A PANEL PAINTING BY THE MASTER OF FEMALE HALF-LENGTHS ANALYSED BY PORTABLE XRF

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Abstract
A panel painting representing *The Pietà* by an anonymous author known as The master of Female Half-Lenghts was analysed by a non-destructive XRF technique. *The Pietà* forms an important part of the permanent exposition of the Fine Arts Museum of Seville. An unusual brownish tonality of the painting led to a research on pigments and their possible changes during the centuries. Portable XRF equipment was used directly "in situ" in the exhibition room. The results showed the use of pigments, commonly applied in that century, lead white, lead-tin yellow, yellow and red ochre, cinnabar or vermilion, some copper based green pigment, azurite and smalt, as well as some organic red and black pigment. The chemical alteration of smalt, whose blue colour is not observed by the naked eye anymore, led to a brownish aspect of the painting. Also several retouches by modern pigments were found, which were first examed by UV light. The comparison to other Flemish paintings held in the permanent Museum's collection was also carried out.

Keywords Panel Painting, Flemish Art, Pigments, Chemical Changes, X-Ray Fluorescence

Research Aims
The Pietà presents an unusual brownish appearance, which called the attention of the restorers of the Fine Arts Museum in Seville, which holds the painting as a part of its permanent exhibition. This appearance led to a research on pigments used by the Master of the Female Half-Lengths. Any possible chemical changes of applied pigments, which would cause to the present state of the panel painting, as well as the original palette of the Master, were principal objectives of this research. Also the identification of later interventions was of interest, some of which can be observed already by the naked eye. Previously, some other Flemish paintings in the Museum's collection have been analysed, so the results would form part of the data base on Flemish painters from the 15th and 16th centuries in the Seville's Museum.

Introduction
An anonymous Flemish painter known as The Master of the Female Half-Lengths (Maestro de las Medias Figuras) was active in the first half of the 16th century. His auxiliary name was chosen on the bases of numerous women portraits he carried out, which present richly dressed ladies in half-lengths. The elegance of his models and the poetical elements which inspired some of his works show that he might have been active in Mechlin, in the highly cultured circle around Margaret of Austria, who lived in Netherlands from 1518 to 1530. The Master of the Female Half-Lengths was probably trained in the studio of Bernard van Orley, a painter who was very close to Margaret; nevertheless his later style is closer to Ambrosius Benson and Adriaen Isenbrandt. Among his works are not only female portraits, but also religious scenes, interspersed in picturesque landscapes in which the influence of Joachim Patinier is clearly seen. This situates the anonymous master also in Antwerp between 1527 and 1540 (Valdivieso González 1993).

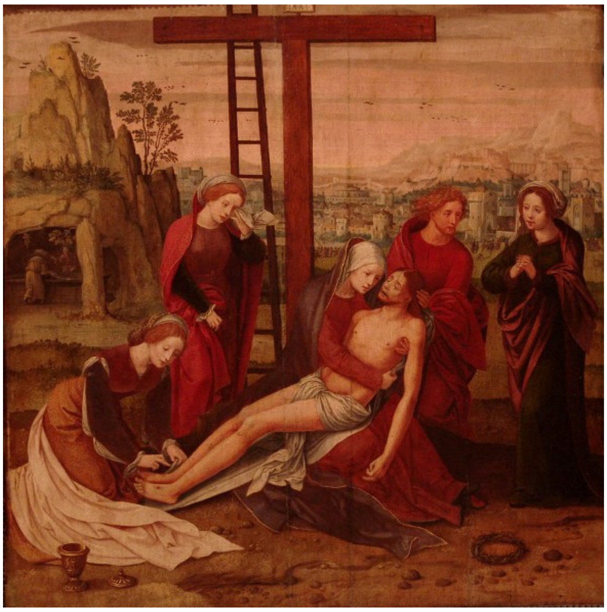

Figure 1. ***The Pietà*** **by The Master of the Female Half-Lengths (towards 1550).**

Experimental procedures
The Pietà is part of the permanent collection and was not in a restoration process at the time of this research, so no micro-samples were extracted. For this reasons, the non-destructive portable X-Ray Fluorescence was considered the most suitable technique for the selected objectives. This technique is very useful in the non-destructive study of materials, especially in art. It allows the first

exam of an artwork, identifying inorganic pigments and helping to discover possible later interventions. XRF gives elemental results, revealing chemical elements present in a radiated point. By this technique it is not possible to identify molecular compositions nor organic pigments, because usual XRF systems do not detect elements with Z lower than 13 or 14 (Gómez 2000, Volpin and Appolonia 2002, Seccaroni and Moioli 2004, Deming Glinsman 2004).

