

*Title:*

Towards an experiment-based protocol for extracting and identifying residues

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*Abstract:*

Residue analysis has recently become a widely applied method in reconstructing the lifecycle of prehistoric stone tools. The identification of residues is traditionally based on the distinctive morphologies of the residue fragments by means of light microscopy. The majority of residue fragments, however, tend to have an amorphous structure and are therefore not easy to identify. In addition, some residue categories can only be detected by using transmitted light microscopy, which requires the extraction of residues from the tool's surface. Yet another challenge is to determine whether the residues were deposited on the tool's surface as a result of use or due to other processes.

Here we present the results of an experimental study that addresses these methodological issues. Stone tools from a new experimental reference collection were used to test 6 different analytical methods: the observation of residues on stone tools with incident light microscopy, dry sampling using tweezers and brushes, wet sampling with micropipettes using distilled water and a tri-mixture of acetonitrile, ethanol and water, and extraction with an ultrasonic scaler or bath. The experiments demonstrate that the choice of a particular extraction procedure may influence the amount and types of residues that are extracted. This implies that the analytical method has an impact on the results of a residue analysis. Building on these data, we designed a new protocol, which was subsequently submitted to blind testing in order to test its accuracy and precision. Certain key attributes were also identified that may prove useful in distinguishing between use-related and natural residues. We discuss the importance of adapting analytical protocols to the research question of the study.