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Examination of Paintings by Physical and Chemical Methods

Ian N.M. Wainwright

Abstract: *Communication between scientist, curator, and conservator can be hampered by the specialized language used to describe analytical instruments and procedures. As a contribution to overcoming this barrier, synopses are presented of chemical and physical methods employed in the examination of paintings and other works of art. The aim is to clarify the terminology, briefly describe each method, and indicate areas of application. Methods described include infrared, ultraviolet and fluorescence photography, infrared reflectography, x-radiography and electron emission radiography, light and scanning electron microscopy, x-ray spectrometry including x-ray secondary-emission (fluorescence) spectrometry and x-ray microanalysis, x-ray diffraction and Fourier transform infrared spectroscopy. Sampling and cross-sections are discussed. A bibliography of related references is appended.*

Introduction

Physicists and chemists use a specialized terminology to exchange information. When the subject is the analysis of art and archaeological materials, this can present, quite unintentionally, a barrier to effective communication with the curator, art historian, archaeologist, or conservator with whom the scientist is working. There are a number of reasons for this. The fields of conservation science and archaeometry¹ encompass a great number of scientific disciplines, each of which has its own terminology. No one can be expected to stay abreast of them all, and yet new developments, in such areas as remote sensing, computers, and instrumental analytical methods, continue to find new applications in the study of art and archaeology. Numerous methods are currently applied in archaeometry and they are typically quite complex and employ sophisticated instrumentation.

A second reason for this difficulty in communication is that the terminology used to describe methods, instrumentation, and techniques for the analysis of artifacts and works of art can be confusing or inconsistent. This is compounded by the frequent use of acronyms and other shorthands. From a physical and chemical perspective, the analysis of artifacts consists of studying the interaction of electromagnetic radiation—light, x-rays, infrared and ultraviolet radiation—and sub-atomic particles with artifacts or their constituents. These interactions can be studied on a macroscopic, microscopic, molecular, or atomic scale, giving rise to a number of interrelated methods of analysis which have very similar names but which produce quite different data or images and explore quite different questions.

Many reviews, articles, and books have been written on particular aspects of archaeometry, providing clear descriptions of analytical results for the non-specialist.² A representative bibliography has been appended to this paper as a guide to further reading in this area. The references relate primarily to the examination of paintings or to methods of analysis employed in the Analytical Research Services laboratory (ARS) of the Canadian Conservation Institute (CCI), with an emphasis on microscopy and x-ray spectrometry.

The aim of this paper is to describe some of the instruments and methods of examination which are routinely used in the ARS laboratory of CCI to study paintings and painting materials. The bibliography includes references to a number of additional methods which have been used to study works of art. These include neutron activation autoradiography,³ dendrochronology,⁴ lead isotope mass spectrometry,⁵ particle-induced x-ray emission spectrometry (PIXE),⁶ and gas chromatography.⁷ Digital image processing and machine vision are also becoming increasingly widespread. ARS has been collaborating with the Autonomous Systems Laboratory of the National Research Council of Canada (Institute for Information Technology) on applications of a laser scanner to recording and replication of museum objects.⁸

A number of long-standing methods such as optical emission spectroscopy (implemented as the "laser microprobe"⁹), atomic absorption spectrophotometry, and microchemical tests continue to find important applications.

Visual Observation and Measurement

A careful visual examination of a painting or artifact is as important to the conservation scientist as it is to the curator or conservator. Projects have a far greater chance of success if all participants have an opportunity to examine a painting together, and to reach a consensus on which analytical questions the scientist will attempt to answer. When this is not possible, a clear statement of the nature of the problem, with a detailed technical history of the painting, is essential.

If an examination will involve the removal of particles or cross-sections for analysis, it is important to choose sites which are representative, and where samples can be removed most safely and inconspicuously. Occasionally, a question can be resolved without the need for sampling because underlying structure is already revealed near areas of damage or loss.

Photography in Normal, Raking, and Transmitted Light

Photography is a high priority in conservation for recording the initial condition of an object and subsequent stages in its treatment. In the scientific examination of paintings, photography is undertaken both to document the location of samples which may be removed for analysis and to record areas of discontinuity, overpaints, alterations, and pentimenti which will later be examined with infrared and ultraviolet techniques, x-radiography, and x-ray spectrometry for comparison.

ARS and other laboratories have developed standard procedures with respect to film size and format, colour and black-and-white film materials, and technique. Selection depends on the size and complexity of the object and the nature of the investigation.¹⁰ Paintings are illuminated with electronic flash lamps for both conventional photography in black-and-white and colour, and for scientific photography.¹¹ Electronic flash facilitates reproducibility of colour rendition within the limitations imposed by variations in film and film processing. Tungsten lamps are employed for transmitted infrared photography, infrared reflectography, and special illumination cases.

When the lamps are arranged at very shallow angles to the surface of a painting—called raking light—surface defects, craquelure, impasto, and distortions of the support are often revealed with great clarity. Paintings on canvas can be illuminated from behind (transmitted light) to reveal severe paint loss. Transmitted light can be applied in other situations, for example in the study of signatures, overpaints, crack patterns in wooden panels, and alterations to works of art and documentary artifacts on paper supports.

Close-Up Photography, Photomacrography, and Magnified Drawings

Close-up photographs of paintings at a magnification range of 1:10 to 1:1, and photomacrographs at magnifications from 1:1 to 50:1, are occasionally taken of areas of interest.¹² Macro lenses on a conventional camera, a photomacrography bellows camera, or a camera in conjunction with a stereomicroscope, can be used. Magnified drawings can be produced using a drawing attachment similar to a camera lucida with a stereomicroscope or other compound microscope (see below). This approach, which dates from the earliest days of microscopy, is still of great value in deciphering obscure signatures and in interpreting and annotating paint cross-sections.

Infrared Photography (Reflected and Transmitted)

The region of the electromagnetic spectrum which is visible to the human eye (i.e. light) lies in the wavelength range of about 400 to 700 nanometers (abbreviated nm). Infrared radiation is electromagnetic radiation with wavelength longer than 700 nm and infrared films are available which are sensitive in this region of the spectrum up to about 900 nm. Most commercially available infrared films have a maximum response at approximately 850 nm.¹³ Infrared photography is widely utilized in such fields as medicine, forensic science, and in remote sensing in the earth sciences and archaeology.¹⁴

Infrared photography can often reveal or clarify obscured or faded painting, overpaints, and underdrawings executed with charcoal or graphite. Under favourable conditions, carbon in the black pigment of an underdrawing (charcoal black, lamp black, graphite etc.) absorbs infrared preferentially, resulting in an enhancement of the drawing. This effect can be obscured by pigments such as azurite and malachite. On the other hand, lead white and cadmium red, which are radiopaque (see below under X-Radiography), are very transparent in the infrared. Consequently, infrared can reveal information which is obscured by lead white grounds in x-radiographs.¹⁵ Damages and other discontinuities are often revealed by infrared photography.¹⁶

Infrared photographs are exposed on Kodak High Speed Infrared Film 4143 with a Kodak Wratten Filter No. 87 over the camera lens to filter out unwanted visible light from the tungsten-halogen lamps or flash.¹⁷ Photographs can be taken with the camera either in front of the painting (reflected) or, where applicable, behind the painting (transmitted), with the positions of the lamps changed accordingly.

