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What Lurks in the Martian Rocks and Soil? Investigations of Sulfates, Phosphates, and Perchlorates

A temperature-controlled sample stage for in situ micro-X-ray diffraction: Application to Mars analog mirabilite-bearing perennial cold spring precipitate mineralogy†

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Abstract

A temperature-controlled sample stage with an operational range of ~60 °C above or below ambient laboratory temperature (~ –35 to 85 °C) was constructed for in situ X-ray diffraction of minerals and materials using a Bruker D8 Discover diffractometer with θ-θ geometry. The stage was primarily designed for characterizing mirabilite-bearing samples from a Mars analog High Arctic perennial cold spring at an in situ temperature. Operation of the stage was demonstrated through the analysis of a synthetic sample of the hydrated sodium sulfate, mirabilite ($Na_2SO_4 \cdot 10H_2O$). Mirabilite was held at –25 °C for approximately two hours without significant dehydration and then incrementally warmed to ambient laboratory temperature at 5 °C intervals, during the acquisition of in situ diffraction data. At ambient laboratory temperature, the mirabilite dehydrated and only polycrystalline thenardite (Na_2SO_4) remained. Preliminary analysis of the cold spring precipitates demonstrates that when mirabilite is present in the sample the dehydration reaction is occurring between collection and analysis at ambient laboratory temperature. This temperature-controlled stage was designed for versatility and ease of X-ray access, with applications that can extend to many geological and planetary settings, including Mars analog environments.

Keywords: Micro-X-ray diffraction, sodium sulfate dehydration, Peltier-effect, thermoelectric temperature-controlled stage, mirabilite, thenardite, Mars analog