internal strains, lattice mismatches between coherent phases, residual stresses and to observe the in-situ formation of intermetallic precipitates in implanted metals and semiconductors.
The commissioning of such a diffractometer at ITN enables the team to carry out locally highly specialized experiments that will impart substance to collaborations with foreign teams and the training of young scientists.
The controlling software to perform the several experiments procedures (θ–2θ powder, ω-scan, ω–2θ scans, etc.) the remote control of the generator, temperature control, data acquisition and some of the data analysis were also developed by the team.
As a result form this work a paper was submitted to an international conference to be published in the journal *Materials Science Forum*.

Further work

Currently, additional software is being developed to enlarge the range of experimental procedures available on the Hotbird.

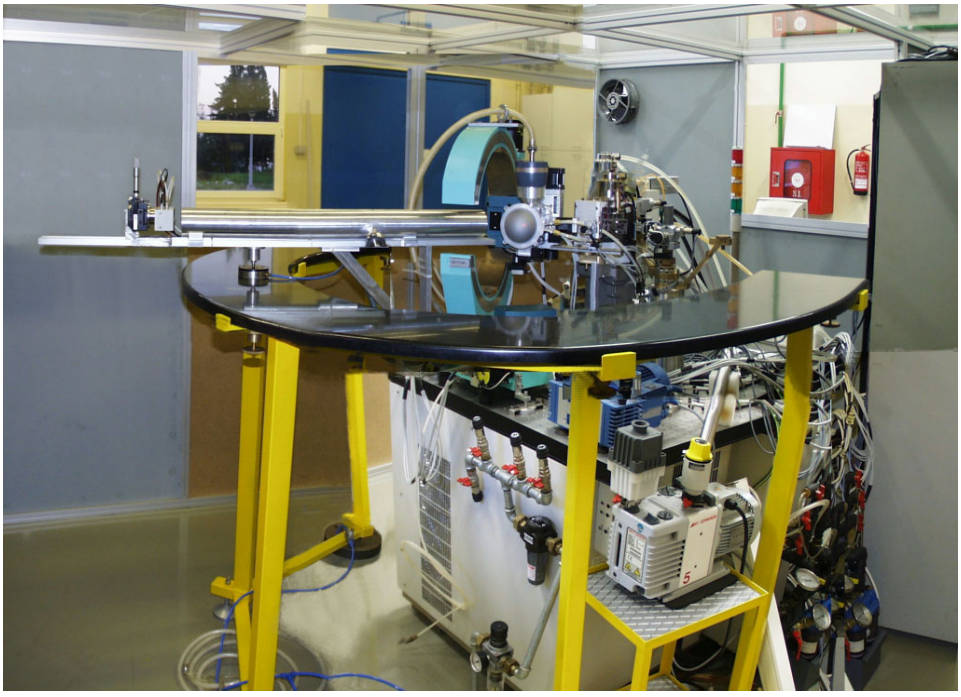


Fig. 1. A general view of the core of the Hotbird.

[1] Centro de Física Nuclear, Av. Prof. Gama Pinto 2, 1699 Lisboa, Portugal.

Low Temperature and High Magnetic Field Facility

Cryostat (0.3 K-300 K) with a 18 T magnet

Among the several low temperature and high magnetic field facilities available in ITN the cryostat with 18 T magnet offers the most extreme conditions. Two different inserts can be fitted in this cryostat allowing either a temperature range from 1.6 K up to 300 K (^{4}He flow) or from 0.3 K up to 300 K (^{3}He cryostat).

The ^{4}He insert is easy to use and various type of probe can be fitted. Our probes have been used for magnetotransport measurements in quasi-1D or 2D materials, in intermetallic compounds and in High Tc superconductors. A very precise rotating sample holder was built at the ITN to measure the variation of the magnetoresistance as a function of the angle between the magnetic field and the cristallographic axis or the electrical current. The angle is measured by an Hall sensor, the resolution is better than 0.01°. The rotation is obtained by a endless screw moved at room temperature by a step-by-step motor controlled by computer. This probes allows resistivity, magnetoresistivity and Hall effect measurements vs angle, vs temperature or vs magnetic field (1.6 K-300 K; 0-18 T). This probe is used to determine the anisotropy parameter in High Tc multilayers as a function of the angle between the magnetic field and the *a,b* plane (Fig. 1). Recently, a rotating cantilever magnetometer was installed, allowing magnetisation measurements up to 18 T in various directions.

The ^{3}He insert is equipped with a rotating sample holder (2° precision, manually operated) for resistivity, magnetoresistivity and Hall effect. This probe was used to study localisation effects and Shubnikov-de Haas oscillations in quasi-2D materials (Fig. 2).

