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Author(s): E. Rene de la Rie

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FLUORESCENCE OF PAINT AND VARNISH LAYERS (PART I)

E. René de la Rie

Abstract—As part of a study of fluorescence of paint and varnish layers under ultraviolet light, the fluorescence of a number of pigments has been measured with a fluorescence spectrometer. Of the pigments studied, only zinc white, cadmium pigments and genuine madder appear to possess fluorescence of considerable intensity. The physico-chemical background of fluorescence in general and of fluorescence of pigments in particular is discussed.

1 Introduction

The ultraviolet lamp is a common tool for the examination of painted surfaces of art objects such

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as easel paintings and wall paintings [1–13]. Ultraviolet light excites fluorescence* of certain materials on the surface. Different materials may exhibit different colours and intensities of fluorescence, which can provide information on the condition of the paint surface. Later additions may be observed in this way, because new retouchings and overpaints often appear dark under ultraviolet light,

*As long as the nature of the emission of painting materials is not known it would be better to speak of *luminescence* since this term covers fluorescence as well as phosphorescence. However, the word fluorescence has been used ever since the ultraviolet lamp has been applied to the examination of pictures. Therefore, in this publication this term has been maintained. Moreover, most of the investigated emissions turned out to be fluorescence.



Figure 1 Oil painting in normal light (a) and in ultraviolet light (b). The strong fluorescence of the varnish layer is clearly visible. At the lower part of the inner oval the portrait was presumably partly cleaned, resulting in a less strong varnish fluorescence. The strong fluorescence of white lead containing oil paint (collar, hair and flesh colours) appears through the varnish layer. The dark spots in the collar are retouchings. Portrait of Casper Barlaeus, probably a seventeenth century copy of a miniature by Gerard ter Borch. Oil paint on panel, 17 × 12.5cm. Published by courtesy of the University of Amsterdam. Photography by E. Klusman, Central Laboratory.

whereas old paint layers may show considerable fluorescence. Old varnish layers generally fluoresce strongly; thus, the use of an ultraviolet lamp during their removal can be helpful (Figure 1). For documentary purposes, fluorescence is often recorded photographically [12–15].

The ultraviolet lamp has been applied to the examination of old pictures ever since it became commercially available around 1925. Oddly enough, no studies of interest on the fluorescence of painting materials have been published since the pioneer works of Eibner and others appeared around 1930 [2–4, 16–20]. Because the fluorescence of painted surfaces can be very complex and little is known about its nature, the fluorescence spectra of painting materials and picture surfaces were chosen as a field of study at the Central Research Laboratory for Objects of Art and Science in Amsterdam. Since the range of materials that could be studied had to be limited, it was decided first to aim at a greater insight into the fluorescence of oil paintings. For this purpose the following materials were studied: linseed oil, some natural resins, pure pigments and pigments ground with linseed oil. The fluorescence of some picture surfaces was also measured, and in some cases measurements were made before and after cleaning. Although this study cannot be considered exhaustive, the results obtained are worthy of publication. The measurements on pigments will be discussed in this paper while the other materials will be dealt with in subsequent articles. As the theory of fluorescence does not seem to be widely understood in the field of conservation, a few remarks may be made on this subject before dealing with the results of this study. For detailed information on fluorescence in general, however, the reader is referred to the literature [21].

2 The phenomenon of fluorescence

When electromagnetic radiation, such as ultraviolet light, impinges upon matter, part of it can be absorbed, causing electronic transitions in some of the molecules of the material. Such *excited* molecules rapidly lose their excess electronic energy by conversion into other forms of energy, such as vibrational energy, or it may be partly emitted as radiation. Simplified, this process can be described as follows (see also the diagram in Figure 2). Most molecules have a singlet ground state (S_0), that is, a ground state with paired electron spins, and absorption takes place from this ground state to a singlet excited state (S_1 , S_2 , etc.). Superimposed on these electronic states are the vibrational states. Normally, an excited molecule returns immediately and without emission of radiation to the lowest vibrational

