Experimental Report

17/02/2015

Proposal: **9-10-1394** **Council:** 4/2014

Title: FLUID TRANSPORT AND PORE ACCESSIBILITY IN PROTEROZOIC AND EARLY PALEOZOIC SHALES

This proposal is a new proposal

Researh Area: Other...

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Samples: 3 natural shales, Georgina Basin, Australia
3 natural shales, Cooper Basin, Australia
3 natural shles, McArthur Basin, Australia

Instrument	Req. Days	All. Days	From	To
D11	2	2	16/10/2014	18/10/2014

Abstract:

We propose to investigate the pore size dependent adsorption and desorption of geological fluids in several Paleozoic and Proterozoic sedimentary rocks using SANS and USANS. The rock samples were sliced off a solid core extracted from various depths in three geologically old Australian basins. Selected samples were characterised using standard geochemical methods and contain small amounts of organic matter of various thermal maturity.

The pore space topology of such rocks is often fractal on the linear scale from nanometers to tens of micrometers. As a surprising result of only few previous experiments similar to the one proposed here, it transpired that a significant fraction of pores may not be accessible to invading fluids, with no obvious trend with the pore size. Also, there are indications that in the smallest of nano-pores the adsorption mechanism is pore size dependent and dominated by the condensation phenomena.

We propose to systematically investigate the sorption mechanisms and pore accessibility in a carefully selected set of rock samples across the entire pore size range (1 nm to 20 microns), using the contrast matching technique with pressurized

Report for experiments CRG-2102 (S18), CRG-2171 (S18) and 9-10-1394 (D11)

CD_4 was used as a contrast match fluid in the pressure range from 0 (vacuum) to 650 bar. There were three types of experiments performed: (1) SANS and USANS at p=1 bar, (2) seeking contrast match at a fixed Q-value using stepwise increased gas pressure and (3) SANS and USANS at the contrast match (zero average contrast, ZAC) pressure. For some samples we also monitored sorption kinetics during a stepwise increase of pressure from the vacuum to 50 bar. This was the first time that SANS and USANS experiments at such high gas pressure were performed at ILL. Out of the planned 9 rocks samples, full sets of SANS and USANS data were obtained for 6 samples. Both SANS and USANS data were isotropic. The data were acquired, reduced and analysed using standard methods. In particular, the polydisperse spheres (PDS) model implemented in the PRINSAS software was used to fit the combined SANS and USANS curves.

Here we present the most significant interpreted results for sample Baldwin1_217129. Owing to space limits, descriptive text is mostly limited to figure captions. Full text (7 pages) is available on request.