Figure 2. Analysis *in situ* of the panel painting by portable X-Ray Fluorescence.

Our portable equipment uses an X-ray generator RX38 with W Anode from EIS Company and a silicon drift detector (SDD) with energy resolution of 140 eV. An Al filter of 1mm was coupled to the tube to suppress the W peaks from the anode in the X-ray spectra obtained during the radiation. The diameter of the radiated spot was 3mm. The analyses were carried out *in situ* during the days that the museum is closed to the public (Figure 2). The panel painting was radiated in 125 different points, selecting different colours, tonalities, shadows and highlights. All measurements were done under the same fixed conditions: 80μA of cathode current, 29.5 kV of applied high voltage and 300s of preset live time. This permitted a comparison of the spectra among them and also supported semi-quantitative results. The pigments were identified according to the characteristic energy (keV) of the X-ray peaks in each obtained spectrum, which correspond to specific chemical elements (Seccaroni and Moioli 2004, Deming Glinsman 2004, Matteini and Moles 2004). The spectra were compared with a pigment database that was elaborated at CNA, analysing commercial pure pigments from old traditional recipes. Also the wide bibliography on pigments was consulted (Wehlte 1967, West Fitzhugh et al. 1987-2007, Knoepfli et al. 1990, Montagna 1993, Schram and Herling 1995, Brachert 2001, Eastaugh et al. 2004).

Results and discussion

In general, the most important chemical elements detected in the panel painting are Ca, Mn, Fe, Cu, Hg and Pb, however, in some areas also Mg, Al, Si, Co, Ni and As were found. Their presence as well as their individual net peak area count numbers (or cps) in a spectrum vary according to the pigment applied. Consecutively, this information also shows their relative quantity in the radiated point in comparison to other chemical elements in the same point. This allows us, in some cases, to estimate, if a certain pigment belongs to the superficial layer (higher net peak areas) or it can be found in one of the lower layers (lower net peak areas) of the painting.

Lead and calcium compounds

A presence of Pb was discovered in all analysed points, however in very different count numbers responding to Pb Lα net peak areas, which go from 6 cps on the rocks of the background up to 927 cps in white Mary's wimple. In general, the highest count-rates can be found on white colour and highlighted areas of the painting, while the lowest ones in the darker parts, as expected. The presence of lead all over the painting shows an important role of some lead compound, that could be lead white (basic lead carbonate), yellow litharge or massicot (lead oxide) or orange-red minium (lead oxide). With XRF technique it is not possible to distinguish between these different compounds (West Fitzhugh et al. 1987-2007, Seccaroni and Moioli 2004). However, the colour of the analysed area can, in some cases, help with the identification of the pigment applied. In *The Pietà* panel, mostly lead white must have been used, first in the preparation layer, and secondly also as white pigment for white draperies, carnations and highlights. One of these Pb compounds, probably lead white or litharge, could have been applied also as a dryer for other pigments. High Pb peaks in some red draperies as the St. John Evangelist's coat show also a possible use of minium in this painting. On the other hand, low Sn peaks in some highlighted yellow areas reveal presence of lead-tin yellow, normally used to recall gold.

The other chemical element that is richly presented in almost all spectra is Ca, whose Kα count numbers go from 1 cps in St. Mary Magdalene's white drapery to 23 cps in the Christ's hair. In most cases, it is difficult to find out to which chemical compound it belongs, but in panel paintings calcium can be basically found in gypsum or calcite preparation and in animal glue, used in preparations or as the binding media for pigments (Wehlte 1967, Knoepfli et al. 1990, Brachert 2001, Matteini and Moles 2004). It can also belong to some earth pigments or organic black pigments. The variations of Ca content are mostly due to the thickness or to the composition of the preparatory layers and not to the concentration of this element in a certain area (Seccaroni and Moioli 2004).

