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# Multispectral Analysis of Paper Objects – The Amalgamation of Archaeometry and Conservation

**Abstract:** This paper is dedicated to the material science analysis of drawings using imaging techniques commonly referred to as multispectral analysis. Multispectral here means examination with electromagnetic radiation from the range of UV, visible and NIR light. Radiation diagnostics are extended by the use of X-ray fluorescence analysis which uses distribution images to depict the spatial arrangement of characteristic elements on the drawing surface. Such analyses not only provide general insights into the type and composition of the materials used; in the field of drawing research insights into the genesis of the work can be gained by locating preliminary drawings, determining revisions and corrections, while sometimes it is also possible to draw conclusions about the provenance of the pencils, crayons or inks used on the basis of characteristic trace elements. Furthermore, multispectral analysis provides essential insights when examining palimpsests in order to make erased or deleted parts visible. The determination of the colorants possibly allows dating and thus enables the detection of later copies or even forgeries. The non-invasive examinations provide a comprehensive insight into the materiality of paper objects and thus form the basis for the development of sustainable conservation and restoration campaigns.

## 1 Introduction

Drawing has been established as an independent art genre alongside painting since the early modern period. Drawings are not only a sketch or draft but also a work of art in their own right. As with paintings, questions of original, copy, or replica can be answered not only by cultural-historical but also material-scientific investigations. In his standard work on drawing analysis Joseph Meder points out the impor-

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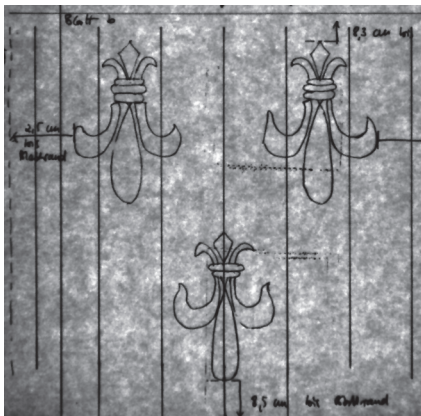
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tance for interpretation of knowing the materials used in drawings and emphasises the scope and versatility of the drawing materials used.<sup>1</sup>

In this article the primary question to be addressed is to what extent material-scientific investigations can contribute to – more art-scientific – connoisseurship.<sup>2</sup> Knowledge of art history and methods of material science combined will possibly constitute a higher level of connoisseurship. It is undisputed that materials – including drawing materials – change over time. As early as the late eighteenth century the colour change in iron gall inks was pointed out: inks with too much iron vitriol turn yellow to red while the more stable inks mixed with too many gallstones soon turn brown.<sup>3</sup> It can therefore be assumed that the originally black iron gall inks have only partially retained their original colour and have browned. Damage caused by ink corrosion or wood pulp paper is far more serious – here, material analysis plays an important role in preserving the cultural heritage.

## 2 Methods

Dating and proving the authenticity of a work are classic questions addressed to material analysis. If the substrate is paper analysis of the watermarks (Figure 1) and documentation of the paper structure are essential starting points for a temporal and spatial classification of the medium which in turn provides clues for dating or



**Fig. 1:** Characteristic watermarks from Germany, three fleurs-de-lis without further designation, Joh. Michael Stoß (1738–1821), Arnstadt, paper mill. Watermark Collection DNB/DBSM/WZ, reference number DE4815-DNB-L-WZ-0000523-wm1. Image: German National Library

<sup>1</sup> Meder 1919, 33.

<sup>2</sup> Ketelsen and Hahn 2022, 289.

