Test schedule for Inter-lab testing – Perm/Strength/Vel...
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Dear colleagues,

Here is the revised protocol for Inter-lab testing of Strength/Permeability/Wave Speed/Resistivity/Conductivity. Thank you all for your input in preparing this test procedure. Unless any new questions arise, we should conduct our first round of test in accordance with the following descriptions.

Please note that there was an error in the initial proposal: samples should be nominally 1 inch in diameter (see below).

Also, for thermal conductivity tests, we will only measure water-saturated samples (no dry tests).

Please contact me if you have further questions.

regards,
David
1) General comments for all tests

1.1) Please provide a brief description of how calibrations of pressures, displacements, etc. are carried out and how these can be traceable, for example, to NIST (in USA) or some equivalent agency.

1.2) If appropriate, a description of corrections should be provided. For example, in our high temperature friction test, Cu jackets have measurable shear strength that must be corrected for. Seal friction, when using an external load cell, is another example.

1.3) Estimates of uncertainties should be provided for all measurements. This will be useful when we compare results from different labs and different techniques.

1.4) Time history of stresses, displacements and related parameters should be recorded as appropriate for the types of measurement performed. We will decide at a later time how these will be included in final results.

1.5) Basic information should be recorded for all samples including:
   - dimensions
   - mass
   - dry bulk density
   - Basic determination of connected porosity should be performed on some portion of the samples (e.g., submerged, saturated weight test).

1.6) Samples should be preserved in case follow-up tests (i.e., thin section work) are wanted.

1.7) Note. Sample types can be identified as follows:
   - Crab Orchard sandstone – fine-grained, banded, 5% nominal porosity
   - Berea sandstone – lightly banded, 20% nominal porosity
   - Wilkeson sandstone (to be delivered) – medium grain size, 10% nominal porosity
   - Carrera marble – marble
   - Sierra white granite – medium-grained granite (to be delivered)
2) Strength Tests

Eight samples of each rock type should be prepared (cored parallel to up/down direction). Nominal sample dimensions: right cylinders, 2.5 inches long x 1.0 inch diameter. [This is our standard dimension. Other sample diameters are acceptable, i.e., 30 mm or 40 mm. An aspect ratio of 2.5:1 is preferred. Aspect ratio should be greater than 2.0:1.]

2.1) Crab Orchard sandstone: (DRY tests)

- Vacuum dry samples for 24 hr at 60°C (or 48 hr at room temperature). Run all tests room-dry – that is, equilibrated with relative humidity in laboratory.
- Perform constant strain rate tests at $10^{-5}$ sec$^{-1}$ axial shortening rate. (For example, this would be 0.635 micron/sec for a sample length of 2.5 inches.)
- Determine peak strength and (if possible) residual friction.
- One test each at constant confining pressures of: 0, 10, 20, 50, 150 MPa
- 3 repeat tests at constant confining pressure of 100 MPa

2.2) Carrera marble: (DRY tests)

Same sequence as Crab Orchard

2.3) Sierra granite: (DRY tests)

- Vacuum dry samples for 24 hr at 60°C (or 48 hr at room temperature). Run all tests room-dry.
- Perform constant strain rate tests at $10^{-5}$ sec$^{-1}$ axial shortening rate. (For example, this would be 0.635 micron/sec for a sample length of 2.5 inches.)
- Determine peak strength and (if possible) residual friction.
- One test each at constant confining pressures of: 0, 20, 60 MPa
- 5 repeat tests at constant confining pressure of 40 MPa

2.4) Berea sandstone: (WET tests)

- Vacuum dry samples for 24 hr at 60°C (or 48 hr at room temperature).
- Saturate with distilled water
- Perform constant strain rate tests at $10^{-5}$ sec$^{-1}$ axial shortening rate. (For example, this would be 0.635 micron/sec for a sample length of 2.5 inches.)
- Determine peak strength and possibly residual friction.
- Record volume change during deformation (if possible).
- Run all tests at constant pore pressure of 2 MPa.
- One test each at constant confining pressures of: 12, 22, 52, 152 MP
- 3 repeat tests at constant confining pressure of 102 MPa

2.5) Wilkeson sandstone: (WET tests)

Same sequence as Berea
3) Permeability Tests

Prepare 3 samples of each rock type (cored parallel to up/down direction). For now, I propose testing of only 1 or 2 samples for each rock type. Nominal sample dimensions: right cylinders, 1.969 inches long x 1.0 inch diameter. [Use different diameter samples if necessary; i.e. 30 mm or 40 mm]. Please prepare standardized sample lengths of 50 mm (1.969 inch) for all tests, regardless of sample diameter.