Pigments

(a) White colour

The palette of The Master of Female Half-Lengths was composed by pigments, commonly applied by the painters in that period of time (Wehlte 1967, West Fitzhugh et al. 1987-2007, Knoepfli et al. 1990, Montagna 1993, Schram and Herling 1995, Brachert 2001). The results can be seen in Table 1. The white pigment, as said above, is lead white, revealed by high Pb peaks in spectra obtained from the radiation of white areas, as white draperies or highlights. Its major concentration can be observed on wider white areas, where count numbers of Pb Lα net peak areas go from 343 cps on the shadow of Christ's white shroud to 927 cps in white Mary's wimple.

(b) Carnations

Lead white was used also as the principal pigment for carnations, where its concentration varies depending on a lighter or darker skin tone. Count numbers of Pb Lα peaks in carnations go from 90 cps up to 670 cps. The painter mixed lead white with a red colour, obtained, according to the analysis, by different pigments – cinnabar/ vermilion, red ochre or hematite and maybe even an organic red lake.

(c) Red colour

The major red pigment in this painting is cinnabar or vermilion. They are both mercury sulphides, but the first one is natural, obtained from the mineral, while the second one is a synthetic reproduction of the mineral (Eastaugh et al. 2004). Both of them were well known and used in the 16th century, when this panel painting was made. They can be distinguished by PLM technique, but not by XRF. Cinnabar/ vermilion was applied, as said, already in the colour of carnation. On lighter areas its concentration is lower (Hg Lα: 4 cps), while on reddish parts like cheeks or hands and legs the concentration decreases (Hg Lα: 27 cps), which shows that the painter added more of this pigment. A very intense colour was used for lips, where the major concentration was detected on the female saint next to the cross (Hg Lα: 41 cps). Differently, red Christ lips were modelled by blue azurite, as shown respectively by Hg and Cu peaks in the spectrum from this area. This blue colour also creates the aspect of the dead Christ's body. Cinnabar/ vermilion was used in its purest form for red draperies. The highest Hg peaks can be observed on St. John Evangelist's tunic (Hg Lα: 155 cps). On the other hand, the analysis of his red coat gives much lower Hg count numbers (Hg Lα: 10 cps), so, probably, the colour layer is thinner or the pigment is more diluted. The same red pigment was found in high concentration as well on the cross in the background, in small figures in the left corner and in some other elements of the background.

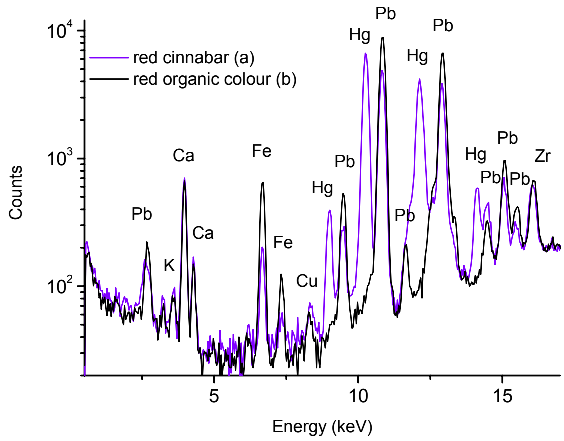

Figure 3. Comparison of two XRF spectra showing cinnabar red (Hg) from St. John Evangelist's coat (a) and an organic red colourant (no characteristic chemical elements) from St. Mary Magdalene's dress (b). In both spectra also the presence of red ochre can be seen (Fe).

In all red areas where cinnabar/vermilion was detected, also Fe peaks appear (Fe Kα: 2 cps), showing that this pigment was mixed with red ochre or hematite, which was a normal procedure (Wehlte 1967, West Fitzhugh et al. 1987-2007, Knoepfli et al. 1990, Brachert 2001). Natural red ochre and hematite are both iron oxides, the second one is a mineral, while the first one is a clay material which contains hematite. The presence of silicates can help to distinguish between both of them, however with XRF technique this is not possible (Eastaugh et al. 2004).

