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# Dating a composite ancient wooden artefact and its modifications. A case study

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**Summary.** — The recent restoration of the large wooden doors of the medieval St. Ambrogio Basilica in Milan gave the opportunity for a broad project of scientific examination, including dating with radiocarbon and dendrochronology. The two doors represented a puzzle for art historians, because of the non-documented modifications occurred during their history. In fact, they are composed of various wooden elements, some of them surely modified with the 1750 jubilee restoration, assembled into three layers. The large number of these elements suggested not to restrict the samples only to the main carved scenes, but to extend them to frames and backboards, in order to obtain a consistent set of data and a more reliable chronology. Analytical campaign oriented to dating, coordinated by Quartana Restauri and LAM (University of Bergamo), took into account the different characteristics of woods, needing 24 samples carefully extracted from hidden areas to be radiocarbon dated by AMS (Accelerator Mass Spectrometry) at CEDAD (Centre for Dating and Diagnostics, University of Salento, Lecce, Italy) laboratories, and two of them dated at LABEC (Laboratory of nuclear techniques for cultural heritage, University of Florence and INFN, Italy) for comparison. The results obtained for the vertical woods of the back, belonging to Renaissance period, suggested further studies employing dendrochronological analyses based on high-resolution photographs, carried out by CUDaM (University of Milano-Bicocca). ED-XRF (Energy Dispersive X-ray Fluorescence) analyses were also carried on, by LAM, with portable instruments, in order to study the composition of the residues of polychromy, where present.

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The obtained results show a radical alteration of an original work being divided and recomposed inserting more recent parts, as well as—surprisingly—older ones. At least five different ages can be recognized in the woods. The most ancient parts, thought to belong to the 4th century AD according to stylistic exam, appear to be of the first century of the Christian era. The interdisciplinary approach, analytical methods and main results are discussed together with some historical implications.

PACS 89.90.+n – Other topics in areas of applied and interdisciplinary physics.
PACS 82.80.Ms – Mass spectrometry (including SIMS, multiphoton ionization and resonance ionization mass spectrometry, MALDI).
PACS 82.80.Ej – X-ray, Mössbauer, and other $\gamma$-ray spectroscopic analysis methods.

## 1. – Introduction

Archaeometrical studies usually need an interdisciplinary approach, trying to develop a dialogue between historians and scientists, and to construct projects which can be significant in respect to the studied object. The recent restoration—realized by Luca Quartana Restauro—of the large wooden doors of the medieval St. Ambrogio Basilica in Milan (fig. 1) gave the unusual occasion to plan and carry out a large project of scientific examination, coordinated by a team including the restorer, physicists, chemists and entomologists. The analytical campaign had the double goal to improve the conservation conditions and the knowledge of the object, also verifying the hypotheses made by historians made during the last century, about the original aspect of the doors and the influence of later modifications, additions and restorations [1].

The door consists of three layers: the external one with ten square carved scenes with many and differently decorated frames (up to five orders or typologies), attached to a second layer of horizontal boards, again protected with vertical ones facing the church interior (fig. 2). The two wings represented a puzzle for art historians and archaeologists, being composed of various wooden elements, some of them stylistically incoherent with the main scenes, and others surely modified during the 1750 jubilee restoration, as attested by two bronze nameplates reporting the phrase: "Quod religio peregrinorum imminuit / restituitur anno jubilaei MDCCL". According to historical-stylistic comparison, the older parts of the door, the main scenes, were intended to belong to the 4th century AD, with supposed modifications occurred between the 6th and the 11th century, then in the 18th one.

This complex case of a composite wooden artefact, with traces of polichromy and a long conservative history, justifies an extensive analytical campaign and the need of a large number of samples to obtain a reliable description of the ages of its different parts.

Analyses were carried out with non-invasive—where possible—as well as micro-invasive or micro-destructive techniques, including: Energy Dispersive X-Ray Fluorescence (ED-XRF) and stratigraphic analyses to study the remains of the polychrome decoration, ED-XRF analyses intended to study the composition of modern and ancient metals and alloys, studies on the wood conservation to prevent future deterioration, environmental and thermographic analyses to assess heat distribution in order to realize the optimal glass protection, and radiocarbon and dendrochronology to date the different woods.