Infrared Reflectography and Infrared Transmittography

Examination of paintings in the infrared can be accomplished using an infrared vidicon¹⁸ system. In infrared reflectography, a method first developed by J.R.J. van Asperen de Boer, paintings can be illuminated from the front or back as in infrared photography. The reflected (or transmitted) infrared radiation is detected by an infrared vidicon, an electronic device which is sensitive to longer wavelengths than infrared photographic emulsions. As noted above, infrared films are typically most sensitive at wavelengths around 850 nm; infrared vidicons are responsive to wavelengths of up to 2000 nm, or higher. Because some paints are more transparent at these longer wavelengths, infrared reflectography has been found to yield a better image than infrared photography, for example in the case of underdrawings executed with carbon-containing pigments on white grounds.

The infrared vidicon employs a tube with a photoconductive material (e.g. a compound of selenium or lead) which allows an infrared image to be converted to an electronic (television) signal. In our laboratory, a Hamamatsu Type N214 infrared vidicon tube, with a spectral response up to approximately 2100 nm, is incorporated in a Dage 800 camera with a Canon zoom lens TV-16 (25-100 mm; 1:1.8; a 72 mm closeup lens can be substituted). A tungsten-halogen lamp is positioned, as in photography, to provide reflected or transmitted illumination, and a Kodak Wratten Filter No. 87 is placed over the camera lens to filter out unwanted visible light.¹⁹ The amount of infrared radiation from the lamp is controlled with a variable transformer. The television signal is viewed on a monitor or it can be photographed or recorded on tape to provide a permanent record. To obtain the best spatial resolution possible, paintings are recorded as a series of overlapping detailed reflectographs which must be photographed and then assembled into a montage. Digital image processing can achieve the same result more quickly and without the planar distortion which occurs in hand-made montages.²⁰

Infrared Luminescence Photography

The physics of infrared luminescence²¹ photography is similar to ultraviolet fluorescence photography although the results are different in a practical sense. In both techniques, the painting is irradiated (excited) with electromagnetic radiation resulting in emission of radiation of longer wavelength. In ultraviolet fluorescence photography (see below), the exciting radiation is ultraviolet and the emitted radiation is visible light. In the case of infrared luminescence photography, the exciting radiation is blue-green visible light and the emitted radiation is in the infrared region of the spectrum and is recorded on black-and-white infrared film. Infrared luminescence photography is applicable when thick layers of varnish are present. Ultraviolet fluorescence of later varnish layers interferes with fluorescence from the original paints and surface coatings. Blue-green light can penetrate such yellow varnish layers and induce infrared luminescence in the original materials.

To obtain blue-green light, we enclose a 10% solution of copper sulphate in water in a Plexiglas[®] cell which is placed in front of the electronic flash lamps. The copper sulphate solution transmits a broad band of light in the approximate range 400 to 540 nm. A Kodak Wratten filter No. 87 (or sometimes an 87C) is placed over the camera lens to eliminate visible light reflected from the painting and the recording is conducted in total darkness.

Ultraviolet Fluorescence Photography

Ultraviolet fluorescence photography records the visible light emitted by an object which is irradiated with ultraviolet radiation—electromagnetic radiation of wavelength just below the short wavelength end of the visible range (below 400 nm). Fluorescence can also be produced by short wavelength (violet or blue) visible light. Overpaints, partially cleaned areas, damages, and other discontinuities often differ markedly from original paints in their fluorescence which allows them to be clearly distinguished. For this reason, a fluorescence photograph is frequently an essential guide to choosing locations to remove samples for analysis.

Photographs are recorded on black-and-white or colour film. Illumination is from a complement of four Balcar electronic flash units with four bi-tube flash heads. A Corning ultraviolet transmitting glass exciter filter is placed on each bare tube head.²² These are large glass filters which transmit ultraviolet radiation in the range 320-400 nm. A Kodak Wratten No. 2E barrier filter is placed on the camera lens. This combination permits ultraviolet radiation to strike the object but only visible light of wavelength greater than 415 nm to reach the film.

In addition to the 2E barrier filter, a light yellow filter (Kodak Wratten No. 3), and Kodak Wratten colour compensating filters CC10C (cyan) and CC20M (magenta) can be placed in front of the camera lens. This combination attenuates unwanted blue light and produces excellent results with Kodak Ektachrome Film 6176.

It should be emphasized that the purpose of fluorescence photography, as well as other methods described here, is primarily to provide qualitative differentiation of areas within a painting. Comparisons between paintings in questions of provenance and art history can also be accomplished. It is therefore necessary to be aware that differences in colour rendition and surface detail can arise as a result of the use of different sources of illumination, camera lenses, filters, films, and film processing.

Ultraviolet Reflection Photography

A photographic image can be made of the ultraviolet radiation which is reflected by an object. This method is often effective in enhancing faded documents since residual ink may still absorb ultraviolet. In ultraviolet reflection photography, the Balcar electronic flashes are not filtered and a Carl Zeiss B57-UG 11, or a Kodak Wratten 18A filter, is placed on the camera lens. The 18A filter transmits only ultraviolet radiation in the range 320-380 nm. The Zeiss filter transmits in the range 260-380 nm. These filters prevent visible light caused by fluorescence from reaching the camera. The ultraviolet radiation reflected is recorded on Kodak Tri-X, T-Max 400 or T-Max 100 film using a Hasselblad 500C/M camera with a UV-Sonnar quartz lens which transmits ultraviolet radiation.

X-Radiography

X-rays undergo absorption when they pass through an artifact such as a painting.²³ The extent to which they are absorbed by various materials depends on an atomic property called the mass absorption coefficient²⁴ which is a function of x-ray wavelength and atomic number. It is a measure of the ability of a material to stop x-rays and, at a given x-ray wavelength, it increases with the average atomic number of the chemical elements present. For a given element, or combination of elements, the mass absorption coefficient increases with x-ray wavelength because longer wave x-rays are less energetic and less penetrating.

In practice, the transmission of x-rays depends on paint layer thickness, pigment density, the ratio of pigment to vehicle,²⁵ and the thickness and density of the support (canvas, panel, stretcher, etc.). Assuming equal paint thickness and ratio of pigment to vehicle, paints containing pigments composed of chemical elements of higher atomic number, such as lead white, vermilion, lead-tin yellow, and lead antimonate (Naples) yellow, absorb x-rays much more strongly than pigments of lower average atomic number, such as azurite and green earth, or umbers, ochres, and siennas. Organic materials such as the canvas itself, varnishes, organic pigments, lakes, and dyes are virtually transparent to x-rays because they are composed of carbon, hydrogen, nitrogen, and oxygen which are all elements of low atomic number.

Lead white is a common constituent of grounds, and tends to concentrate in the interstices of the canvas threads making the pattern of the canvas visible in x-radiographs. Stretcher garlands—a scallop pattern at the edge of a canvas originating at the time when it was first stretched—are revealed in this way. Their presence or absence can help determine if a canvas has been cut down in size in the past. Lead white is one of the most common paint pigments and is frequently the pigment which is most critical in the formation of radiographic images.

