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PGAA, INAA and luminescence to trace the "history" of "The Panoramic View of Lisbon": Lisbon before the earthquake of 1755 in painted tiles (Portugal)

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Abstract "The Panoramic View of Lisbon" is a unique masterpiece of Portuguese glazed tiles which depicts the city before the earthquake of 1755. Compositional analysis was done by PGAA, INAA and XRD. The first absolute dating by luminescence was done, including a reconstruction of the history of radiation exposure of the pieces. Compositional patterns of the tiles suggest the use of carbonated clays from downtown Lisbon to manufacture the ceramic bodies at high firing temperatures (900–1100 °C). Luminescence dating results point to the manufacture of "The Panoramic View of Lisbon" in the late 17th–early 18th centuries.

Keywords "The Panoramic View of Lisbon" · Glazed tiles · Prompt-gamma activation analysis · Instrumental neutron activation analysis · X-ray diffraction · Luminescence dating

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Introduction

In the last five centuries glazed tiles ("azulejos") have been a decorative art of greatest significance in the Portuguese culture, art and architecture. The National Glazed Tiles Museum (*Museu Nacional do Azulejo*—MNAz) represents itself the importance of this type of art in Portugal. Among the numerous tiles panels, the highlight of the MNAz is "The Panoramic View of Lisbon" (commonly known as "Vista"), a blue and white illustration of the city of Lisbon before the destruction caused by the earthquake of 1755. In fact a notable change occurred by the reconstruction that followed the earthquake so the iconography of this panel is fundamental to know the architecture and cityscape of Lisbon in the first half of 18th century. According art historians it is a masterpiece because it is the closest possible approximation of an 18th century panoramic photograph, showing 14 km of riverfront, depicting churches, palaces, convents and houses.

The blue and white style of decorating tiles was introduced to Portugal from the Netherlands in the second half of the 17th century. Workshops in Amsterdam created large tile panels with historical scenes for Portuguese clients. However when King Pedro II of Portugal stopped all imports of *azulejos* between 1687 and 1698, local workshops such as Gabriel del Barco, took over the production. Soon large home-made blue-and-white figurative tile panels, designed by academically trained Portuguese artists, became the dominant fashion.

"Vista" is one of the most remarkable Portuguese panels, both for the rarity of its iconography [1] and for its huge dimensions (~23 m × 1 m). The panel is not signed and dated. Several dates have been attributed to the manufacture of the panel: 1725 by Simões [1] and 1730 by Vieira da Silva [2]. More recently José Meco for stylistic reasons has attributed the panel to the Spanish tile painter Gabriel del Barco (1648–1703?) dating it to 1700, during the height of the popularity of “azulejos”, from about 1690 to 1750, when many exterior and interior walls were faced by complex continuous picture tiles [3].

Since their production, the glazed tiles of the precious “The Panoramic View of Lisbon” panel have been in several locations under different conditions. Firstly, the panel was ordered to decorate an inner room of the Santiago Palace, in Lisbon. The glazed tiles were removed by the owner, and sold in 1876 to the Royal Academy of Fine Arts. In 1903 the glazed tiles of the panel were transferred to the *Museu Nacional de Arte Antiga* (MNAA), and in 1961 transferred to the MNAz. A significant conservation intervention of the tiles was performed in the conservation laboratory of MNAz [4] in order for the panel to be transported and exposed in EUROPALIA exhibition which was held in September–October, 1991. The two outer parts of the panel were not treated and were kept in the museum depository.

Compositional studies of Portuguese glazed tiles, including tiles from the “Vista”, have been performed in order to get a better insight of the “history” of this famous panel, and for the establishment of conservation strategies of ancient tiles in general [5–10]. In the present work two glazed tiles of the “Vista”—one from the exhibition room and submitted to the conservation treatment of 1991, and one from the depository (not treated in 1991)—were selected for a detailed compositional study using prompt-gamma activation analysis (PGAA), instrumental neutron activation analysis (INAA) and X-ray diffraction (XRD). These methods have been found very useful in characterization and provenance studies of archaeological and historical materials, and raw materials [11–14]. PGAA provides concentration data mostly for major components and for some minor and trace elements with high neutron absorption cross-section. On the other hand, INAA is much more sensitive for a series of trace elements. While INAA requires sampling of the object, during PGAA, the object is irradiated by external neutron beam, as a whole and the result is characteristic for the irradiated volume. Previous compositional studies of clay materials of the Lisbon region have been done including clays historically used for ceramics production in Lisbon [15]. In this way a comparative study with previous chemical and mineralogical results of clays from Lisbon is also done in this paper. Despite the absence of signature and date in the masterpiece, a narrow time range according to historical evidence can be assumed. Luminescence dating techniques [16–18] were applied for the first time to obtain absolute date of the “Vista”. In order to attain a better chronological accuracy a detailed environmental dosimetric reconstruction was performed according to historical evidence of the several locations of the panel till the present day.

