

XRD_LLNL_2023_FORGE_5_2428.docx

These powder X-ray diffraction (XRD) data were obtained from samples prepared from subcores taken from four depths from FORGE well 16A(78)-32 core. Desired core lengths were selected from available core photos (on GDR), provided by FORGE personnel, and subcored at Lawrence Livermore National Laboratory (LLNL) at the following four discrete downhole-measured depths: (1) 5477.2 ft depth; (2) 10955.4 ft depth; (3) 10960.6 ft depth; and (4) 10974.5 ft depth. See Figures 1-4 for core log photos, subcore sample photos, and diffractogram patterns.

The XRD scans were collected in May 2023 at Lawrence Livermore National Laboratory as pre-experimental characterization data for these subcores, which will be used in core-flooding experiments at LLNL and in triaxial direct shear experiments at Los Alamos National Laboratory (LANL) as part of DOE Project 5-2428. Waste cuttings were first crushed to sub-2mm size fraction by hand and then pulverized using a McCrone micronizing mill at 10 minutes in ~2mL isopropyl alcohol and corundum media. No glycolation was performed on these samples. The powders were allowed to dry and then, immediately prior to loading, were agitated in a 50-mL centrifuge tube with corundum spheres and ~0.5mL of Vertrel to better disperse clay-like and platy minerals. Samples were then immediately side-loaded into sample holders to avoid preferential sample orientation and loaded into the X-ray diffractometer.

The XRD data were collected at LLNL on a Bruker D8 advance XRD with CuK α radiation using a LynxEye detector set at 10mm, scanning over a range of 3.5-70 2 Θ (two-theta) angle with a step size of 0.012, and using a fixed knife edge to suppress unwanted low-angle scattering. The raw datafiles consist of intensity versus 2 Θ angle for the four samples. See Figure 5 for stacked diffractograms. Initial phases were identified using Bruker's DIFFRAC.EVA software, and further phase identification was carried out during Rietveld refinement analysis carried out in freely available Profex software (open source software; Dobelin et al., 2015; www.profex-xrd.org). See Figure 6 for an example of q-XRD analysis. These analyses are forthcoming and results will be shared in this same submission.

Prepared by LLNL under Contract DE-AC52-07NA27344.