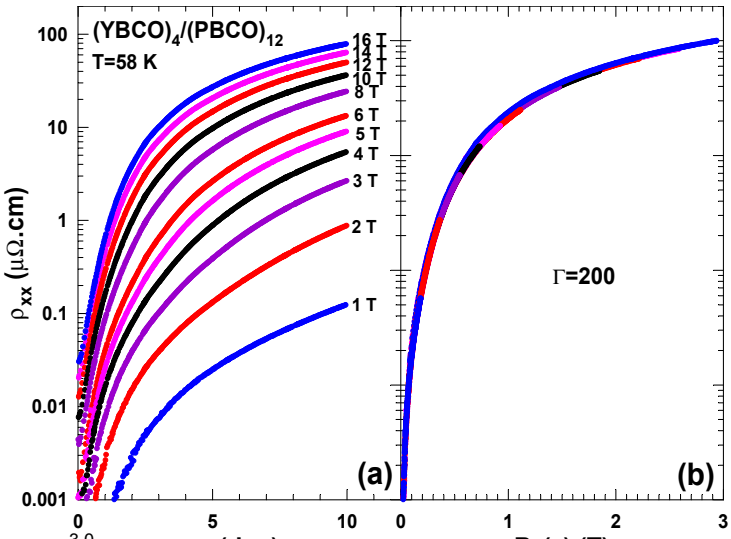
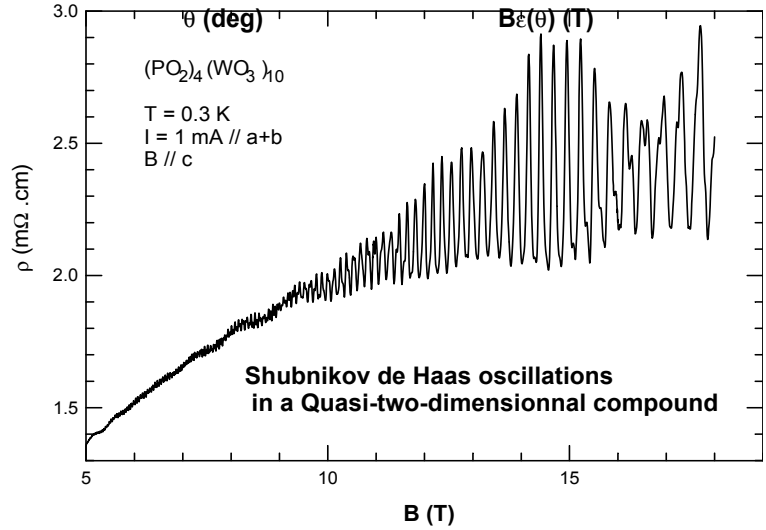


Figure 1:
Rotating sample holder 1.6 K-300 K, 0-18T
- **Left**: Resistivity of a YBaCuO/PrBaCuO multilayer as a function of the angle Q between the magnetic field B and the ab plane for various magnetic fields. Data obtained with the home-made rotating sample holder.
- **Right**: Verification of the scaling law for anisotropic superconductors: All the data of the left part are plotted as function of the reduced field:

B_{red}==$B(\sin^2Q+\cos^2Q/\Gamma^2)^{1/2}$.

The perfect superposition confirms the validity of this scaling law and allows the determination of the anisotropy parameter G



- Figure 2:
Electrical transport at very low temperature Magnetoresistance of a quasi-two-dimensional compound at 0.3 K. The Fourier transform of these oscillations (Shubnikov-de Haas effect) allows the determination of the Fermi surface.

Multipurpose specific heat and magnetic characterization

System *MagLab 2000* *

Under a special project submitted to PRAXIS by ITN and partners at two universities for the upgrade and purchase of new magnetic characterization equipment*, a commercial system (*MagLab 2000* from Oxford Instruments) capable of multipurpose characterization measurements at low temperatures and under fields up to 12 T, was acquired. The system comprises a variable temperature insert, operating in the range 1.5-400 K inside a 12 T magnet with a 50 mm bore with several easily interchangeable probes.

- One type of probe allows either magnetisation measurements by an extraction technique or AC-susceptibility measurements.

- Another type of probe, allows specific heat measurements of small samples (~5mg) in the range 2-200 K using a relaxation technique and under different magnetic fields up to 12 T.

All these measurements are, highly automated in a user-friendly environment. These characteristics allow either routine magnetic measurements complementary to those obtained with other techniques, for instance with a SQUID magnetometer, or easy specific heat determinations in small samples.

This equipment, installed at ITN, is shared with the University of Lisbon and the system is expected to serve different teams of researchers involved in a variety of research projects where the magnetic properties are relevant. The system became recently operational having already a long waiting list of experiments of benefit to research projects involving teams at ITN, FCUL, University of Coimbra or IST in different subjects such as:

Magnetic phase transitions in Intermetallics,
Heavy Fermion systems
Molecular Conductors
Molecular magnets
Magnetic oxides and magnetic particles

* Funding: Contract PRAXIS/PCEX/P/FIS/1/96 (50×10^6 PTE- 40×10^6 PTE to ITN)

Faraday Balance

The Faraday System for magnetisation measurements is a facility enabling determinations in the range 2-300 K, using fields up to 7 T and gradients up to 10 T/m. The force is measured with a microbalance with a sensitivity of 0.01 μg, making it particularly appropriated for susceptibility measurements of powder samples. It has been used mainly to study molecular materials and compounds, that are being prepared and studied by ITN initiative or,on a scientific collaboration basis, different compounds prepared by other research laboratories such as the Institut de Ciencia de Materials de Barcelona, ITQB, IST, and UNL.