X-radiographs can reveal wood grain, cracks, defects, voids, joins, pentimenti, additions, alterations, and composite canvases or panels. Insect damage, caused by borers such as the death watch beetle or common furniture beetle, shows up in x-radiographs if holes have been filled in with lead white containing putty. Double paintings can often be detected.²⁶ Conversely, an x-radiograph can demonstrate that a painting is in excellent condition or free of alterations.

An x-ray tube is aimed at the painting and the radiographic image is recorded on film which is placed closely behind it. The spectrum of x-ray wavelengths emitted by the tube is determined by the electrical potential at which it is operated, usually in the range 10 to 25 kilovolts (kV). Better resolution can be achieved if the film can be placed adjacent to the painted surface and irradiation is from the back. An overhead suspension system allows the tube to be moved orthogonally over large paintings when it is necessary to expose a mosaic of overlapping films. The standard film size is 14" x 17".²⁷

Electron Emission Radiography

Electron emission radiography was first described by Trillat in the 1940s and has been called "reflection microradiography" and "photoelectron autoradiography."²⁸ Its application to panel paintings and a painting on a copper support was first described by Charles Bridgman of Eastman Kodak and others.

In electron emission radiography, x-rays of higher energy are required than are needed for the conventional radiography of paintings. We use a Philips G300 x-ray tube operating at a potential as high as 300 kV. A 12 mm thick copper sheet filters out low energy x-rays from the emission spectrum of the tube which would otherwise register on the film and interfere with the image.²⁹ When a painting is irradiated with such energetic x-rays, electrons are emitted. These are recorded on film placed on the same side of the painting as the x-ray tube. The image results from exposure to electrons ("photoelectrons") which are emitted from the object and whose intensity depends on the average atomic number of the elements present in the area irradiated.

The method requires that the film be in intimate contact with the surface of the painting; this can be achieved by enclosing the painting and film together in a plastic envelope in which a moderate vacuum is drawn.³⁰ This is not undertaken if there is incipient paint loss or other structural weakness such as cleavage, or insect damage. As an alternative, films can be lightly pressed against the painting using an x-ray transparent material, although usually with inferior results.

One advantage of electron emission radiography is that the canvas weave pattern, stretcher bars, cradles, tacks and other interfering elements which appear in conventional radiographs are not recorded. Because only the uppermost paint layers contribute to the image, the method is useful in revealing design changes, overpaints, and previous losses. Electron emission radiography can be used for paintings executed on a radiopaque support such as copper when conventional x-radiography cannot.

Light Microscopy

Because of the great diversity of materials and problems encountered in the examination of artifacts, the scientist and conservator employ a number of different types of microscopes and microscopical methods. Both light microscopes and electron microscopes find application in this field. The four light microscopes in most common use in conservation and archaeometry are the stereomicroscope, the polarizing microscope, the fluorescence microscope, and the metallograph. They belong to a family of microscopes called compound microscopes because resolving power and magnification is accomplished by a combination of an objective lens and an ocular (or eyepiece). Where they differ is in their design, function, and application. For example, a metallograph is a compound microscope specifically designed for studying polished and etched samples of metal alloys using reflected light. The crystalline structure of historic bronze, iron, and other alloys revealed in this way provides information on casting, forging, and annealing as well as patination and corrosion.³¹ The stereomicroscope, fluorescence microscope, and polarizing microscope are discussed below.

Stereomicroscopy

Stereomicroscopes are used for examination and treatment tasks requiring magnifications of about 3 to 50 times. Widely employed in medicine, industry, and other fields, the stereomicroscope consists of two separate compound microscopes which are housed within one body and focused at the same point but at slightly divergent angles. The microscopist sees an upright, three-dimensional image which makes the stereomicroscope the ideal instrument for the precise removal and manipulation of samples from paintings. Typically, the stereomicroscope is mounted on a table stand for small artifacts and sample preparation. Floor stands with articulated arms, of a kind designed for surgical applications, allow larger as well as inclined surfaces to be examined. We have mounted one stereomicroscope on a reciprocating carriage of our own design which allows it to be moved over a surface 1.8 x 1.1 metres in conjunction with an x-ray spectrometer.³²

Sampling: Cross-Sections, Particles, and Fragments

Before discussing polarized light and fluorescence microscopy, as well as other analytical methods, it is appropriate to briefly discuss sampling. Samples fall into several categories which include particles and fragments, cross-sections, fibre and wood samples. The validity of all subsequent analytical steps depends upon the quality of the initial samples and the extent to which they are representative of the structure and materials under investigation. Samples in general, and cross-sections in particular because they tend to be larger, are taken only when they are essential to the solution of a conservation or archaeometry problem.

Where possible, samples are removed from inconspicuous areas of the painting or polychrome, for example, from below the rabbet of a framed painting. Sampling at areas of actual or incipient loss is often appropriate except where there is concern about interference from previous restorations in the area. Sample size varies, depending on the nature of the investigation and the method of analysis, from particles which are

difficult to see with the unaided eye, to cross-sections which may include a paint surface area of about 0.5 to 1 millimetre across.

Samples are removed with scalpels, surgical blades, razor blade fragments, hypodermic needles, sharpened tungsten needles, or other appropriate microtools.³³ Pigment and corrosion samples for x-ray diffraction are taken directly onto a fine glass fibre for insertion in the x-ray diffraction camera (see below). It is usually preferable, but not essential, to work under a stereomicroscope while sampling. Depending upon the location of the sample, its proximity to an area of loss, and the brittleness of the surface coating and design surface layers, cross-sections may be pried, sliced, or cut out of position. For the study of accretions, interpretative varnishes, glazes, blooms, and other surface phenomena, samples can be scraped or peeled off.

Sample locations are documented by marking them on a photograph or sketch, by reference to a pictorial feature, or by measurement in centimetres from the left and bottom edges of the stretcher or canvas margin in the case of rectangular, or nearly rectangular, paintings.

Cross-sections are miniature cores of material removed from an object. One side is ground and polished flat to reveal the sequence of structural and design layers of a painting or artifact. Sometimes cross-sections consist of, or include, the basic structural material or support of an artifact. They are prepared by embedding samples in a liquid casting resin—usually a polyester or epoxy resin—which has been poured into small, square rubber forms. The resin is allowed to harden before one transverse face is ground flat with silicon carbide papers. This face is then polished smooth on synthetic velvet using polishing compounds. The procedure is not unlike that of a petrographic laboratory for preparing thin sections of rocks. In some studies, it is advantageous to cut sections with a microtome—a high precision mechanized knife—or a diamond impregnated circular saw. The advantages of the standard grinding and polishing method are in the precise control of speed, depth, and orientation of the section.

Cross-sections in which only one face is ground and polished are sometimes called thick or opaque sections because they can be examined in a microscope only using incident, reflected light. In the study of rocks, minerals, ceramics, and many other materials, the standard procedure is to prepare thin sections which can be observed in transmitted polarized light. Thin sections are prepared by adhering the polished cross-section to a glass microscope slide and grinding and polishing the other side to a thickness which permits observation in transmitted polarized light. Sometimes this approach is advantageous for samples from paintings as well. These sections have to be made very thin (less than 20 micrometers) to permit light transmission, especially when lead white predominates.