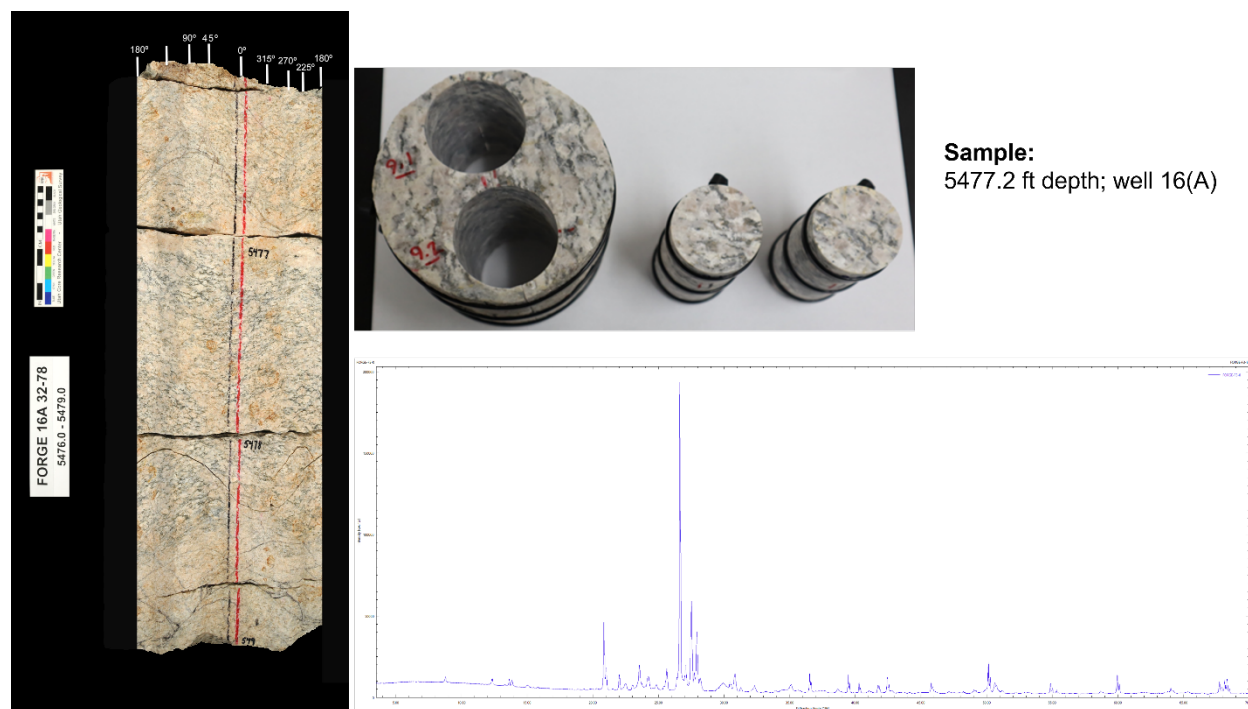


Figure 1: Core log photos (left), subsample photos (center top), and powder X-ray diffractogram results for sample prepared at depth of 5477 ft, from well 16(a).

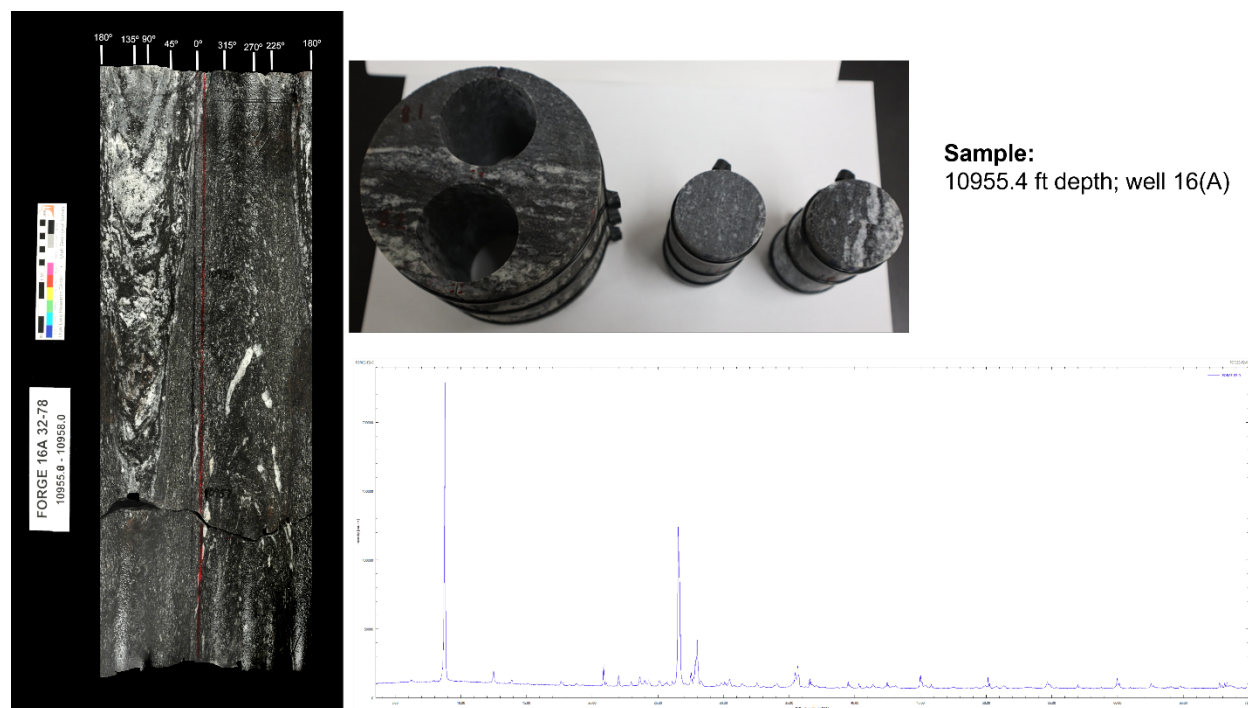


Figure 2: Core log photos (left), subsample photos (center top), and powder X-ray diffractogram results for sample prepared at depth of 10955 ft, from well 16(a).