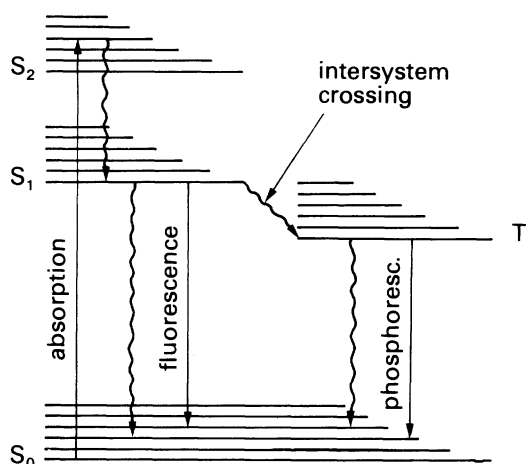


Figure 2 Scheme showing absorption and emission processes. \rightsquigarrow = radiationless transition. This diagram is highly simplified. The radiationless transitions do not occur in one step as might be concluded from the picture. See the text for an explanation.

state of the first excited singlet state S_1 (within about 10^{-12} sec). Transition from S_1 to S_0 may take place through the emission of radiation, the process being called *fluorescence* (occurring within about 10^{-8} sec).

Because of this, fluorescence is always of longer wavelength than the absorbed radiation (Stokes rule). Moreover, fluorescence in one compound takes place at a fixed wavelength, independent of the wavelength of the radiation that is absorbed.

It should be pointed out that most substances do not fluoresce, which implies that the transition S_1 – S_0 can also occur without the emission of radiation. A third possibility occurs when so-called 'intersystem crossing' from S_1 to T_1 , the first excited triplet state (a state with parallel electron spins), takes place. This state is between S_1 and S_0 . From this triplet state, too, the molecule returns to the ground state S_0 , without or with the emission of radiation, which is then called *phosphorescence*. The process of phosphorescence is much slower than that of fluorescence and can last up to several seconds or even longer, resulting in a visible afterglow.

Most fluorescent compounds are organic compounds; in inorganic compounds fluorescence is a rare phenomenon.

In organic molecules π -electrons and, occasionally, nonbonding (n-) electrons, especially those in conjugated and aromatic systems, are the ones involved in absorption and emission processes in the near ultraviolet (above 200nm) and visible region. Phosphorescence only occurs in rigid media, that is at low temperatures, since at room temperature the

energy of the triplet state is readily lost to surrounding molecules.

Inorganic crystalline compounds may show fluorescence or phosphorescence, although this is very often associated with irregularities in the crystal structure such as vacancies and impurities. These act as luminescence centres, and can be created on purpose in the manufacture of phosphors. These phosphors are used, for example, in fluorescent lamps.

Strong fluorescence occurs in a great number of metal chelates, which are inorganic ions combined with organic ligands. The formation of these fluorescent chelates is used in analyzing many inorganic ions.

Fluorescence, if in the visible part of the electromagnetic spectrum, can be observed with the naked eye. It must be clear, however, that for more precise information the fluorescence spectrum should be recorded.* Descriptions of fluorescence spectrometers can be found in the literature [21]. Basically, they are composed of a unit which produces monochromatic exciting radiation and a unit which analyses the fluorescent light. The equipment used in this study is described under the heading 'Experimental'. Recorded fluorescence spectra are distorted by different sensitivities of the detection system at different wavelengths. Fluorescence spectra, therefore, generally are reproduced with the notation 'corrected' or 'uncorrected'.

3 Results of measurements on pigments

Although fluorescence colours of a large number of pigments have been described by some authors [20, 22], only a few pigments with a fluorescence of considerable intensity were found during the present study. These pigments are: zinc white, cadmium yellows, oranges and reds and genuine madder. All other pigments that were studied lacked fluorescence or showed a very weak fluorescence. These pigments include: ochres, siennas, umbers, verdigris, copper resinate, minium, bone black, viridian, malachite, azurite, Prussian blue, green earth, white lead and titanium white.†

The fluorescence of zinc white is yellow-green. The spectrum consists of a broad, unstructured band (Figure 3a). Between samples of various origin little

*It must be emphasized that many hand-lamps available for fluorescence analysis are equipped with unsuitable filters that transmit a considerable portion of the visible light (mainly violet) coming from the lamp. This greatly influences the fluorescence colour or gives the impression of fluorescence where it does not exist.