<sup>3</sup> Ribeaucourt 1797, 28.

attribution to an artist or workshop.<sup>4</sup> Whereas in the past, until the 1980s, elaborate stationary procedures were used to reveal watermarks<sup>5</sup> today a diverse and variable set of instruments is available that allows not only watermarks but also the structure of the paper to be digitised and thus made available for comparative research.<sup>6</sup>

Characterising the drawing materials used is far more complex. In addition to questions of age and authenticity the focus here is more on explaining the genesis of a drawing. How and with what material was the drawing or copy made? What ink was used for the final drawing? Can revisions, corrections, or possible later revisions be made visible? Can connections be drawn between the drawing and possible inscriptions? For a first approach to these questions looking through a microscope to classify the materials is very helpful – but an exact material characterisation cannot be done with the naked eye.<sup>7</sup> Detecting trace elements, which provide clues to the provenance of a mineral drawing material (Figure 2),<sup>8</sup> and determining minor components in iron gall inks enabling the differentiation of the inks by determining the so-called fingerprint (Figure 3)<sup>9</sup> require more elaborate material-scientific examinations. Finally, there are imaging methods which are becoming increasingly important. These methods, nowadays referred to as multispectral imaging analysis (MSI), are derived from infrared reflectography (IRR) which was developed in the 1960s to reveal preliminary drawings<sup>10</sup> and are increasingly becoming part of today's archaeometric and restorative research due to their diverse analytical possibilities in combination with effort and costs.

Most of the analytical procedures can be summarised under the term radiodiagnostic examinations. In such procedures light of a certain wavelength or energy interacts with the matter to be examined. In this process, known as excitation, portions of energy are transferred,<sup>11</sup> orders are briefly disturbed and individual particles are removed from groups. The interaction depends on the type of excitation light – visible (VIS), ultraviolet (UV), infrared (IR), lasers of different wavelength excitation (Raman), or X-ray light (X-Ray) – and the structure or composition of the material under investigation. After a short disturbance the system returns to its

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<sup>4</sup> Dietz 2011, 281.

<sup>5</sup> Schoonover 1987, 154.

<sup>6</sup> Dietz and Wintermann 2015, 19.

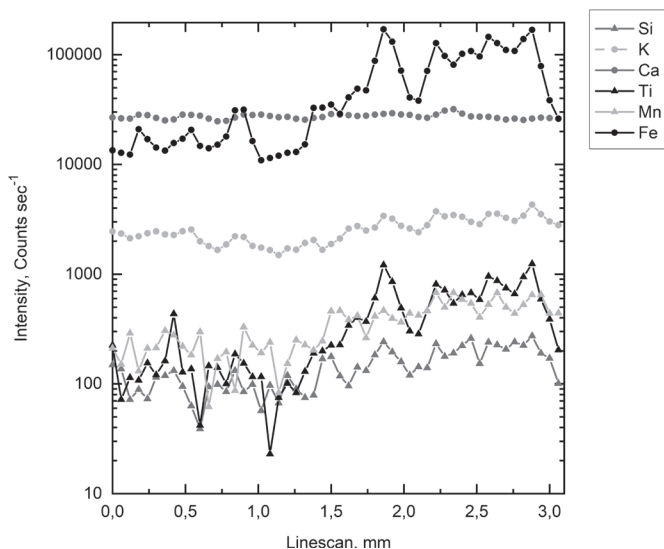
<sup>7</sup> Wintermann and Melzer 2022, 156.

<sup>8</sup> Nöller 2012, 61.

<sup>9</sup> Malzer et al. 2004, 229.

<sup>10</sup> Asperen de Boer 1972.

<sup>11</sup> In most cases, this energy transfer occurs from the exciting light to the material under investigation.



