A survey of the possibilities of various radiographic techniques for the non destructive examination of cultural heritage artefacts

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With a large contribution of the image libraries of my former colleagues & friends
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Bernard Rattoni (CEA) & Elisabeth Ravaud (C2RMF)

International Conference on Applications of Radiation Science & Technology
(ICARST 2017)
IAEA - Vienna - April 24-28th 2017
Content of the presentation

1 - Specific situation of cultural heritage examination and analysis

2 – Physical basis of the different radiographic techniques

3 – Interest of radiographic examination for the study of a cultural heritage artefact

4 - Some examples of application of radiographic techniques

5 - Conclusion
1 - Specific situation of cultural heritage examination & analysis

- For the examination, characterisation & analysis of cultural heritage artefacts or art objects & their component materials, the conservation scientist needs a palette of non-destructive techniques:
  - to improve understanding of their manufacture, their evolution and/or degradation during time
  - to give a rational basis for the restoration and/or the conservation of the objects

- For non-destructive testing (NDT), the existing scope of methods includes photography, endoscopy, **radiography**, holography, liquid penetrant testing, magnetic testing, Eddy current, thermography, acoustic emission, ultrasonic testing…

- These methods are widely used in industrial control & generally as complementary ones.

- We will discuss on the different techniques which covers the general term **radiography** & see what they can provide to the study of cultural heritage artefacts
Uses of ionising radiation techniques for the examination & analysis of cultural heritage artefacts & their component materials

- X-ray radiography
- Gamma radiography (\(^{192}\)Ir & \(^{60}\)Co) of large metal (mainly bronze) & stone statues
- Accelerator radiography
- Beta radiography (\(^{14}\)C) or secondary electron radiography of paper, manuscripts, drawings, prints… to underscore watermarks, paper manufacturing processes…
- Neutron radiography & neutron induced autoradiography
- Computed assisted tomography
- X-ray fluorescence analysis: pigments in easel paintings, mural or parietal paintings, ceramics, enamels, glass…, metals in coins, medals, statues, ethnographic artefacts, jewellery…, minerals in gemstones, various archaeological findings… (in laboratories or in situ: museums, restoration workshops, excavations, on historical building scaffoldings…)
- X-ray diffraction
- Neutron diffraction
- Activation analysis (neutron & particle induced)
- Prompt gamma neutron analysis (PGNA)
- Ion beam analysis (IBA): PIXE, RBS, ERDA…
- Synchrotron radiation based analytical methods
- Thermoluminescence dating
- Carbon 14 accelerator mass spectroscopy (AMS) dating or liquid scintillation dating
Uses of ionising radiation techniques for the conservation/restoration of cultural heritage artefacts

- Desinfestation or sterilisation by irradiation (\(^{60}\)Co or accelerator)

- Resin impregnation & in core polymerisation by irradiation (\(^{60}\)Co)

* Topics that have been treated in session A09
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<thead>
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<th>Rank</th>
<th>Technique</th>
<th>Frequency</th>
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<td>Standard Colorimetry</td>
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<td>Low HV (&lt;150 kV) X-ray Radiography</td>
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<td>Spectro-Photo-Colorimetry</td>
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### Table I - LabS TECH - FREQUENCY OF THE DIFFERENT TECHNIQUES (26/02/2009)

N.B.: 114 different techniques are indicated by the 151 participating institutions

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<th>RANK</th>
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<th>FREQUENCY</th>
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<td>High voltage (150 &lt; HV &lt; 450 kV) X-ray Radiography</td>
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<td>X-ray Fluorescence Analysis - X-ray Tube - Portable</td>
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<td>Atomic Emission spectrometry (ICP-AES)</td>
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<td>Atomic Absorption Analysis (AAA)</td>
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<td>Environmental Natural Weathering Tests (Outdoor)</td>
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<td>36</td>
<td>Pyrolysis Gas Chromatography (Py-GC)</td>
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<td>38</td>
<td>Particle Induced X-ray Emission (PIXE)</td>
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<td>Pyrolysis Gas Chromatography - Mass Spectroscopy (Py-GC-MS)</td>
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<td>40</td>
<td>Environmental Scanning Electron Microscopy (ESEM)</td>
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<td>Inductively Coupled Plasma Mass Spectrometry (ICP-MS)</td>
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<td>Ultra-Sound Testing</td>
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<td>Rutherford Backscattering Spectrometry (RBS)</td>
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<td>Specific Surface Area Measurement (BET)</td>
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<td>Contact Angle measurement</td>
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<td>Carbon 14 Accelerator Mass Spectroscopy (AMS) Dating</td>
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A survey of the results of three successive research projects (2000-2015) funded by the European Commission (LabS TECH, EU-ARTECH & CHARISMA) has been recently published:

