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Scientific Methods Applied to the Study of Art Objects

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Abstract. Scientific methods applied to the study of works of art are varied (examination, analysis and dating) and must be chosen with care in accordance with the object of the research. This paper briefly sets out the state of the art highlighting the development and applications of non-destructive methods. Such methods come under either the use of electromagnetic or particle radiation for the formation of images based on shape, development technique, or, on the other hand, the quantitative determination of elements down to and including their traces, isotopes or microsurfaces for the purpose of identifying the matter, its date, origin and development.

Key words: works of art, examination methods, atomic spectroscopy, nuclear spectroscopy, image processing.

Today, the application of scientific methods to the study and conservation of works of art is no longer merely a matter of interdisciplinary curiosity but rather a genuinely scientific discipline in itself adopted by numerous museum laboratories, institutes specialising in each material as well as state and private research laboratories active in areas as diverse as nuclear techniques [1, 2], non-destructive examination [3, 4], microbiology, the manufacture of new products (pigments, adhesives, strengtheners, etc.) and the study of their durability. These researchers' aims are not only to improve the accuracy of their knowledge of the artistic heritage, but also to ensure its conservation i.e. its intelligibility and preservation.

Indeed, all materials, both organic and inorganic, change and are permanently attacked and degraded by many different factors including the environment (light, humidity, temperature, pollution, vibration, etc.), nature (floods, earthquakes), accidents (fire), and man himself (handling, transporting, theft, the general public).

The multiplicity of problems, approaches, materials, and technical and scientific means makes it necessary to narrow the subject down to a few applications arising from current technological trends. A museum laboratory's assignments can be broken down into five aims:

— Research into ancient techniques: their dating, origin and workmanship. Such research is reported on in publications and specialised conferences under the heading "archaeometry", which includes aerial and land prospecting of archaeological sites.

— Enlarging collections: problems of authentication, attribution and state of a work that determine its evaluation.
— Conservation-restoration: drawing up a work's medical file by a laboratory
— to distinguish the original parts from later additions;
— to determine changes in appearance and in physical properties due to ageing processes;
— to discover the causes of deterioration and the mechanisms involved prior to the restorer's diagnosis and action.
— Preservation: study of conditions conducive to the durability of collections both in permanent and temporary exhibition halls and in reserve collections.
— Training and communication: imparting by researchers of their acquired knowledge of art works to students, restorers and curators, as well as exhibitions showing the latest results of research, the most suitable way to inform the general public.

Generally speaking, museums may be differentiated from each other by type of collection: there are fine art museums and archaeology museums. Laboratories are thus specialised according to the museums to which they belong. Our research institute, *le Laboratoire de Recherche des Musées de France*, has the advantage of being at the service of 1200 museums in France. Hence the need to have a range of scientific equipment of working on all categories of art objects in collaboration with curators, art historians and restorers.

The means available can be classified in three groups: methods of examination, analysis and dating [5]:

— *Methods of examination* are based on the recording of images from different spectral zones (visible, ultra-violet, infra-red, X-rays, beta, gamma, electrons, etc.) in adequate experimental conditions for revealing information invisible to the naked eye.
— *Methods of analysis* and of microanalysis (atomic or nuclear, isotopic, vibrational and structural, surface or bulk, panoramic or sequential, directly on the work itself or on a sample, destructive or non-destructive). These very different and often complementary techniques sometimes call for major investments, making it necessary to work with teams equipped with equipment whose development is extremely costly. Few techniques make it possible to work directly on works of art. Museum laboratories have to try to adapt them to their requirements while taking into account the constraints due to the need to respect the integrity of such objects [6, p. 1]. Transfers of technology are indispensable, as the works are not supposed to leave the museum except for exhibitions.
— *Absolute dating methods* are, to our knowledge, all destructive (except dendrochronology). There are four groups of such methods [5, p. 151]:
(a) those based on the material's internal radioactivity (carbon-14, potassium-argon, disequilibrium of the uranium group) or on the side-effects of such radioactivity (thermoluminescence, fission traces, paramagnetic electronic resonance);
(b) those based on surface alteration (obsidian hydration), the substitution in bones of chemical elements (uranium, nitrogen and fluorine), as well as isomorphic changes (racemization of amino acids);
(c) those based on the direction (paleomagnetism) or on the orientation of the earth's magnetic field (thermoremanence);
(d) finally, the size of tree age-rings (dendrochronology) relative to seasonal cycles. Absolute dating methods have to be combined with *relative chronology methods* based on the periods of use of certain materials with specific compositions (rock, metal, glass or ceramic) and on object shaping techniques characteristic of certain periods (melting, assembling or decorating). Examples abound and the subject is well documented.