The XRF sensitivity for Si is too low to detect it, especially in mixed materials. The distinction therefore can not be made, so further on we will refer to the pigment as red ochre, being a wider term. Fe peaks are much higher on darker red areas (Fe Kα: 13 cps), revealing that shadows were carried out by red ochre, to which also smalt was added (Co, Ni, As, Bi peaks). The analysis of the upper part of St. Mary Magdalene's dress show no Hg peaks, only Fe ones (Fe Kα: 10 cps). The painter has used only red ochre, which he again mixed with smalt. According to the dark colour it is also possible, that in some areas of carnations or draperies the Master applied an organic red lake, which can not be confirmed by XRF (Figure 3). However, relatively high Ca peaks (Ca Kα: 9 cps) could suggest that calcium was used as a substrate for an organic red lake, maybe carmine, and as such applied on the painting. High Pb peaks (Pb Lα: 700 cps) in the red coat of the female saint standing next to the cross show towards the possibility of the use of minium, but we can not be sure, as explained above.

(d) Violet colour

Spectra of violet colour identify Ca, Fe, Co, Ni and Cu peaks, showing that the colour is a mixture of red ochre (Fe), smalt (Co, Ni), azurite (Cu) and maybe an organic red lake, as suggested by relatively high Ca peaks.

(e) Yellow colour

Yellow pigment is natural or burned yellow ochre, identified by lower or higher Fe peaks in the spectra, depending on the lighter or darker colour. It could have been applied also for darker carnations, where low Fe peaks appear. However, with XRF technique it is not possible to distinguish between yellow and red ochres, having only Fe Kα peaks as the relevant information. Only in areas which are visibly yellow as hair of several figures (Fe Kα: 4 cps) or the drapery of kneeling St. Mary Magdalene (Fe Kα: 12 cps) the earth pigment can be identified as yellow ochre. On the other hand, in some spectra of highlighted yellow coloured areas low, not always well defined peaks of Sn can be observed (Sn Kα: 1 cps). The presence of Pb and Sn together reveal the use of tin-lead yellow, as said before. There are two types of lead-tin yellow, type I and type II. They are both lead-tin oxides, however the type II may contain free tin oxide and additional silicon (West Fitzhugh et al. 1987-2007, Montagna 1993, Eastaugh et al. 2004). In order to distinguish them, the presence of Si should be confirmed, which is not possible by XRF due to the low sensitivity for chemical elements under $Z=13$. Also the ratio between Pb and Sn could give some answers. But in *The Pietà* lead is present in every analysed point of the panel in high concentrations and it is impossible to know which part of Pb belongs to the yellow pigment and which to the preparation or dryer, so the ratio between Pb and Sn can not be calculated. The use of lead-tin yellow can be observed in the ochre skirt of St. Mary Magdalene, on the figure in yellow in the background, on some architectural elements, on the cross and maybe even in some carnations.

(f) Blue colour

Blue colour was obtained by two different pigments, azurite and smalt. Azurite, identified by Cu peaks was applied in smaller areas as blue lips of the dead Christ, shadows on white and violet draperies, in several areas of the architecture in the background, while low peaks can be detected also in the colour of the carnation of the dead Christ. Count numbers of Cu Kα net peak areas vary from 1 cps in carnations to 95 cps in the violet tunic of a female saint on the right, depending on the analysed area. However, the prime blue pigment in the painting is smalt, which was applied for wider surfaces as Virgin Mary's coat, architecture in the background, the mountains and the sky that cover the upper part of the panel. It was also used, together with red ochre, for shadows of red draperies and, mixed with azurite, for shadows on some white clothes. In all these areas high peaks of Co, Ni, As and Bi can be observed, chemical elements characteristic for this blue pigment.

However, by the naked eye no blue colour can be observed in most of these areas and its presence was revealed only by XRF analysis. The pigments must have overcome some chemical changes during the centuries. Smalt has an intense blue colour at the beginning, but tends to loose its strength and can become totally transparent or it can turn brownish (West Fitzhugh et al. 1987-2007, Knoepfli et al. 1990). There can be several reasons for this chemical change of the pigment: it can be influenced by discoloration of the paint medium or by saponification products, which are normally result of cobalt ions or excised potassium that comes in contact with the oil (Eastaugh et al. 2004). In the case of *The Pietà* panel it could not be determined which process led to the discoloration of the smalt. Nevertheless, due to this chemical change the painting has today the brownish aspect. Different tonalities of brownish-greyish colour correspond to relatively higher (Co Kα: 35 cps) or lower (Co Kα: 3) amount of the pigment, in the mixture with lead white (Figure 4).