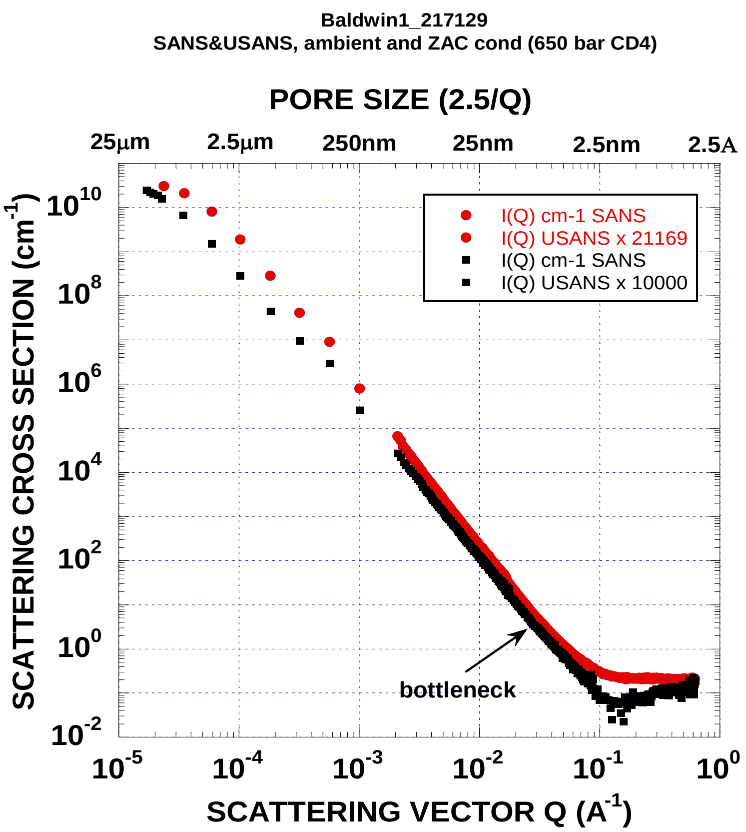

Figure 1. Combined SANS and USANS data, reduced to the absolute scale. Red circles – ambient conditions, black squares – ZAC condition at the room temperature. Scattering intensity at ZAC decreases by a factor of up to about 6, depending on the Q-value; this indicates that a significant part of the pores is not accessible to the invading CD_4 fluid. Note that both the abscissa and ordinate are presented on the logarithmic scale. The top horizontal axis is labelled with the pore sizes which contribute most to the scattering intensity at the corresponding Q-values.

Fraction of accessible pores determined from data in Fig.1 is presented in Figure 2.

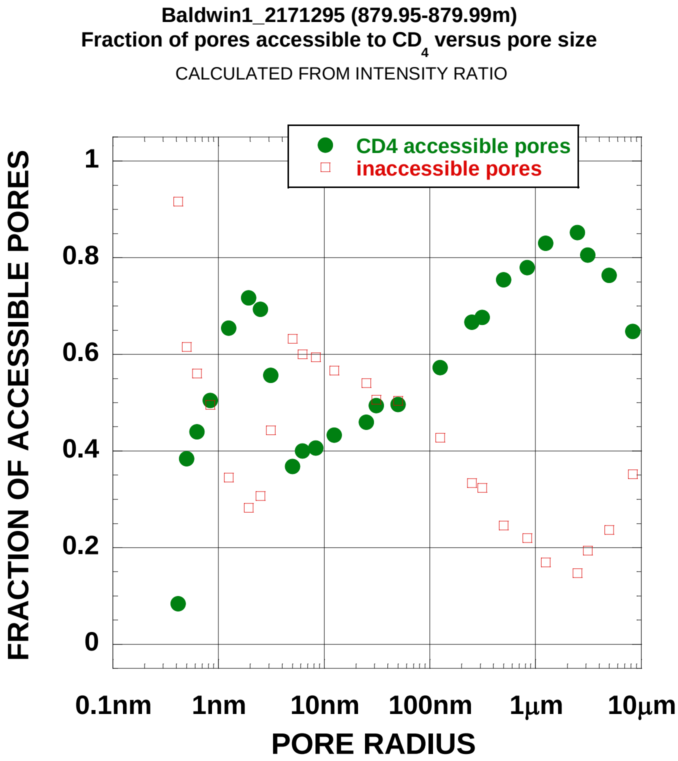

Figure 2. Fraction of pores accessible (and inaccessible) to CD_4 in sample Baldwin 1.