We shall briefly present a few of these methods in their order of importance for museum laboratories, including techniques for working directly on works of art (known as "non-destructive"), those that use samples but without consuming them (also known as "non-destructive"), and, finally, those that destroy samples.

The aim being to present the state of the art, the examples mentioned will not be elaborated on, as they have been published. We shall give the reader some useful references. We particularly wish to draw your attention to the existence of a biennial publication that summarizes 3000 articles on our discipline: "The Art and Archaeology Technical Abstracts" [7].

The catalogue of the exhibition organized in 1987 by Mr. J. Riederer, Director of the Rathgen Forschungslabor in Berlin also contains a detailed thematic bibliography of books and specialized periodicals [8].

Techniques for Working Directly on a Work of Art

Examination Methods

To begin with, there is examination by reflected white light or such light being transmitted through the work [3, p. 114], an overall or partial view more or less enlarged depending on the use of a binocular magnifying glass or of a camera alone. In this latter case [9, pp. I/6.1 and I/5.1], the five to fifteen degree declination relative to the painting's plane reveals by means of shadows: the relief of the network of craquelures due to the varnish, the paint layer, the primer, swellings (scales, blisters), touch-ups or thickenings (e.g. Van Gogh), but also distortions of the support (split wood, canvas seams) and preparatory incisions (e.g. Raphael's preparation of the composition).

Ultra-violet lighting of an object causes the emission of fluorescence in the visible spectrum of its surface elements. The emission's heterogeneity reveals changes that often arise from occasional restorations.

Examination in the *near infra-red* spectrum requires sensitive film with wavelengths of up to a μm and CCD cameras with a sensitivity of up to 1.1 μm or cameras with a videcom (PbS-PbO) tube sensitive up to 2 μm as well as the interposition in front of the lens of a filter that absorbs the visible spectrum [10, p. 79]. Reflected infra-red radiation, which is less absorbed than the visible spectrum by the object's surface, can, under favourable conditions, reveal unreadable inscriptions or paintings masked by natural patina [11, p. 84-1-75] or even by underlying drawings applied to the white primer covered by the paint layer and the varnish [11, p. 84-1-38]. The infra-red rays are reflected by the surface of the primer and absorbed by the black line or pounced drawing (using a pounce) which can then be compared with the preparatory studies. The shadow technique, particularly when used by the primitive school of art, made up of parallel or crossed lines, the use of a pounce, of ink, of charcoal or lead pencils, the presence of squares, the identification of inscriptions that make it possible to locate colours are all new elements for analysing artists' picture techniques.

Holographic interferometry has been used to show up, on a painted wooden panel, the parts that are sensitive to climatic variations [9, p. I/14.3], as well as the distortions under stress of a metal horse at the Capitole museum [9, p. I/20.1].

Radiography (X-ray photography) produces more information as the X-rays penetrate the object [3, p. 170; 11, pp. 84-1-65, 84-1-69]. The radiographic image of

a painting reveals, for instance, the craquelure network, the opaque sketch that has been done using lead white and the reserved areas (outlines, shadows) but also the state of the support (weakened wood panelling, canvas texture, canvas seams), the presence of gauze immersed in the primer by some members of the primitive school, as well as changes made by the artist (modifications, second thoughts) or by others at a later date (reduction or increase in the size of the painting). Even more surprising is the showing up of dual composition i.e. the presence of two superimposed layers of paint on the same support (a canvas reused by the same artist or 19th century paintings improperly painted over in the 20th century). Finally, there also appear in-depth restorations (mastic) which hardly absorb rays at all.