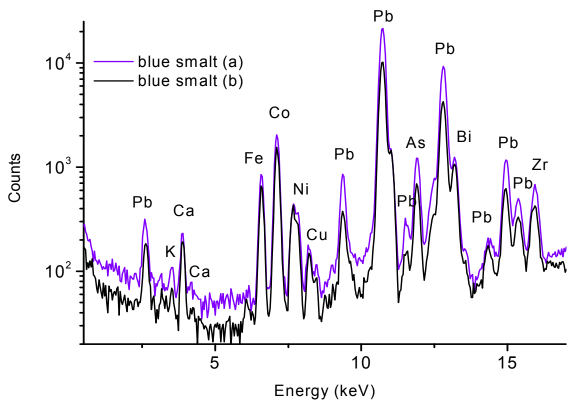

Figure 4. Comparison of two XRF spectra, showing blue smalt (Co, Ni, As, Bi) on the Virgin Mary's dress, in lighter (a) and darker (b) tonality, with more/ less Pb white added.

(g) Green colour

High Cu peaks in green areas (some parts of vestments, trees and grass) reveal the use of some copper based green pigment. Its highest quantity can be found in the green tunic of the female saint on the right border (Cu Kα: 332 cps), while less pigment was applied on trees or grass (Cu Kα: 130 cps). The palette of copper based green pigments is very wide, but with XRF it can not be identified more precisely, because it does not give the molecular composition of the pigment. Among the most used in the 16th century Flemish painting were malachite, verdigris and copper resinates (Wehlte 1967, Knoepfli et al. 1990, Montagna 1993), so the Master of the Female Half-Lengths probably used one of those. Different tonalities were obtained by adding lead white or some natural or burned ochre to the green colour. This mixture was used above all for the brownish floor of the scene. Low Mn peaks (Mn Kα: 2 cps) observed together with Fe peaks on some spectra obtained from rocks and floor show that also natural or burned brown umbra was applied, especially for the darker tones. The black pigment, used above all for details, fine lines and some shadows, is of organic origin and can not be identified by XRF.

Interventions, retouches

In various areas of the panel painting the presence of chemical elements Ti, Zn and Ba was observed already by the naked eye and then confirmed by the inspection under the UV light. They reveal the use of modern pigments titanium and zinc white, which appeared not before the middle of the 19th century, but entered in regular use only at the beginning of the 20th century (Wehlte 1967, West Fitzhugh et al. 1987-2007, Knoepfli et al. 1990, Montagna 1993, Eastaugh et al. 2004). Therefore can not form part of the original painter's palette and belong to later interventions, which are not documented.

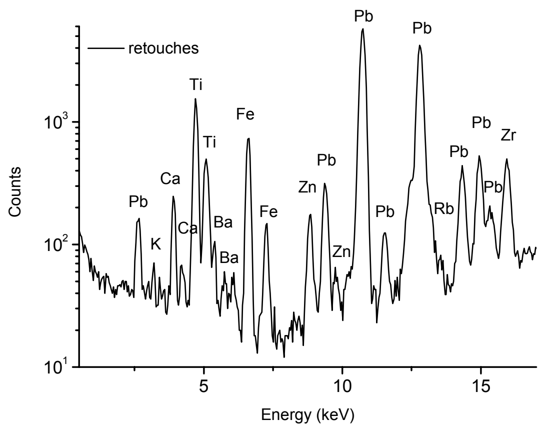

Figure 5. The XRF spectrum of the Christ's leg, showing important retouches, made by Ti-Zn white, mixed with some ochre.

