

## **Reduction in piston-cylinder experiments: The detection of carbon infiltration into platinum capsules**

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### **ABSTRACT**

Problems associated with intermittent and variable degrees of sample blackening are often reported for studies involving the preparation of CO<sub>2</sub>-bearing silicate glasses in piston-cylinder apparatus. This phenomenon is generally attributed to H infiltration, which leads to the reduction of CO<sub>2</sub> and the precipitation of graphite with the concomitant formation of water. In this study we demonstrate that carbon diffusion into platinum capsules may be a common cause of blackened glasses and this process may be detected using fourier transform infrared spectroscopy (FTIR) to identify the presence of CO without elevated H<sub>2</sub>O contents. The simulated infiltration of <sup>12</sup>C from a graphite furnace into a <sup>13</sup>C-bearing sample is illustrated using secondary ion mass spectroscopy (SIMS) and micro-FTIR analysis.

Careful FTIR monitoring of variable sample reduction has helped to identify the precautions required to reduce C (and H) infiltration in solid media assemblies and it appears that physical barriers can be more important than the chemical buffers traditionally employed.