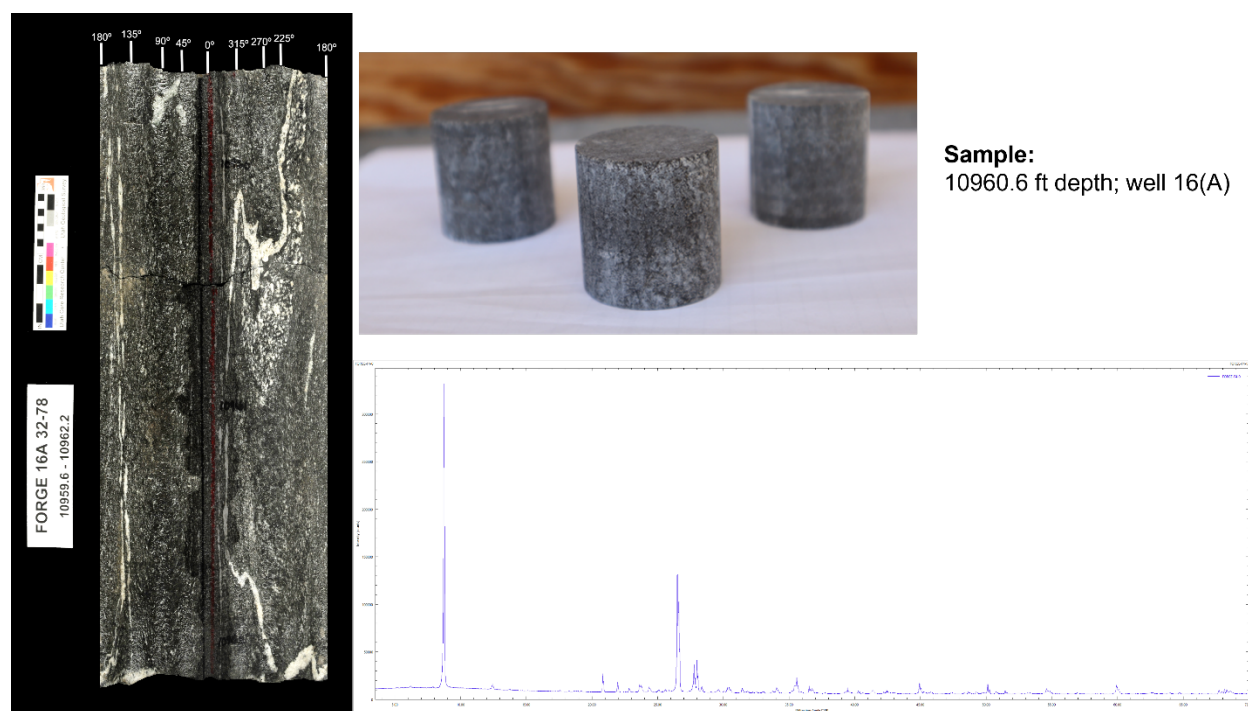


Figure 3: Core log photos (left), subsample photos (center top), and powder X-ray diffractogram results for sample prepared at depth of 10961 ft, from well 16(a).

