EUREKA PROJECT E!2363 - EUROCARE SURFACE MONITOR

1. General description

Project	E! 2363 - EUROCARE SURFACE MONITOR	**Status**	Announced - 28-JUN-2001
Title	**Development Of A Portable X-Ray Spectrometer For Diffraction And Fluorescence Analyses**		
Class	Sub-Umbrella	**Technological area**	Environment
Start date	01-DEC-2000	**End date**	01-DEC-2004
Duration	48 months	**Total cost**	3.6 Meuro
Partner sought	Yes		
Summary	An Innovative, Portable And Rapid X-Ray Spectrometer For Diffraction And Fluorescence Analyses Is Proposed.It Will Be Tested And Optimized Via In-Field And Laboratory Analyses Of Different Ancient Artefacts.		

Budget and duration

Phase	Budget(Meuro)	Duration (Months)
Definition phase	2.3	22
Implementation phase	1.3	14
Total	**3.6**	**48**

Member contribution

Member	Contribution	Position	Since
Italy	**60.00%**	**Contact Member**	**13-SEP-2000**
France	.00%	Notified Withdrawn	10-JUN-2003

Participants

Company	Country	Type	Role
Assing S.P.A.	**Italy**	**SME**	**Main**
Istituto Centrale Per Il Restauro	Italy	Governm./Nat. Admin.	Partner
C.N.R. Istituto Di Chimica Dei Materiali	Italy	Research Institute	Partner

2. Project outline

Project description

It is well established that X-ray fluorescence (XRF) and X-ray diffraction (XRD) are highly effective laboratory techniques for non-destructive analysis and characterization, without sampling and with no contact with the objects to be examined which is why these techniques are used in the laboratories for the restoration and conservation of ancient artefacts.
In many cases, it is very hazardous to carry out these analyses in a laboratory and portable apparatus could be very useful for analysing ancient artefacts without moving or sampling them. Unfortunately, the state-of-the-art of commercial X-ray portable equipment only allows us to obtain the X-ray fluorescence analysis of materials characterized by flat and large surfaces with a simple geometry and furthermore, it is not possible to obtain reliable and reproducible quantitative analyses or perform structural investigations.
Bearing these considerations in mind, in order to make up this lack of analytical capability, a portable, reliable, non-intrusive and commercially viable X-ray spectrometer for diffraction (XRD) and fluorescence (XRF) analyses (also for chlorine and sulphur) with analytical features comparable to laboratory equipment, will be developed. It will be possible to operate this modular system in laboratoriers as well as on different sites such as museums, galleries, historical buildings, archaeological excavations.
This innovative equipment will use a new X-ray micro-generator and optic system for the collimation of X-rays, that will make it possible to considerably reduce the weight by up to 4 kg and produce a miniaturized, silicon-drifted detector cooled with a thermoelectrical Peltier system, thus ensuring good energy resolution and reduced weight. The existing source XRF will be modulated to optimize the energetic range in accordance with applications.
Furthermore, the new equipment will be characterised by:
- a good spatial resolution with an analysed area whose diameter ranges from 0.2 mm to 1.5 mm;
- a specially adapted imaging system for viewing the analysed area;
- a computer-controlled pantograph system for scanning the surface of the materials with the X-rays and simultaneously acquiring the resulting X-ray emission, thus obtaining both microchemical and microstructural maps of the materials combined with the image of the analysed area. This capability will be very useful in order to mark an already analysed area and to analyse it again as a function of conservation treatments and time;
- an external microwave telemetric focusing system for the analysis of materials with a complex geometry;
- a theta-theta geometry for the vertical and horizontal analysis:
- a laser source to remove the surface layer by layer in order to obtain the depth profiling analysis of the materials.
In particular, the innovative design of this instrument