The specific objectives of this work are: (i) the chemical (major, minor and trace elements) and mineralogical characterization of the ceramic body of two tiles of the “Vista”—one from the exhibition room (treated in 1991) and one from the depository (not treated); (ii) a contribution for the identification of the raw materials used and the production technology to make the ceramic bodies; and (iii) luminescence and dating of the tiles as a contribution for a better establishment of the manufacture date of the panel and for the assignment of the artist. Thus, a better knowledge of the materials and techniques involved in the manufacture and the date of “The Panoramic View of Lisbon” panel is expected in this work.

Materials and methods

“The Panoramic View of Lisbon” panel and samples studied

The panel is a remarkable blue-and-white Portuguese panel, with huge dimensions (~23 m × 1 m and more than 1200 tiles with 14 cm × 14 cm). One part of the panel exposed in the MNAz is shown in Fig. 1.

The sampling of the ceramic body was done in two tiles of the “Vista”: (i) one from the nowadays exhibited part at MNAz (sample VLE) and (ii) one from the depository part (sample VLD) (Fig. 2). The ceramic body samples were obtained in the rear of the tile, after cleaning the outer surface in order to avoid any contamination including from mortar and glue residues. Only two tiles were sampled due to the high value of the panel.

Chemical analysis

The chemical (element) composition was obtained by prompt-gamma activation and instrumental neutron

Fig. 1 Partial view of “The Panoramic View of Lisbon” panel exhibited in the MNAz (Lisbon, Portugal)

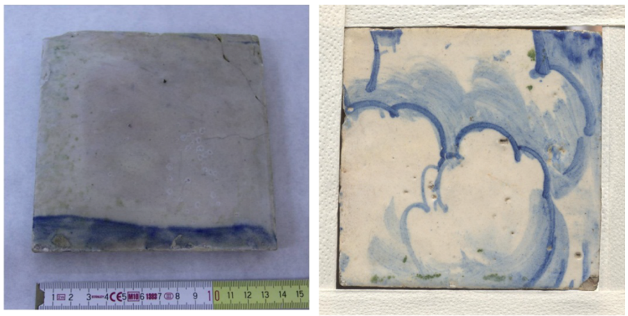

Fig. 2 Images of the ceramic tiles of the panel "The Panoramic View of Lisbon" studied **a** tile exposed in MNAz (treated), and **b** tile from the panel segment stored in the depository (not treated)

activation analysis (PGAA and INAA, respectively). Relative precision and accuracy are, in general, within 5 %, and occasionally within 10 %. The details for PGAA are presented in Table 1.

Prompt-gamma activation analysis of the ceramic body has been performed at the PGAA instrument of the Budapest Neutron Centre [19]. The PGAA instrument operates on a 7.6 × 10^7 cm^{-2} s^{-1} intensity guided horizontal cold neutron beam. The samples have been irradiated with a beam collimated to 24 or 44 mm^2. The prompt- and delayed gamma photons emitted after neutron capture were detected with a HPGe detector in Compton-suppression mode. The typical acquisition time varied between 2300 and 8300 s, in order to collect statistically significant peak areas in the gamma-ray spectra. In one particular case, a sample have been measured overnight with thermal neutron beam, which is used during the "cooling period" of the Cold Neutron Source in the beginning of a reactor campaign. The collected spectra have been evaluated with the Hypermet PC software, the element identification and calculation of concentrations are based on our PGAA library. Details of the method can be found elsewhere [20–22]. Quantitative compositional data of SiO_2, TiO_2, Al_2O_3, Fe_2O_3, MnO, MgO, CaO, Na_2O, K_2O, H_2O, SO_3, Cl and B were obtained.