**Une Europe de la recherche en sciences du patrimoine**

**(A European research in cultural heritage science)**

Integrated Platform for the European Research Infrastructure ON Cultural Heritage

http://www.iperionch.eu
2 – Physical basis of the different radiographic techniques
The various radiographic techniques

- Microradiography (focal spot: some μm – some kV to 100 kV)

- Classical X-ray radiography (from some kV up to 450 kV) – Various detectors: classical silver halide industrial radiographic films, storage phosphor imaging plates, solid state digital detectors, fluoroscopy...

- Gamma radiography (\(^{192}\)Ir & \(^{60}\)Co)

- Accelerator radiography (2 MV up to 30 MV)

- Beta radiography (\(^{14}\)C) or secondary electron radiography of paper, manuscripts, drawings, prints... to underscore watermarks, paper texture & manufacturing process...

- Secondary electron radiography (as a substitute of beta radiography)

- Neutron radiography

- Electron emission radiography

- Neutron induced autoradiography

- Computed assisted tomography

- Synchrotron radiation based radiographic or tomographic methods
Schematic diagram of a radiographic exposure. The darker region of the film represents the more penetrable part of the object; the lighter region, the more opaque.
Experimental relative intensity vs. energy (keV) distribution for 100, 200, 300 & 400 kV X-rays generators with tungsten anticathode.
Schematic drawing of a storage-phosphor imaging plate.
The image generation is separated into two steps. First, the image plate (IP) is exposed to the X-ray energy, part of which is stored within the detective layer of the plate. Second, the imaging plate is scanned with a laser beam, so that the stored energy is set free and light is emitted. An array of photomultipliers collects the light, which is converted into electrical charges by an analog-to-digital (A/D) converter.
Schematic drawing of an amorphous silicon–based digital radiography detector.

The X-ray energy is converted into visible light in a scintillator layer. The emitted light is then converted into electrical charges by an array of silicon-based photodiodes and read out by a TFT array.
3 - Questions to be solved by radiography. Some examples

For certain categories of objects and/or materials, one has to solve different problems.

A - PAPER, SUPPORT OF DRAWING OR TEXT

Visualisation of the texture of the paper, of local variation of mass per unit area, of the watermarks, whatever can be the drawings or the texts, manuscript or printed, on it.
Solution: beta radiography or radiography with secondary electrons.

B – EASEL PAINTINGS

The classical structure of an easel painting is a multilayer one:
- plane support: canvas (linen, hemp, cotton), wood (panel of different species, cut according to different manners), metal (copper), stone (obsidian)
- preparation layer (chalk, gypsum (or gesso), lead white)
- animal glue
- eventually underdrawing (carbon black)
- paint layer: adjacent and/or superimposed spots of organic or mineral pigments suspended in organic media
- varnish.
One wants to put in evidence the texture of the backing and of the paint layer, the characteristic "touche" of the author, the eventual existence of an underlying painting, the pentimenti, the alterations, the restorations, the modifications of frame, the linings, the transpositions...
Solution: low energy X-ray radiography and/or electron emission radiography
C – ENAMELS

Glassy layer loaded with mineral pigments, on a metallic backing (generally copper alloy). One wants to determine contrasts in the composition in the enamel layer, its alterations…
Solution: X-ray radiography, sometimes at different energy (selective filtration), and electron emission radiography.