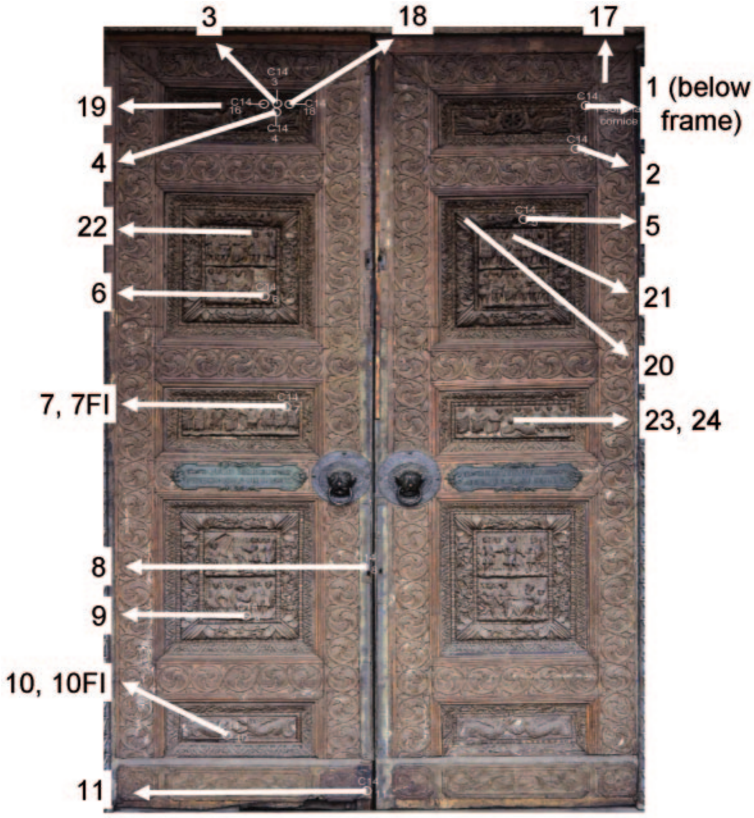


Fig. 1. – The doors of St. Ambrogio Basilica, Milan (front). Position of samples.

Dendrochronology, giving the year of the last ring sampled, was chosen because it is particularly effective in dating also recent woods, being complementary to $^{14}$C method, which has an intrinsic limit in the analysis of samples younger than about 200 years [2]. Actually, due to the mutual interference of fossil fuel burning after the industrial revolution and atmospheric nuclear detonation tests carried out after the Second World War, samples whose radiocarbon age is less than 200 years BP (Before Present) are referred as "modern". This limits $^{14}$C reasonable datable samples to the middle of the 17th century.

The intrinsic uncertainty in radiocarbon measurements (typically about $\pm 40$ years on the conventional radiocarbon age, for samples belonging to historical periods), however, cannot be sufficient to solve problems in recent times, *e.g.*, during Middle Ages or Renaissance. These are the reasons why radiocarbon dating analyses have been integrated, where possible, with dendrochronological data.

Dendrochronology or tree-ring dating is based on the analysis of tree-ring growth patterns, specifically on the difference in ring thickness, which varies as a result of climate, environmental and seasonal changes. Conventionally, a graph representing the difference in thickness of the rings is produced in order to compare the trend of different trees visually, or by software analysis [3]. A known-age reference curve for the considered specie of wood is used as reference in order to estimate the age of the samples. Not all the species of trees can be dated with this method due to the different response in growing rate of different species, which can be more or less sensitive to the environmental