When a painting is reinforced by parquetry, or when a polyptych panel is painted on both sides, it is possible to reduce the ray absorption of the awkward area by means of *stratiradiography*. The painting is placed horizontally on a support. The X-ray tube moves under the support on a semi-circular cradle. The naked film is applied to the picture surface whose definition is thereby enhanced.

For three-dimensional (3D) objects, such as metal (and thus non-absorbent) statues or statuettes, high-voltage (420 kV) *radioscopy* is used [11, p. 84-1-58]. Direct vision on a monitor screen of the X-ray image permits the complete examination of the object by remote control and the recording of a radiographic snapshot as soon as a favorable angle has been detected revealing, for instance: the assembly technique (soldering, rivetting) or the melting technique (lost wax with or without a reinforced nucleus, the location of spout holes or the nucleus fixing nails, as well as casting defects (air bubbles, thinning of surfaces) or the lack of homogeneity of alloys (lead nodules in bronze objects), traces of hammering, straw in terracotta, and the structure of wax objects [11, p. 84-1-58]. Finally, damage to metal resulting from more or less deep corrosion and sometimes ancient, even original, restorations are located without harming the object. *Endoscopy* is a useful complementary aid for interpreting photos of hollow objects [3, p. 182] as is *stereoradiography*, which permits a view in relief of the inside of an object.

Xeroradiography is perfectly suited to ceramics (china and terracotta) whereas *betagraphy* is used for studying texture and paper watermarks [3, p. 34].

Gammagraphy is indispensable only for the exceptional cases of highly absorbent objects such as statuary, and *neutrography* for showing low-density compounds (organic matter) in a matrix with a low neutron capture cross-section.

When a picture has been painted on copper or stone, the support's opacity masks that of the paint layer. So *emissioradiography* is used [10, p. 53]. High-energy X-rays (>300 kV) are filtered by several millimeters of copper [12, p. 75]. The film (preferably single-layer) is applied to the paint layer facing the incidental radiation. This only slightly shows up but stimulates the paint layer which emits fluorescent radiation and electrons arising from its chemical composition. So we were able to detect the incised and gilt preparatory drawing of the Plantagenet enamel which is kept at the Tessé museum in Le Mans [11, p. 84-1-54].

Other interesting techniques are *tomography* [3, p. 61, 69; 11, p. 84-1-77] and the *scanner* for studying hollow and closed objects whose contents have had to be identified, such as ceramic particles, the first elements of accounting found in Mesopotamia in the IVth millenium [11, p. 84-1-62]. This technique has also been used to monitor the impregnation of wooden frames by resin [12, p. 99].

Finally, *autoradiography* [3, pp. 87, 95; 13, p. 3] as applied in Boston and Berlin mainly to easel paintings permits the obtention of a chemical map of the paint layer. The painting, which is protected by a plastic coating is irradiated by a flow of neutrons in a nuclear reactor, and then exposed by contact to an assembly of radiographic films covering the whole of the work. This is repeated several times so that the images obtained can reveal the overall distribution of the pigments used, depending on the period of the radioelements present. As lead is not sensitive to neutrons, its almost systematic presence will not adversely affect the images obtained, which are complementary to the radiographic images of the paintings on which white lead is often the most absorbent element.

"Elementary" Spectroscopic Analysis

As X-rays do not destroy inert matter, we first of all concentrated on developing elementary analysis equipment specifically designed for works of art: "*micro-fluorescence X*" [13, p. 44; 14]. A wavelength dispersive apparatus acquired in 1972 was adapted to a goniometer that permitted open air work on objects placed vertically. The beam of primary X-rays excites the surface of the object which emits X-ray fluorescence radiation; collimated by linear slits, this radiation is dispersed by a crystal analyser (LiF), and then detected by a mobile scintillation counter (NaI). So we obtain the chemical composition of the object's surface (in the case of easel paintings, the stimulation reaches the primer) by working on surface areas of between 30 and 0.1 mm^2.