High Temperature Synthesis and Crystal Growth Laboratory

The High Temperature Synthesis and Crystal Growth Laboratory is a facility that has been implemented in ITN primarily for the preparation and single crystal growth of uranium intermetallic compounds. This facility has been supporting national and international projects and is the only place in Portugal where single crystals of intermetallic compounds can be grown. Furthermore it has all the safety requirements for work with U.

Main equipment

- Induction furnace equipped with cold crucibles and accessories for the growth of single crystals by the Czochralski, Bridgman and Floating Zone methods.
- Arc furnaces.
- Resistive furnaces (up to 1600 C).
- Laboratory precision cut-off machine for small dimension materials
- Low damage spark erosion unit

Main Users

This facility has been used either in the framework of projects co-ordinated by the ITN Solid State group in the Chemistry Department or by demand of other researchers inside and outside ITN:

- Preparation and single crystal growth of intermetallic compounds based on Fe and *f*-block elements (collaboration with Dept. Physics, Faculty of Sciences, Univ. Lisboa; Dept. Physics, FCT, Univ. Coimbra; Institute for Transuranium Elements, EC JRC, Karlsruhe, Germany; Laboratoire de Chimie du Solide et Inorganique Moléculaire, Université de Rennes 1, France).
- Preparation and single crystal growth of intermetallic compounds of the series $(U_{1-x}Th_x)_2M_2X$
- Preparation of the $(U_xDy_{1-x})Ni_2B_2C$ intermetallic compounds.
- Single crystal growth of B20 compounds (Dept. Physics, FCT, Univ. Coimbra).
- Targets for ion implantation.
- Preparation of RM_x (R = *f* element; M = *3d* metal) for catalytic studies.

Mössbauer Spectroscopy Facility

Mössbauer spectroscopy is a nuclear technique that has been developed in ITN for applications in ITN coordinated research on Solid State Science and Earth Sciences. This facility has also been supporting, since its very first steps, research programs from other national or foreign Research Institutes and Universities. This interaction with other institutions has always been performed on the basis of scientific collaboration and formation of young scientists (graduate and post-graduate students) since expert interpretation of the Mössbauer spectra based on the electronic structure of the studied samples is necessary.

Main equipment

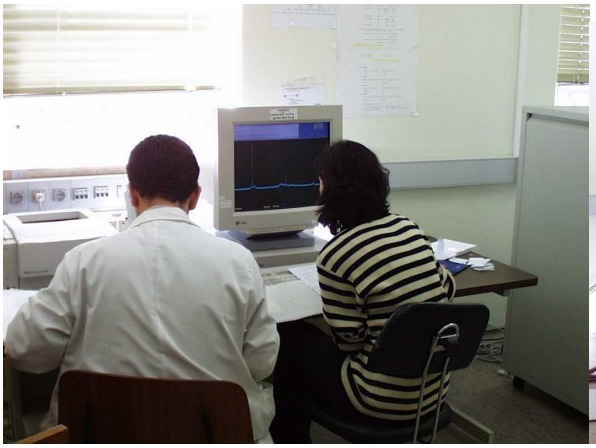
Two Mössbauer Spectrometers, in either transmission or back-scattering geometry. These spectrometers may be coupled to a continuous-flow liquid-He cryostat or a bath cryostat equipped with a superconducting split-pair magnet enabling studies with the absorber in the temperature range 2-350 K and under magnetic fields up to 5 T.

Sources existing and currently used are for ^{57}Fe and ^{151}Eu Mössbauer spectroscopy. Other sources such as ^{119m}Sn may be acquired if a research program justifies it. In other cases, as for same rare earths, the sources can be prepared at the RPI.

Examples of on-going research:

- Structural characterization and study of the magnetic interactions of the Fe sublattices in magnetic intermetallics based on Fe and *f*-block elements (collaboration with Dept. Physics, Faculty of Sciences, Univ. Lisboa; Dept. Physics, FCT, Univ. Coimbra; Institute for Transuranium Elements, EC JRC, Karlsruhe, Germany).
- Characterization of perovskites, materials for Selective Oxygen Separation Membranes (collaboration with Unidade de Investigação em Materiais Cerâmicos da Universidade de Aveiro).
- Characterization of Fe-complexes of tetraazamacrocycles (Instituto de Tecnologia Química e Biológica da Univ. Nova de Lisboa).
- Eu oxidation state and magnetic relaxation in organometallics containing aryloxide and alkoxide ligands (University of California, Irvine, USA).
- Alteration mechanisms of ferromagnesian minerals in order to establish the causes and mechanisms of degradation of igneous rocks in monuments from Minho, Alentejo and Açores (Dept. Earth Sciences, Univ. Minho, or Lab. Nacional de Engenharia Civil, Lisboa).

Nuclear Magnetic Resonance Spectrometer



Nuclear magnetic resonance spectrometry is a fundamental equipment in any laboratory of chemistry. It is one of the most important techniques used by the chemist to the study of molecular structures of organic and organometallic compounds.

It is operated by the Inorganic and Organometallic Chemistry Group of ITN, being also used by other research groups of the Chemistry Department and from the Technical University of Lisbon.

Our Laboratory is equipped with a Unity Inova Varian 300 Mhz multinuclear spectrometer with pulsed field gradient (PFG) probes.