Specialized techniques and knowledge are needed to identify the wood and other plant species which serve as painting supports and in the manufacture of paper.

Incident Light Microscopy

Cross-sections are first examined in a microscope with incident light. Examination of cross-sections in this way is frequently all that is needed to answer questions concerning overpainted areas, whether a paint layer is original or not, or whether an artist has used a particular ground, an imprimatura, a glaze, or a pigmented varnish. A cross-section sequence of a painting might include one or two ground layers applied to a sized canvas, several paint layers, an old varnish layer, a thin layer of grime, a layer of overpaint, and one or two more recent applications of varnish. It is not unusual for cross-sections from buildings, furniture, outdoor sculpture, industrial and utilitarian objects, ships' figureheads and the like to reveal a complex original finishing process or a great number of repaintings.

Histochemical stains are occasionally used to differentiate organic components of paints, although specificity and reproducibility can be difficult in the case of paint vehicles, coatings, and other complex mixtures. Microchemical tests with chemical reagents can also be used to identify chemical elements and

ions present in pigments and other painting components. For most but not all studies, the analysis of cross-sections by microchemical methods has been superseded by x-ray microanalysis and other techniques.³⁴

Fluorescence Microscopy

It is always worthwhile to examine cross-sections of paintings by fluorescence microscopy. A fluorescence microscope is essentially an incident light microscope in which the incident illumination is not white light but rather a region in the electromagnetic spectrum chosen to cause fluorescence of paint components. This is achieved using a lamp—usually a high pressure mercury vapour lamp—which strongly emits ultraviolet and visible light. A wavelength range is then selected using combinations of filters for ultraviolet, violet, blue, and green bands in the spectrum of different widths.³⁵ Special filters and mirrors allow the exciting radiation to irradiate the sample but only visible fluorescent light to be observed at the eyepiece.

The term autofluorescence describes materials which fluoresce by themselves without the introduction of other reagents. Autofluorescence of paint layers is often effective in differentiating layers or improving visual contrast between layers or zones within a layer. A simple example is the case in which the aim is to determine whether there is an old, thin varnish layer between two paint layers which would prove that the cross-section is from an area which has been overpainted. An aged varnish layer usually fluoresces strongly in the microscope but can be difficult to decipher in normal light.

A promising area of research in this area pertains to the use of fluorescent dyes to characterize vehicles and coatings in paintings, furniture, and other artifacts.³⁶

Polarized Light Microscopy

The polarizing microscope is a versatile instrument for the study of the materials encountered in art and archaeological objects. As noted already, rocks, minerals, glasses, ceramics, and paint layer sequences of painted objects can be studied by preparing polished thin sections. Pigments, fibres, corrosion products, accretions, and other particles—both naturally occurring and synthetic—are dispersed in a viscous medium of known refractive index for examination with transmitted polarized light.³⁷

The microscopist characterizes a material, mixture, or compound on the basis of a number of morphological and optical properties which include size, shape, cleavage, homogeneity, colour, transparency, pleochroism, refractive indices, and birefringence. This last property accounts for the exquisite interference colours observed with the polarizing microscope. With some experience, pigments, fibres, and other materials can be quickly and unequivocally identified by polarized light microscopy, based on a set of unique optical and physical properties. In other cases, it is necessary to confirm the identification by another technique.

X-Ray Spectrometry—Terminology

The term "x-ray spectrometry" encompasses many basic methods and techniques, and an even greater number of instrument configurations and designs. It is therefore not surprising that the nomenclature is inconsistent, and that multiple terms often describe essentially the same technique.

The purpose of x-ray spectrometry is to detect the presence of chemical elements in objects, or in samples removed from objects. This is accomplished by measuring the x-rays which are emitted by the object or sample when it is irradiated with either x-rays or a beam of particles, such as a beam of electrons. The energies, or wavelengths, of the x-rays emitted by each chemical element are characteristic of that element. This process of detecting the elements present is referred to as *qualitative* analysis. Depending on the aim of the analysis, it may be *necessary to estimate the concentrations of elements (semi-quantitative analysis)* or to determine their concentration accurately (*quantitative analysis*).

The term "x-ray secondary-emission spectrometry" has been recommended for methods in which emission of x-rays from an object or sample is excited by x-rays from an x-ray tube or a radioactive source.³⁸ The more conventional term is "x-ray fluorescence spectrometry."³⁹ When x-rays in a sample are excited by an electron beam, the term "x-ray primary-emission spectrometry" is appropriate but rarely encountered in the literature. Instead, most authors prefer a generic term such as electron beam x-ray microanalysis, or they refer to one of the two instruments designed for this purpose—the electron microprobe or the scanning electron microscope (SEM).⁴⁰ The electron microprobe and SEM differ in their historical development, specific design criteria, and application but are, in essence, the same instrument.

X-rays emitted by the object or sample can be dispersed, detected, and measured in several ways. X-ray lines can be separated on the basis of their wavelength or their energy, depending on the type of detector (there is a simple relationship between the two parameters). This has led to the coining of terms like wavelength spectrometry, energy spectrometry, as well as common variants such as energy-dispersive x-ray spectrometry (EDX or EDS).⁴¹ We devised the term radioisotope-excited x-ray energy spectrometry (which we abbreviate as REXES) to describe a method of x-ray secondary-emission (fluorescence) spectrometry in which excitation is by x-rays from a radioactive source and detection of the characteristic x-rays is achieved with an energy spectrometer.

X-Ray Secondary-Emission (Fluorescence) Spectrometry Radioisotope-Excited X-Ray Energy Spectrometry (REXES)

The chemical elements present in a painting or artifact can be identified using x-ray energy spectrometry. We employ a method in which selected areas of the object, a few millimetres in diameter, are irradiated by a radioactive source of x-rays. X-rays emitted by the source impart energy to the atoms in the paint, which in turn emit x-rays. Each chemical element emits x-rays which are characteristic of it. X-rays are absorbed by a detector, and electronic pulses are generated whose amplitude is proportional to the x-ray energy. These pulses are amplified, sorted, and counted by an x-ray spectrometer, resulting in a spectrum in which the number of x-rays counted at each x-ray energy interval is displayed as a histogram.⁴² The chemical elements present and their relative concentrations are determined by a judicious interpretation of the spectrum and comparison with spectra from reference materials.

The technique is advantageous for museum objects because it is relatively fast and does not require sample removal. The radioisotope is mounted inside a cylindrical brass holder which is positioned on the end of a stainless steel shaft which houses the x-ray detector. The detector itself is located inside the end of the shaft. The source and detector are mounted on a positioning carriage which allows them to be moved horizontally over large paintings. There is no physical contact between the instrumentation and the painting or artifact.