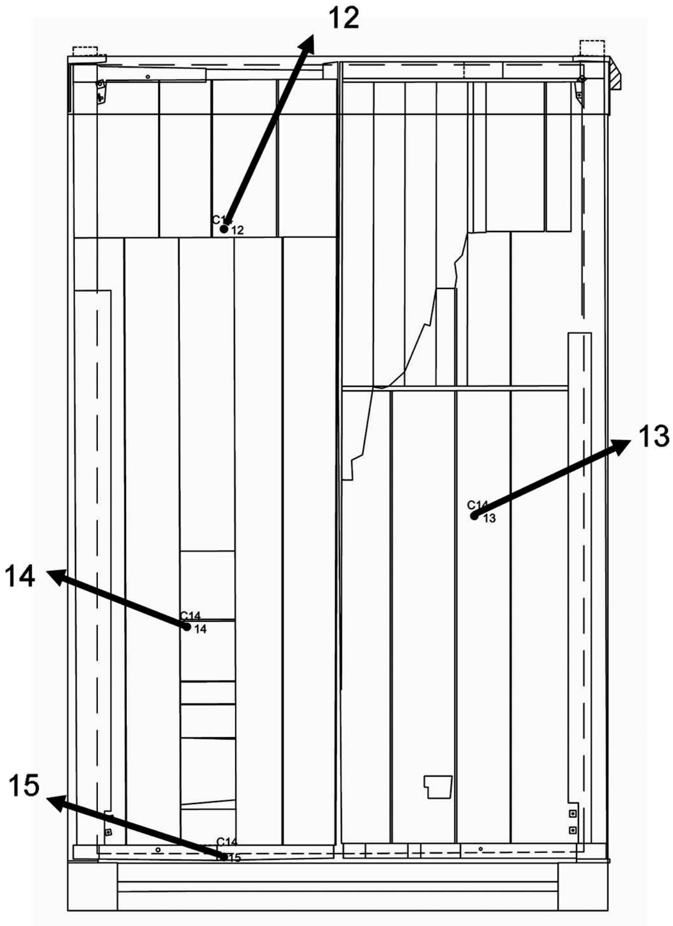


Fig. 2. – The doors of St. Ambrogio Basilica, Milan (scheme of the back). Position of samples.

parameters. Measurements in Italian region are mainly performed on pine (*Pinus pinea*), fir (*Abies alba*), larch (*Larix decidua*), and oaks (*Quercus*) [4]. The method can be applied to woods, boards and generally to all kinds of objects in which it is possible to collect at least a set of 30 consecutive rings [4]. For historic and precious objects like paintings, furniture and doors the normal sampling through drilling (generally a 16 mm hollow corer which produces a core approximately 10 mm in diameter) must be avoided and substituted, if possible, with careful high-resolution photographs. This restriction allowed us to study by dendrochronology only the back boards (third layer) of the door.

Radiocarbon dating, on the contrary, could be applied to a larger selection of woods (26 samples) from all the three layers of the door.

## 2. – Materials and methods

The samples for radiocarbon analyses were taken from the wood using chisel and scalpel, at about 1 cm of depth, taking care to reject the most external layer of wood to avoid contaminations.

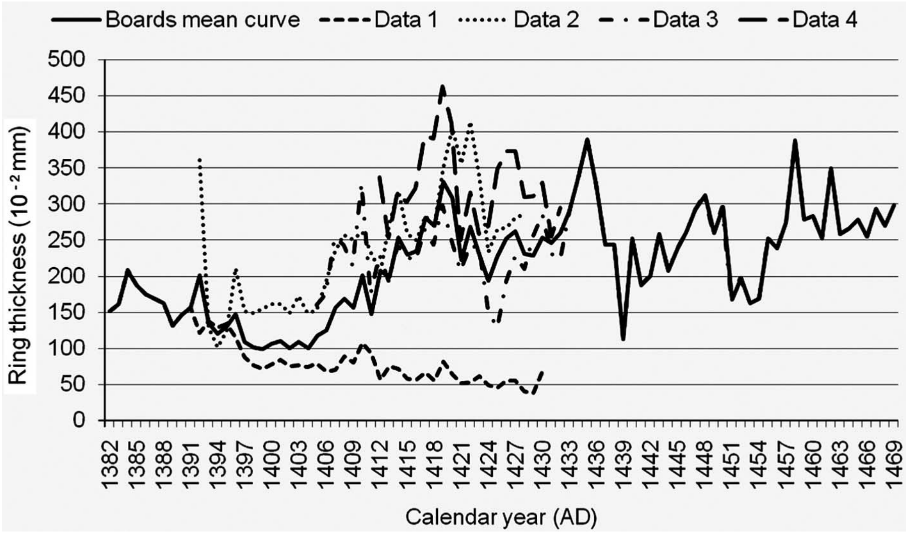


Fig. 3. – Creation of the mean chronological curve.