The presence of low Ba peaks could identify the use of barium sulphate, which can be found as the pigment base in Ti white or together with Zn sulphur in lithopone, also applied in 20th century retouches. Net peak areas of Ti, Zn and Ba are extremely variable, in some cases can not even be determined, while in the others can get up to 78 cps or even 600 cps as in the case of Zn Kα peak on the Christ's body. These pigments were mixed with others, mostly ochres, to obtain the appropriate colour of the retouched area. Where Zn peaks are very high, normally also Fe peaks are strong.

In some cases, retouches can be seen by the naked eye, especially on the junctions between the panels. As the wood was drying, the panels were separating. The gaps between the junctions were restored and overpainted, but these interventions help us see where these junctions are, without having to take the painting off the wall. Among the figures in the painting, the dead body of Christ has many retouches with Ti-Zn white (Figure 5), an important intervention was discovered also in St. John Evangelist's face and in some saint's vestments, while many small retouches can be found all over the painting (Figure 6).

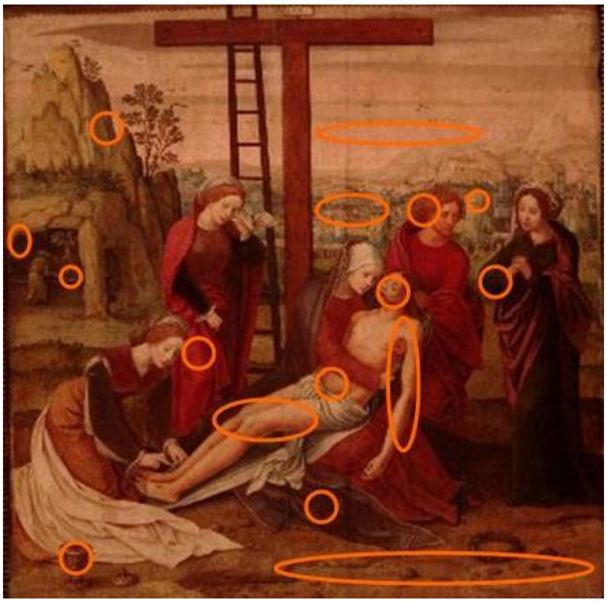

Figure 6. ***The Pietà*** **by The Master of the Female Half-Lengths. Several retouched areas with Ti-Zn white.**

Table 1. The pigments applied on *The Pietà* panel painting, according, to colour and chemical elements. Elements given in red type are present in high concentration, while (tr) indicates trace amount

Colour	Chemical elements	Pigments
White pigment	Ca, Fe, Cu (tr), **Pb**	Lead white
Carnations	Ca, Mn (tr), Fe, Cu (tr), Hg, **Pb**	Lead white + cinnabar/ vermilion + red or yellow ochre + red lake (?)
Yellow pigment	Ca, **Fe**, **Pb**, Sn (tr)	Yellow ochre + lead-tin yellow
Red pigment	Ca, Mn (tr), **Fe**, **Hg**, Cu (tr), **Pb**	Cinnabar/ vermilion + red ochre + red lake (?)
Blue pigment	Ca, Fe, **Co**, Ni, **Cu**, **Pb**, As (tr), Bi (tr)	Smalt + azurite
Green pigment	Ca, Mn (tr), Fe, **Cu**, **Pb**	Copper based green pigment
Brown pigment	Ca, Mn, **Fe**, Cu, **Pb**	Burned yellow ochre + umbra
Black pigment	/	Organic black pigment
Interventions	Ca, Ba, Ti, **Zn**, Fe	Titanium white + zinc white + barium sulphate (lithopone?)

Conclusions

The panel painting *The Pietà*, made by the anonymous Master of the Female Half-Lengths towards 1550, belongs to the permanent collection of the Fine Arts Museum in Seville. Because of its unusual brownish tonality it was analysed by the portable non-destructive X-Ray Fluorescence technique, directly *in situ*. The original palette of the Master as well as possible alterations of pigments applied were the main object of this research. The painting was analysed in 125 points, always under the same measurement conditions. The pigments applied are common in the 16th century, lead white, yellow and red ochre, cinnabar, azurite, smalt and some copper based green pigment. Also a possible presence of lead-tin yellow and minium was detected, while some results led to the conclusion about the use of some organic red lakes and black pigments, which can not be identified by XRF. The brownish tonality of the painting is a result of chemical alterations of blue pigment smalt which lost its blue colour and turned brownish and transparent. This is why the sky, the mountains and the vestments that should be blue, appear brown, like in other Flemish paintings in the Museum's collection. On the other hand, a lot of retouches were found, made by modern pigments Ti and Zn white, especially in the junctions of the wood panels and in the Christ's figure, but also many small ones were detected (Table 1). There are no informations about earlier interventions; this is why these results are of special interest for restorers and conservators.