High Resolution Fourier Transform

Ion Cyclotron Resonance Mass Spectrometer

Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR/MS) is a technique that is specially suited to study the chemistry of ion-molecule systems in the gas phase, due to the possibilities it offers in terms of manipulation of ions and neutrals. As a result of the applied magnetic (3 Tesla) and electrical fields, it is possible to trap the ions for long periods of time compared with conventional mass spectrometry, and in those periods it is possible to perform complex sequences of operations: reagent introduction, ionisation, selection, acceleration, ejection, collision, reaction and detection. To different event sequences correspond different types of experiments in which the structure of the ions can be probed, reagent/product sequences can be determined and kinetic and thermochemical studies can be made.

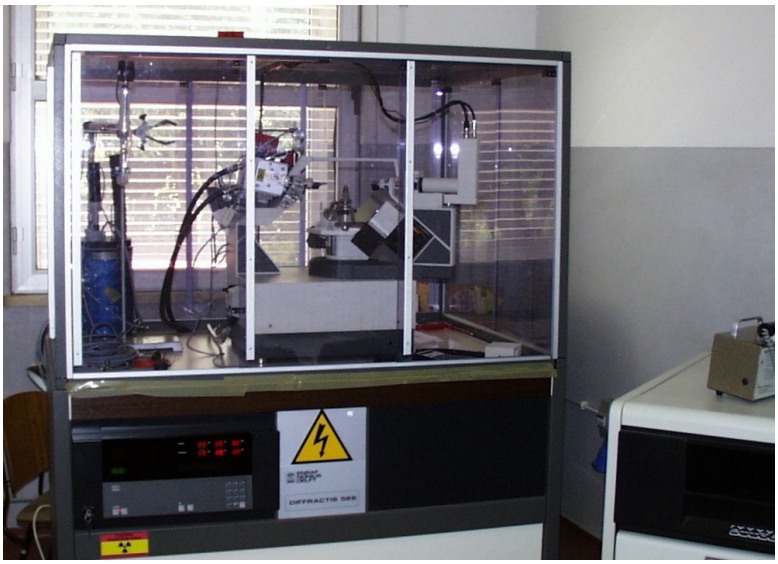
The FT-ICR/MS technique is also a very powerful analytical tool, being capable of very high mass resolution and exact mass measurements, and due to the available ionisation methods, namely laser desorption/ionisation (LDI), is adequate for the analysis of numerous types of materials and compounds.

The FT-ICR/MS instrument, a Finnigan FT/MS 2001-DT, is unique in our country and was installed in October 1991 as a result of the "Ciência" Programme, being shared by ITN, IST and FCUL. It is run by the Inorganic and Organometallic Chemistry Group of ITN, that has been mainly using it in studies of the gas phase ion chemistry of lanthanides and actinides, and also in the mass spectrometric characterization of lanthanide, actinide and rhenium compounds. The other users are the Thermochemistry Group of IST, that has been using the instrument to study organic and organometallic thermochemistry, and the Mass Spectrometry Group of FCUL, that has been using the instrument to study d transition metal ion chemistry and to characterize organic compounds of biological interest.

Due to its analytical capabilities, the FT-ICR mass spectrometer has also been used for the characterization or the identification, specifically through the determination of exact masses, of a large number of organic and organometallic compounds, as requested by research groups in national and international universities or institutes.

Supported by the "Praxis XXI" Programme, a new station for instrument control and data treatment and a quadrupolar axialization module were installed and became fully operational in the beginning of 1999.

Single Crystal X-Ray Diffractometer



Single crystal X-ray diffraction analysis is a powerful technique for the determination of crystal structures. It is considered today as a routine technique for the structural characterization in organic, inorganic and organometallic chemistry research.

The Enraf-Nonius CAD4 Diffractometer is equipped with a Kappa-axis 4-circle goniometer, a high-stabilized 3Kw generator, a scintillation detector and a low-temperature device.

The equipment was installed in Sacavém in July 1986, and it is shared with the Technical University of Lisbon. At that time, it was the unique diffractometer in Lisbon.

It is run by the Inorganic and Organometallic Chemistry Group of ITN, being also used by other research groups of the Chemistry Department and from the Technical University of Lisbon.

This facility has been used in the determination of crystal and/or molecular structures compounds of f block elements and of rhenium, molecular compounds for conducting and magnetic materials, intermetallics containig 5f elements and in general for coordination compounds of d transition elements.

Actinide Chemistry Laboratory

This Laboratory, with a special ventilation system, is equipped with several gloveboxes for handling actinides. It has been used for neptunium 237 chemistry and one glovebox is now being adapted for technetium 99 chemistry.
We intend in a near future to handle or prepare in this Laboratory targets of actinide metals or oxides to be used in our Fourier Transform Ion Cyclotron Resonance Mass Spectrometer for ion molecule reactions.