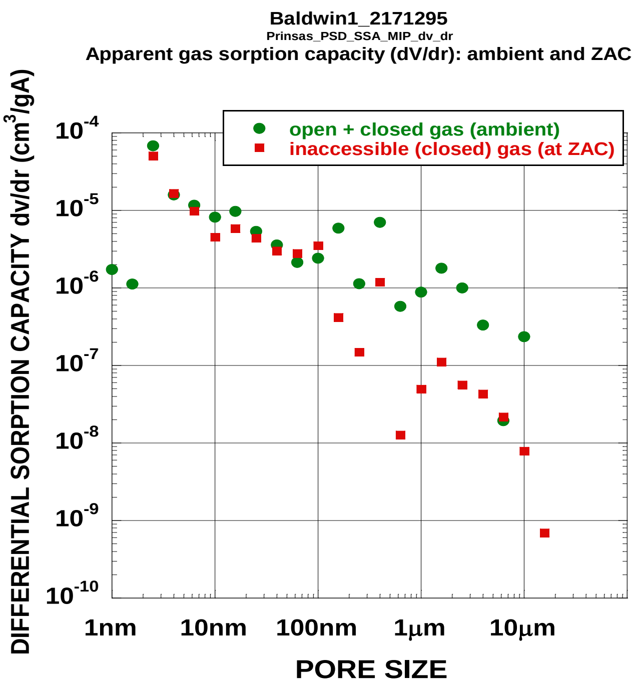

Figure3. Total gas sorption capacity (green circles) and inaccessible gas sorption capacity (red squares) in shale sample Baldwin 1_2171295. The actual (real) differential sorption capacity is the difference between the green circles and red squares. For this sample, for pores smaller than 100 nm, most of dV/dr resides in closed pores and cannot be produced.

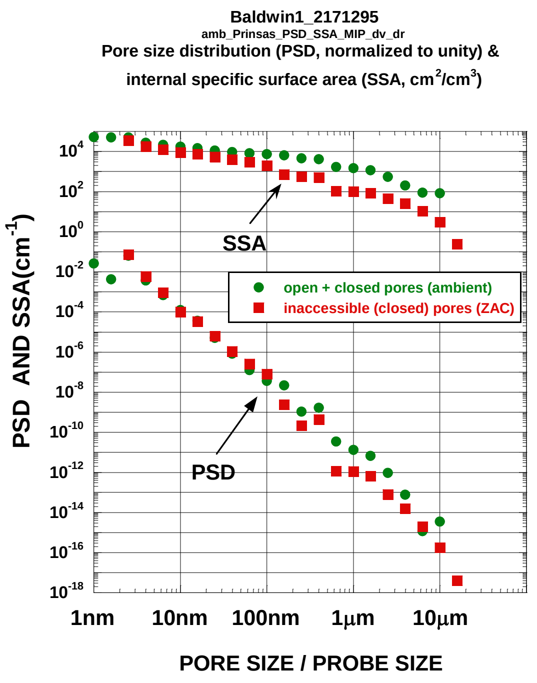

Figure 4. PSD versus pore size (normalised to unity) and SSA versus probe size (in absolute units) computed from the SANS/USANS data acquired from sample Baldwin1_2171295.

Figure 5 illustrates what the MIP curve (based on the pore body size) would have looked like if (1) all the pores were accessible (green circles) and (2) the inaccessible pores only were accessible. The utility of curve (1) is obvious for rocks with generally open pore structure (e.g. some reservoir sandstones), but not always for tight shales.

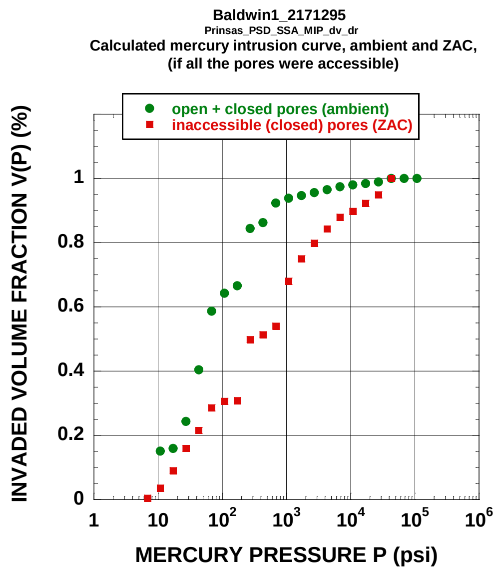

Figure 5. Hypothetical mercury intrusion curve for all of the pores (green circles) and the inaccessible pores (red squares) present in shale sample Baldwin1_2171295.