The 24 wood samples submitted to CEDAD for AMS radiocarbon dating analyses underwent a standard AAA chemical processing consisting of alternate acid (37% HCl), alkali (1% NaOH) and acid (1% HCl) attacks [5]. Purified sample material was then combusted to $CO_2$ in sealed quartz tube at 900 °C together with copper oxide and silver wool. The extracted carbon dioxide was then cryogenically transferred to the graphitisation lines and catalytically reduced at 550 °C to graphite by using hydrogen as reducing agent and iron powder as catalyst [6]. $^{14}$C concentration was measured at the CEDAD AMS beam line using IAEA (International Atomic Energy Agency) C6 sucrose as reference standard. Measured $^{14}$C concentrations were corrected for isotopic fractionation effects by using the $\delta^{13}$C term measured with the accelerator, and for machine and sample processing background [7].

The largest between the radioisotope counting error (Poisson Statistics) and the standard deviation of the repeated measurements carried out on each sample was used to estimate the final uncertainty of each measurement.

On the two samples dated for comparison, analogous preparation treatment—normally employed to analyze wood samples—and dating method were performed, respectively, in the CUDaM laboratory in Milan and in the LABEC AMS facility in Florence [8].

Conventional $^{14}$C ages were then calibrated by using the OxCal Ver. 4.0 [9] software, and the last INTCAL04 atmospheric dataset [10].

Dendrochronology was based on a series of high-resolution photos of the heads of nine boards from the inner layer of the door. In fact these areas of the timber were not damaged, allowing the rings to be accurately measured. Optical microscopy observation allowed the characterization of the species of the trees used in this part of the door, all being *Larix Decidua*. Nine series of rings were collected from the boards covering the inner layer of the door. The data were acquired using CooRecorder Ver. 5.3.11 software, obtaining a curve for every single board. We supposed the trees belonging to the same wood, placed in the north of Lombardy, where Milan diocese possessed many areas.

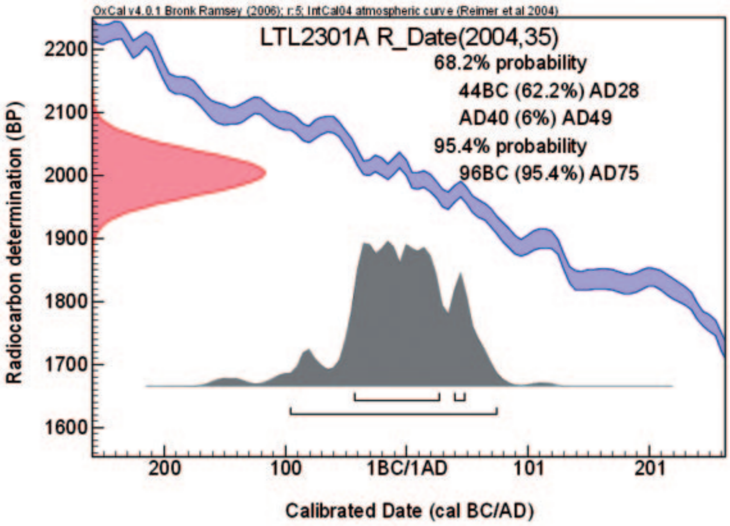


Fig. 4. – Calibration of conventional $^{14}$C date of sample 7 (CEDAD lab.).

Under this condition, it was possible to create a single mean curve, searching the points of connection of the single curves with each other and using the average value of the linking part of the curves. In fig. 3 a step of this phase is shown. The dotted curves represent the single board curves, whereas the solid one represents the mean curve. The values of thickness in $y$-axis are given in a linear scale, while the comparisons were made using a logarithmic scale, in order to reduce random variations in the slope, and to stress similar behaviours. The result was a single curve which extends through 88 years of life