For INAA the samples and standards were prepared for analysis by weighing 200–300 mg of powder into cleaned high-density polyethylene vials. Two standards from the Institute of Geophysical and Geochemical Prospecting (IGGE) GSR-4 (sandstone) and GSS-1 (soil) were used. Two aliquots of each standard were used for internal calibration (Quality Assurance). Short (1 min) and long irradiations (6 h) were performed in the core grid of the Portuguese Research Reactor (CTN, IST) at a thermal flux of 3.96 × 10^{12} n cm^{-2} s^{-1}; $\phi_{epi}/\phi_{th} = 0.01$ %; $\phi_{th}/\phi_{fast} = 29.8$ [23]. Different cooling times were selected to determine radionuclides with different half-lives. Corrections for the spectral interference from U fission products in the determination of Ba, REE and Zr were made. Details of the analytical method may be found elsewhere [24]. The contents of Sc, Cr, Co, Zn, Ga, As, Br, Rb, Zr, Sb, Cs, Ba, La, Ce, Nd, Sm, Eu, Tb, Dy, Yb, Lu, Hf, Ta, W, Th and U were obtained.

Table 1 Chemical results obtained by PGAA and INAA of the ceramic body of two tiles belonging to "The Panoramic View of Lisbon"

Method	PGAA				INAA					
Sample	VLE		VLD			VLE	VLD		VLE	VLD
	Conc. (wt %)	Rel. unc. (%)	Conc. (wt %)	Rel. unc. (%)		Conc. (mg/kg)	Conc. (mg/kg)		Conc. (mg/kg)	Conc. (mg/kg)
SiO_2	44.1	1.3	48.6	1.1	Sc	10.8	10.5	La	27.9	28.7
TiO_2	0.978	2.0	0.862	2.5	Cr	74.3	71.5	Ce	58.2	61.0
Al_2O_3	10.9	1.7	10.8	1.7	Co	10.3	10.2	Nd	28.0	28.3
Fe_2O_3 (t)	4.28	2.6	3.70	2.3	Zn	69.7	64.7	Sm	4.99	5.15
MnO	0.064	6.0	0.046	2.1	Ga	13.4	15.1	Eu	1.13	1.11
MgO	3.5	5.0	3.7	5.0	As	9.05	17.0	Tb	0.632	0.596
CaO	32.5	1.7	27.1	1.8	Br	1.6	1.2	Dy	4.03	3.89
Na_2O	0.91	4.0	1.01	4.0	Rb	66.3	70.8	Yb	1.91	1.81
K_2O	1.27	1.9	1.91	1.7	Zr	191	141	Lu	0.299	0.282
H_2O	1.10	1.4	1.16	1.2	Sb	0.863	3.29	Hf	4.87	4.31
SO_3	0.28	6.0	1.00	3.7	Cs	5.12	4.6	Ta	1.68	1.65
Cl	0.021	2.6	0.096	1.6	Ba	151	150	W	1.15	1.66
B	0.00689	1.2	0.00701	1.1				Th	8.22	8.67
								U	3.54	2.40

Mineralogical analysis

Mineralogical analysis of the ceramic body of the tiles was performed by X-ray diffraction (XRD) of non-oriented aggregate powders using a Philips X' Pert Pro diffractometer, CuKα radiation at 45 kV and 40 mA, a step size of 1° 2θ/min from 2 to 70° 2θ. The mineral proportions were determined by semi-quantitative XRD diagnosis of peak areas [25, 26].

Luminescence and dating

Samples for luminescence analysis were cut from the rear faces of the tiles. 4–11 µm quartz grains were obtained by repeatedly dissolving <63 µm fragments of the tiles in HCl (10 %) + HF (10 %). Thermally- and optically- stimulated luminescence (TSL, OSL) measurements were made on Riso DA15 and DA20 readers with calibrated $^{90}Sr/^{90}Y$ irradiators [27, 28].