**Fig. 2:** Element scan of red chalk on paper carried out on a line by means of X-ray fluorescence analysis. From the element courses it can be concluded that red chalk not only contains the main component iron (Fe), but also other components such as silicon (Si), potassium (K), titanium (Ti) and manganese (Mn), which correlate with the iron. Only the course of calcium (Ca) is constant – it derives from the paper. Image: Federal Institute for Materials Research and Testing

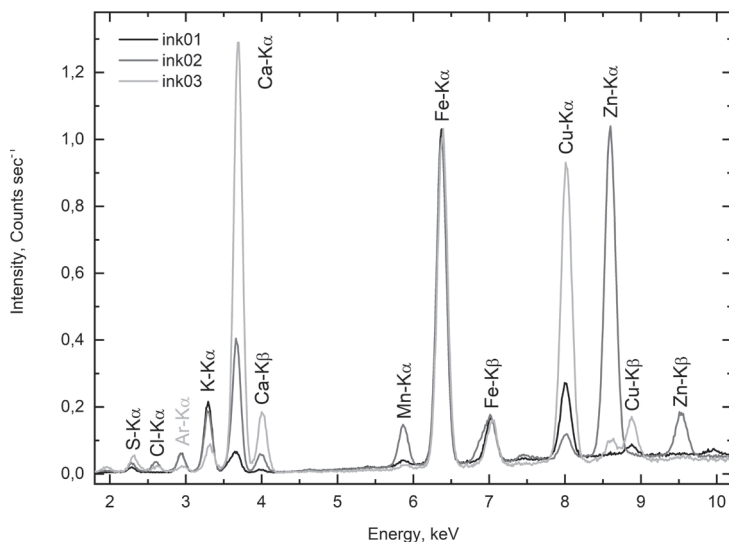
original ground state. This relaxation process leads to the release of characteristic radiation which, interpreted as a spectrum, provides information about the type and composition of the material. In this context the term spectroscopy still needs to be explained: it refers to scientific procedures that differentiate radiation according to a certain property.<sup>12</sup>

Many of the radiation diagnostic examination methods are named after the type of excitation radiation used. In this article we will not describe all methods in detail.<sup>13</sup> Only X-ray fluorescence analysis, infrared reflectography and Raman spectroscopy will be briefly touched upon here since these methods are indispensable as basic technologies in the material-scientific analysis of written material and graphics. It should be particularly emphasised in this context that these methods are non-invasive, i. e. non-destructive, methods of analysis that do not require the removal of samples.

<sup>12</sup> These properties include wavelength or energy.

<sup>13</sup> Detailed descriptions can be found, for example, on infrared spectroscopy in diffuse reflection in Steger et al. 2018, 103 and on Raman spectroscopy in Centeno 2016, 9.





**Fig. 3:** X-ray fluorescence analysis of three iron gall inks with different compositions. Besides the main component iron (Fe), other components such as potassium (K), manganese (Mn), copper (Cu) and zinc (Zn) can be detected. The element calcium (Ca) results from the paper substrate. Image: Federal Institute for Materials Research and Testing

## 2.1 X-Ray Fluorescence Analysis (XRF)

X-ray fluorescence analysis is one of the standard methods for determining elemental compositions and is an integral part of archaeometric investigations.<sup>14</sup> The material to be examined is irradiated with X-rays. Due to the interaction between X-ray light and matter, individual electrons are knocked out of their original environment in the atom. The atoms, excited in this way, return to their ground state while emitting X-ray light, with electrons from shells further filling out the hole created. This relaxation process is associated with the emission of characteristic radiation because each atom has a special electron configuration – i. e. a structure of its electron shells. The height of the X-ray peaks generally obtained allows conclusions to be drawn about the quantity of the element contained.<sup>15</sup> Usually the examinations are carried out as individual measurements or line scans. An X-ray fluorescence portal scanner enables an entire drawing in the area to be analysed.

<sup>14</sup> Bronk et al. 2001, 307.

<sup>15</sup> Hahn-Weinheimer et al. 1995, 70.

To perform the XRF scans presented here the Jet Stream from Bruker Nano GmbH with a rhodium X-ray tube was used. During the scan the measuring head containing the excitation tube (Rh, 50 kV, 0.6 mA) and the detector (Xflash™ detector) is moved over the object at high speed (diameter of the excitation beam: 50  $\mu\text{m}$ , step size: 25  $\mu\text{m}$ ; measuring time: 2 ms per measuring point). A complete mapping of the elements within the different drawing sections is obtained. The device is designed to measure in air. The penetration depth of the radiation is between  $10^{-6}$  and  $10^{-1}\text{ cm}$  depending on the matrix.