Laboratories for handling radioactive materials

Fully equipped laboratory for the storage, handling and preparation of radioactive materials. This facility, unique in Portugal, is supplied with (1) a ventilation system designed to maintain a negative pressure relative to the surroundings areas and to provide an appropriate number of air changes with filtration of the replacement air. (2) Four radiochemical exhaust hoods with adequate airflow to allow a rapid air movement and to prevent back flow of gases from the hood. (3) Four Perspex glove boxes suitable for handling low-energy radiation radioactive materials. (4) Five lead wall remote controlled hot cells to manipulate high-energy radiation radioactive materials. (5) Equipment, such as HPLC preparative systems, used for the purification and determination of chemical and radiochemical purity of radiolabeled materials. (6) Equipment for assessing chemical and radiochemical purity using chromatographic or electrophoretic separations, such as a radiochromatographer and an electrophoresis apparatus. (7) Instruments for radiation detection and measurement, namely ionization chambers, gamma-counters and dose radiation monitors. (8) A UV/VIS spectrophotometer. (9) Safety devices and facilities for protection of personnel including a changing room equipped with a shower.

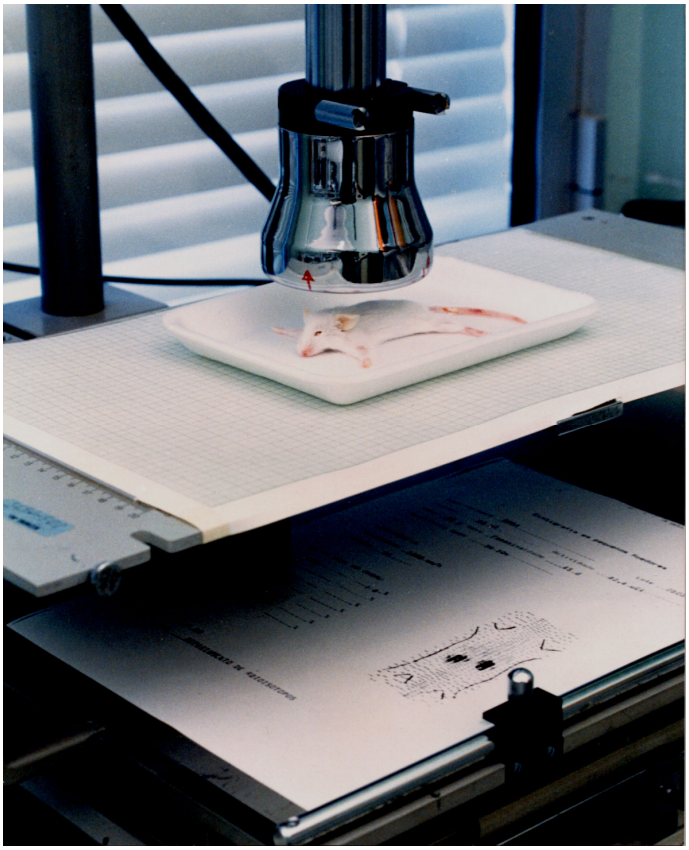
For some years the radiolabelling of compounds either for radiopharmaceutical production or radiopharmaceutical research had been developped in this laboratory. More recently, radiolabelling of biological active molecules, included in the research programme of receptor-binding radiopharmaceuticals based on peptides and steroid molecules for tumour scintigraphy, has also been performed.

Clean Room For Radiopharmaceuticals Preparation

The preparation of sterile radiopharmaceuticals involves special requirements to avoid the risk of microbial contamination and, for injectable preparations to prevent particulate and pyrogen contamination. Thus sterile radiopharmaceuticals should preferably be prepared in a contained work station which is located inside a clean area accessible to personnel and goods through airlocks.

According to these GRP requirements our group is provided with a fully equipped clean room (class 1000) with 2 laminar flow workbenches (class 100) for in solution and suspension or biomolecule based radiopharmaceuticals production respectively. 2 lyophilizers with capacity up to 1000 vials are also available. Relying on these facilities it is possible to produce radiopharmaceuticals both in a lyophilized or solution form suitable for clinical application.

Laboratory for animal experiments



Fully equipped laboratory for: Biodistribution and scanning studies of radiopharmaceuticals in small experimental animals (mice and rats); sterility and apirogenicity tests and tissue culture cells.

In the past this laboratory was used to give support to the radiopharmaceuticals production insuring the required biological quality control of the final products to be supplied for clinical use.

Nowadays these facilities has been used to the evaluation of the *in vivo* stability, biological distribution and pharmacokinetics studies on the radiolabelled molecules developped under the different radiopharmaceutical research projects running in the group, namely:

- 17α-[^{125}I]Iodovinylsteroids Substituted at 7α: Investigation of a New Series of Iodine[^{125}I]-Labelled estrogens as Potential Imaging Agents for Estrogen Receptor-Positive Breast Cancers;
- Development of ^{99m}Tc labelled somatostatin and evaluation of their radiochemical and biological behaviour;
- Chelating Properties Towards Gallium and Biological Evaluation of Two *N*-substituted 3-Hydroxy-4-pyridinones;
- Synthesis and Characterization of Biguanide Complexes with Technetium.

X-Ray Fluorescence Spectrometry Laboratory

The Energy-Dispersive X-Ray Fluorescence spectrometry (EDXRF) is a multielemental technique, allowing the detection of elements from Na to U with high accuracy and reproducibility. The procedure requires only a small amount of sample (1-2g), which can be analyzed with little or even without any pre-treatment.