Thermal activation characteristics of the "110 °C" TSL peak of the samples were measured on one aliquot each. The samples were given a predose of 35 Gy, heated to temperatures between 200 and 650 °C at 10 °C s^{-1}, and the TSL response to a test dose of 0.7 Gy was then measured at 1 °C s^{-1}, with a quartz-window between sample and PMT. Absorbed dose (Gy) was estimated from 2 aliquots of each sample, using a single regenerative dose, multi-stimulation measurement sequence with detection in the UV (IRSL, OSL, TSL [28]). Alpha efficiency (k_{eff}) was evaluated by using this measurement sequence before and after irradiation in a Littlemore unit. Dose rate (mGy a^{-1}) to 4–11 µm grains, from each tile matrix and their external environments, were calculated based on PGAA (K_2O) and INAA (Th and U) results from the tile matrices, average as-received and drained water contents of 28 similar tiles, and time-weighted averages of 19 field gamma spectrometry (FGS) measurements in historic locations of the "Vista". Cosmic ray dose rate was calculated based on an overburden of 50 cm of masonry. Ages and hence calendar dates were estimated by dividing dose by dose rate.

Results and discussion

The mineralogical composition obtained by XRD of the ceramic bodies showed that both samples are mainly composed of quartz, gehlenite, and wollastonite; small amounts of feldspars, hematite and cristobalite were also found. The mineralogical assemblage points to high firing temperatures (1000–1100 °C) [25].

The element concentration results obtained by PGAA and INAA are given in Table 1. The results obtained for major and minor elements agree in general with those previously obtained for the ceramic body of a different tile of the Vista panel [4]. According to these authors, a similar chemical composition has been found for many ceramics of the seventeenth–eighteenth centuries, particularly the high calcium oxide and low silica contents. Concerning trace elements, significant differences in the contents of some elements (higher than 10 %, the upper limit for analytical errors of both PGAA and INAA) of the two ceramic bodies studied in this work were found, namely for Hf, W, Br, Zr, As, Sb and Cl (in increasing order). These differences may be partially due to different proportions of minerals where these elements are incorporated in the raw material used, contamination of the ceramic body during manufacturing and/or treatment procedures.

Nevertheless the rare earth elements (REE) contents variations between the two ceramic bodies are lower than 10 %, and the patterns are similar (Fig. 3a). Concerning clayey materials, REE are well known as good fingerprints of geological contexts (source of sediments, and the depositional and post-depositional environments), particularly the distribution curves including the degree of fractionation between light, intermediate, and heavy REE, as well as Ce and Eu anomalies [29, 30]. Based on REE patterns, the two ceramic bodies appear to be produced with the same type of clay, which could be expected if one assumes that most of the ceramic bodies of the panel tiles may have been produced using the same type of raw material before the preparation to be painted by the artist.

The production of ceramics in Lisbon is well known for many centuries, and the hypothesis of using clays from Lisbon region to produce the ceramic body of the "Vista" tiles may be expected. So, in this work a comparative study using REE patterns (Fig. 3) and the mineralogical composition of the ceramic body of the tiles, and nine Lisbon clays previously analysed by the same methods [15] was done. These clayey materials comprise nine Miocene samples collected in Lisbon area (seven clayey fine sands from the Formation M^2_{IVa}—"Argilas do Forno do Tijolo"; and two from the Formation M^1_{II}—"Areolas da Estefânia" [15]. The mineral phases found in the ceramic body of the tiles studied in this work agree well with the minerals present in these clays (quartz, calcite, feldspars, phylosilicates, anatase), pointing to high firing temperatures of the tiles with the complete transformation of calcite (1000–1100 °C) [25]. The REE patterns suggest the use of clays from the "Argilas Forno de Tijolo" (AFT) formation, with a good match with one particular sample (AFT-9, see Fig. 3c). Thus, despite the reduced number of tiles analyzed, the results obtained point to the use of clays from downtown Lisbon to produce the ceramic body of the tiles to be further painted with the remarkable view of Lisbon before the earthquake of 1755.