Acknowledgements

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TECHNOLOGIES FOR THE CONSERVATION AND VALORIZATION OF CULTURAL HERITAGE (CSD-TCP): SPAIN SHOWS ITS PRIORITIES THROUGH A NEW CONSOLIDER RESEARCH PROGRAM

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In December 2007 the Ministry of Science and Innovation (MiCInn) placed its wager on cultural heritage. It approved the project called *Research programme on technologies for the conservation and valorization of cultural heritage* (TCP), designed to create a strong cluster of research groups around the theme of applying technology to the study, protection and valorization of cultural heritage. This cluster, once consolidated, should be able to compete with similar networks on an international level. The funding necessary was provided by the new Consolider-Ingenio 2010 program, designed for large-scale support of scientifically competitive networks.

Due to different administrative problems the CSD-TCP project has not entered full functioning mode until 2009, which has not impeded the streamlining of cooperation between the partners. These include 15 research groups (Figures 1-3) from different institutes of the Consejo Superior de Investigaciones Científicas (CSIC) and the universities of Jaén (UJa), Politécnica de Madrid (UPM), País Vasco (UPV-EHU) and Santiago de Compostela (USC), as well as one private company (NECO). These partners enjoy funding up to 5 million € in 5 years, which has helped continued research, contracting of experts and other service providers and, generally, ensuring the highest quality in heritage research available on the market.

The team members and their affiliations are detailed in Table 1. This large team includes some of the best Spanish specialists in cultural heritage studies from the diverse disciplines involved in studying and protecting this valuable social asset. The team uses current trends in the field, availing itself of top of the line resources and technologies. Collaboration between the teams is the main objective so that a solid network can be created for the future. This cooperation stems from the previous experience of the teams involved, which has only grown tighter as the program has advanced.

Table 1. Member groups of the CSD-TCP team

Member	Coordinator	Institution
ArqBio	M. Moreno	CCHS, CSIC
ArqueoMetal	A. Perea	CCHS, CSIC
CERVITRUM	M.A. Villegas	CCHS, CSIC
EST-AP	I. Sastre	CCHS, CSIC
GIPSE	J. Vicent	CCHS, CSIC
GFYT	S. Ormeño	UPM
CAAI	A. Ruiz	UJa
EPEC	A. Martínez Cortizas	USC
SINCRISIS	M.V. García Quintela	USC
GPAC	A. Azkarate	UPV-EHU
LANAPAC	M. Castillejo	IQFR, CSIC
LaPa	F. Criado Boado	IEGPS, CSIC
MICROPATH	C. Saiz-Jimenez	IRNAS, CSIC
PAP	R. Fort	UCM-IGE, CSIC
PATRYMAT	M.T. Blanco-Varela	ICCET, CSIC
NECO	C.A. González	NECO Information Technologies

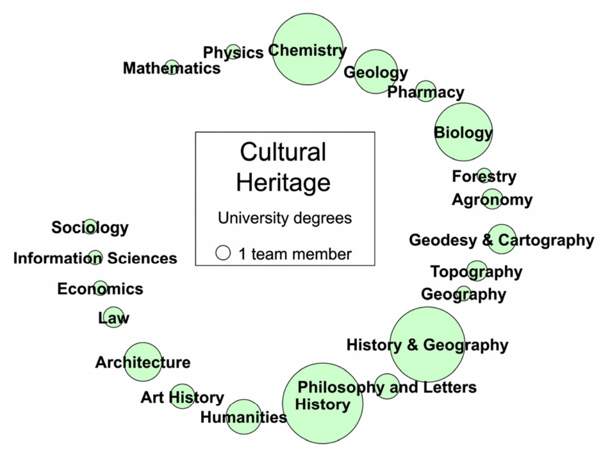

Figure 1. Team composition (I)