From a knowledge of the chemical elements present and the colour of the paint, it is frequently possible to deduce which inorganic pigments, fillers, or paint extenders are most probably present. The area irradiated is usually too large to permit analysis of only one colour of paint. The more detailed the execution of a painting, pastel, or watercolour, of course, the more difficult it is to find a broad area of one colour to analyze. The analyst must take into consideration paint mixtures, underlying paint layers, and the existence of pigments that are made up of the same elements but have different chemical compositions. For example,

while the detection of lead (Pb) in paint usually indicates the presence of lead white (basic lead carbonate; $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$), other pigments such as lead sulphate (PbSO_4), basic lead sulphate ($\text{PbSO}_4 \cdot \text{PbO}$), red lead (Pb_3O_4), or litharge (PbO) could also account for the lead. Elemental analysis cannot distinguish between such closely related pigments as emerald green ($\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{Cu}(\text{AsO}_2)_2$) and Scheele's green (CuHAsO_3), or between the historically notable pigments lead tin yellow type I (Pb_2SnO_4) and lead tin yellow type II ($\text{Pb}_2(\text{Sn}, \text{Si})_2\text{O}_6$).

Because x-rays of lower energy are absorbed by air, or by a thin beryllium window which protects the x-ray detector, only elements above and including calcium (atomic number 20) in the periodic table can be analyzed. This means that chemical elements with lower atomic number which occur, for example, in clays and other minerals (sodium, magnesium, aluminum, silicon, etc.) are not detected, and it becomes necessary to remove a microscopical sample for analysis by another technique such as electron beam x-ray microanalysis or x-ray diffraction (see below). The elements comprising pigments, dyes, and other organic materials (hydrogen, carbon, oxygen, and nitrogen) are not detected.

In spite of these limitations, REXES has proven to be extremely effective. It can be very helpful in establishing that a painting is a copy or forgery because pigments which were discovered and produced only after certain periods—especially after the Industrial Revolution or later—frequently are composed of chemical elements which could never occur in a painting executed before that time. REXES can reveal that a painting is in fact a painted photograph, through the detection of silver in the original photographic emulsion.

Scanning Electron Microscopy and X-Ray Microanalysis

The scanning electron microscope (SEM) has found application in a wide range of biological, medical, and materials science applications since it became a routine tool in the 1960s. Its function is twofold: the SEM was first developed as an imaging tool to study morphology and structure at a range of magnifications with much higher spatial resolution than is possible with a light microscope, and with much greater depth of focus. Second, because characteristic x-rays are emitted from materials when they are irradiated with electrons, the SEM—like an electron microprobe—can also function as an x-ray primary-emission spectrometer. This is accomplished by incorporating energy or wavelength detectors in the column of the instrument. When these two functions are combined, it is possible to determine the distribution of chemical elements in cross-sections, particles, and fragments from artifacts and works of art.

The sample to be analyzed must be small enough to fit on the holder of a goniometer stage which allows it to be moved horizontally and vertically, as well as tilted and rotated.⁴³ A beam of electrons is focused to a fine spot on the surface of the specimen. The beam can be made to stand still in one spot to study the composition of a single feature such as a pigment particle. It can be moved along any line across the specimen, for example, to determine variation in concentration of chemical elements in the layers of a cross-section. The beam can be scanned, line by line, over a rectangular area on the surface of the specimen. At every point at which the beam strikes the specimen, any of a number of interactions can occur, resulting in the emission or backscattering of electrons, and the production of x-rays, light, and so on. These are detected and displayed on a screen as images of varying intensity. With our detector, x-rays from elements having atomic number greater than or equal to 11 (sodium) in the periodic table can be detected.

X-Ray Diffraction

Many artists' pigments, especially ancient and historical pigments, are finely ground, naturally occurring minerals. Ochres, umbers, and siennas are mixtures of iron and manganese oxide and hydroxide minerals like haematite and goethite, and also contain clays and other minerals. Other well known pigments of colourful natural origin are lazurite (lapis lazuli), cinnabar, malachite, azurite, and green earth. Early in the history of pigments, but especially after the Industrial Revolution, synthetic analogues of natural minerals were

developed as well as synthetic pigment compounds which do not exist in nature. A majority of these inorganic pigments are characterized by having a crystalline structure, that is, they have a highly ordered arrangement of atoms. The technique of x-ray diffraction is ideally suited to the study of such materials. Painting grounds, some organic pigments such as indigo, the products of metal corrosion, and other chemical compounds can be precisely identified with this method.

Microscopical paint fragments or particles are removed from carefully selected areas of a painting using surgical scalpels and fine tungsten needles. The sample is then mounted at the end of a finely drawn glass fibre which is positioned in a Debye-Scherrer x-ray diffraction camera for analysis by x-ray diffraction. Our camera is equipped with a Gandolfi mechanism which rotates the sample about two axes during exposure to a beam of x-rays. This permits excellent x-ray diffraction patterns to be obtained from extremely small samples. The pattern of diffracted x-rays is recorded on film wrapped around the interior circumference of the camera. Because of the small sample size, exposures of as long as 24 hours or more can sometimes be required for each sample.⁴⁴

X-ray diffraction patterns consist of a series of dark lines of varying intensity on a film. The position and intensity of these lines depend on the spacing between the regular planes of atoms which make up the crystalline structure of the material being studied, and on the nature of the atoms themselves. Comparison with patterns from reference materials and with published data allows mixtures of pigments, fillers, grounds, and other materials to be identified.

Fourier Transform Infrared Spectroscopy

Organic materials (gums, oils, resins, varnishes, proteins, organic pigments, fibers, detergents), inorganic materials (minerals, pigments, fillers, extenders, corrosion products), polymeric materials (plastics, rubbers, adhesives, paint vehicles) and their mixtures can all be identified by infrared spectroscopy.

Materials absorb different amounts of infrared radiation at different wavelengths, depending on the vibrations of the chemical bonds that are present. Spectra showing the intensity of absorption at various wavelengths are interpreted to determine the type of bonds in the sample. This provides clues to the composition. Precise identification is made by matching a sample spectrum with spectra of reference materials of known composition.

To acquire a spectrum, a sample is placed in the beam of the infrared spectrometer using a variety of sampling techniques and accessories, depending on the size and nature of the specimen. In our laboratory, high pressure and low pressure diamond anvil micro-sample cells are most often used. These require sample sizes on the order of only a few micrograms, and no preparation is required other than placing the small sample on the diamonds.⁴⁵ One of our instruments integrates a light microscope with a spectrometer in such a way that spectra can be obtained from a portion of the sample visible in the field of view of the microscope.⁴⁶ Other devices such as silver chloride and sodium chloride transmission plates, internal and diffuse reflectance accessories, and gas and liquid cells are used as required.

Conclusions

It would be unfortunate if the reader were left with the perception that it is the instruments and methods themselves which alone provide solutions to problems of examination and analysis in conservation and archaeometry. True, scientists now have available more sophisticated instruments than was the case, say, in the 1930s. This allows for greater accuracy and precision with smaller samples. It must be acknowledged, though, that today's scientist relies heavily on the work of earlier researchers who had fewer tools but were ingenious in the ways in which they applied them. The solution to a problem depends on the skill and experience of those involved in the interpretation of the data collected as much as it does on the instruments available. There is also a risk that the procedures followed in the examination of paintings and artifacts will

be seen to be completely impartial and free of bias. This is certainly true up to a point. The scientist tries to be rigorous in experimental design and execution but must also, on the other hand, exercise considerable judgement in ensuring that the reported analytical results accurately address the question asked by the curator, art historian, archaeologist, or conservator.