## 2.2 Raman Spectroscopy (Raman)

For the chemical analysis of many organic and inorganic materials Raman spectroscopy has been a common method alongside infrared spectroscopy for years. In contrast to investigation methods for element-specific characterisation, vibrational spectroscopy exploits the effect of electromagnetic radiation on atomic bonds.

If molecules are irradiated with monochromatic light (e. g. laser light) the irradiated light is scattered. This scattering is based on a transfer of energy from irradiated light to the molecules and molecular lattices, which are always vibrating, or the reverse process.

After decomposition of the scattered light additional spectral lines appear next to the intense spectral line of the primary beam of the light source which are shifted with respect to the frequency of the light source. The latter lines are called Raman lines. Raman spectroscopy is used in particular for the investigation of minerals and pigments.

The investigations were carried out with a Raman spectrometer from Renishaw. The instrument was specially designed for archaeometric applications and is equipped with glass fibre optics. The excitation is done with a laser of 785 nm wavelength. The spectral range is 3600 to  $100\text{ cm}^{-1}$ . The maximum spectral resolution is  $4\text{ cm}^{-1}$ .<sup>16</sup>

## 2.3 Infrared Reflectography (IRR)

Infrared reflectography, which was first used to examine paintings in the 1960s, is based on the characteristic interaction between infrared light and the object to be examined. The object is irradiated with infrared light (IR) and an infrared-sensitive

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<sup>16</sup> Centeno 2016, 9.

camera captures an image of the radiation not visible to the human eye. The substances absorb the IR light in a way that is characteristic of their chemical composition. Substances containing elemental carbon appear black in the IR image while other materials – for example colourants – appear transparent. With this method preliminary drawings in paintings can be successfully made visible again if they were executed in charcoal or black chalk.

The method was further developed by the introduction of band-pass filters which split the infrared light into defined sub-areas. The band-pass filters are placed in front of the infrared camera so that it always detects only a discrete section of the IR light. A continuous change of the band-pass filter allows a much more detailed determination of the absorption behaviour. This makes it possible to clearly differentiate between different writing and drawing materials such as soot inks, iron gall inks and plant inks.<sup>17</sup> Further diversification within a type of writing material is possible only to a limited extent with this method since processes that have occurred in the course of the transmission – such as restorations – falsify the picture.<sup>18</sup>

Infrared reflectography and the band-pass filter infrared reflectography that emerged from it are fundamental for the development of multispectral analysis, which extends the range of excitation light beyond the visible light to UV light.

### 3 Case Studies

As already mentioned at the beginning of this chapter, the result of the scientific analysis is based on the interaction – the interplay – between stimulating light and illuminated material. And so the comparison of the two sheets *Rebecca and Eliezer at the Well* (c. 1640), originally attributed to Rembrandt (1606–1669), and *The Old Man Reading* (c. 1630–1631) by Ferdinand Bol (1660–1680) shows the influence of materiality on the visualisation of preliminary drawings or underdrawings. The material-scientific characterisation of the drawing *The Levite and the Dead Concubine* (c. 1650–1652) provides information about corrections and revisions that the drawing has undergone. Furthermore, examining the two drawings *Handing of the Keys to Peter* and *The Striding Male Figure* makes it possible to distinguish between charcoal and mineral chalk. The possibilities of colour analysis will be explained using the example of two compositions by Andor Weininger (1899–1986).

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<sup>17</sup> Mrusek et al. 1995, 68.

<sup>18</sup> Different quantities of one and the same prescribed material as well as different paper substrates can also lead to a misinterpretation of the results. Here, further investigations are necessary, such as the already mentioned element analysis using the fingerprint model.



