Materials with applications in gas storage and separation are of enormous interest across many disciplines of science. This has been driven in large part by advances in carbon sequestration technologies in line with global government targets for cleaner energy and the reduction of carbon dioxide (CO2) emissions. Potential applications in this field include the removal of CO2 from flue gas mixtures before it enters the atmosphere, for example. Porous metal-organic frameworks (MOFs) are currently seen as one of the most promising types of materials for this purpose since they combine the desired features of high porosity, thermal stability and chemical versatility. Fundamental to such research is the requirement to optimise the quantity of gas that can be stored in the MOF. Evaluation of gas storage capacities is usually performed using time-consuming gravimetric or calorimetric analyses over a range of temperatures and pressures, and often requiring gram-scale quantities of material. Here, we present a novel method for gas delivery and calculation of gas storage capacity using a perfluorocarbon (PFC) liquid. PFCs have long been recognised as useful due to their predisposition for dissolving large volumes of gases such as CO2 and oxygen. They are chemically inert hydrocarbons in which the hydrogen atoms have been replaced with fluorine. We have used a PFC containing dissolved CO2 as a pressure-transmitting liquid in an in situ high-pressure single-crystal X-ray diffraction experiment. Application of industrially-achievable pressure within a diamond anvil cell causes the CO2 to be squeezed out of the liquid into a host crystal of a porous MOF as the system seeks to fill empty space and redistribute density upon contraction. Diffraction data from a crystal of Sc2BDC3 (BDC = 1,4-benzenedicarboxylate)[1] has allowed us to determine its maximum CO2 storage capacity, which is in perfect agreement with other methods. This result is the first of its kind and the technique has several notable advantages over other methods: it requires just one experimental step, can be performed at room temperature, and requires only one single crystal of solid material.


**Keywords:** metal-organic framework, high-pressure crystallography, carbon dioxide