†Generally, no analysis tests have been done on these pigments. The list is based on data from manufacturers and labels on old pigment samples in a collection of the Central Laboratory.

difference could be discovered. A sample of reagent grade zinc oxide in practice fluoresces in much the same way as the zinc white samples of various origins. On some picture surfaces, however, a fluorescence originating from zinc white has been found which has a considerably higher intensity than that of the samples.

The fluorescence of cadmium pigments falls in the red and infrared part of the electromagnetic spectrum.* The fluorescence spectra consist of relatively narrow bands (Figures 3b, c). The bands shift with the colour of the pigments; those of the cadmium reds lie at longer wavelengths than those of the oranges, which occur at longer wavelengths than those of the yellows. However, in samples of different origin, different fluorescence maxima and marked variation in the intensities of the fluorescence were found. Moreover, some cadmium pigments as well as reagent grade cadmium sulphide did not fluoresce. The compositions of the zinc whites and the cadmium pigments were checked by X-ray fluorescence and X-ray diffraction analysis (Table 1).†

Genuine madder, prepared from the madder root, fluoresces with a red colour. The spectrum consists of a relatively narrow band (Figure 3d). Among samples of various origin, small differences in the wavelength of the fluorescence maxima and great differences in the fluorescence intensities were found. In order to obtain some clarity in the fluorescence of madder a few tests were done with reagent grade alizarin and purpurin. Both did not fluoresce as such. However, purpurin solutions in ethanol, diethyl ether and acetone fluoresced. When a drop of such a solution was applied to filter paper, a fluorescent spot remained after evaporation of the solvent. Also, a weak fluorescence was observed in a solution of purpurin in 0.1M NaOH. When aluminium hydroxide was added to this deep red coloured solution, the fluorescence increased somewhat. Subsequent lowering of the pH by the addition of dilute hydrogen chloride caused an initial disappearance of the fluorescence. However, at a pH of about 3 an orange-coloured and strongly fluorescent suspension was formed. All these tests gave negative results as far as fluorescence is concerned when they were done with alizarin.

To date there has been no indication that changes occur in the course of time in the fluorescence characteristics of pigments. The fluorescence spectra

*The wavelength area in which the emissions of the cadmium pigments fall explains the results obtained when photographing this fluorescence with infrared sensitive film [23].

†The luminescence of zinc white and the cadmium pigments had lifetimes in the order of 100 μ sec. These are the only long-living emissions that were found during this study.