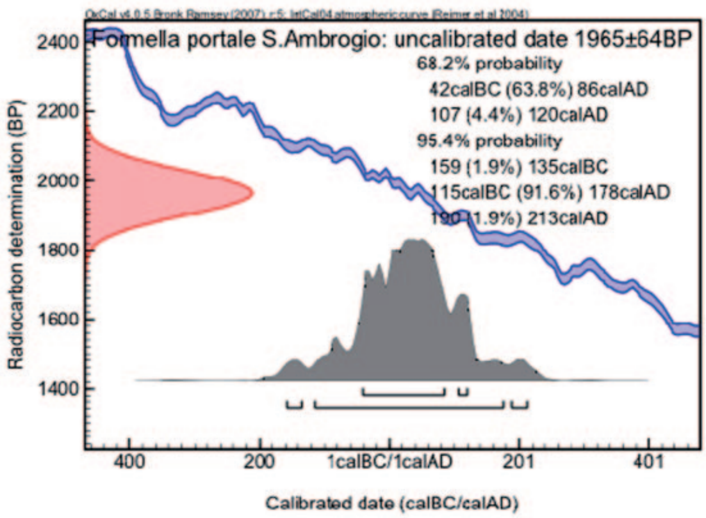


Fig. 5. – Calibration of conventional $^{14}$C date of sample 7FI (LABEC lab.).

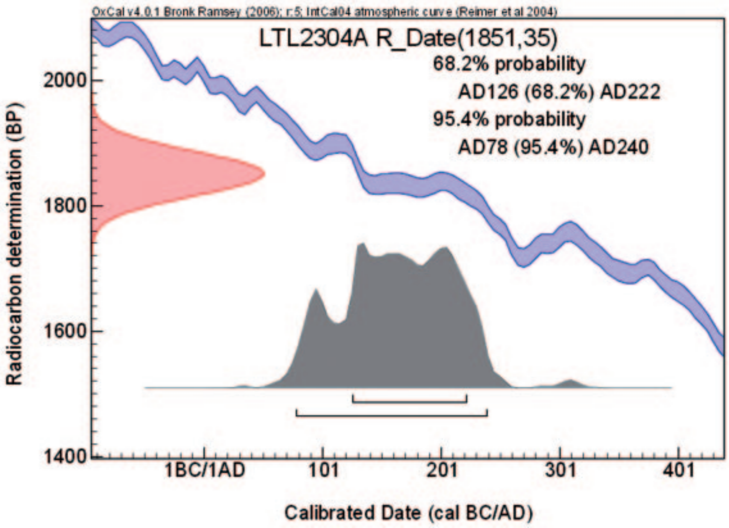


Fig. 6. – Calibration of conventional $^{14}$C date of sample 10 (CEDAD lab.).

of the trees used to build the door.

For the comparison with a known-age curve, the master chronology of *Larix Decidua* from Berchtesgaden (Germany) was chosen [11], considering the similar environmental conditions between this area and the supposed origin area of the samples. The statistical parameters (mean value, standard deviation, autocorrelation coefficient, mean sensitivity), Gleichläufigkeit coefficient, and the t-test value for crossdating operations were calculated using PAST4 software [12]. From the latter, values the Cross Date Index (CDI) was calculated, and it was taken as a reference to select the best date for the younger ring of the local curve.

In order to better understand the interventions occurred during the centuries to the woods belonging to the front part of the doors, where discontinuous residues of pigments survive, non-invasive ED-XRF exams have been performed *in situ*, with a portable spectrometer Niton XLt 797 X. The system has a Mo target and 3 mm spot diameter; X-Ray tube operating conditions were 40 kV, 35 $\mu$A; the acquisition time was 40 s.

## 3. – Results and comments

Sampled points are marked in fig. 1 and 2 and results of radiocarbon dating are shown in table I, where the calibrated ages are presented in the $2\sigma$ range.

At least five sets of ages can be recognized in the woods:

– 50 BC–100 AD (class A = Antiquity);

– 100–400 AD (class MC = Milan Capital);

– 1480–1650 AD (class R = Renaissance);

– 1650–1800 AD (class 1750 = 1750 Jubilee);

– 19th–20th century (modern).