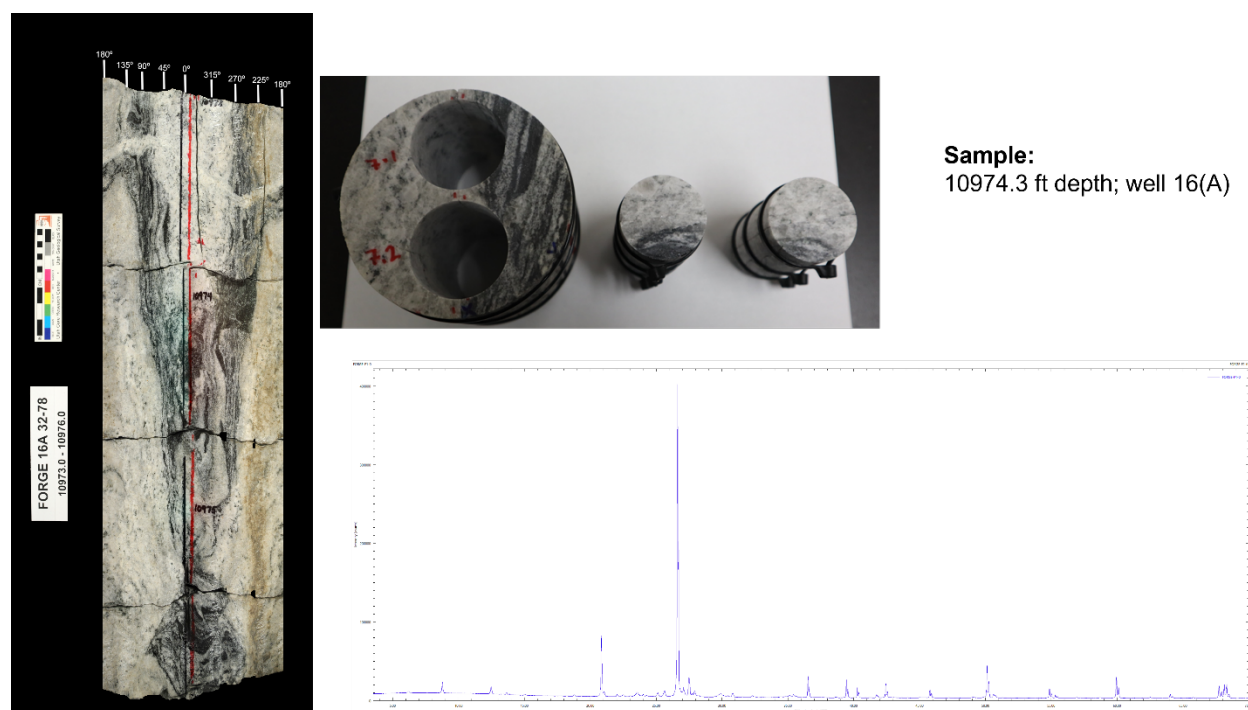


Figure 4: Core log photos (left), subsample photos (center top), and powder X-ray diffractogram results for sample prepared at depth of 10974 ft, from well 16(a).