In 1975, our acquisition of an energy-dispersive system, which we connected up to a microcomputer, enabled us to reduce significantly the time required for analysis (from 40 to 2 min). However, the energy resolution of the solid-state detector (Si-Li) is much less than that of the wavelength dispersive system. In 1978, the following stage consisted of developing a *cross-section analyser* for analysing from time to time, with the same equipment, polished micro-samples centred under microscopes using micrometric screws situated on the sample support. This equipment very quickly replaced the microchemical analysis of metallic elements in painting sections, which is often difficult to carry out. As the analysis did not require any metallization, the state of the sample's surface remained unchanged.

The *particle accelerator* (synchrotron or Van de Graff radiation) grew out of these laboratory techniques whose sensitivity was limited, especially for micro-analysis [6; 11, p. 84-1-34; 9, pp. II/8.1, II/11.1]. As early as in the eighties, a Van de Graaff mini-accelerator of the 2 MeV tandem type was acquired. AGLAE (the Grand Louvre's Elementary Analysis Accelerator) built by Electrostatics International Corporation USA (Pelletron 6 SDH-2 model) was installed in the basement of the Louvre museum in 1987. Today, this apparatus has made it possible to carry out studies in PIXE, PIGE and RBS modes, either in the open air using an extracted beam [13, pp. 18, 51; 15, p. 120], or in a vacuum on conditioned samples. In the years to come, the development of a nuclear microprobe is planned with the same benefits as a scanner electronic microscope, but even more sensitive. In the more distant future, a new beam line for dating C-14 by mass spectrometry will also be installed. Such equipment will obviously improve the quality of current research. So the conservation of samples is of great importance as it is often impossible to replace a sample obtained when a work is being restored. Sample banks have an

essential role to play in future research. This is why the spectrometric methods used in archaeometry, which generally destroy the samples taken from an abundance of shards, are to be banned from the study of museum objects.

The development of microanalytical methods of surface analysis has permitted a greater understanding of the heterogeneity of the composition of materials. Thus, thanks to the *scanning electron microscope* connected to a wavelength dispersive analyser, it is possible to quantify inclusion grains in metals, glass and ceramics characteristic of their development, to study accurately changes in a corrosion product's composition so as to explain how it was formed. It has also been possible to identify the types of assembly techniques (soldering, brazing and copper salts) used in antiquity for gold jewellery [16] and to prove that copper salt soldering was already practised in Iran as early as in the 13th century i.e. before the Etruscans. Another study of Etruscan jewellery kept by the Berlin Archaeological museum led to the discovery of fakes made by Roman goldsmiths in the second half of the 19th century [9, p. II/5.1]. The scanning electron microscope revealed obvious differences in the techniques used.

Proton activation is particularly suited to the analysis of museum objects as the energy of highly accelerated particles (>7 MeV) makes them penetrate deeply into the matter thereby reducing the causes of errors due to contamination of the surface of archaeological objects (gold, glass, bronze) [6, p. 133]. It is possible to reduce the lack of surface homogeneity by using a *source of Americium 241* whose 60 KeV radiation is more penetrating than rays from an X-ray tube.

The use of a *laser* beam as an excitation source for a *UV-visible spectrometer* also permits the specific evaporation by a thermic effect of a microquantity of matter on an object's surface. However, this method is little used [11, p. 84-1-27; 17, p. 157, 189].

Reflectance spectrophotometry [3, p. 120] with external integration sphere permits on-the-spot measurement of the reflectance spectrum in the visible and infrared spectra and thus identification of certain modern synthetic organic pigments [18]. As the primary spectra of pigments of the same hue are mostly very similar, the identification has been done by higher order derivative spectrophotometry. This method also permits the confirmation and measurement of differences observed on a work by means of examination methods. For example, it permitted the rejection of a few ink additions to drawings by Rembrandt [19] or the monitoring of varnish removal in the course of a restoration.