We try to obtain as much useful information as possible without sampling, and never sample if the integrity of the object would suffer as a result. Sample size and location impose severe limitations on analysis, and it requires care and experience to select appropriate locations and to draw conclusions from analysis of the samples. Because of sample size restrictions, one of our goals is to perform as many analyses as possible on the same sample, and to correlate the findings carefully with the photographic and x-radiographic images. A knowledge of similar paintings, images, and data is essential for comparative purposes, particularly in questions of provenance and authenticity. Fortunately, there now exists a considerable body of published literature in conservation and archaeometry, and a willingness among individuals and institutions to share the information they have gathered.

Notes

1. Archaeometry has been defined as ". . . the application and interpretation of natural science data in archaeological and art historical studies." Jacquelin S. Olin, ed., *Future Directions in Archaeometry* (Washington: Smithsonian Institution, 1982), p. 19.
2. *Art and Archaeology Technical Abstracts* (AATA), or the larger computerized bibliographical database (BCIN) of the Conservation Information Network, are especially useful. An excellent short review is J.R.J. Van Asperen de Boer, "An Introduction to the Scientific Examination of Paintings," in: *Scientific Examination of Early Netherlandish Painting*, eds. J.R.J. van Asperen de Boer and J.P. Filedt Kok, Netherlands Yearbook for History of Art, vol. 26 (Bussum: Fibula-van Dishoeck, 1976), pp. 1-40. [Refer in particular to Notes 44-69, 76-78.]. A classic text in the field is Rutherford J. Gettens and George L. Stout, *Painting Materials: A Short Encyclopaedia* (New York: Dover Publications, 1966). This is a 1966 edition of information previously published between 1936 and 1939 in the journal *Technical Studies in the Field of the Fine Arts*. Detailed studies of pigments have also been published as a series of articles in the IIC journal *Studies in Conservation* and in the book *Artists' Pigments: A Handbook of their History and Characteristics* (Robert L. Feller, ed.) and these are listed together in the bibliography. Recent reviews include F. Mairinger, and M. Schreiner, "New Methods of Chemical Analysis—A Tool for the Conservator," *Science and Technology in the Service of Conservation*, Preprints of the Contributions to the Washington Congress, International Institute for Conservation of Historic and Artistic Works, 3-9 September 1982, pp. 5-15; and Richard Newman, "Roles of Scientific Examination in the Study of Works of Art," *Museum Studies Journal*, vol. 3, no. 2, 1988, pp. 20-32.
3. See, for example, Edward V. Sayre and Heather N. Lechtman, "Neutron Activation Autoradiography of Oil Paintings," *Studies in Conservation*, vol. 13, no. 4, 1968, pp. 161-185; Peter Meyers et al. "The Application of Neutron Activation Autoradiography in the Study of Paintings by Rembrandt and Related Artists," in: *Science and Technology in the Service of Conservation*, Edited by N.S. Brommelle and G. Thomson, Preprints of the Contributions to the Washington Conference, 3-9 September 1982 (London: International Institute for Conservation of Historic and Artistic Works, 1982), pp. 165-168.
4. For a discussion of dendrochronology see Peter Klein, "Age Determinations Based on Dendro-chronology," Chapter XII in: *Scientific Examination of Easel Paintings, PACT, Journal of the European Study Group on Physical, Chemical and Mathematical Techniques Applied to Archaeology*, no. 13, 1986, Roger van Schoute and Hélène Verougstraete-Marcq, editors (Strasbourg: Council of Europe—Parliamentary Assembly, 1986), pp. 225-237.

5. For a discussion of lead isotope ratio analysis see Bernard Keisch and Robert C. Callahan, "Rubens's The Gerbier Family: Investigation by Lead Isotope Mass Spectrometry," *Studies in the History of Art* (Washington: National Gallery of Art, 1973), pp. 75-78.
6. PIXE is described in Timo Tuurnala, Aimo Hautojärvi and Kirsti Harva, "Examination of Oil Paintings by Using a PIXE/PIGME Combination," *Nuclear Instruments and Methods in Physics Research*, vol. B14, no. 1, *Proceedings of the International Workshop on Ion Beam Analysis in the Arts and Archaeology*, Pont-à-Mousson, France, 18-20 February 1985, Proceedings editors Ch. Lahanier et al. (Amsterdam: Elsevier, North-Holland, 1986), pp. 70-75.
7. Raymond White, "The Characterization of Proteinaceous Binders in Art Objects," *National Gallery Technical Bulletin*, 1984, pp. 5-14.
8. The laser scanner and its applications have been described in a number of journals and proceedings including J.M. Taylor, I.N.M. Wainwright, F.R. Livingstone, M. Rioux, and P. Boulanger, "Applications of a Laser Scanner to the Recording and Replication of Museum Objects," *Proceedings, 8th Triennial Meeting, ICOM Committee for Conservation*, Sydney, Australia, 6-11 September 1987, pp. 93-97.
9. See F. Brech and W.J. Young, "The Laser Microprobe and Its Application to the Analysis of Works of Art," in: *Application of Science in Examination of Works of Art, Proceedings of the Seminar*, 7-16 September 1965 (Research Laboratory, Museum of Fine Arts, Boston), pp. 230-237.
10. A Linhof Kardan B1 System camera serves for 10.2 x 12.7 cm (4" x 5") negatives and a Sinar p camera for 20.3 x 25.4 cm (8" x 10"). 35 mm and 120 mm cameras are used for colour transparencies (slides) and special applications.
11. Balcar electronic flash units are the mainstay of our documentation and technical photography system. These are A5000 W/s electronic flash units with four bi-tube flash heads.
12. The Eastman Kodak Company of Rochester, New York has published a number of excellent Technical Publications on close-up photography, photomacrography and photomicrography.
13. For a review of infrared to visible conversion methods, see J.R.J. van Asperen de Boer, "Reflectography of Paintings Using an Infrared Vidicon Television System," *Studies in Conservation*, vol. 14, no. 3, 1969, pp. 96-118.
14. The work of William McLennan on Northwest Coast artifacts at the Museum of Anthropology, University of British Columbia, is an excellent example of the application of infrared photography. See Margaret Munro, "Indian Art Comes to Light," *Canadian Geographic*, vol. 108, no. 4, 1988, pp. 66-70.
15. See Dan E. Kushel, "Applications of Transmitted Infrared Radiation to the Examination of Artifacts," *Studies in Conservation*, vol. 30, no. 1, 1985, pp. 1-10.
16. For a discussion of the application of infrared photography and reflectography see J.R.J. Van Asperen de Boer, "An Introduction to the Scientific Examination of Paintings," in: *Scientific Examination of Early Netherlandish Painting*, eds. J.R.J. van Asperen de Boer and J.P. Filedt Kok, Netherlands Yearbook for History of Art, vol. 26 (Bussum: Fibula-van Dishoeck, 1976) pp. 1-40.
17. The spectral transmittance of the Kodak Wratten filters used can be found in Eastman Kodak Company, *Kodak Filters for Scientific and Technical Uses*, Kodak Technical Publication No. B-3H (Rochester: Eastman Kodak Company, 1970).
18. Vidicon = video + iconoscope or video image convertor.