KEVEX DELTA XRF ANALYST

This Labotarory is run by the Environmental Analytical Chemistry Group. Due to its analytical capabilities, the EDXRF is particularly suitable for studies involving the quantification of an high number of chemical elements in a large number of samples, such as: pollution and geochemical studies.

The EDXRF unit allows the analysis of solid, powder and liquid samples with elemental concentration varying from mg/kg to 100%. The system is computer controlled and its automatic 16-position sample changer allows a fast throughput of the samples.

Moreover because of being completely non-destrutive, EDXRF is also the ideal technique for the analysis of objects with museological or archaeological interest.

Supported by a Technical Cooperation Program (International Atomic Energy Agency) a project has been approved to ugrade the spectrometer.

Mass Spectrometry Laboratory

The isotopic ratios determination by mass spectrometry of $^{18}O/^{16}O$, $^{13}C/^{12}C$ and $^{2}H/^{1}H$ in solid, liquid and gas samples are being applied in Water Resource studies, Palaeohydrology, Palaeoclimatology, Palaeoceanography and Archaeometry, either in the framework of financed research projects or services.

SIRA 10 VG ISOGAS

Mass Spectrometry for Light Isotopes Laboratory is run by the Environmental Analytical Chemistry Group. Studies are mainly based in the understanding of the natural abundance variations of the light element isotopes in Nature. Interpret\ation is made by comparison with international standards and with regional isotopic variations.

This technique is mainly being used in the investigation of hydrogeologic problems, such as, groundwater salinization mechanisms, identification and quantification of the degree of mixing between hydrological systems, definition of recharge areas, dynamic characterization of hydrological systems and characterization of paleoenvironments by isotopic signals encoded in the materials (waters, sediments, etc).

Tritium Dating Laboratory

The Tritium Dating Laboratory operated by the Environmental Analytical Chemistry Group is dedicated to the evaluation of the tritium concentrations in precipitation, groundwater and surface water samples. These determinations are being currently used in the characterization of different environments and in pollution studies, in the framework of research projects, international collaborations and services.

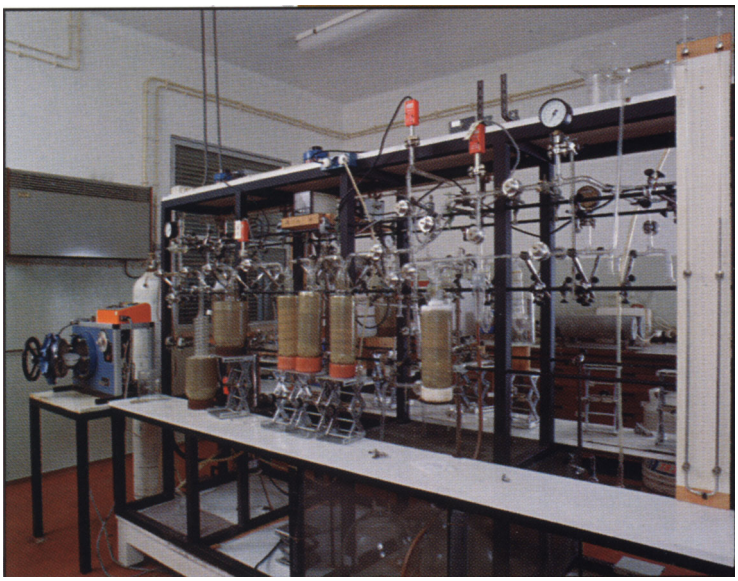
PACKARD TRI-CARB LSC AND ELECTROLYSIS PROCEDURE

The Liquid Scintillation Counting (LSC) is the method applied in the detection and quantification of tritium concentrations in the water samples. Prior to the counting process, the samples are enriched in tritium by an electrolysis procedure to improve the overall detection limit.

RADIOCARBON UNIT

The ITN Radiocarbon Dating Laboratory was implemented in 1986, and is unique in Portugal. This laboratory is associated with the "Cultural Heritage and Sciences" research group.

The sample analysis procedure can be divided in 3 steps: sample chemical treatment, benzene synthesis in a vacuum line and, ^{14}C activity measurements with a liquid scintillator counter.



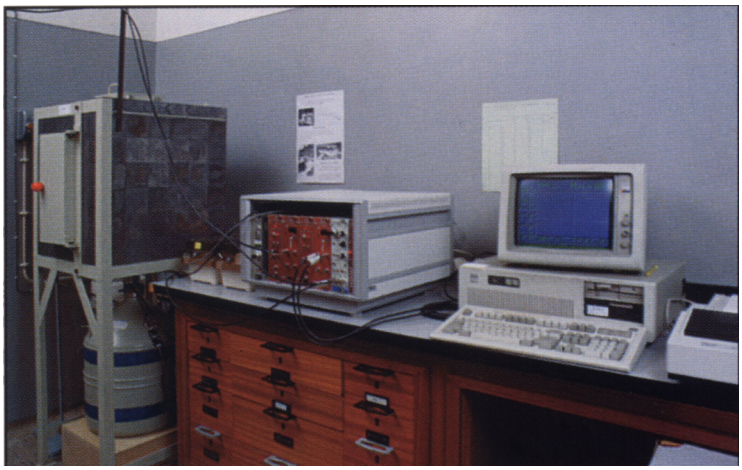
Regarding the first two steps of the procedure, the final purpose is to obtain, from the various forms of carbon contained in the samples, benzene with a high degree of chemical purity, to be fed in the liquid scintillator counter for the measurements of ^{14}C activity.