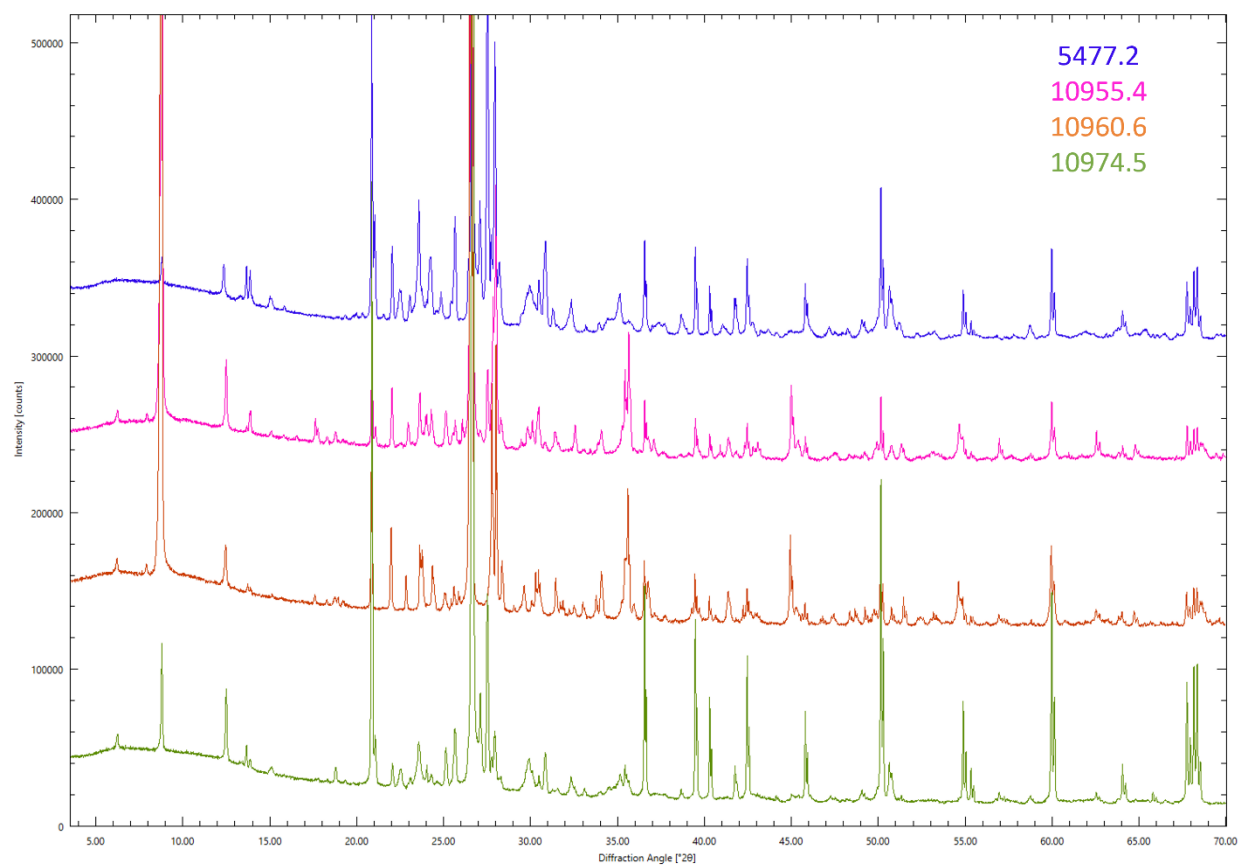


Figure 5: Peak intensity (counts) versus diffraction angle ($^{\circ}2\theta$) for the four samples. Sample depths are identified in the top right-hand region and are color-coded to individual scans, which are vertically offset by an arbitrary value to allow visual differentiation.

Sample:
5477.2 ft depth; well 16(A)

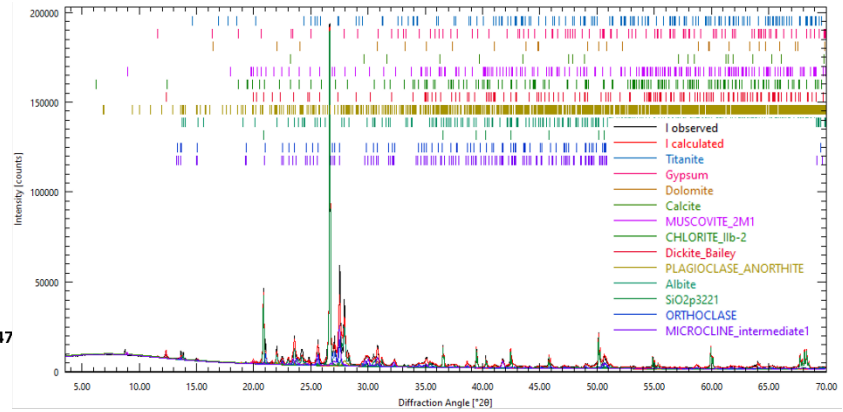
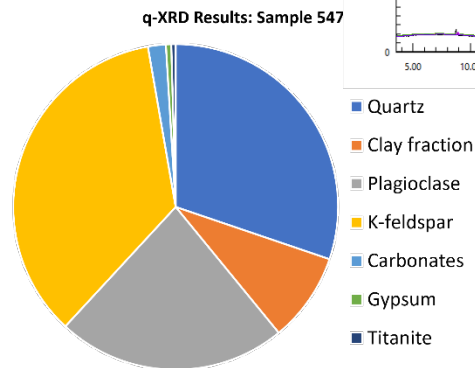


Figure 6: Illustrative example of q-XRD refinement results of analysis of XRD data from sample from 5477' depth. Core photo (bottom right) is shown for illustration; X-ray diffractogram show in top right with mineral phases selected for peak refinement. Results shown in bottom left in pie chart.