Photoacoustic and photothermic spectrometry are of interest as they permit not only the open air study of opaque and diffusing bodies, but also their in-depth analysis. Hence the measurement of the thickness of superficial layers [10, p. 234; 11, p. 84-1-79]. The Raman microprobe permitted, for example, the identification of pigments by specific work on illuminations. However, the energy specifically dissipated in absorbent materials can affect fragile compounds.

Radiocrystallography also permits on the spot identification of chemical compounds. Rigaku has developed a diffractometer specially designed for working directly on objects with a flat surface [12, pp. 55, 59].

Techniques Applicable to Samples

In addition to traditional X-ray diffraction techniques [12, p. 37; 13, pp. 55, 99; 15, p. 182; 17, p. 217], the *Gandolfi chamber*, used mainly by museum laboratories,

permits precision work on microsamples. The sample is positioned on a mobile support inclined at 45° with two rotation axes [20]. Such dual rotation makes it possible to orientate the sample in all directions resulting in a Debye-Scherrer diagram with thin unbroken stripes. However, the exposure time generally amounts to 20 h.

The *scanning electron microscopy* can refine the microscopic studies of metallographic sections, thin slices of ceramic or stone, stratigraphic sections of picture material [13, p. 79] not only by local analysis, but also by mapping the distribution of chemical elements [15, pp. 34, 45]. It has thus been possible to identify two crystallographic forms of the same pigment, lead-tin yellow, on stratigraphic sections on the basis of the presence of silicon systematically combined with one of them. This observation has made it possible to reveal the existence of quite a short transition period in the middle of the 15th century separating the use of one type of pigment from the other of the same nature [9, p. II/12.1; 11, pp. 84-1-22, 84-1-44].

Similarly, the *Raman microprobe* has permitted the identification on microsamples of 20th century paint of two types of titanium white (anatase and rutile shapes) and the discovery of the dates on which they were first used [9, p. II/6.1; 12, p. 25]. Such identification is also possible by scanning electron microscopy using *cathodoluminescence.*

The *transmission electron microscope* combines elementary and structural analysis on microsamples. Its use requires ultra-thin microtome-prepared sections with a thickness of less than a micrometer [9, p. II/15.1; 12, p. 79; 21]. This technique was used for a refined but local study of the picture matter of Edouard Manet's "La serveuse de Bocks" and its comparison with that of other paintings by the same artist, revealing slight differences of composition.

UV fluorescence spectrometry has made it possible to analyse the media (egg, oil, varnish) on micro areas of each paint layer of a stratigraphic cross-section [15, p. 168]. The *FTIR microscope* has been applied to metallic objects to identify coatings on an Edo period Japanese palanquin and lacquers. In the first case, shellac was identified, and, in the other, a urushi resin which is insoluble and thus difficult to identify [9, p. I/30.1].

Analysis of non-metallic seals by *C-13 Fourier transform nuclear magnetic resonance* has revealed [13, p. 62; 17, p. 201] that all seals of the Middle Ages are made of bee wax and have not suffered any chemical deterioration whereas modern seals (since the 18th century) contain wax and resin (mainly rosin) [12, p. 87].

For the study of the origins of objects, neutron activation plays a major part. Fine identification of the composition of the materials used (terracotta, glass, bronze, silver, etc.) including the trace elements has permitted the obtention of "fingerprints" corresponding to each origin of clay or minerals. For certain elements, its great detection power of $< 1\ \mu g/g$ makes it possible to determine on powder solid about thirty chemical elements while significantly reducing the preparation of samples [15, p. 139].

Techniques That Destroy Samples

In addition to traditional atomic spectroscopy techniques, to which we shall not refer, two techniques are worth mentioning for museological purposes.