TABLE I. – *Radiocarbon AMS results. In the last column the results are classified in different ranges of age: A = antiquity; MC = Milan Capitol; R = 16th–17th century; 1750 = 18th century restoration; MO = modern.*

| Sample no. | Radiocarbon age (BP) | $\delta$13C (per mille) | Calibrated date | Probability, 2 sigma (%) | Age group |
|---|---|---|---|---|---|
| 1 | $1967 \pm 30$ | $-30.5 \pm 0.2$ | 50 cal BC–90 cal AD | 94.4 | A |
| | | | 100–120 cal AD | 1.0 | |
| 2 | $325 \pm 35$ | $-25.1 \pm 0.6$ | 1470–1650 cal AD | 95.4 | R |
| 3 | $1781 \pm 45$ | $-24.8 \pm 0.3$ | 120–390 cal AD | 95.4 | MC |
| 4 | $1767 \pm 50$ | $-23.8 \pm 0.3$ | 130–390 cal AD | 95.4 | MC |
| 5 | $1826 \pm 55$ | $-25.7 \pm 0.4$ | 60–340 cal AD | 95.4 | MC |
| 6 | $209 \pm 35$ | $-28.0 \pm 0.3$ | 1640–1700 cal AD | 29.4 | 1750 |
| | | | 1720–1820 cal AD | 48.9 | |
| | | | 1910–1960 cal AD | 17.1 | |
| 7 | $2004 \pm 35$ | $-27.9 \pm 0.3$ | 100 cal BC–80 cal AD | 95.4 | A |
| 7FI | $1965 \pm 64$ | $-11.5 \pm 0.5$ | 159–135 cal BC | 1.9 | A |
| | | | 115 cal BC–178 cal AD | 91.6 | |
| | | | 190–213 cal AD | 1.9 | |
| 8 | $313 \pm 30$ | $-30.1 \pm 0.3$ | 1480–1650 cal AD | 95.4 | R |
| 9 | $203 \pm 30$ | $-26.1 \pm 0.4$ | 1640–1690 cal AD | 26.6 | 1750 |
| | | | 1720–1810 cal AD | 50.9 | |
| | | | 1920–1960 cal AD | 17.9 | |
| 10 | $1851 \pm 35$ | $-24.7 \pm 0.4$ | 70–240 cal AD | 95.4 | A-MC |
| 10FI | $1740 \pm 67$ | $-20.3 \pm 0.4$ | 91–99 cal AD | 0.4 | MC |
| | | | 125–432 cal AD | 95.0 | |
| 11 | $307 \pm 35$ | $-26.5 \pm 0.2$ | 1480–1660 cal AD | 95.4 | R |
| 12 | $282 \pm 30$ | $-24.6 \pm 0.5$ | 1490–1670 cal AD | 93.7 | R |
| | | | 1780–1800 cal AD | 1.7 | |
| 13 | $294 \pm 35$ | $-24.6 \pm 0.5$ | 1480–1670 cal AD | 95.4 | R |
| 14 | $491 \pm 45$ | $-25.2 \pm 0.5$ | 1310–1360 cal AD | 9.4 | <R |
| | | | 1380–1480 cal AD | 86.0 | |
| 15 | $267 \pm 30$ | $-27.2 \pm 0.4$ | 1510–1600 calAD | 37.7 | R-1750 |
| | | | 1610–1670 cal AD | 48.5 | |
| | | | 1780–1800 cal AD | 8.2 | |
| | | | 1940–1960 cal AD | 1.0 | |

TABLE I. – *Continued.*

| Sample no. | Radiocarbon age (BP) | $\delta$13C (per mille) | Calibrated date | Probability, 2 sigma (%) | Age group |
|---|---|---|---|---|---|
| 16 | $21 \pm 50$ | $-24.2 \pm 0.6$ | modern | | MO |
| 17 | $1842 \pm 40$ | $-24.5 \pm 0.4$ | 70–260 cal AD | 95.4 | A-MC |
| 18 | $1868 \pm 40$ | $-25.0 \pm 0.1$ | 50–240 cal AD | 95.4 | A-MC |
| 19 | $145 \pm 35$ | $-35.6 \pm 0.3$ | modern | 95.4 | MO |
| 20 | $100.86 \pm 1.20$ pMC | $-35.1 \pm 0.6$ | modern | | MO |
| 21 | $1941 \pm 45$ | $-22.5 \pm 0.1$ | 50 cal BC–170 cal AD | 94.2 | A |
| | | | 190–210 cal AD | 1.2 | |
| 22 | $1778 \pm 60$ | $-44.7 \pm 0.3$ | 80–110 cal AD | 1.3 | MC |
| | | | 120–400 cal AD | 94.1 | |
| 23 | $1591 \pm 50$ | $-23.6 \pm 0.4$ | 340–590 cal AD | 95.4 | MC |
| 24 | $101.23 \pm 1.60$ | $-25.8 \pm 0.8$ | modern | | MO |

