

Noble gas extraction and purification from oil/water mixtures

D.J. HILLEGONDS^{1*}, R.L. TYNE¹, P.H. BARRY^{1,2}, D.J. BYRNE¹, J.T. KULONGOSKI², C.J. BALLENTINE¹

¹ Dept. of Earth Sci., University of Oxford, Oxford, UK

² USGS, California Water Sci. Center, San Diego, CA, USA

*Correspondence: darren.hillegonds@earth.ox.ac.uk

The isolation and analysis of noble gases - derived from water samples - is routine in several mass spectrometry laboratories, however oil-water mixtures present unique challenges. We have developed a method for isolating clean noble gas aliquots from water and oil/water mixtures collected in Cu tubes, using gas expansion into known-volumes and clamped Cu tubes in the place of traditional valves, to minimize vacuum system oil contamination.

The Oxford noble gas extraction system consists of two 2.75" conflat (CF) unions, each with a blank flange at one end and a CF-3/8" Swagelok (SL) adapter on the other. These are connected via Cu tubing and SL tees to a Cu sample tube, and to a 1.33" CF vacuum line including a titanium sponge getter and a 10 torr capacitance manometer. Samples are inlet into the first CF union while it is isolated from the rest of the system by (vacuum-tight) clamping; the head space gas is then expanded into a second CF union by unclamping the Cu tube and re-clamping at a second location. The aliquot of the original sample gas in the second CF union contains water vapor (but very little of the liquid water), plus a fixed proportion of reactive and inert gases present in the Cu tube, based on relative volumes and gas solubility in the sample liquid. After exposure to titanium sponge at 950°C and room temperature, the second CF union is sealed by clamping and transferred to the mass spectrometer [1] for analysis of noble gas concentrations and isotope ratios. CF unions are subsequently disassembled and cleaned while clamped Cu tubes are discarded.

First results from air saturated (standard) waters demonstrate reproducibility of 2-3% for He, Ne, Ar, Kr, and Xe abundances, 2% for ³He/⁴He, and <0.5% for Ne, Ar, Kr, and Xe isotope ratios. Produced water and casing gas samples from the same well were similar in ²⁰Ne/²²Ne (by ±2%), ⁴⁰Ar/³⁶Ar (by ±4%), ⁸⁶Kr/⁸⁴Kr (by ±3%), and ¹³²Xe/¹³⁰Xe (by ±3%). These results suggest the sample extraction system is amenable to the study of subsurface noble gases in the context of oil and gas production activities [e.g., reference 2]. [1] Barry et al., (2016) *GCA*, 194, 291-309. [2] Barry et al. (2017) *Goldschmidt Abstracts 2017*.