Mass spectrometry is mainly used to measure the isotopic ratios of lead, carbon, oxygen and sulphur (see [1]). The isotopic ratios of these elements vary according to the geographical origins of the minerals. Lead is used mainly to differentiate bronze, pigments and types of glass, whereas carbon and oxygen isotopes are looked for in Greek, Italian and Turkish marble; sulphur permits the differentiation of lapis-lazuli from Afghanistan and Chile [1, p. 97] and Almaden cinnabar mined in Antiquity [10, p. 252]. White lead also deserves a mention: the value of its 206/204 isotope ratio is stable until the beginning of the 19th century in line with European reserves, and then diminishes sharply, most probably owing to contamination by lead from sources outside Europe.

(High-pressure) liquid chromatography techniques and techniques in gaseous phase with pyrolysis equipment combined with the mass spectrometer are mainly used to identify organic compounds:

— on the one hand, amino acids in leather, textile dyes and certain media (egg, glue, and gums) [13, pp. 9, 95; 15, p. 212];
— on the other hand, fatty media and modern synthetic paints [15, p. 41; 22].

Thermal techniques also permit identification of organic media and complement traditional microchemical tests carried out on thin sections [13, p. 57].

All these methods produce numerous and diverse results. Indeed, the inflation in the quantity of information available now poses problems of data management and exploitation. Computers and statistical methods have become indispensable to the management and processing of this great amount of data so as to define rules whose application would be managed by expert and computer-aided decision-making systems.

Image Processing and Analysis

For more than half a century, European museums have been building up a truly unique collection of documents including 1.5 million photographs and X-ray photographs, mainly of easel paintings. This collection has been growing at the rate of around 100000 images per year. To ensure the durability of the collection, to facilitate its management and to renew its exploitation by means of image processing and analysis, several museum laboratories have joined forces within the European Community (DG XIII IMPACT) to set up by 1991 a very high definition digital image data-bank within the framework of the NARCISSE project. The data inputting, compression and storage equipment needs are being defined to ensure reading and communication compatibility between document data bases of increasing economic, scientific and cultural importance. Very high definition data inputting implies the use of high resolution CCD (charge coupled device) scanners permitting the capture, correcting, collating, compressing and storing of digitized documents [23]. These documents meet the needs of art professionals or institutions for the purposes of research, teaching and dissemination of European culture. The fragility and uniqueness of the documents and the methods of access to them are not suited for the increasing demand. Stored in pouches or envelopes, chronologically inventoried by painting and by category, it is difficult to gain access to these documents, particularly in the large photo libraries, which are also the most visited ones. Permanent access to these documents, which are dispersed all over Europe, will be

a major asset for research into the history of art. Very high definition colour computer inputting is being carried out within the framework of another European project (DG XIII Esprit II) called VASARI. New image processing and analysis techniques will lead to considerable improvements in the reading of scientific images. Applications developed for X-ray shots and photos of paintings already permit:

— automatic mapping of digitized images;
— improved contrast or easier reading [15, p. 131];
— toning down of the support image in relation to the paint layer, be it wood, canvas or parquetry;
— artificial reintegration of a painting's gaps prior to its restoration (to solve the problem of colour matching when touching up paintings) [15, pp. 110, p. 116];
— improvement in the image to be found underneath where there is an artist's change of mind by comparing the radiographic image with the visible image.

Historically the first technical approach of works of art, scientific images will undoubtedly enjoy renewed favour and will tend to become a new way of assessing artists' styles using digitized images.

Conclusion

Thus, once again, science is bringing method and matter together. The artist's gestures, his instinctive or controlled touch, his pictorial writing are being analyzed in the visible spectrum on images obtained in spectral ranges he could not perceive or from a digitized number of colour hues he could not possibly distinguish.

Similarly, the matter he methodically worked on is being analysed with such finesse that the correlations brought to light between the physical and chemical data—which permit the identification of origin, date, development technique—greatly surpass the know-how of craftsmen such as potters and glass or metalwork artists, but make it possible to identify their production and, more generally, to reveal the evolution of techniques from antiquity to the present day.

Matter ages, so, it being impossible to safeguard everything, it is urgent to extract and preserve the knowledge contained in objects that bear testimony to the history of humanity.

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