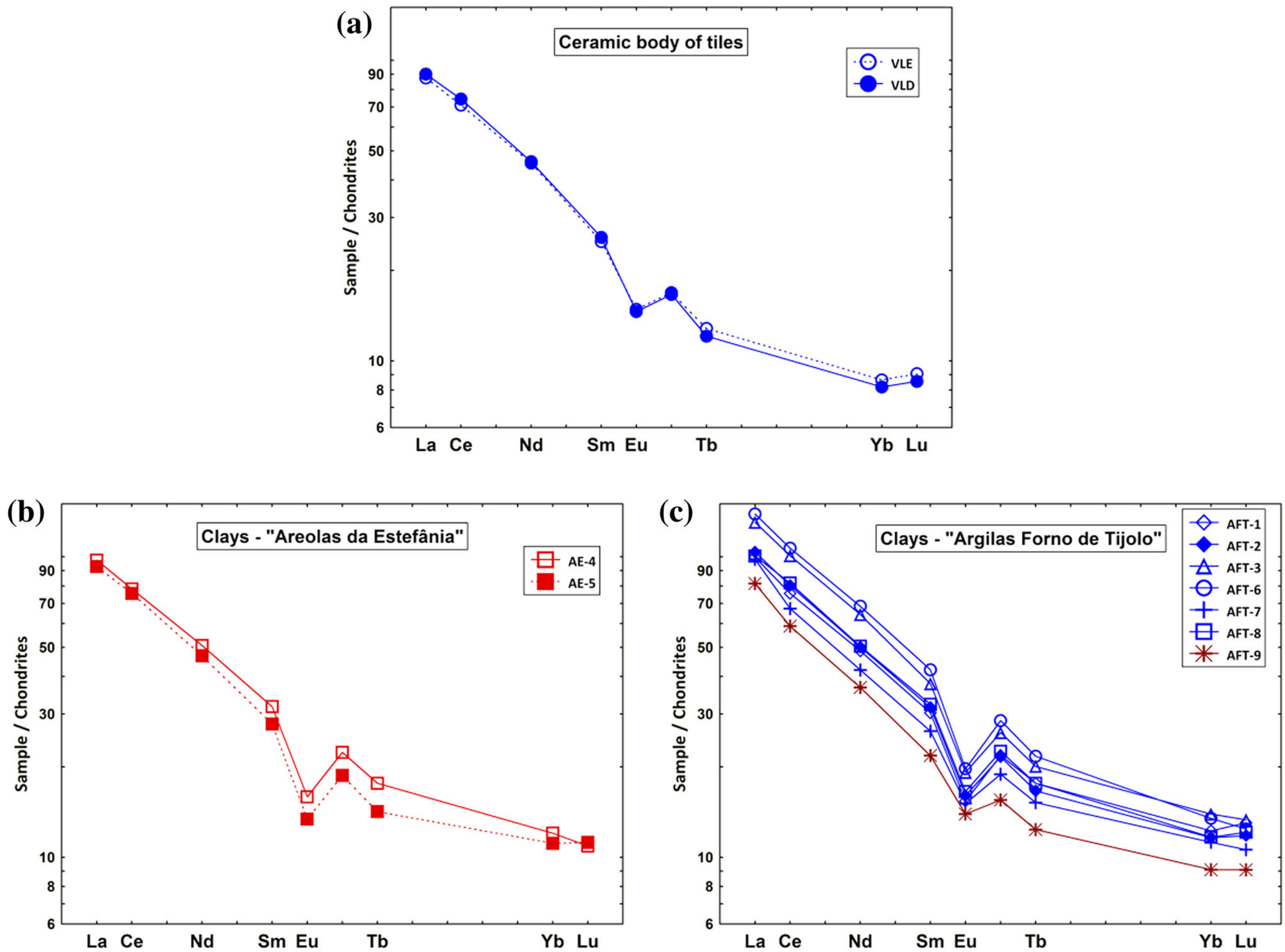

Fig. 3 Chondrites-normalized REE patterns of the ceramic body of **a** the two tiles of the panel "The Panoramic View of Lisbon"—VLE and VLD; and clays from Lisbon region—**b** "Areolas da Estefãnia" formation, and **c** "Argilas do Forno de Tijolo" formation

The timeline for location of the Vista, and gamma dose rate estimates for luminescence dating are given in Table 2.

Weak increase in "110 °C" TSL dose response between activation temperatures of 300 and 400 °C (Fig. 4) is considered to represent activation of the 35 Gy predose. Continuous strong increase above 450 °C is consistent with observations from quartz from high-fired ceramic materials [31], and the greater increase observed for VLE was coincident with high temperature mineral phases identified by XRD.