19. Infrared reflectography and transmittography are described in detail in the references by van Asperen de Boer and Kushel.
20. See G. Wecksung, R. Evans, J. Walker, M. Ainsworth, J. Brealey and G. Carriveau, "Infrared Reflectogram Assembly by Digital Image Processing," in: *Preprints, 15th Annual Meeting, American Institute for Conservation of Historic and Artistic Works*, Vancouver, 20-24 May 1987, pp. 163-167.
21. Luminescence has been defined as ". . . the emission of light resulting from all processes except incandescence . . . The fundamental law of luminescence states that the wavelength of the fluorescent light is longer than the wavelength of the exciting radiation. Fluorescence usually involves the absorption of invisible radiation in the ultraviolet part of the spectrum and the simultaneous emission of this energy as visible light with a longer wavelength. The absorption of visible light may result in emission in the infrared part of the spectrum—invisible radiation of longer wavelength, undetectable except by special equipment sensitive to infrared radiation. This is known as infrared luminescence," L.G. Berry, and Brian Mason, *Mineralogy* (San Francisco: W.H. Freeman, 1959), pp. 198-199. Infrared luminescence and its application to the study of paintings is the subject of a paper by Charles F. Bridgman and H. Lou Gibson, "Infrared Luminescence in the Photographic Examination of Paintings and other Art Objects," *Studies in Conservation*, vol. 8, no. 3, 1963, pp. 77-83.
22. The Pyrex® ultraviolet shells on the Balcar flash heads are removed for fluorescence and ultraviolet reflection photography. An ultraviolet exciter filter can also be made with a saturated solution of cobalt chloride.
23. For a discussion of the early application of x-radiography see Charles F. Bridgman "The Amazing Patent on the Radiography of Paintings," *Studies in Conservation*, vol. 9, no. 4, November 1964, pp. 135-139 where we learn that x-radiography was applied to the examination of paintings and painting materials shortly after Roentgen's publication of his discovery of x-rays in 1895. In March 1896, a friend of Roentgen's wrote that "In a wooden color box the tablets containing metallic pigments, such as cinnabar, chrome yellow, Berlin blue, and the like, can be separated from those tablets which do not contain metallic pigments, for example carmine and gamboge, with the cover closed. The first-mentioned color tablets gave shadow pictures; the latter did not." That same year König radiographed an oil painting and in 1897 this statement was published in *The Electrical Review*: "This application of the roentgen rays [x-rays] may prove of considerable value to picture dealers and others in detecting fraudulent imitations of valuable paintings."
24. The mass absorption coefficient of a compound or mixture is equal to the weighted sum of the mass absorption coefficients of the elements of which it is made. Attenuation of polychromatic x-radiation can also be expressed in terms of a half-value thickness or half-thickness which is the thickness of a material required to reduce the intensity of incident x-rays by half. For calculations of the half-value layers for several pigment-vehicle mixtures, see S[tephen] Rees Jones, "Physics and Painting," *Bulletin of the Institute of Physics*, vol. 11, 1960, pp. 157-165.
25. Vehicle: the preferred term for the film-forming and binding material of paint also referred to as medium, binding medium, or binder.
26. See, for example, M. Ruggles, "The Interpretation of Double Paintings," *AIC Preprints of Papers Presented at the Fourth Annual Meeting*, Dearborn, Michigan, 29 May–1 June 1976, pp. 102-110.
27. Typical operating conditions for x-radiography: Philips MG 152 Be x-ray tube operating at an accelerating potential of 25 kV; 0.4 mm² focal spot; 100 cm film-focal distance; tube current 4.5 mA; 200 second exposure; Kodak SR 5 film; GBX developer, 5 minutes at 21°C. Many artifacts, for example bronzes, require a more penetrating x-ray beam for which we have available a Philips G300 x-ray tube with a maximum tube voltage of 300 kV.

28. See the article by D.T. O'Connor and W.R. Maddy, "Radiography and Autoradiography by Photoelectrons," *ASTM Papers on Radiography*, 1950, pp. 20-33; Charles F. Bridgman, Peter Michaels and Harold F. Sherwood, "Radiography of a Painting on Copper by Electron Emission," *Studies in Conservation*, vol. 10, no. 1, 1965, pp. 1-7; Charles F. Bridgman, Keck, Sheldon, and Sherwood, Harold F., "The Radiography of Panel Paintings by Electron Emission," *Studies in Conservation*, vol. 3, no. 4, 1958, pp. 175-182.
29. Typical operating conditions for electron emission radiography: Philips G300 x-ray tube operating at an accelerating potential 290-300 kV; tube current 3-5 mA; film-focal distance of 100-120 cm; 12-17 minute exposure; Kodak SR5 film; GBX developer 5 minutes at 20 °C.
30. Charles F. Bridgman, Sheldon Keck and Harold F. Sherwood, "The Radiography of Panel Paintings by Electron Emission," *Studies in Conservation*, vol. 3, no. 4, 1958, pp. 175-182.
31. See for example Rutherford J. Gettens, *The Freer Chinese Bronzes*, Volume II, *Technical Studies* (Washington: Smithsonian Institution, Freer Gallery of Art, 1969).
32. See Marilyn E. Laver and Ian N.M. Wainwright, "A Positioning Carriage for Stereomicroscopy and X-Ray Spectrometry," *Journal of the International Institute for Conservation-Canadian Group*, vol. 10 & 11, 1985/1986, pp. 28-30.
33. Rutherford Gettens was an early refiner of sampling methods. See R.J. Gettens, "A Microsectioner for Paint Films," *Technical Studies in the Field of the Fine Arts*, vol. 1, no. 1, 1932, pp. 20-28. When paint samples are extruded from a syringe, they have a tendency to break apart. Sometimes this problem can be overcome by direct insertion into a mounting medium.
34. One of the key contributors to the study of cross-sections of paintings has been Joyce Plesters of the National Gallery, London. See Joyce Plesters, "The Preparation and Study of Paint Cross-sections," *Museums Journal*, vol. 54, no. 4, 1954, pp. 97-101; Joyce Plesters, "Cross-sections and Chemical Analysis of Paint Samples," *Studies in Conservation*, vol. 2, no. 3, 1956, pp. 110-157. More recently, in David Bull and Joyce Plesters, *The Feast of the Gods: Conservation, Examination, and Interpretation*, *Studies in the History of Art*, no. 4, Monograph Series II (Washington: National Gallery of Art, 1990).
35. Several companies manufacture systems which make selection of the appropriate exciter filters and barrier filters convenient. The Leitz (Wetzlar) vertical fluorescence illuminator is based on a configuration developed by J.S. Ploem. It permits a selection of exciter filters which correspond to the ultraviolet, violet, blue, and green regions of the spectrum. Dichroic beam splitting mirrors and matched barrier filters permit selection of the appropriate visible light fluorescence. Zeiss, Olympus, and other manufacturers have similar systems.
36. See Richard Wolbers and Gregory Landrey, "The Use of Direct Reactive Fluorescent Dyes for the Characterization of Binding Media in Cross Sectional Examinations," *Preprints, Fifteenth Annual Meeting, American Institute for Conservation of Historic and Artistic Works*, Vancouver, 20-24 May, 1987 (Washington: American Institute for Conservation of Historic and Artistic Works, 1987), pp. 168-202.
37. Walter C. McCrone of the McCrone Research Institute, Chicago and his colleagues have developed techniques for studying pigments as well as many other classes of materials by microscopy. McCrone has also been a long time promoter of the use of the polarizing microscope in conservation science. A recent review of microscopical identification and properties of about 50 historically important pigments is Walter C. McCrone, "The Microscopical Identification of Artists' Pigments," *Journal of the International Institute for Conservation-Canadian Group*, vol. 7, nos. 1 & 2, 1982, pp. 11-34. Other references by McCrone et al. are given in the bibliography.