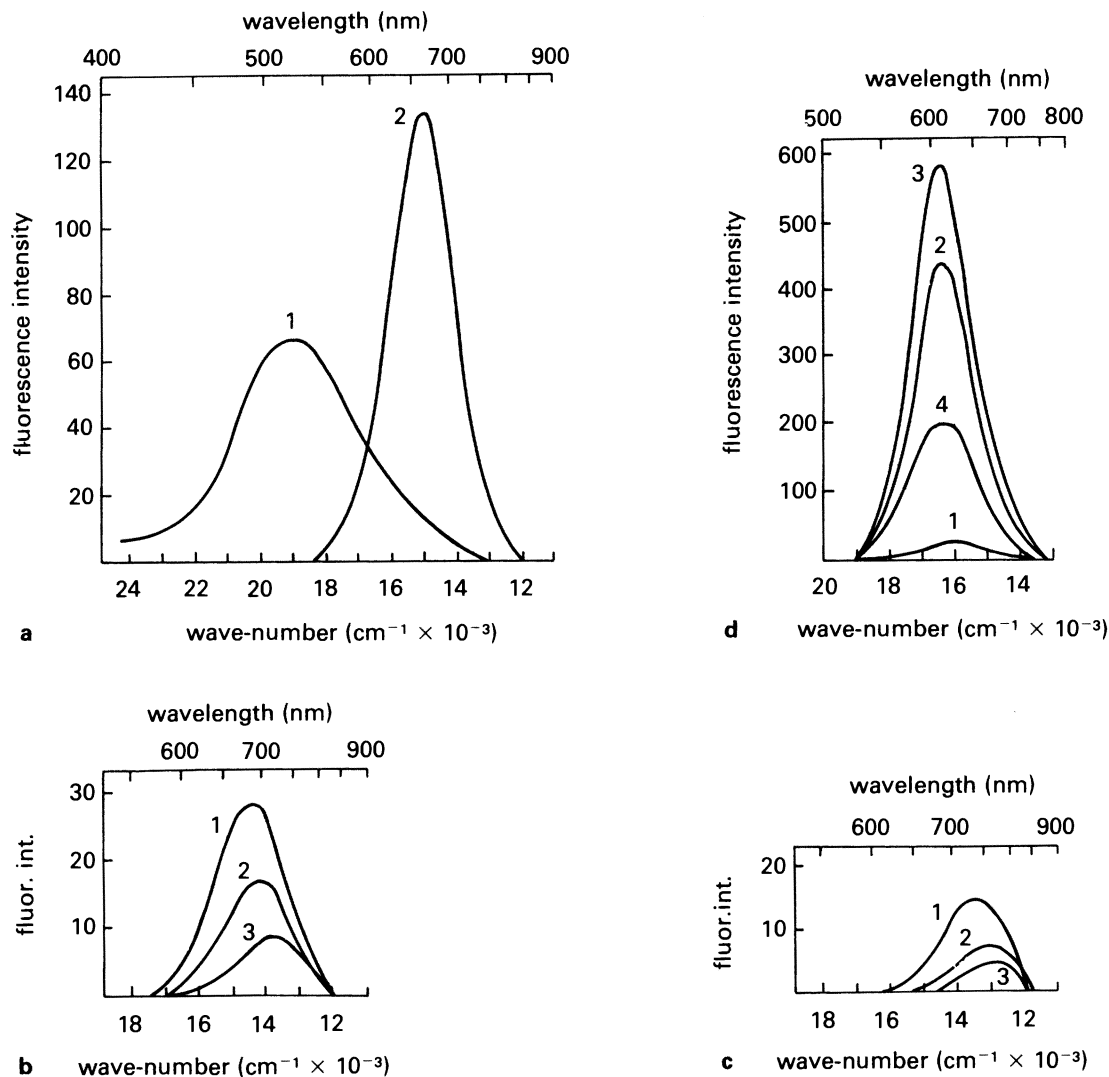


Figure 3 Fluorescence spectra of pigments: (a) zinc white from van der Linde, Amsterdam (1) and cadmium yellow lemon from the Oudt Hollandse Olieverven Makerij (OHOM), Scheveningen, Holland (2); (b) cadmium yellow light (1), middle (2) and dark (3) from OHOM; (c) cadmium orange (1), cadmium red light (2) and dark (3) from OHOM; (d) rose madder (1) and pink madder (2) from C. Roberson & Co. Ltd, London, 1935, madder lake prepared by A. P. Laurie (3) and rose madder prepared by M. Cowell (4). (All the madders were kindly supplied by the Courtauld Institute of Art in London.) Wavelength of excitation 365nm.

of the pigments are independent of the wavelength of excitation, which means that pigments fluorescing at long wavelengths—madder and cadmium pigments—can also be excited by visible light.

4 Discussion

It is not surprising that most pigments, being inorganic compounds for the greater part, do not fluoresce. The question arises as to why some of

them do show fluorescence.

The main component of zinc white is zinc oxide (ZnO); cadmium yellow, orange and red mainly consist of cadmium sulphide (CdS) or cadmium selenium sulphide, a complex compound of cadmium sulphide and cadmium selenide (CdS/CdSe) (Table 1). Zinc oxide and cadmium sulphide belong to a class of compounds that are known to be made easily luminescent [24]. In pure form the crystals do not fluoresce. Very small quantities of impurities

