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The Benefits of Consistently Orientated Samples for Coincident Data Collection

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As technology has improved and scientific instrumentation has become increasingly widespread and accessible, it is now possible to analyze the same sample by a wide variety of techniques. Such multi-technique data collection benefits scientists by allowing charaterisation of a wide range of sample aspects, including phase identification, distribution, elemental concentration, crystallography, deformation, grain size, grain shape and much more. For true integration of the datasets, the sample's orientation must be tracked at all points to ensure the data can be reoriented and directly compared.

In recent years, new designs for sample preparation have emerged that increase throughput such as the development of trans-vertical moulds to optimize the SEM chamber geometry by reducing excess epoxy, which allows for increased capacity within the holder [1, 2]. In this project, we developed a systematic routine for the preparation of 25 mm diameter round samples, where each sample contains a permanent sample orientation discriminator. A sample collar was produced that is embedded into the sample during the preparation process (figure 1). As the collar is located around the edge of the sample, it does not block the transmitted light path. The most important aspect of the collar is the orientation line that allows the sample to be correctly reoriented from one instrument to the next, ensuring that all data are collected in the same reference frame. The collar also contains the sample number or name and can also include other provenance information such as an international geo sample number (IGSN) or quick reference (QR) code, which can be scanned by a mobile phone app and linked to the metadata record for the sample. The collar is being used for mineral liberation analysis samples at Curtin University in Perth [3, 4].

For this project, we analyzed rock sample, D43, collected from the North Qaidam ultrahigh-pressure terrane, Western China (figure 2A). Part of the sample was powdered for X-ray diffraction (XRD) (figure 2F) and then placed onto Kapton tape in a 25mm Struers FixiForm 2-part mounting cup. The collar (examples are shown in figure 1), which was pushed inside the cup and fits firmly around the edge. EpoFix resin was applied using a Struers CitoVac vacuum impregnation unit (VIU). The pressure inside the VIU chamber was cycled to drive the air bubbles out of the resin. Once the resin had cured, the sample was polished with a Struers Tegramin 30 that uses DiaPro water based diamond suspensions (figure 2B). The sample was then imaged using reflected light on an automated stage Zeiss Axio imager (figure 2C), then carbon coated for backscattered electron (BSE) (figure 2D) imaging and energy dispersive spectroscopy (EDS) (figure 2 E) mapping using a FEI Quanta 250 scanning electron microscope (SEM). Finally, the sample will be analyzed using a LA-ICP-MS (ESI NWR 193 µm laser coupled to an Agilent 8900 triple quad inductively coupled plasma mass spectrometer). All analyses were performed at the Murdock Research Laboratory within the Department of Geological Sciences at Central Washington University. All data have been collected on the exact same area of the sample because reorientation is easy due to the sample collar, therefore finding the same analysis sites is no longer a chore. This allows easy integration of the optical and SEM data, which, in turn, can be used to help interpret the XRD data and ensure accurate phase matches are chosen.

References

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Figure 1. Simple collar design for embedding into 25 mm diameter resin sample. Each label is 78 mm, long including the 4 mm overlap area, and 9 mm tall. These measurements fit the label firmly into the Struers FixiForm 2-part mounting cup. The collar design can be manipulated to display a variety sample information, as well as the orientation line which is what allows the samples to be reoriented in each analytical instrument.

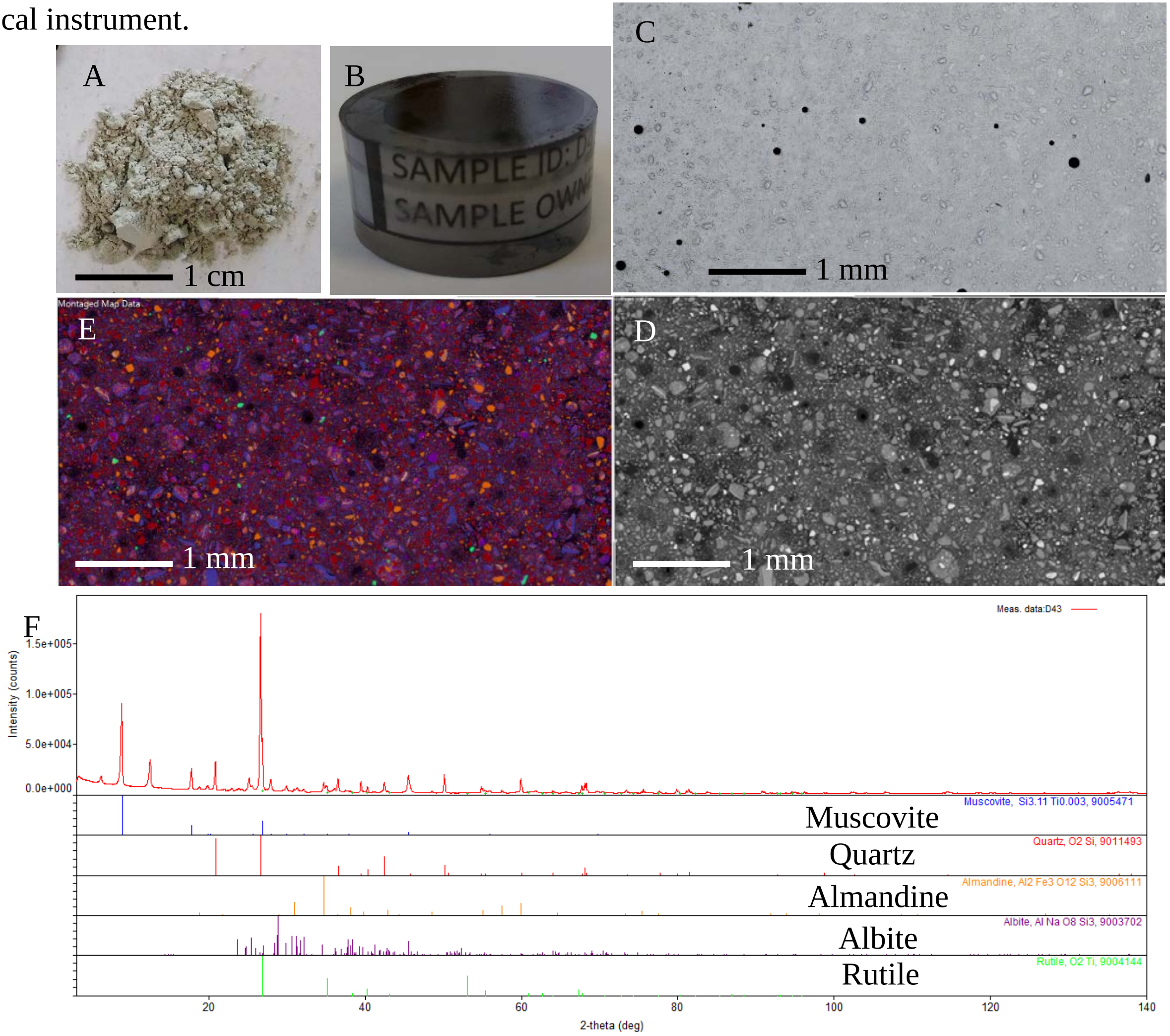

Figure 2. (A) D43 XRD powder. (B) 25 mm diameter mount. (C) Reflected light image of analysis area. (D) BSE image of analysis area. (E) Phase EDS map of the analysis area where Blue = Muscovite, Red = Quartz, Orange = Almandine, Purple = Albite, Green = Rutile. (F) XRD spectra of the powdered rock.