The MC range corresponds approximately to the period in which Milan was the capital of the Western Roman Empire (286–402 AD). In particular, Ambrosius—the founder and dedicatory of the church—was the bishop of Milan and Lombardy between 374 and 397 AD.

For samples 6, 9 and 15, we preferred to report the calibrated age—being around the limit of 200 years of age, so in the range 1650–1800 AD—in spite of the fact they could be considered, in some respects, modern.

Comparative $^{14}$C dating on two samples (7, 7FI and 10, 10FI) in two different labs shows a good agreement (figs. 4, 5 and 6, 7). In particular the samples 7 and 7FI, from the background of the small carved scene at the centre of the left door, are dated (with about 60% probability) in the 45 BC–30 AD and 42 BC–86 AD age range, respectively: before the supposed 4th century and also before Milan was established as the capital of the Roman Empire.

For the samples 10 and 10FI, from the drakes in the lower scene of the same door, the $1\sigma$ ages belong to the ranges 125–225 AD and 230–393 AD, respectively, although these scenes were commonly believed to be realized during the Jubilee restoration, thirteen centuries later [1, 13].

About the back part of the door, radiocarbon results (samples 12-15) show the only attestation of a late Medieval intervention (sample 14), perhaps only structural, without insertion of carved wood, because it seems limited to the horizontal boards of the core layer on which the front scenes are nailed. On the vertical boards of the third layer (samples 12 and 13) the calibrated dates belong to the Renaissance period, or a little later, between 1480 and 1670 (about 90% probability). The best date obtained on these woods by dendrochronology is 1469 AD, representing the younger ring in the sequence (figs. 3, 8, and table II). It must be taken as a *post quem* term, due to the absence of

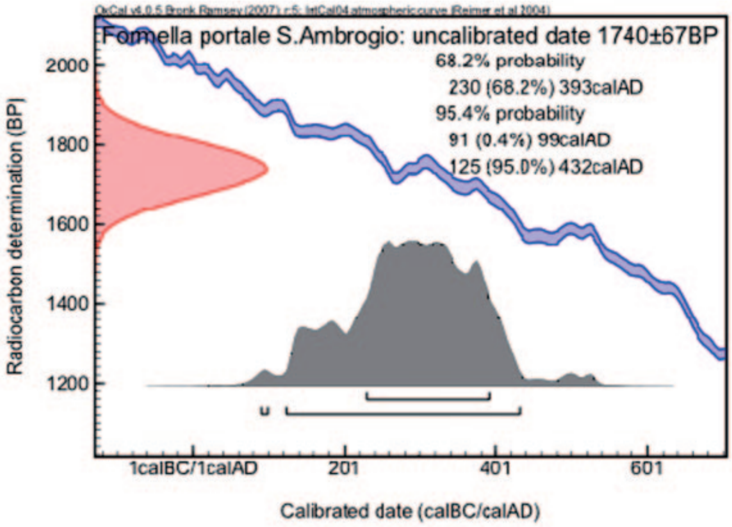


Fig. 7. – Calibration of conventional $^{14}$C date of sample 10FI (LABEC lab.).

bark and sapwood [3, 14]. Anyway, the dendrochronological result is coherent with the range of dates expressed by radiocarbon analyses, and, as supposed, it helps to obtain a better precision in dating wood younger than the 15th century.