**Fig. 4:** *Rebecca and Eliezer at the Fountain*. Copy after Rembrandt Harmenszoon van Rijn? Klassik Stiftung Weimar, Graphische Sammlungen, Inv. no. KK 5511, ID 447184. Image: Klassik Stiftung Weimar, Graphische Sammlungen, photo: Carsten Wintermann

### 3.1 Recovery of Underdrawings

The first sheet shows *Rebecca and Eliezer at the Fountain*, originally attributed to Rembrandt (Figure 4). The brown-toned colour impression suggests that the drawing was executed with iron gall ink. A more precise visual analysis with the microscope reveals indications of a very precise preliminary drawing, which was obviously executed with a dry drawing material. The comparison of the infrared reflectography (Figure 5) with the original drawing illustrates the accuracy of the preliminary drawing; even the contours of the broad ink strokes are pre-drawn. Infrared reflectography cannot differentiate whether this is charcoal which consists solely of pure carbon or, for example, stone chalk which contains additional mineral components;<sup>19</sup> an elemental analysis is necessary here.<sup>20</sup> However, the analysis

<sup>19</sup> At this point it should be noted that a microscopic observation could be used to differentiate. The sharp-edged mineral components can produce scratch marks in the abrasion of the stone chalk; these are missing in the stroke of the charcoal.

<sup>20</sup> Further analysis using X-ray fluorescence analysis suggests bone black.



**Fig. 5:** *Rebecca and Eliezer at the Fountain*. The carbonaceous preparatory material is clearly visible in infrared reflectography, while the overlying iron gall ink disappears. Image: Klassik Stiftung Weimar, Graphische Sammlungen, photo: Carsten Wintermann

suggests that this is a copy that has been transferred with the carbon-containing pigment.

In the case of the *Reading Old Man* by Ferdinand Bol (Figure 6) it is quite the contrary.<sup>21</sup> The naked-eye view shows that in the area of the head, especially in the beard and on the back of the old man, a sketchy pen drawing was executed with light brown ink. Most of the drawing – the face, the coat and the book the old man is reading as well as the chair and the stone wall in the background – was executed with a brush in grey and black. In addition, there are ornamental details in red chalk and revisions in opaque white. The exact interplay of pen and brush, i. e. preliminary drawing, execution and final reworking, is not clearly discernible at first.<sup>22</sup>

Only through material analysis does the genesis of the sheet become understandable. For example, X-ray fluorescence analysis which was carried out as an element scan and makes its distributions on the sheet visible clearly shows a prelim-

<sup>21</sup> Ketelsen et al. 2021, 498.

<sup>22</sup> Hahn 2022a, 117.





**Fig. 6:** *Reading Old Man*, Ferdinand Bol, c. 1630–1631, pen and brown ink, brush and grey and black ink, red chalk, opaque white. Klassik Stiftung Weimar, Graphische Sammlungen, Inv. KK 5496, ID 502508. Image: Klassik Stiftung Weimar, Graphische Sammlungen, photo: Carsten Wintermann

inary drawing in iron gall ink: the underdrawing for the entire work (Figure 7). The spatial heterogeneity of the preparatory drawing which gives an indication of the templates used for the motif will not be discussed here.<sup>23</sup> This preparatory drawing could not have been made visible with infrared reflectography since substantial parts of the drawing were executed with carbonaceous pigments that overlay the preparatory drawing in iron gall ink.

<sup>23</sup> Ketelsen et al. 2021, 499.



**Fig. 7:** *Reading Old Man*. The element distribution of iron (Fe) determined with an X-ray fluorescence scanner indicates a signature with an iron-containing material, iron gall ink. Image: Klassik Stiftung Weimar, Graphische Sammlungen, graphics: Uwe Golle

The two case studies concerning the visualisation of preparatory drawings document impressively that there is no universal method for a specific question from the field of connoisseurship that can be applied in principle. This may sound trivial at first but it is a significant fact. The diversity of drawing materials – soot inks and iron gall inks, metal, graphite and coloured pencils, charcoal and crayons, as well as inorganic pigments and organic dyes – requires an equal diversity of material science methods. Only the interaction of the individual findings provides the overall context of the drawing.



