Goldschmidt 2026 Abstract
https://doi.org/10.46427/gold2026.33127

Non-destructive study of protected sites by portable X-Ray Fluorescence: application to colored speleothems in Cigalère Cave (France)

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With the development of cutting-edge analytical technologies, the study of fragile or protected sites is becoming increasingly feasible with *in situ* and non-destructive analytical techniques. Among these techniques, portable X-ray fluorescence (pXRF) spectrometry has seen tremendous advances in both precision and reliability over the past few decades, with growing applications in various fields of study, including (geo)archaeology, geochemistry, planetary field geology, and environmental contamination. Here, we propose a non-destructive characterization of variably colored speleothems from the well-protected Cigalère Cave (Ariège, France), exclusively using pXRF analysis [1].

The Cigalère Cave (French Pyrenees) is well-known for its sulfate mineralizations, linked to the overlying Pb–Zn SEDEX deposits. In this cave, several speleothems exhibit colorations, most likely caused by the presence of metal-rich compounds within the speleothems. However, because of strict preservation policies, few studies have investigated these remarkable elements of geoheritage. Thanks to the non-destructive and portable nature of pXRF, we propose new insights into the causes of coloration in three colored zones of the Cigalère Cave.

The Cascade Noire zone is a 20-meter-long flowstone mainly composed of gypsum, exhibiting yellow-orange and black hues. This structure was three-dimensionally modeled and analyzed by pXRF, revealing the presence of Fe, most likely as Fe sulfates and Fe oxides, producing orange and black colorations, respectively. The Chapelle de Donnea zone is a small, remote chamber hosting velvet-black stalactites and stalagmites. Our pXRF analysis revealed that the black coloration is caused by the presence of Mn, probably incorporated within oxides overlain by a thin layer of gypsum. Finally, the Blue zone hosts several stalactites with a blue-colored core and an uncolored, well-crystallized gypsum cortex. Our data suggests that the blue coloration is linked to the presence of Cu^{2+} in substitution, along with some Zn-rich amorphous compounds, which may also contribute to the coloration, as suggested by a previous study conducted in Malaval Cave [2].

1. Vlieghe, M. *et al.* Non-destructive characterization of variously colored gypsum and aragonite/calcite speleothems from the Cigalère Cave. *Int. J. Speleol.* **55**, (2025).
2. Vlieghe, M. *et al.* Polymetallic interactions of Zn-Pb-Cu in blue/green-colored speleothems from Malaval Cave (France). *Geochemistry* **85**, 126285 (2025).



