# THE SUITABILITY OF CERAMIC STANDARD TILES IN MICROSPECTROPHOTOMETER CALIBRATION: HOMOGENEITY OF THE TILE SURFACE

Bart DECRUYENAERE, Gilbert de ROY, Filip GOVAERT, Dominique ZIERNICKI

*National Institute of Forensic Science, Brussels, Belgium*

**ABSTRACT:** The suitability of ceramic colour standards in microspectrophotometer calibration was examined using a set of ceramic colour standards obtained from CERAM RESEARCH and a Zeiss Axiotech 100HD microscope equipped with a J&M TIDAS 300–1100 nm Diode Array detector. Spectra were obtained using different objectives and apertures. These tests show that surface inhomogeneity only influences the obtained result when using a small spot size.

**KEY WORDS**: Microspectrophotometry; Ceramic colour standards; Microscopy.

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## INTRODUCTION

Objective colour determination of paint samples is gaining importance in forensic comparison casework. The reliability of the measurement sequence is therefore important and should be documented by calibration of the apparatus used. Reference standards should be used to calibrate and check the colour response of the microscope-spectrometer system. This poster reveals some drawbacks of currently available standards.

## MATERIALS AND METHODS

A set of ceramic colour standard tiles (# 9912) was obtained from CERAM RESEARCH (Stoke on Trent, UK) as well as the white reflectance standard reference tile (# 2).

All spectra where collected using a Zeiss Axiotech 100HD equipped with a 100W halogen lamp and a J&M TIDAS 300–1100 nm Diode Array detector operated in the range 380–780 nm at 1 nm spectral resolution. The microscope was fitted with dark field objectives (5×, 10×, 20×, 50× and 100×). The detector was equipped with a variable entrance slit in order to select the measuring area. Spectral processing was performed using the Spectralys© software (J&M).

Reference spectra were collected at ten sample intervals using the white standard tile. Mean results were obtained for each sample by averaging at least 20 full range spectra.

Pictures were obtained using the same microscope equipped with a 1/2" colour CCD camera (JVC) linked to an image archiving system (Micro, Olympus).

## RESULTS AND DISCUSSION

Ceramic tiles are very stable but exhibit some colour inhomogeneity on their surface (Figure 2), which is much more pronounced than a regular paint (Figure 1). In order to assess the influence of this effect varying surface areas of the tiles were measured for 30 times and averaged.

Fig. 1a. Orange paint, 10× objective.

Fig. 1b. Orange paint, 100× objective.

Fig. 2a. Orange tile, 10× objective.

Fig. 2b. Orange tile, 100× objective.

Figure 3 demonstrates that this should be achieved by regulating the measuring aperture to the detector and not by changing to another objective giving the same irradiated area.

Fig. 3. Same sample measured with progressively smaller spot sizes.

Figure 4 demonstrates the effect of the measuring slit size on the spectra obtained. 30 spectra were recorded at different spots on the tile using the 10x objective and slit sizes yielding a measuring spot of 0.8 mm (a) and 0.08 mm (b) respectively. Clearly the variation in spectral response is higher at the lower spot size. This variation has been plotted as standard deviations in Figure 5.

Fig. 4a. Difference green tile measured at 30 locations using a 0.8 mm spot size.

Fig. 4b. Difference green tile measured at 30 locations using a 0.08 mm spot size.

Fig. 5. Difference green tile measured at 30 locations, standard deviation of spectra.

The use of spot sizes in the range 0.2–0.8 mm does not have much influence on the statistical variation of the spectral curves. This variation is somewhat larger than the reproducibility obtained at one single measuring spot on the tile. An effect of the surface heterogeneity of the tile is therefore observed.

Deviations at the smaller spot size of 0.08 mm can originate from the mixed effect of surface inhomogeneity and detector insensitivity.

## CONCLUSION

Reference standards should be used to calibrate and check the colour response of the microscope-spectrometer system. These standards should be stable, traceable and amenable to analysis in the same conditions as the casework samples. Ceramic tiles offer these advantages but in our work some limitations as to their use have become obvious.

Ceramic tiles are less homogeneous than solid paint samples. Measuring a sufficiently large portion of the tile surface, or alternatively by measuring a sufficiently large number of smaller spots should average out these inhomogeneities. In this way spot sizes down to 0.2 mm on the sample could be used. At smaller apertures however detector sensitivity problems occur.