**Fig. 8:** *The Levite and the Dead Concubine*, originally attributed to Rembrandt Harmenszoon van Rijn, c. 1650–1652, Klassik Stiftung Weimar, Graphische Sammlungen, Inv. no. KK 5497, ID 502523. Image: Klassik Stiftung Weimar, Graphische Sammlungen, photo: Carsten Wintermann

### 3.2 Corrections and Pentimenti

The drawing *The Levite and the Dead Concubine* (Figure 8), previously attributed to Rembrandt, has several corrections whose design poses a mystery. First, the analysis of the drawing inks revealed an iron gall ink in two different concentrations. With the first ink, whose appearance is now light brown, the centrally positioned donkey and the four figures were outlined with a narrow pen and joined together to form an overall composition. In the original arrangement the donkey's head faced the old man standing under an arched doorway. The donkey's head facing inwards was then painted over with white lead. This can clearly be seen in the corresponding element distribution picture (Figure 9). Later on, the donkey's head was changed with a broad pen and brush using the same iron gall ink in a stronger concentration,





**Fig. 9:** *The Levite and the Dead Concubine*, elemental distributions of iron (Fe) and lead (Pb). Image: Klassik Stiftung Weimar, Graphische Sammlungen, graphics: Carsten Wintermann

which has a darker effect. The iron gall ink was mixed with the opaque lead white here; this resulted in the darker appearance of the ink visible today. With the same second ink further corrections were made to the drawing. Furthermore, a second white pigment that is not lead white was also used for corrections which probably originates from a later retouching. The framing of the sheet was also done later. The reconstruction of the individual work steps suggests that the original arrangement on the sheet was changed. The question arises whether this sheet is a workshop drawing.<sup>24</sup>

<sup>24</sup> For a detailed discussion of this topic, see Ketelsen et al. 2022, 503.

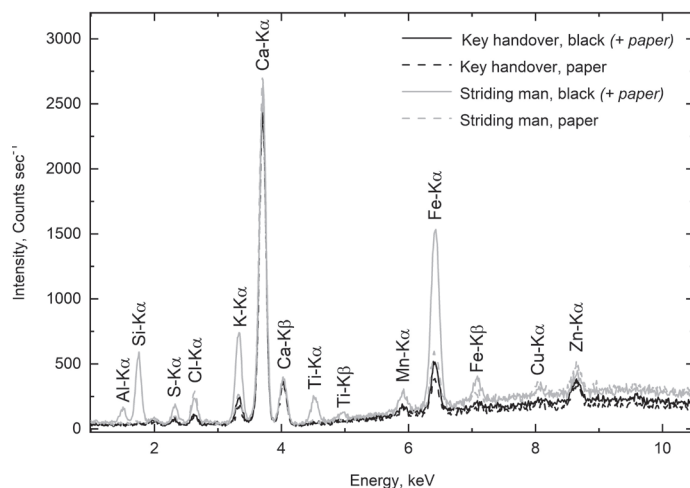


**Fig. 10:** (left) detail *Handing of the Keys to Peter*, Städel Museum, Frankfurt, Inv. n. 7063. Image: Städel Museum; (right) detail *Striding Male Figure*, Kupferstich-Kabinett, SKD, Inv. no. Ca19BI26. Image: Kupferstich-Kabinett der Staatlichen Kunstsammlungen Dresden

### 3.3 Material Analysis of Black Drawings

As already indicated, infrared reflectography or multispectral analysis can be used to distinguish carbon-based drawing materials such as soot, charcoal, graphite, or natural chalk from other drawing materials, but further distinctions are not possible. Based on two drawings, the *Handing of the Keys to Peter* from the Städel Museum in Frankfurt and the *Striding Male Figure* from the Kupferstichkabinett in Dresden, X-ray fluorescence analysis will be used to make such a differentiation (Figure 10).