38. Eugene P. Bertin, *Principles and Practice of X-Ray Spectrometric Analysis*, 2nd edition (New York: Plenum Press, 1975).
39. X-ray fluorescence spectrometry (XFS) is frequently abbreviated as simply x-ray fluorescence (XRF); ARS had in use, for many years, a Philips Vacuum Spectrometer with the capability of producing a very fine x-ray beam (50 μm) and moving samples across the beam to determine element concentration as a function of depth in painting cross-sections. See John M. Taylor, "An Improved X-ray Macroprobe Technique for Cross-section Analysis of Museum Objects," Paper presented at the *10th Congress, International Institute for Conservation of Artistic and Historic Works*, Lisbon, 9-14 October 1972, 21 pp.; Nathan Stolow, James F. Hanlan, and Raymond Boyer, "Element Distribution in Cross-sections of Paintings Studied by the X-ray Macroprobe," *Studies in Conservation*, vol. 14, no. 4, 1969, pp. 139-151.
40. Terms used for the same method and instrument: electron microprobe; electron beam microanalysis; electron-probe microanalysis; electron-microprobe analysis; electron-probe microanalyzer (EPMA); electron-probe x-ray primary-emission spectrometer; electron probe analyzer (EPA).
41. The terms EDX and EDS are so common in the literature that the following quotation is included to try to clarify the terminology: "Historically, the technique of using diffraction to selectively analyse X-rays based on their wavelengths has been termed "dispersive". This label finds its basis in optics where dispersion is the process of deflecting the monochromatic components of a polychromatic beam through varying angles as a function of their wavelengths. Detectors not employing the diffraction technique are called "nondispersive". Until the advent of the Si(Li) detector, the energy resolution of nondispersive detectors was not adequate to provide an energy spectrum with usable element separation. In an attempt to emphasize the improved resolution of this new member of the nondispersive detector family, the Si(Li) energy spectrometer was termed "energy dispersive". This use of the word "dispersive" no longer fits with the original term from optics. It serves instead to indicate that the energy spectrometer sorts detected X-rays of different energies into an energy spectrum presentation with usable atomic-element separation. A more recent trend is to refer to the two types of spectrometers on the basis of the coordinate used to identify peak positions in the spectrum. In this scheme the dispersive, Bragg-diffraction instrument is called a "wavelength spectrometer" (WS), whereas the nondispersive Si(Li) detector system is termed an "energy spectrometer" (ES). In addition to the latter terms being less awkward, they are not in conflict with the correct meanings of the words "dispersive" and "nondispersive". Furthermore, they indicate the type of spectrum being produced. The WS and ES terminology will be used in the remainder of this article." From D.A. Gedcke "The Si(Li) X-Ray Energy Spectrometer for X-Ray Microanalysis," Chapter 12 in: *Quantitative Scanning Electron Microscopy*, Edited by D.B. Holt et al. (London: Academic Press, 1974), p. 407.
42. Radioisotopes in use are iodine-125, americium-241 and iron-55. Other sources such as gadolinium-153 and cadmium-109 have also been used for this purpose. Two x-ray spectrometer systems are currently in use. One uses a Gamma-Tech LS-15 lithium drifted silicon—Si(Li)—detector and a Tracor Northern NS-750 x-ray spectrometer. The other uses a Princeton Gamma-Tech IG 25 intrinsic germanium detector and a Tracor Northern NS-880 x-ray spectrometer incorporating a Digital Equipment Corporation PDP 11/05 minicomputer.
43. Our instrument is an Hitachi S-530 scanning electron microscope with a Tracor X-ray Microtrace Hypersense lithium drifted silicon detector. The spectrometer is a Tracor Northern TN-2010 with a Digital Equipment Corporation LSI 11/73 microcomputer. The system permits digital control of the electron beam scan.
44. A Philips PW 1130 x-ray generator and a cobalt tube (with an iron beta filter to eliminate the Co K_{β} line) have been used with a 114.6 mm diameter Debye-Scherrer x-ray diffraction camera and Gandolfi mechanism for small particulate samples and fragments. Samples are irradiated with cobalt K-alpha

radiation and patterns are recorded on CEA 25 film. Larger samples—mainly reference materials, large samples of corrosion etc.—were analyzed with a Philips PW 1050/76 diffractometer. Recently, ARS has acquired a Rigaku Corporation x-ray diffraction system consisting of a position sensitive proportional counter x-ray micro diffraction unit to provide greater precision and speed for small samples. For other samples, the system includes a traditional x-ray diffractometer (goniometer) and a port for Debye-Scherrer x-ray diffraction (cameras with Gandolfi mechanism).

45. For further information on the diamond anvil micro-sample cells, see Laver, Marilyn E. and Williams, R. Scott, "The Use of a Diamond Cell Microsampling Device for Infrared Spectro-photometric Analyses of Art and Archaeological Materials," *Journal of the International Institute for Conservation-Canadian Group*, vol. 3, no. 2, 1978, pp. 34-39.
46. Recently, ARS has acquired a Bomem MB-120 Fourier transform infrared spectrometer is integrated with a Spectra-Tech IR-Plan Research Microscope. The spectrometer is equipped with DTGS, MCT, and photoacoustic detectors. A Nicolet 5DX FTIR spectrometer is also in use. The Fourier transform infrared spectrometer is an integrated system of optical and computer components. As with all of today's modern laboratory equipment, data is obtained and stored in digital format which facilitates all data manipulation and interpretation processes. For a more complete discussion and comparison of dispersion and Fourier transform infrared spectroscopy see Low, M. J. D. and Baer, N. S., "Application of Infrared Fourier Transform Spectroscopy to Problems in Conservation. I. General Principles," *Studies in Conservation*, vol. 22, no. 3, 1977, pp. 116-128.
47. Elements which are often of interest with respect to their occurrence in pigments or alloys of historical interest are highlighted.

Periodic Table⁴⁷

	I																VIII	
1	H																He	
2	Li	Be								B	C	N	O	F		Ne		
3	Na	Mg								Al	Si	P	S	Cl		Ar		
4	K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
5	Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
6	Cs	Ba	La*	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
7	Fr	Ra	Ac**															
			*	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	
			**	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr	

Bibliography: Pigments

[References to a series of nine articles on pigments which were published in *Studies in Conservation*, and to ten chapters on pigments in the book *Artists' Pigments: A Handbook of their History and Characteristics*, Robert L. Feller, Editor, vol. 1 (Washington: National Gallery of Art, 1986) are listed in alphabetical order. These are part of a continuing international effort to publish research and reviews of artists' materials.]

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