Table 1 Compositions of cadmium pigments and zinc whites

Pigment	Origin ¹	X-ray fluorescence ²	X-ray diffraction ³	Fluorescence ⁴
Cadmium yellow lemon	a	Cd +++ Zn ++	CdS-g ZnS	++
Cadmium yellow light	a	Cd +++ Zn +	CdS-g BaSO ₄	+
Cadmium red light	a	Cd +++ Se +++	CdS/CdSe? BaSO ₄	-
Primrose cadmium	b	Cd +++ Zn +	CdS-h CdCO ₃	-
Cadmium dark	b	Cd +++ Zn + Pb +	CdS-h ZnS	-
Cadmium yellow lemon	c	Cd +++ Zn +	CdS-g BaSO ₄	-
Cadmium yellow lemon	d	Cd +++ Zn +	CdS-g BaSO ₄ ZnS	+
Zinc white	a	Zn +++	ZnO-z	+
Zinc white	e	Zn +++	ZnO-z	+

Notes

¹ a = Oudt Hollandse Olieverven Makerij, Scheveningen, Holland; b = L. Cornelissen & Son, London, England, ± 1935 (kindly supplied by the Tate Gallery, London); c = Maimeri, Milan, Italy; d = Schminke, Düsseldorf, West Germany; e = Van der Linde, Amsterdam, Holland.

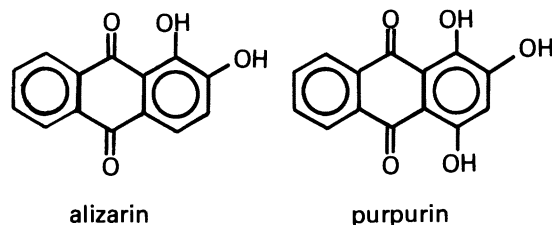
² Results of X-ray fluorescence analysis. The relative amounts of the elements were roughly determined from the peak heights: +++ = much, ++ = average, + = little.

³ Results of X-ray diffraction analysis: CdS-g = cadmium sulphide, greenockite; CdS-h = cadmium sulphide, hawleyite; ZnO-z = zinc oxide, zincite. Since no reference data are known for cadmium selenium sulphide, the result is given with a question mark.

⁴ Fluorescence under ultraviolet or visible light: ++ = strong, + = medium, - = weak or not present.

make the compounds strongly fluorescent. No regular differences appear in the composition of the fluorescent and the non-fluorescent cadmium pigments. However, impurities at concentrations much below those that can be detected by X-ray fluorescence analysis can make cadmium sulphide fluorescent.

The fluorescence of genuine madder is of a different nature. Its main colouring matter is alizarin, although other compounds, of which purpurin is the most important, are present as well. Alizarin and purpurin are organic dyestuffs of the hydroxy-anthraquinone family.



Lake pigments are prepared by precipitation of the dyestuffs on an inorganic base such as calcium sulphate, barium sulphate or aluminium hydroxide.

Since the end of the last century alizarin has also been prepared synthetically. Because pigments prepared with alizarin do not show fluorescence, it has been stated that the fluorescence in madder originates from purpurin [2]. The tests of this study reveal that purpurin is able to fluoresce when combined with other substances, such as a solvent, a paper substrate or an inorganic base such as Al(OH)₃. The fluorescence of genuine madder, therefore, most likely originates from purpurin chelates. Metal chelates of hydroxy-anthraquinones are known for their ability to produce a strong and pH-dependent fluorescence [21b]. The observed differences in the fluorescence spectra of madder samples of different origin might well be caused by the use of different bases and by varying purpurin content.

5 Experimental

Observation of fluorescence is usually done at an angle of 90° relative to the incident light. This is the best arrangement to prevent scattered exciting light entering the detection system when working with solutions, but another arrangement must be made when working with solid samples. For this research

an arrangement was used which allowed observation along the normal of the surface of the sample while the incident light was at 30° to this normal (Figure 4).

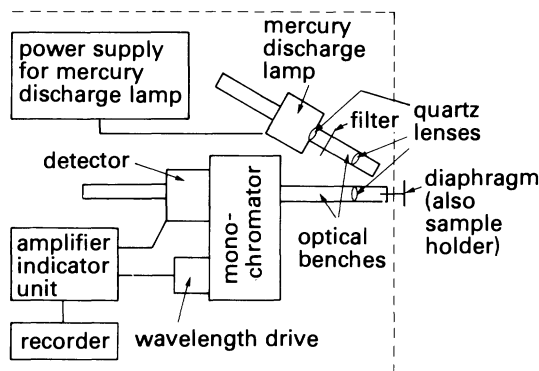


Figure 4 Scheme of the fluorescence spectrometer in the Central Laboratory.