Figure 11 shows the respective X-ray fluorescence spectra of the black drawing material of the handing over of the key and the striding man. In addition, comparative spectra of the respective papers are included to estimate the influence of the background on the elemental analysis of the drawing material. It becomes clear that in the *Handing of the Keys to Peter* the spectrum of the black drawing material and the corresponding reference spectrum are identical which means that it is a purely organic drawing material. In the *Striding Male Figure*, however, additional peaks appear for the elements aluminium (Al), silicon (Si), potassium (K) and titanium (Ti). In addition, the peak for iron (Fe) is clearly elevated. The



**Fig. 11:** X-ray fluorescence spectra of the black drawing material of the *Handing of the Keys to Peter* and the *Striding Male Figure*. In addition, comparative spectra of the respective papers are included to estimate the influence of the background on the elemental analysis of the drawing material. Image: Federal Institute for Materials Research and Testing

elements indicate mineral components: in the case of the striding one, the drawing material is stone chalk, i. e. clay slate, or alumina coloured with carbon. The material, which contains the silicate components common to clay, is found in deposits all over Europe.

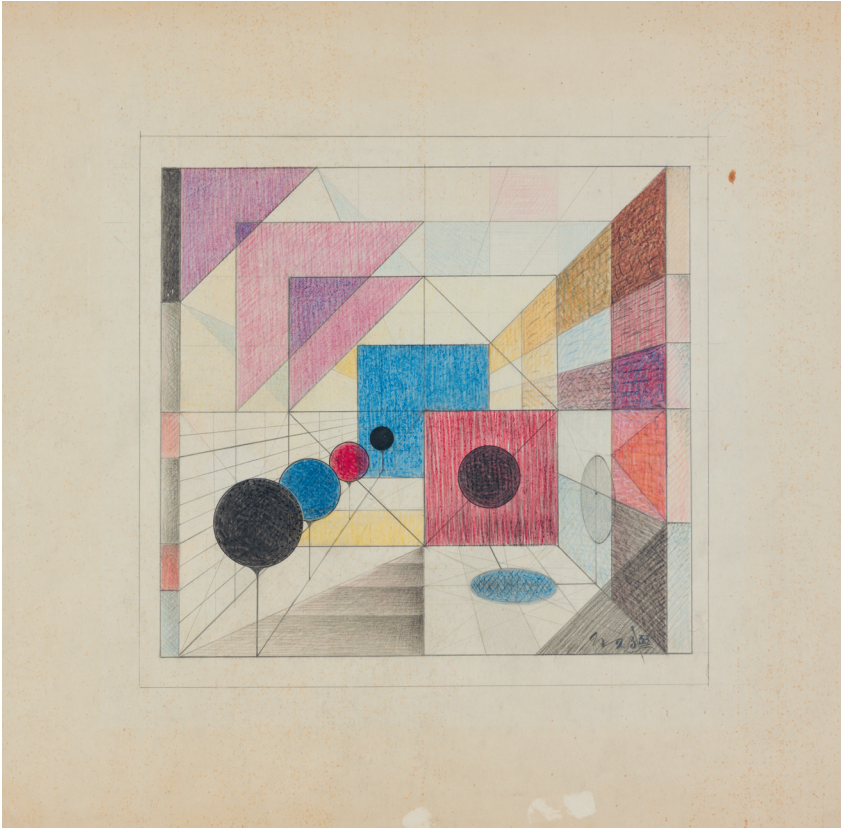
### 3.4 Investigation of Colourants

The question of copy or original arises particularly in the case of artists or workshops that were active over a long period. In the Bauhaus holdings of the Klassik Stiftung Weimar [Foundation of Weimar Classics], there are several pairs of sheets with clear motivic similarities; here it can be assumed that one drawing was created at the time of the Bauhaus, while the other is to be regarded as a copy of the original by the same artist. As an example, two compositions by Andor Weininger are discussed here.<sup>25</sup> Weininger began his studies in Weimar in 1921 and devoted himself to various techniques and genres during his time as a student, developing a particular affinity for theatre. In 1968, Weininger participated in the

<sup>25</sup> Wintermann 2022, 64.