D - WOOD

The goal is to visualise the characteristic texture of the wood species, the assembly techniques, the alterations (flaws, brittleness areas), the infestations, the restorations, the eventual metallic inserts…
Solution: X-ray radiography

E – STONE

One wants to determine the texture of the stone material, its homogeneity, the eventual cleavage planes, the eventual repairs or previous restorations and eventual metallic inserts…
Solution: high energy X-ray radiography or gamma radiography.
F – FOUNDRY (METAL)

- Determination of the internal structure of the object: hollow / fill, thickness & steadiness of the walls, homogeneity / heterogeneity of the constitutive materials, presence of structural defects (porosities, shrink holes, inclusions, cracks, slag, bubbles, voids, delaminations, corroded or altered zones…)
- assembling features, nails, screws, welding, brazing, armatures, core pins…
- repairs: inserts, secondary casting, screws…
- presence of eventual previous restorations.
Solution: high energy X-ray radiography, gamma radiography or radiography with an accelerator.

FOR ANY TYPE OF ARTEFACTS AND/OR ANY MATERIALS

This may also give pertinent information regarding the conservation state & arguments for eventual consolidation and / restoration procedures.
Some examples of classical X-ray radiography taken among various collections: easel paintings, manuscripts, bronze statues, stones statues, cult objects, archaeological objects, industrial heritage...
Complete set of examination of a wax painting – Ptolemaic Egypt (320 – 30 BC) - Louvre Museum (C2RMF)

<table>
<thead>
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<th>UV fluorescence</th>
<th>Infrared</th>
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<td>Electron emission radiography (negative)</td>
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<tr>
<td>X-ray radiography</td>
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<tr>
<td>Electron emission radiography (regular)</td>
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</tbody>
</table>
Colour, visible light
False colour, ultraviolet fluorescence
X-ray radiography
Electron emission radiography, left positive, right negative
La Belle Ferronnière, Léonard de Vinci, Musée du Louvre
Mikhail Larionov, *Small Jewish shop at the Vitebsk market*, Centre Georges Pompidou (Paris), colour visible light photography, colour ultraviolet fluorescence, raking light photography, X-ray radiography (C2RMF)

NB: one of the first example of the use of agglomerate wood panel as a substrate for easel painting
The radiograph discloses the join between the head & the trunk. It is constituted of a welding bead punctuated by a sequence of basins shaping a necklace.
Dimension H = 192 cm

X-ray radiography 420 kV
High definition radiographic film
+ copper filtration
+ lead anti backscattering protection

58 exposures
DSD = 3 m
**Hawtar’athat**, bronze sculpture, H = 140 cm, South Arabian period, Nashqum (presently Al-Bayda), Yemen, end of VII\(^{th}\)-VI\(^{th}\) Cent. BC, lead bronze (primary castings: \([Sn] = 6\) to 12\% , \([Pb] = 7\) to 17\% / secondary castings: \([Sn] = 7\% , [Pb] = 12\%) + lead (eyes), Sanaa National Museum
Joining techniques - large variety

Examples: Khmer techniques – Musée Guimet (Paris)

Ganesa, Bayon, 26 cm

Lokesvara, Bayon, 42 cm

Male Divinity, Angkor Vat, 63 cm
X-ray radiography (before restoration) of the Blériot XI which made the first flight across the Channel in 1909
Musée des Arts & Métiers - Paris
X-ray radiography of the wing & the fin of the Blériot XI
T. Borel (C2RMF)
Radiography with other detectors than films
Moche style whistling ceramic pot in the form of a parrot, Northern Peru, between 100 BC & AD 700, British Museum London. Xeroradiograph revealing details of construction, including the hollow whistling mechanism located within the head of the bird
Example of radiography of a bronze statue (Metropolitan Museum New-York) with (a) film – 320 kV, (b) with a-Si Vidisco digital detector, 230 kV
Some examples of gamma radiography
Gamma radiography (iridium 192 - Kodak Définix film) of the Gold mask of Toutankhamon, at the occasion of the exhibition «Toutankhamon et son temps» - Petit Palais Paris (1967) - © SGS - Qualitest
Venus Genitrix (or Aphrodite) – Roman marble replica (1st Century AD) of a bronze Greek original (5th Century BC) - Louvre Museum
Examination before restoration
B. Bourgeois (C2RMF – Versailles),
B. Rattoni (CEA – Saclay)
& D. Bagault (C2RMF – Paris)
Gamma radiograph showing previous metallic restoration armatures
One example of radiography with an accelerator
Apollon du Belvédère, copy of the Primatice original by Keller (1695), h = 215 cm
Château de Fontainebleau
Radiograph with a MINAC 6 MV accelerator (© EDF-Valectra)
Beta radiography & secondary electron electron radiography
The three techniques: soft X-ray radiography, secondary electron radiography & electron-emission radiography can be compared using a postage stamp as a specimen (Bavarian stamp of 1920)

