

CHSOS Application note # 2

May 2021

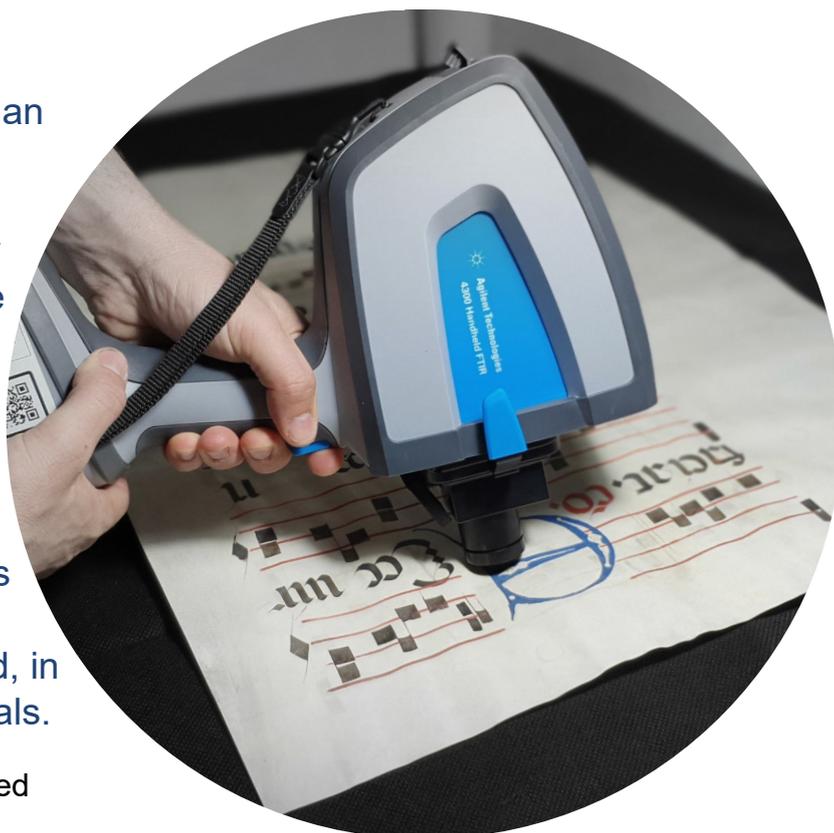
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FTIR Diffuse Reflectance. Pigments Checker Database

We perform molecular analysis with an [Agilent 4300 Handheld FTIR spectrometer](#). This system allows to analyze art and archaeology directly on their surface without any damage or sampling. This method requires a special probe and it is called FTIR Diffuse Reflectance Spectroscopy. The results can be considerably different from standard FTIR spectroscopy and, consequently, it is necessary to build new spectral databases of historical pigments and, in general, art and archaeology materials.

Standard FTIR spectroscopy has been used extensively for art and archaeology examination [1]. The interpretation of the spectra acquired with this method is quite straightforward because in this case the IR radiation is just absorbed or transmitted. On the other hand, FTIR Diffuse Reflectance theory is much complex because the radiation is also scattered, other than absorbed and transmitted. Scattering is a more complex phenomenon and can vary extensively even across samples of the same materials [2]. The consequence is that the spectra can be very different from those available in standard FTIR databases. Therefore, the need to build new databases specifically for FTIR Diffuse Reflectance



spectroscopy. CHSOS contributes to this effort with the spectra of the pigments collected in the [Pigments Checker](#), our collection of historical pigments, ranging from antiquity to the early 1950'. The colors are laid with an acrylic binder on a cardboard support. We collected the spectra of the pigments and that of the binder alone on the cardboard. All these spectra are available online on the [Pigments Checker webpage](#).



Figure 1. Diffuse Reflectance FTIR analysis on Pigments Checker.

Pure powder pigments

The FTIR-DR spectrum of a pigment in Pigments Checker, [figure \[1\]](#), is the result of the contribution from the pigment itself but also from the binder (acrylic) and the cardboard, used as support (100% cotton paper). Consequently, we cannot really see what are the absorbance bands of each pigment. [Figure \[2\]](#) show, for example, the spectrum of the pure [azurite](#) pigment and the azurite swatch of color in the Pigment Checker. We understand that having the spectrum of the pure powder pigment is important for the correct interpretation of the painted surface.

For this reason we built a spectral database of the pure powder pigments. This can also be downloaded, together with the other databases, from the [Pigments Checker webpage](#).