The apparatus was built up with units of the Zeiss PMQ3 fluorescence spectrometer.* A medium-pressure mercury lamp was applied as a light source, which yields a line spectrum with the principal lines at 313, 365, 405, 436, 546 and 578nm,† one of which could be selected by means of an Oriel interference filter or a Zeiss monochromatic filter. The light was focused with quartz lenses on to an opening in a vertical plate (diaphragm and sample holder at the same time). An opening size of 9 × 3mm was usually used, but measurements of smaller areas were possible. Since the plate projected somewhat beyond the edge of the table on which the apparatus was placed, any spot on an object could be brought before the opening. A frame specially built to hold paintings was attached to the table. By means of a lens, part of the diffuse fluorescence was focused on the entrance slit of a quartz prism monochromator. The detector behind this monochromator (a Hamamatsu R446 photomultiplier) was sensitive from 200–850nm. The apparatus further consisted of an amplifier-indicator unit, a wavelength drive and a recorder.

*Manufactured by Carl Zeiss, 7082 Oberkochen, West Germany.

†The portable mercury lamps available for fluorescence analysis generally contain a filter which transmits the 365nm line. Often, however, and especially for photographic purposes, high-pressure lamps are used as well. These lamps yield, in addition to the line spectrum, a continuous spectrum, and give off light between approximately 300 and 400nm. Fluorescent lamps for ultraviolet examination also produce ultraviolet light in this same broad wavelength area. Low-pressure mercury lamps are not useful for the examination of paint and varnish layers, since they mainly yield radiation of 254nm, which is outside the wavelength area in which excitation of fluorescence occurs in painting materials.

One madder sample was used as a standard throughout the study, to which the apparatus was calibrated before each measurement. The fluorescence spectra reproduced in this publication are noted in arbitrary intensity units and are uncorrected.

For the determination of the fluorescence spectra the pigments were put into quartz cuvettes which were tapped on the table in order to pack down the pigment particles. This was continued until a constant fluorescence signal was obtained. The cuvettes were held against the plate by means of a clamp.

Tests for phosphorescence were done at the Laboratory of Organic Chemistry, University of Amsterdam, with a Spex Fluorolog.*

X-ray fluorescence and X-ray diffraction data were obtained with equipment housed in the Central Laboratory [25].

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*Manufactured by Spex Industries Inc., 3880 Park Avenue, Metuchen, NJ 08840, USA.

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E. RENÉ DE LA RIE studied physical chemistry at the University of Amsterdam; joined the Central Laboratory in Amsterdam to work on a subsidized research project; worked during the last few years for a newly established training programme for conservators in Amsterdam; and recently joined the Paintings Conservation Department of the Metropolitan Museum of Art in New York as research chemist. *Author's address: Metropolitan Museum of Art, Paintings Conservation Department, Fifth Avenue at 82nd Street, New York, NY 10028, USA.*

Résumé—Dans le cadre d'une étude portant sur la fluorescence des couches de peinture et de vernis sous lumière ultraviolette, on a mesuré la fluorescence d'un certain nombre de pigments à l'aide d'un spectromètre de fluorescence. De l'ensemble des pigments examinés, seuls le blanc de zinc, les pigments au cadmium et la laque de garance ont une fluorescence très importante. On trouvera discuté la physico chimie de la fluorescence en général et celle des pigments en particulier.

Zusammenfassung—Als Teil einer Untersuchung der Fluoreszenz von Farb- und Lackschichten unter ultraviolettem Licht wurde eine Anzahl von Pigmenten mit einem Fluoreszenz-Spektrometer gemessen. Von den untersuchten Pigmenten scheinen nur Zinkweiß, Kadmiumpigmente und echter Krapp Fluoreszenz von beachtlicher Intensität zu besitzen. Der physikalisch-chemische Hintergrund von Fluoreszenz im allgemeinen, und von Fluoreszenz der Pigmente im besonderen wird diskutiert.