A - Photograph. The design is green and the "Deutsches Reich" overprint is black.
B - Soft X-ray radiograph. Details of both design and paper are visible. Design is "negative," indicating absorption of the X-rays by the mineral ink.
C - Secondary electron radiograph. Only the details of the paper structure including the "wavy line" watermarks are shown.
D - Electron emission radiograph. The design is "positive" indicating that the green ink has a high electron emission and hence that it contains some heavy elements which has a relatively high electron emission. The overprint (an organic black ink) cannot be seen.
Examples of beta radiographs of manuscripts showing various watermarks using a plane sheet source of $^{14}$C radiolabelled plastics (poly-methylmethacrylate). ($E_\beta \text{ max} = 156 \text{ keV} \ ; \ T = 5730 \text{ y}$) & a fast monolayer film. This permits to accurately determine the paper structure and specially its watermark.
Watermarks of British stamps from Queen Victoria to Queen Elizabeth II

Photography & beta radiography – A. Lemonnier (CEA – Saclay)
Filippo Baldinucci (1625-1696)
Portrait of a man

Beta radiography of a XVII\textsuperscript{th} C. Italian drawing of the Louvre Museum collection
A. de la Chapelle, A. Le Prat
Neutron radiography
Variation of mass attenuation coefficients for X-rays (100 & 250 keV) & thermal neutrons vs atomic number.
1 - Block excavation done in Kanton Zug (CH) prepared for radiographic examination.
2 – Neutron radiograph showing bones & leather (PSI – Villigen - CH)
3 – X-ray radiograph showing sword, knife & metallic decoration
Electron emission radiography
Schematic diagram of the technique for making electron emission radiographs. More electrons are emitted from the areas of the specimen’s surface that contain materials of high atomic numbers.
Electron emission radiography - Variation of the film density according to the atomic number & the mass per unit area of the superficial material
Electron emission radiography - Case of enamels on copper alloy substrate - Bishop’s cross, champlevé enamel, Limousin, Middle Age, Louvre Museum. Visualisation of surface micro-cracks & bubble areas in the vitreous layer.
Electron emission radiography - Cult object, Byzantine art, cloisonné enamel, Cluny Museum
Autoradiography
Application of autoradiography to paintings

After irradiation, the neutron-induced radioactivity decays with time. About a dozen different light and heavy isotopes – emitting $\beta$- (electrons) and $\gamma$-radiation – are created (the most important isotopes and their half-lives are presented in table 1). The induced $\beta$-decay is used to blacken highly sensitive films or imaging plates to reveal the spatial distribution of the pigments. It is a big advantage of neutrons that different pigments can be represented on separate films. This is due to a contrast variation created by the differences in the half-life times of the isotopes.

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<th>Isotope</th>
<th>Half life</th>
<th>Pigment</th>
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<tr>
<td>$^{56}$Mn</td>
<td>2.6 h</td>
<td>Brown colours, Umber, Ocre</td>
</tr>
<tr>
<td>$^{64}$Cu</td>
<td>13 h</td>
<td>Azurite, Malachite</td>
</tr>
<tr>
<td>$^{76}$As</td>
<td>1.1 d</td>
<td>Smalt, Realgar, Auripigment</td>
</tr>
<tr>
<td>$^{122}$Sb</td>
<td>2.7 d</td>
<td>Naples-Yellow</td>
</tr>
<tr>
<td>$^{124}$Sb</td>
<td>60 d</td>
<td></td>
</tr>
<tr>
<td>$^{32}$P</td>
<td>14 d</td>
<td>Bone-black</td>
</tr>
<tr>
<td>$^{203}$Hg</td>
<td>47 d</td>
<td>Vermilion</td>
</tr>
<tr>
<td>$^{60}$Co</td>
<td>5.3 a</td>
<td>Smalt</td>
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</tbody>
</table>
Autoradiographs from «Girl with a platter of fruits» (c. 1555) by Tiziano Vecellio, called Tizian – Gemälde Galerie (Berlin)
C. Laurenze-Landsberg, C. Schmidt, C.O. Fischer, B. Schröder-Smeibidl
BENSC Report – Berlin (2001)
Fig. 3: 2nd neutron autoradiography, composed of 12 imaging plates, exposure 1d 7h to 1d 11h after activation.