The materials to be dated are wood and other vegetable remains, charcoal, shells, bones, sediments, waters (precipitated carbonates), peat, etc.

During 1999 numerous samples of different types were analysed, related to research projects or as services to the national and international communities.

Instrumental Neutron Activation Analysis Laboratory

The INAA laboratory of the Chemistry Department, operative since the 70's, is based on a comparative method. This laboratory has two γ-spectrometers, including coaxial Ge detectors and low energy photon detectors. These are connected through Canberra 2020 amplifiers to Accuspec B (Canberra) multichannel analyser. The irradiations are made in the Portuguese Research Reactor.



This laboratory is associated with the "Cultural Heritage and Sciences" research group.

This method of analysis is excellent for the accurate and precise determination of numerous trace elements concentrations, and is frequently used in projects related to cultural heritage valorisation and conservation and environment, as well as services to the Portuguese industry.

Radioactivity Measurements Laboratory

This laboratory is equipped for the measurement of α, β and γ activities in environmental samples.

a) α- spectrometry systems

MCB and software for the acquisition and treatment of α spectra (Maestro 3.2 for Windows 95, EG&G Ortec), with 18 silicon semiconductor detectors.

b) γ– spectrometry systems

Five MCBs, connected to one Ge(Li), one LEPS and five HpGe detectors, one of these being a well-type low-background detector.
Software for spectra analyses, MAESTRO I and II (EG&G Ortec), SAMPO 90 (Canberra), SILGAMMA and GAMMAPLUS (Silena), as well as internally developed packages like GAMACTIV.
Spectrometer equipped with a NaI(Tl) detector suited for automatic food samples analyses.

c) Total α/β counting systems

Two low-background gas flow proportional counters, one of them with automatic sample exchanger, and one β low-background (0,5 cpm) gas flow G.M. type counter.

d) Liquid scintillation counter

The Beckman LS 6500 Scintillation System is designed to provide highly accurate automated counting of the level of beta radioactivity in samples containing nuclides such as ^{3}H.

Metrological Laboratory of Ionizing Radiation and Radioactivity

1. Laboratory premises:
Lab.I – 15m × 6m; gamma radiation at ambiental and protection level, x-rays at protection and therapy level;

Lab. II – 7m × 6m; gamma radiation at protection level and radiotherapy level (high activity ^{60}Co source);

Lab.III – 4m × 4m; beta radiation and radioactivity measurements.

Control room, workshop & maintenance room, and two offices.

2.Radiation sources
x-rays equipment (10 – 320 kV)

- Irradiators:
  - gamma sources
    - high activity (1 source of cobalt-60)
    - medium activity (8 sources of cobalt-60, Cs-137, Am-241)
    - low activity (7 sources of Co-60, Cs-137, Am-241)
    - very low activity (7 sources of Co-60 and Cs-137)
  - beta sources of ^{147}Pm, ^{204}Tl and 2 sources of ^{90}Sr+^{90}Y
  - large area alpha and beta radioactive sources (10 sources)
- Standards of measurement
  - Photon radiation
    - primary standard
      - kerma in air for ^{60}Co gamma radiation
    - secondary standards
      - kerma in air for ^{137}Cs, ^{241}Am and x-rays (T10 to T280 and ISO qual.)
      - Kerma in air for diagnostic x-rays (DV50 to 150 and DN40 to 150)
      - absorbed dose in water for x-rays (T10 to T280)
      - absorbed dose in water for ^{60}Co, 6 and 8 MV
  - Beta radiation
    - secondary standard
      - absorbed dose (^{147}Pm, ^{204}Tl, ^{90}Sr + ^{90}Y)
  - Radioactivity
    - activity per surface unit (beta emission)

"Clean-room" Laboratory for Sample Processing

The "clean room" laboratory is a transversal infrastructure, serving different research lines, located at the Physics Department. This laboratory has been developed for sample handling and preparation to nuclear and nuclear related technique trace element analysis.

This facility, originally created to support biomedical applications of the PIXE technique, had permitted the development of R&D activities in other areas of science, such as environment, air pollution and eco-toxicology, as demonstrated by the research projects that make use of this laboratory, the associated equipment and know-how achieved *. At present, the "Clean Room" Laboratory is used by different groups from ITN and from groups of the Faculty of Sciences of the Lisbon University.

During 1999 a proposal to optimise resources and renew infrastructures for sample processing, based on environment controlled areas perspective for "Good Laboratory Practices" (GLP), has been presented by different activity lines from ITN (see Multidisciplinary Laboratory).

Characteristics of the facility

The "Clean room" laboratory enables GLP required for R&D activities as indicated by International Quality Systems. The room is classified as "Class 10000" and has as principal characteristic the existence of a filtered air injection system (HEPA filter with 99.99% efficiency for particles with diameter larger than 0.3 μm) that creates an increase of internal pressure relative to external pressure (measured relative pressure of 8 Pa) and a vertical laminar flow bench, "Class A" with 30% extraction. The laboratory is also temperature controlled. Personnel from a certified company periodically perform the environment control of the facility.