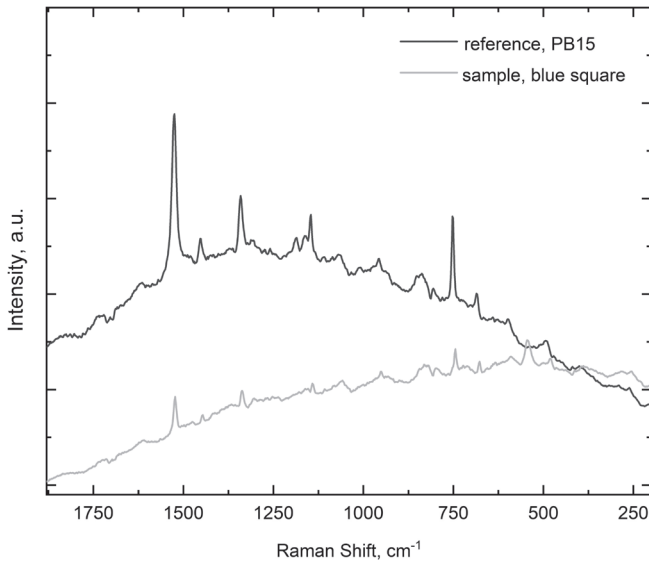




**Fig. 13:** Andor Weininger, *Stage Design*, 1953 (1923), pen and ink in black, graphite, coloured chalks on light green cardboard. Klassik Stiftung Weimar, Graphische Sammlungen, Inv. no.: KK 11557, ID 504368. Image: Klassik Stiftung Weimar, Graphische Sammlungen, photo: Carsten Wintermann

already securely dated with regard to its provenance so that the material-analytical examinations support this claim to authenticity. At the same time, however, they allow conclusions to be drawn about the drawing materials used at the newly founded art school.

The *Stage Design* from 1953 (Figure 13) shows a work in the constructive style of the early Bauhaus years. In contrast to the *Horizontal-Vertical Composition* there are no changes or notes. Both sheets have a very similar, remarkably conventional pigment palette for both 1922 and 1953. In the *Stage Design*, however, an additional clear marker for a time of origin after 1935 could be proven with phthalocyanine blue (Figure 14), thus verifying the date of origin of 1953. The question why Weininger used traditional colourants and not the latest prepara-



**Fig. 14:** Raman spectrum of the medium blue square in the *Stage Design*. The comparison with the reference database clearly shows that the blue colourant is phthalocyanine blue (PB15). Image: Federal Institute for Materials Research and Testing

tions can be answered only in the context of a more comprehensive study of the Bauhaus.<sup>26</sup>

## 4 Conclusion

Even though material analysis – as described here – now offers a variety of approaches to drawing research many publications on the subject do not take it into account. Art historical connoisseurship still relies on comparative stylistic analysis; there is no consideration of the papers used or the drawing materials. One of the reasons for this approach may be the effort required for a careful analysis.

However, the findings from the archaeometric examinations always provide insights into the state of preservation of drawings or even manuscripts. The comparability of these two genres results from the similarity of the materials which were used. The supporting medium is usually parchment or paper; the writing and

<sup>26</sup> Hahn 2022b, 31.

drawing materials are essentially iron gall inks as well as soot inks and more rarely graphite and lead pencils. Damage caused by mould, iron gall ink corrosion and copper corrosion or the fading of writing and drawings have been reported in many publications.<sup>27</sup>

In this way they provide important clues into whether and to what extent conservation interventions are necessary; they generate parameters for planning exhibitions and thus they enable the permanent preservation of cultural heritage.

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<sup>27</sup> Reißland 1999, 167.

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