allows large area (80 cm x 80 cm) multi-point mapping for the determination of the chemical and structural composition by means of a tailor-made, computer-controlled motorised pantograph that combines a laser scanning system and imaging acquisition with X-ray fluorescence and diffraction analyses. Furthermore, the analytical system uses an optical or microwave device that measures and optimizes the working distance for increasing the signal emission and acquisition as well as for turning off the X-ray in the presence of unusual operative conditions. The high-speed signal acquisition in PCMCA format is obtained using a small portable computer whose software also enables data handling with a sophisticated lineshape analysis to eliminate the spurious contribution thus giving reliable and reproducible quantitative XRF results.
The goal of such an instrument will be to carry out on-site, non-destructive and non-contact structural and quantitative chemical analyses of the components of cultural heritage artefacts in order to detect, localise and estimate alteration products or previous, inappropriately restored areas.
The first prototype of the XRD and XRF apparatus will be assembled at the end of the definition phase and immediately tested in order to verify the obtainable results and to optimize equipment design, hardware components and data handling system.
A four-step programme will be applied to reach the above planned objectives:
1) at the beginning of the definition phase the project will be first defined in detail and then the production and the assembling of the different parts of the apparatus will begin. Furthermore, different modern materials such as bronzes, silver alloys, minerals, ceramics, glasses and pigments including ASTM standards, will be collected and characterized from a chemical and structural point of view. These materials will be used as reference materials for preliminary testing and calibrating the XRD and XRF apparatus at the end of the definition phase;
2) during the implementation phase the XRF and XRD portable apparatus will be used for characterizing first the above cited materials and then, also ancient materials such as bronze, iron and silver objects, iron and lead slags, ceramics, glasses and paintings characterized by more complex microchemical structures. As a function of the chemical and physical nature of the material under study and the restoration requirements, the measurements will be carried out both on as-received and powdered materials also taking into consideration the influences of some parameters on the analytical results such as time-dependent stability, temperature, sample volume, grain size and the drifting of measurement results;
3) the chemical and structural characterization of the above cited materials will be carried out during both the definition and implementation phases by means of different laboratory techniques such as XRD, XRF, ICP, SEM+EDS, MO and DTA-TG in order to continuously compare the results obtained using the portable apparatus and the laboratory techniques for optimizing the design of the instrument. In particular, this approach will enable the analytical reliability, minimum data capture time, stability and detection limits of the apparatus to be tested;
4) at the end of the implementation phase, the portable apparatus will be used by conservation experts for

in-field characterizations of artefacts and to study the degradation processes and products (such as efflorescence salts, oxidation products, etc.) of ancient artefacts in some European museums and for the characterization of mural paintings (mortar, plasters, etc.) during a restoration project. Particular attention will be paid to defining the deterioration mechanisms by quantitatively analysing Cl, S, Ca, Cu, etc., on mural paintings, stone materials and bronze patinas.
Keywords: X-ray analysis, restoration, portable apparatus.

Technological development envisaged

The main technological developments envisaged are:
- development of a new, fully computer-controlled, easy-to-use instrument that also performs rapid and reliable chemical and structural characterization of the ancient artefacts in depth and combined with the image of the analysed area;
- development of a new system for the acquisition and measurement of the signal;
- development of a computerised system for the horizontal and vertical scanning of large areas;
- development of new software for XRD and XRF data acquisition and processing tailored to select and control the conservation and restoration procedures of ancient materials;
- possibility of on-site, non-contact and non-destructive structural and quantitative surface chemical analysis of cultural heritage artefacts such as metals, stones, ceramics, glass, pigments of easel or wall paintings, archaeological artefacts, musical, technological or scientific instruments;
- identification of main polluting agents through the detection of chemical compounds currently present in the artefact;
- identification of inadequate previous restoration;
- identification of hidden clues that will make it possible to verify the authenticity of the artefacts.

Markets application and exploitation

The project aims to produce a portable, analytically reliable and low cost instrument for the international market to be used for the characterization of ancient materials as well as quality control and non-intrusive testing.
Apart from the above-mentioned scientific and economic aspects, XRF and XRD in-field analysis with a portable and non-destructive equipment makes it possible to rapidly determine the surface microchemical structure and chemical composition of very different materials and can be used as a screening method to select the damaged areas to be restored and identify in advance the areas to be sampled and subsequently analysed in the laboratory. This approach reduces the possibility of the in-field material arbitrary grouping, allows the classification of samples according to their main and secondary components and finally, reduces the amount and the number of samples to be characterized, the analysis time and therefore, the

costs.
Bearing these consideration in mind, the development of a portable apparatus for X-ray diffraction and fluorescence analysis will be a really commercial innovation for the routine diagnosis, protection and conservation of movable and architectonic European Cultural Heritage such as museums, national or territorial Institutions in charge of historical buildings and archaeological sites, Research and Restoration Centres, service companies working in analysis, restoration or expertise as well as for the mining and metal industries and earth sciences.

