



## NON-DESTRUCTIVE MEASUREMENTS FOR CHARACTERISATION OF MATERIALS AND DATATION OF *CORONA FERREA* OF MONZA

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The "Corona Ferrea" (Iron Crown) of Monza (Italy), a gold alloy crown finely finished and adorned with precious stones and enamels, derives its name from the medieval legend that an iron nail of the Cross was contained in its interior. Without doubt it is one of the most famous masterpieces of late Roman or Longobard origin which, it is claimed, Charles the Great used for his coronation in 800. Historically based datation however traces back only to the year 1530 for the coronation of emperor Charles V. It was later used by Napoleon for his coronation as King of Italy in 1805.

In the 14th centenary of Monza Cathedral, where the Crown is guarded, a series of non-destructive tests was undertaken with the aim of characterising material (metals, glasses, enamels) and obtaining detailed information on the manufacturing technique.

Non-destructive analyses were performed using energy dispersive-XRF portable instrumentation. To irradiate the internal surfaces of the six gold plates which make up the Crown we employed the radioactive isotope americium-241 as the x-radiation source, while to probe the other parts (approximately 200 separate points were studied) we used various types of x-ray tubes equipped with glass capillary to focus the x-rays on single small spots. It was not possible to use monochromatic exciting radiation when analysing the Monza Crown; furthermore, none of its surfaces proved to be flat. This meant that the secondary, concentration-dependent x-ray emission from copper could not be calculated, neither was it possible to calculate the influence of surface irregularities on x-ray intensity. We overcame these difficulties by a method that involved calculating the ratios: copper line intensity to gold line intensity ( $I_{Cu}/I_{Au}$ ) and silver line intensity to gold line intensity ( $I_{Ag}/I_{Au}$ ). We then compared these ratios to the same ratios determined in standard samples of gold alloy whose compositions were accurately known and similar to that of the Crown. In this way the secondary excitation effect of copper was allowed for. The method depends upon the ratio of the intensities of two x-ray emission lines from a metal alloy being relatively insensitive to the geometry of irradiation.

In a second series of measurements attention was turned to smaller metal parts (such as the weld points), as well as the enamel and glass embellishments of the Crown. In this case a special x-ray collimator was used consisting of a glass capillary of internal diameter 0.7mm protected by a rigid brass sleeve. The advantage of this device and the technique (called micro-XRF) is that it focuses the x-radiation more or less to a point. The area (or point) under investigation was identified by laser alignment system. Conditions for detecting each chemical element were optimised by using different types of

x-ray tube: tungsten anode tubes for the heavy metals; tubes with molybdenum and copper anodes for progressively lighter elements.

It was not possible to analyse quantitatively the enamel or glass work present in the Crown, either in terms of light or heavy metals as it was not possible to measure the thickness of these pieces without damaging the Crown and we did not know the exact composition of their matrices. However, qualitative analysis did provide some important information.

The white enamels, considered to be original, were found to have antimony as a major metal component. Given the high level of calcium also, the antimony was probably added as calcium antimonite to render the enamel opaque. In the blue enamels antimony was present in much smaller quantities, sometimes close to the detection limit of our method. In both the white and blue enamels lead was present, probably in oxidised form, and had been added as a brightener. By contrast, the white and brownish enamels, which have a different visual appearance from the rest, were found to have tin as the major component - added to render the glaze opaque, while antimony was completely absent. These white and brownish enamels also contained potassium, presumably added as a fluxing agent. Potassium was not found in the enamels of any other laminae, and this finding is taken to imply that sodium was the flux used for these other enamels, since this is the only practical alternative to potassium. As mentioned previously, our techniques were not able to detect sodium.

The Monza Crown was subjected to radiographic photography to obtain transparent images on film. Twelve photographs were taken: six centred on the plates and six on the junctions between the plates so as to produce a composite image of the entire Crown. The x-ray photography revealed some interesting information, for example the presence of perforations in the plates hidden beneath the glass and enamel work, which were originally made to facilitate attachment to the internal metal ring but were never used.

As to datation, small amounts of wax mixed with clay to fix stones and ornamental external plates, were used for  $C^{14}$  mass spectroscopy analysis. Preliminary results obtained by the ANSTO AMS laboratory gave: radiocarbon age  $1278 \pm 31$  which corresponds to 699-776 AD (1 sigma) and 669-819 AD (2 sigma).

All the results obtained gave new information as well as confirmation of an in-depth examination performed by an international team of history and art experts which produced a basic work for the history of Corona Ferrea.