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A filler-rod technique for controlling redox conditions in cold-seal pressure vessels

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ABSTRACT

A new method has been developed to impose different redox conditions in high-temperature-pressure experiments in cold-seal pressure vessels, at 800 °C and 2000 bars. Experiments were conducted by loading a metallic filler rod into the autoclave together with H_2 sensor capsules, and pressuring the autoclave with H_2O. Rod materials tested successfully were Co, Ti, and C (graphite). The oxidation of these rods produces H_2, but because of diffusive H_2 loss through the walls of the autoclave, the system may not be buffered with respect to H_2. However, f_{H_2} quickly reaches a steady state value, and because f_{H_2} is easily measured by the hydrogen sensor method, the effect of the filler rods on the intrinsic f_{O_2} of the autoclave can be quantified. In order to produce oxidized conditions, Ar was used as the pressure medium and metal oxides, contained in Al_2O_3 tubes, were employed. By using either Ar or H_2O as a pressure medium, a $\log f_{O_2}$ range of NNO –3.9 to NNO +4.6 can be imposed by this method, where NNO is the $\log f_{O_2}$ value of the Ni-NiO buffer. The ability to conduct long-run-duration experiments at high temperature and high f_{H_2} conditions is not possible with the traditional double-capsule technique because the buffer assemblage is consumed too quickly. However, run durations of up to 4 weeks with constant f_{H_2} at reduced conditions have been conducted using the filler-rod technique. This technique has been shown to be an effective method in controlling redox conditions in cold-seal autoclaves, and thus can be applied to investigating redox-dependent reactions in a wide range of geochemical systems.