Despite strong activation of the "110 °C" TSL signal, predose measurements were imprecise. OSL yielded very low, but useable, signals from the unheated sample material. TSL from the OSL source trap was not evident at relevant dose levels, apparently due to thermal quenching. Weighted mean OSL absorbed doses obtained for VLE and VLD were respectively 1.08(±0.24) Gy and 1.12(±0.17) Gy. When combined with K, Th and U concentrations from Table 1, time-weighted external gamma dose rates of 0.64 mGy a^{-1} and 0.77 mGy a^{-1}, time averaged water contents of 2.2 %, cosmic dose rate of 0.21 mGy a^{-1} and k_{eff} of 6.9 %, the luminescence results obtained were: VLD = AD 1650(±60) and VLE = AD 1660(±80). Given the rather low OSL signal levels and their activation through the measurement sequence, systematic effects on absorbed dose estimation are considered possible, within errors. Although the external dose rate was well addressed by historical reconstruction and field gamma spectrometry measurements, the dating results are also highly dependent on how alpha dose rate is taken into account: it should be noted that k_{eff} was evaluated by signal matching to 5 Gy beta, using activated material. The present estimates lie within 1 sigma errors of the turn of the late 17th—early 18th centuries, and are not significantly different (i.e. not out-with 2 sigma) from historical interpretations of manufacture of the Vista.

Thus, the analytical approach used in this work together with historical evidence point to a Lisbon production of the ceramic body of the tiles and reinforce the José Meco

Table 2 Timeline for location of the Vista, and gamma dose rate estimates for luminescence dating

Period	Local	Configuration	Representative measurements	$\dot{D}\gamma$	$\sigma\dot{D}\gamma$	Time fraction
VLE, VLD						
Manufacture–1876	Palácio dos Condes de Tentugal	Exhibited on interior palace wall (with lime mortar?)	FGS, PCT 2014 average of 6	0.61	0.04	0.57
1876–1903	Academia Real das Belas Artes	Boxed reserves	INAA, samples	0.97	0.37	0.09
VLE						
1904–1947	Museu Nacional de Arte Antiga	Exhibited in atrium. Gypsum mount on wood support	FGS, MNAA atrium 2013	0.33	0.01	0.14
1947–1960	Museu Nacional de Arte Antiga	Boxed reserves	INAA, samples	0.97	0.37	0.04
1960–2012	Museu Nacional do Azulejo	Exhibited in dedicated room. (Glue on wood support/plexiglass/Aeroflam)	FGS, MNAz walls 2013 average of 12	0.74	0.01	0.17
1987 & 1991	Foreign exhibitions	ca. 2/3 transported and exhibited	X-Ray during transit?			
VLD						
1904–2012	Various	Boxed reserves	INAA, samples	0.97	0.37	0.35
Time weighted average						
VLE				0.64	0.05	
VLD				0.77	0.16	

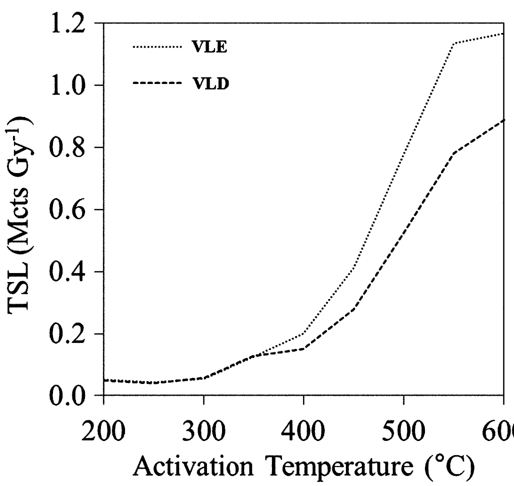

Fig. 4 Thermal activation characteristic (TAC) of irradiated quartz from samples VLE and VLD; TSL is the integral 60–100 °C of the test dose response

hypothesis [3] attributing the panel to the Spanish tile painter Gabriel del Barco (1648–1703?).

Conclusions

The use of PGAA, INAA, and XRD to characterize the ceramic body of two tiles of "The Panoramic View of Lisbon" panel, one from the depository and the other from the part of the panel exhibited in the Museu Nacional do Azulejo (MNAz, Lisbon, Portugal), indicate that the tiles may have been made of carbonated clays from Lisbon region. Gehlenite and wollastonite are the high temperatures phases found in the tiles pointing to firing temperatures around 1000–1100 °C. The luminescence results also indicate a careful production technology at higher temperatures. Reconstruction of the history of radiation exposure of the pieces was accomplished by combining historical records, field, and laboratory analyses. The dating results obtained so far (late 17th, early 18th centuries) are within uncertainties of the expected period for manufacture of the "Vista", and agree with José Meco who for stylistic reasons has attributed the panel to the Spanish tile painter Gabriel del Barco (1648–1703?).

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