The maintenance and use of this environment-controlled facility follow rules in what concerns area cleaning, static mat use at entrance, feet protection, appropriate dressing, type of sample to be handled, and registers of equipment used.

Different equipment is associated to the "Clean Room" Laboratory, mainly related to biomedical and environmental matrices processing for trace element analysis. Sample freeze-drying, powdering at room and cryogenic temperatures (vials of zirconium or agate and Teflon), pelletising, digestion with conventional and microwave ovens (home made digestion and Microwave "Parr" bombs), weighing (10μg precision balance accessible), and target preparation for nuclear analytical techniques are the main methodologies available.

Responsible Personnel

Teresa Pinheiro and Rute Pinheiro

Microbiology Laboratory

The microbiology facility is a well-equipped laboratory where the main work of Radiation Sterilisation Group has been developed and has also been utilised by others external Groups (e.g. Coimbra University). It is divided in two main premises, one at the Physics department and the other one at NUSA (area adapted after the 1st year of project BIOSTER).

A. The premise in Physics Department is divided in three areas

1ST AREA - treatment and preparation of material (e.g: decontamination, washing and sterilisation)
MAIN EQUIPMENT

- Washing machine (Miele); Autoclaves sterilizers; Oven, Millipore water system; Ph meters; Weighing devices; Chemical hotte; balance (Sartorius)

2ND AREA – analysis of "dirty" samples
MAIN EQUIPMENT

- Laminar vertical flux bench, Bio–Hazard (Baker) - Fig. 1; Microscope contrast phase – Leitz/Wild with a Photoautomats – Fig 2; Stereomicroscope (Leitz/Wild); "Stomacher", shakers and other mechanical mixers; Membrane filtration sytems; Incubators; Freezers

3RD AREA – with controlled environmental for analysis of "clean and ultra –clean" samples
MAIN EQUIPMENT

- Laminar horizontal flux bench (Bassaire);

B. NUSA premise

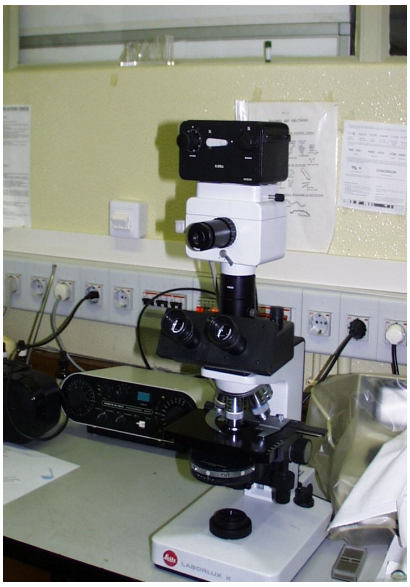
This premise is a large area (approx. 90 m^2) divided in four areas.
MAIN EQUIPMENT

- Laminar vertical flux bench (ASSAB); Analytical balance (Sartorius); super-centrifuge (Beckman); bench centrifuge (Mistral 1000); Incubators; ultra-freeze dryer (ASSAB); Lyophilisation system;

It was proposed a project in 1999 to the Directive Board by four researchers from different activity lines (M. Luisa Botelho, M. Teresa Pinheiro, M. Fátima Araújo, J. Pereira Luis) to adapt this area to a "Multidisciplinary Laboratory" with clean rooms and associated controlled environments (see further on).

Fig. 1 – Bio-Hazard vertical flux bench

Fig.2 – Microscope/photoautomats



Polymer Characterisation Laboratory

The Polymers Characterisation Laboratory (LCP) is a small laboratory where is done the majority of polymeric manipulations; chemical activations and the required operations before and after gamma irradiations.

Main equipment of LCP:

- Vacuum line for preparation of polymeric samples in controlled atmospheres.
- Thermal analysis:
- *DSC:* Differential Scanning Calorimetry
- *TGA:* Thermogravimetry
- Analytical balance
- Hot-house
- Vacuum hot-house
- Polymeric purification unit by discontinuous extraction in a Soxhlet
- Solvent recycling unit by fractional distillation
- IV-Spectrophotometer (FTIR) was repaired during this year.

Fig.1: Thermal analysis equipment. (DSC module and PC controller).

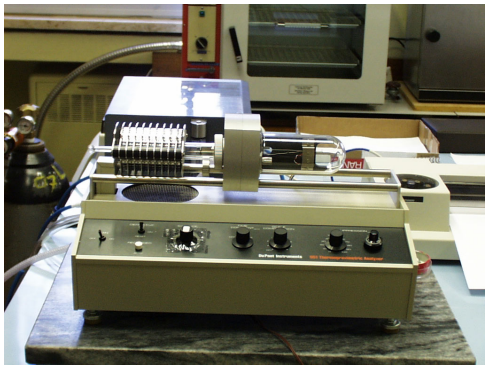


Fig.2: Thermal analysis equipment. (TGA module and vacuum hot-house in background).