Figure 4: 2nd autoradiograph: turned by 180°
Principle of « unrolling » radiography

The object is driven by the moving of the film. So, the image is sharp for the surface details in close contact with the film, the X-ray tube, being at a short distance from the object, gives a fuzzy image of its opposite side from the film, streaming inversely of the rotation direction.
Inkpot of Vaison-la-Romaine, cylindrical, cast and inlayed - Dimensions: \( H = D = 4.4 \text{ cm} \), Roman Empire, 1st Cent. AD, Cu-Zn alloy, Cu, Ag, Au & black bronze inlays

Louvre Museum
Silver  
Copper  
Black bronze & gold

Inkpot from Vaison-la-Romaine, details of the inlays

Modern simulation of the original colors
Computer assisted tomography (or tomodensimetry)
Principle schemes of
a: 2D tomography (linear array detector)
b: 3D cone-beam tomography (2D pixelised flat detector)

Ref.: M. Simon & C. Sauerwein, Quality control of light metal castings by 3D computed tomography,
http://www.ndt.net/article/wcndt00/papers/idn730/idn730.htm
Arm of a Roman Emperor from Essegney (Vosges), bronze sculpture, L = 44 cm, Gallo-Roman, 1st – 3rd Cent. AD, lead bronze (primary castings: [Sn] = 11 %, [Pb] = 6 % / secondary castings: [Sn] = 11 %, [Pb] =: 4 %), Epinal Museum

- The tomography shows the assembly joint of the wax semi-shells & the wax drops in the hands
- The software permits the at will choice of the cross section adapted to the looked for information
- Visualisation of non accessible areas by direct observation, including endoscopy: sedimentation)
The *Leopards weight* from Shahi Tump

The artefact was discovered in a grave, in the Kech Valley, in Balochistan, southern part of Pakistan.

It belongs to the Shahi Tump – Makran civilisation (end of 4th millennium – beginning of 3rd millennium BC). Height: 200 mm; weight: 13.5 kg. The shell \((e = 3 \text{ mm})\) has been manufactured by lost-wax foundry of a copper alloy \((12.6 \% \text{ Pb}, 2.6 \% \text{ As})\), then it has been filled up with lead \((99.5 \%)\) foundry. The shell is engraved with figures of leopards hunting wild goats, made of polished fragments of shellfishes.

No identification of the artefact’s use has been given
The *Leopards weight* from Shahi Tump - Photography
The *Leopards weight* from Shahi Tump – Cross section by tomography with 8 MV accelerator (© CEA Grenoble)
Microradiography, microtomography
Figure 1 – Cross section of a sample of Quercus caducifolia impregnated with a mixture of PEG 1500 at 20% in PPG 425, obtained by μ-CT
Figure 3 - Cross section of a sample of Quercus caducifolia impregnated with a mixture of PEG 1500 al 20% in PPG 425, obtained by SR-CT.

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9th International conference on NDT of Art, Jerusalem Israel, May 25-30 2008
Figure 4 – Three dimensional rendering of a sample of *Quercus caducifolia* impregnated with isoeugenol polymerized, obtained by SR-CT.
Conclusion

I hope that this presentation has shown that radiography offers a relatively large & flexible palette of techniques.

If this palette is used by a competent & skilled technician, & if there is a real dialogue between himself & the curator, or the conservator-restorer, or the art historian, or the historian of techniques, or the archaeologist, or the metallurgist…, it can provide many pertinent elements, contributing to a deeper knowledge of the cultural heritage artefact.
Thank you for your attention