EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:
https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title: Structural investigation of black pigments used on ancient papyri**	**Experiment number**: Hc3833
Beamline: ID22	**Date of experiment**: from: 26/09/2018 to:01/10/2018	**Date of report**: 07/09/2022
Shifts: 15	**Local contact(s)**: Catherine Dejoie	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

P. Martinetto*, Institut Néel, Grenoble

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C. Dejoie*, ESRF, Grenoble

Report:

We have investigated a series of illustrated papyruses from the Champollion museum (Département de l'Isère, Vif, France), combining X-ray powder diffraction (XRD) and fluorescence (XRF) at the ID22 beamline. XRD data were collected using a Perkin Elmer XRD 1611CP3 detector positioned at a distance of 1400 mm from the sample with a beam size of 1 mm x 0.1 mm (h x v) and at a wavelength of 0.4009 Å. X-ray fluorescence spectra were recorded with a Hitachi Vortex 90EX SDD. Papyrus fragments were positioned in the beam using dedicated 3D-printed sample holders (Fig. 1) [1].

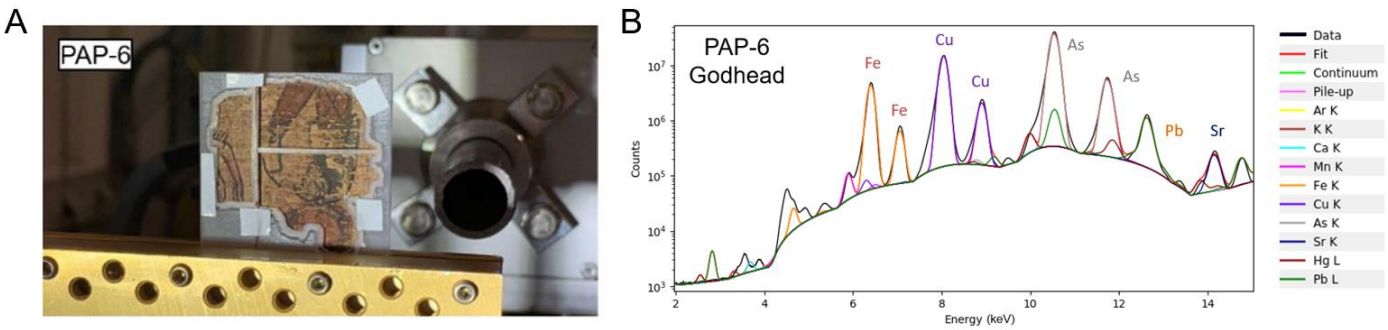

Figure 1. A. View of one of the papyrus fragments (PAP-6) displaying the head of the God Ra (Champollion museum, Département de l'Isère, Vif, France), mounted in dedicated 3D-printed sample holders at the ID22 beamline. B. Sum spectra obtained from the X-ray fluorescence map carried out over the head of the God, with corresponding fit.

The main colors found on the papyrus are blue, green, red, pink, yellow, white and black. Several regions of interest were first investigated, and the fit of the four resulting sum spectra reveals the presence of iron, lead,

copper, arsenic, and mercury as main relevant chemical elements (Fig. 1). Iron is found in the red regions, copper appears in blue and green regions, and arsenic is present in the yellow parts. Then, pigments were identified by XRD along several lines crossing the different regions investigated by XRF. Hematite (α-Fe_2O_3), cuprorivaite ($CaCuSi_4O_{10}$), atacamite ($Cu_2Cl(OH)_3$), orpiment (As_2S_3) and realgar (As_4S_4), and challacolloite (KPb_2Cl_5) were identified in the red, blue, green, yellow and white regions, respectively, in agreement with the results obtained by chemical mapping. Finally, black pigments were identified as carbon-based compounds, and this has been published in [1].

Most of the pigments identified are part of the known Egyptian palette. A few additional phases, such as challacolloite and atacamite, correspond to degradation phases. These results were used to gain insights into the illustration process implement by ancient Egyptians, and are submitted for publication in [2].

[1] Autran P.-O., Dejoie C., Bordet P., Hodeau J.L., Dugand C., Gervason M., Anne M., Martinetto P.. Revealing the Nature of Black Pigments Used on Ancient Egyptian Papyri from Champollion Collection. *Anal. Chem.* **93**, 1135–1142 (2021).

[2] Autran P.-O., Dejoie C., Dugand C., Gervason M., Bordet P., Hodeau J.L., Anne M., Martinetto P. Illustrating papyrus in Ancient Egypt. *Submitted.*