A general analysis of the results allows some preliminary considerations. In agreement to what was stated through historical-stylistic exams, some of the older parts of the door, the main carved scenes, turn out to belong to the 4th century AD, with some exceptions. Some scenes were modified many centuries later adding new parts, usually heads (the

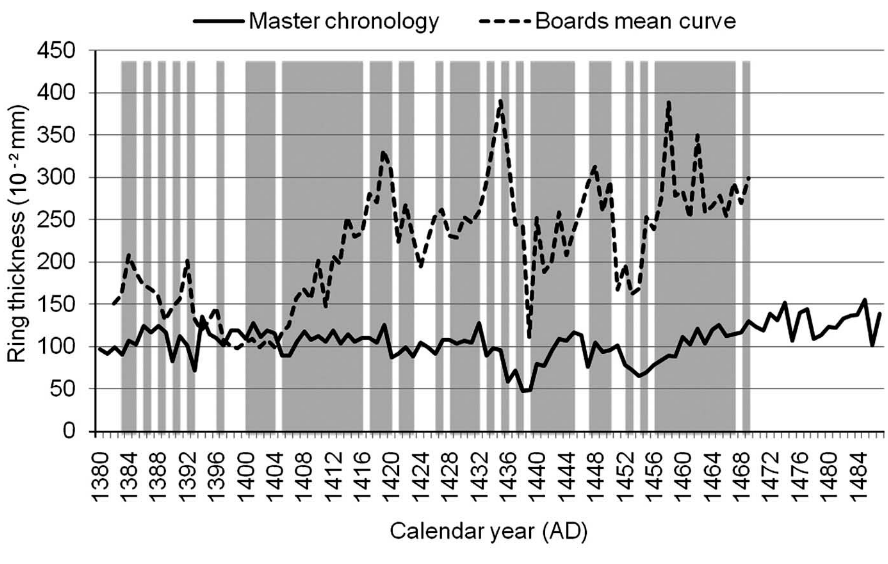


Fig. 8. – Synchronization of the mean local curve with the standard curve.

TABLE II. – *Statistical analysis of the comparison datas: TBP Baillie-Pilchier t-value; THO Hollstein t-value; GLK Gleichläufigkeit, or coincidence parameter; the number ∗ indicates the accordance value between the two curves (# = 95%, ## = 99%, ### = 99.9%); CDI cross date index* [15, 16].

| Date | TBP | THO | GLK | * | CDI |
|---|---|---|---|---|---|
| 1469 | 2.06 | 2.29 | 68.2 | ### | 41.6 |
| 1896 | 3.28 | 2.40 | 64.2 | ## | 34.1 |
| 1881 | 2.48 | 3.17 | 59.7 | # | 30.6 |
| 1846 | 2.98 | 3.08 | 59.7 | # | 29.8 |

original heads of all the human figures seem to have been eliminated). Some parts of the door, like some frames and minor panels, can be dated between 50 BC and 100 AD, an age never hypothesized by historians, and could be referred to a re-use of elder artefacts, such as a door.

We found no trace, in the sampled woods, of the supposed modifications occurred between the 6th and the 11th century; while the presence of woods of the 14th-15th and then 16th-17th centuries should indicate that some modification took place during these periods, mainly in the support structure and in some frames. It seems unlikely that so ancient wood could be inserted in the 18th century interventions. Jubilee restorations were confirmed, especially dedicated to the renovation of the main scenes, representing the Old Testamentary stories of King David.

Such a variety of woods of different periods tried to be harmonized, at a certain age of the door history, with colour. In fact, ED-XRF exams, always performed *in situ* with a portable system, revealed traces of old polychromy, now appearing brownish-white coloured, realized with Pb, Cu and Fe containing pigments. After these, cross-sections studied using Optical and Electronic Microscopy (ESEM + EDS) as well as vibrational micro-spectroscopy (micro-ATR FTIR) confirmed the existence on many areas of the wood of a lead carbonate layer covered with earths and green copper acetate. This relatively homogeneous polychromy is supposed to be a later attempt (between the 16th and the 18th century) to obtain a uniform visual aspect to the assembly of wood of different age and type.

The obtained results show a radical alteration of an original work (probably a door, again) being divided and recomposed inserting more recent parts, as well as—surprisingly—older ones, previous to the age of bishop Ambrosius (4th century). These analytical data should be taken into account by art-historians and archaeologists who will provide a new reading of the history of the door.

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