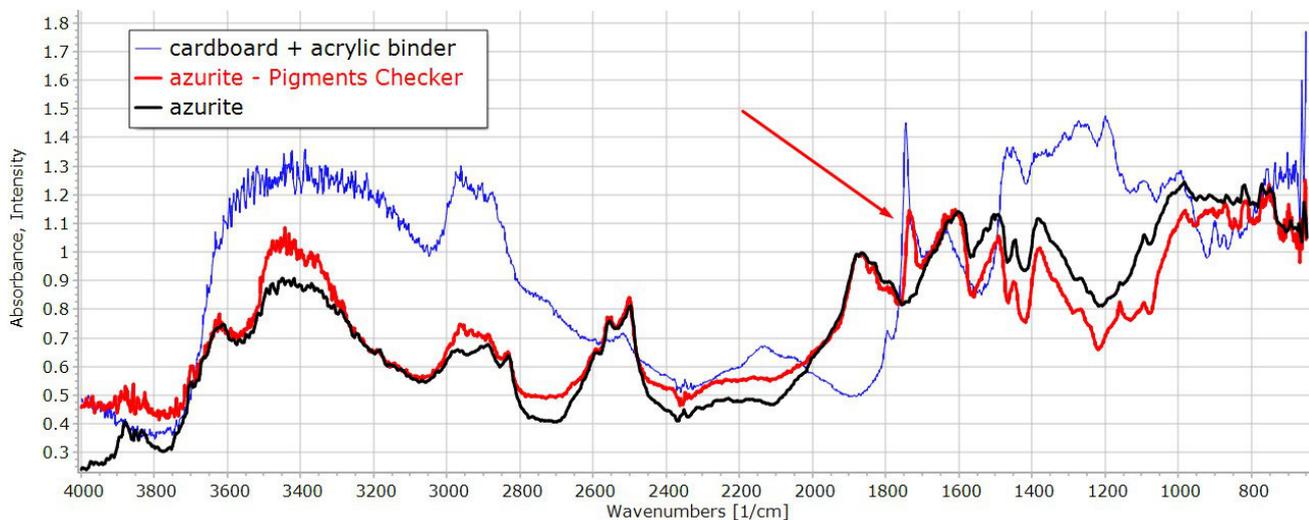


Figure 2. Comparison of the spectra of azurite (pure powder pigment, without any binder); azurite as found in Pigments Checker; the cardboard painted with the acrylic binder. These spectra show that azurite in Pigments Checker show the main features of pure azurite plus the strong peak due to the acrylic binder at 1732 cm^{-1} .

In order not to contaminate the probe, the spectra of the pure pigments were acquired depositing some powder on an IR transparent window, which does not affect the resulting spectrum, figure [4]. This window is then loaded on the probe, figure [5].



Figure [4]. The powder pigment is deposited on an IR transparent window.



Figure [5]. The IR transparent window holding the powder pigment is loaded on the probe for the spectrum acquisition.

Case study: antiphony

We tested a large (68 x 47 cm) [three-color antiphony parchment sheet, 16th century](#), figure[6]. Figure [7] shows the spectra of the parchment, the blue color of a letter and the spectrum of pure azurite in our database. Availability of the pure spectrum of azurite is mandatory for a correct interpretation.

Figure 6. Antiphony parchment sheet, 16th century,

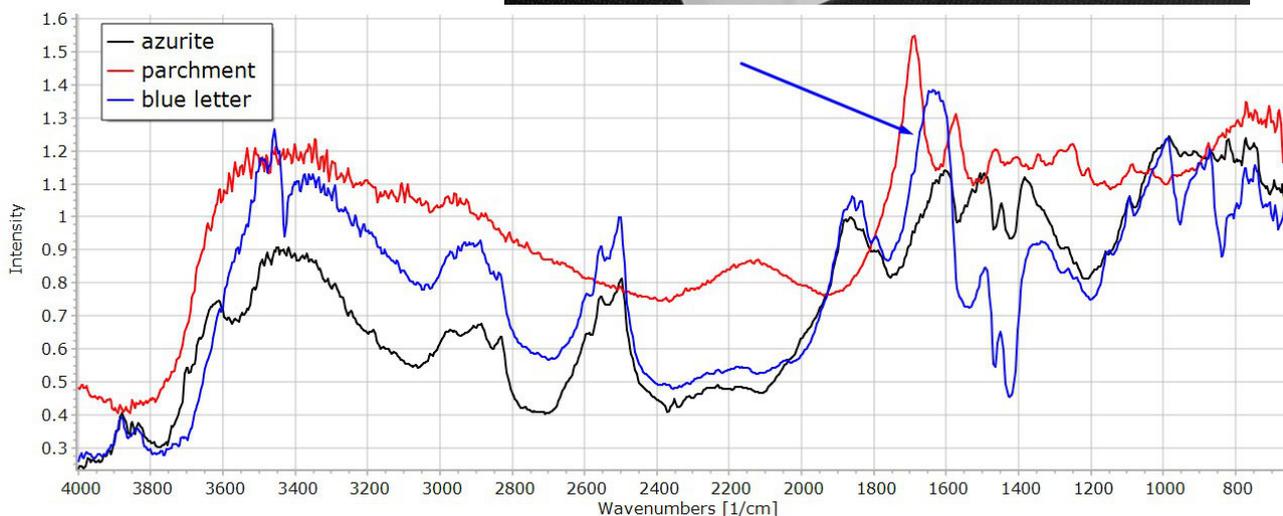


Figure 7. Comparison of FTIR non-invasive Spectroscopy Pigments + fresco technique - Database of the spectra of azurite (pure powder pigment); parchment without any paint; the blue letter. These spectra show that azurite peaks are visible in the blue letter. This example also illustrates what happens with overlapping bands from the support. The parchment has two strong absorption bands at 1692 and 1572 cm⁻¹. These bands, together with that of azurite at 1605 cm⁻¹ results in only one large band centered at 1632 cm⁻¹. This strong band comes also from the contribution of the gum arabic binder.

Conclusions

Non-invasive FTIR with diffuse reflectance probe is a quite novel field. While standard FTIR spectra are easy to interpret, in the case of diffuse reflectance, the use of databases is mandatory. This application note discussed the spectra taken on Pigments Checker, but also the database taken from powder pigments. This helps to understand the effect that the binder has on the spectrum of the pigment. In upcoming application notes we will discuss the effect of other binders on the same pigments.

References

- [1] DERRICK, M. R., STULIK, D., & LANDRY, J. M. (1999). Infrared spectroscopy in conservation science. Los Angeles, Getty Conservation Institute. http://www.getty.edu/conservation/publications/pdf_publications/infrared_spectroscopy.pdf.
- [2] MILOSEVIC, M., & BERETS, S. L. (2002). A REVIEW OF FT-IR DIFFUSE REFLECTION SAMPLING CONSIDERATIONS. Applied Spectroscopy Reviews. 37, 347-364.

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Pigments Checker (TP-MSI calibration card included)

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