Project codes

BSI

AFT	inspection equipment
ATS.K	portable
AUC	surfaces
AUY	conservation
B	measurement, testing and instruments
BJN	x-ray analysis
BQB.E	chemical composition
CN	radiation physics
COO.F	x-rays
ZOO	archaeology

NACE

29	Manufacture of machinery and equipment not elsewhere classified
3320	Manufacture of instruments and appliances for measuring, checking, testing, navigating and other purposes, except indus
33201	Manufacture of electronic instruments and appliances for measuring, checking, testing, navigating and other purposes, e
3330	Manufacture of industrial process control equipment
36632	Other manufacturing not elsewhere classified
73	Research and development

3. Main participant

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Organisation type SME
Participant role Main

Contribution to project

Design/produce hardware components of the X-ray spectrometer to perform simultaneous diffraction/fluorescence analyses, assembling the new X-ray generator, pantograph/optical system with laser ablation for depth profile analysis.

Expertise

Italian medium-sized company devoted to the development and production of high technological plants and advanced analytical equipment for different applications in the enviromental, medical, chemical and earth science fields. Full time staff includes about 70 members (30 graduates) whose deal with technical and financial feasibility projects of industrial plants and micro-analytical instruments, defining executive design and technical support for the manufacturing of plants and equipment. Furthermore, ASSING produces systems for micro-controls and image analysis, developing and assembling tailored hardware components and software applications. ASSING is also the sole agent and distributor for ITALY of some of the world's most important manufacturing companies of high technological and analytical equipment for industrial and research applications (SEM, STM, SCLM, EDS, XRD, XRF, MO, DTA, TGA, densitometry and rheology). In these fields, ASSING ensures the assistance and maintenance of the equipment, employing 20 high-level technicians experienced in electronics and computers.

4. Partner

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Organisation type Governm./Nat. Admin.
Participant role Partner

Contribution to project

Individualization of some technical requirements for projecting/assembling of a really portable, reliable, non-intrusive X-ray spectrometer for diffraction/fluorescence analyses whose results will be compared to lab. results.

Expertise

One of the four Central Institutes of the MINISTERO PER I BENI E LE ATTIVITA CULTURALI of ITALY that constitutes the leading guide for the methodological and operative choices in restoration and conservation and co-ordinates the activity for characterization, restoration and conservation of ancient artefacts in ITALY. Founded in 1939, the ICR's activity is summarized as follows: - development of tailored research for the characterization and conservation of ancient artefacts; - development of methodological research based on the detailed evaluation of the influence of environmental factors on degradation processes; - improvements to the materials, procedures and techniques for restoring different ancient materials such as metals, ceramics, marble, glasses, paintings and frescoes; - study of some innovative methods for conservation and restoration. - scientific consulting for other public Institutes (Superintendences, Museums) of the MINISTERO PER I BENI E LE ATTIVITA CULTURALI of ITALY and for international and foreign Institutions. - teaching activities on restoration methodologies to Italian and foreign students; - restoration of important ancient works of art whose conservation needs high level experience; - standardization of diagnostic procedures.

4. Partner

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Organisation type Research Institute
Participant role Partner

Contribution to project

Construct/characterize a relevant number of ancient artefacts/ modern materials of different classes via lab. techniques such as XRD, XRF, XPS, ICP, SEM+EDS, MO and DTA for use as reference materials for XRD-XRF experiments.

Expertise

ICMAT belongs to CNR, the NATIONAL RESEARCH COUNCIL OF ITALY that is ITALY's largest research organisation. Their research activities are dedicated to the synthesis and chemical physical characterization of materials of technological interest and in the Cultural Heritage field. Their main activities fall into the following main fields: * optoelectronic materials * semiconducting and magnetic materials * functional inorganic materials * Cultural Heritage. In the ISTITUTO DI CHIMICA DEI MATERIALI, the activity of the group (four researchers, two technicians and 1 Ph D. student) coordinated by Dr. Ingo, the scientist responsible for the Institute in this project, is devoted to the microchemical characterization of materials and their relationship with the final properties. This objective is reached by means of the combined use of different surface and bulk analytical techniques such as XRD, GDOES, SA-XPS, SIMS, ICP-MS, OM, SEM+EDS and DTA-TG. Dr. Ingo's background and experience are in Materials Chemistry. He has been involved in research into various aspects of microchemical development of metals and ceramics and how their microstructures are related to the final properties, primarily mechanical, thermal and electronic properties. In the field of the Cultural Heritage studies, their contribution concerns the knowledge and protection of metallic materials and ancient marbles. In particular, by means of the combined use of different analytical techniques, studies to ascertain the origin of metals and marbles have been carried out as have investigations to obtain detailed microchemical and microstructural characterization of ancient materials for their restoration and conservation and to gaining insight into ancient production technologies. Finally, Dr. Ingo is also a Professor of Archaeometry at the Faculty for the Conservation of Cultural Heritage of the UNIVERSITY OF BOLOGNA-RAVENNA.