3.1) Note: Particularly for oscillating flow measurements, there is an optimum range of permeability/sample length/reservoir volume/oscillation frequency. Those of you who have performed these measurements are, no doubt, aware of these constraints. In the Fisher and Paterson (1992) notation, $\gamma$ represents the ratio: (down-stream reservoir storage capacity)/(sample storage capacity). For best results, design the down-stream reservoir for $\gamma$ between 1 and 100. Then, for optimum sensitivity in the inversion, oscillation period should be chosen to provide $\psi$ in the range between 1 and 3 – again, see Fisher and Paterson (1992). Pore pressure drop across the sample should be limited to a maximum of $\pm 5$ MPa.

3.2) Crab Orchard, Berea, Wilkeson, Sierra granite: (use distilled water)

Procedure:
- Fully saturate samples in the following manner: (i) Vacuum dry for 24 hr at 60°C (or 48 hr at room temperature). (ii) Introduce water into sample while still in vacuum chamber. [There are different ways to do this. The point is to avoid trapping air in the sample; especially in low-porosity samples.]
- Assemble sample column and place in pressure chamber.
- Evacuate to remove stray air bubbles and fill pore pressure system with water.

Comments on test conditions:
- Confining and pore pressure only; no differential stress.
- Tests should be run at nominal pore pressure of 10 MPa. For steady flow tests, high-side and low-side pore pressure should be chosen to provide average pore pressure of 10 MPa. (If this is not practical, then down-stream side of sample can be vented to atmosphere.)
- Wait 1 hour at each pressure before measurement to allow sample to relax.
- Care should be taken to avoid over-pressuring sample prior to permeability test to avoid complications from hysteresis of permeability.

Measure permeability in the following sequence of effective confining pressure:

10, 30, 60, 100, 30, 60, 100, 30, 60, 100 MPa
4) Ultrasonic Velocity Tests

Note: This should be about the same procedure as will be used in permeability tests. Prepare 3 samples of each rock type (cored parallel to up/down direction). For now, I propose testing of only 1 or 2 samples for each rock type. Nominal sample dimensions: right cylinders, 1.969 inches long x 1.0 inch diameter. [This is our standard dimension. If this presents problems for you, go ahead and use your own sample configurations. Use the 1.969 inch (50 mm) length if at all possible.] Keep track of NS and EW orientations.

4.1) Crab Orchard, Sierra granite, Carrera marble: **DRY** tests

Procedure:
- Vacuum dry for 24 hr at 60°C (48 hr at room temperature). Conduct tests on room-dry samples (that is, samples equilibrated with relative humidity of laboratory).

Comments on test conditions:
- Confining pressure only; no differential stress.
- Measure shear wave speed polarized in NS direction.

Measure P and S in the following sequence of confining pressure:

5, 10, 30, 60, 100, 30, 60, 100, 30, 60, 100 MPa

4.2) Berea: **WET** tests (use distilled water)

Procedure:
- Fully saturate samples: Vacuum dry for 24 hr at 60°C (48 hr at room temperature). Introduce water into sample while still in vacuum chamber. [There are different ways to do this. The point is to avoid trapping air in the sample; especially in low-porosity samples.]
- Assemble sample column and place in pressure chamber.
- Evacuate to remove stray air bubbles and fill pore pressure system with water.

Comments on test conditions:
- Confining and pore pressure only; no differential stress.
- Pore pressure can be vented to atmosphere.
- Wait 1 hour at each pressure before measurement to allow sample to relax.
- Care should be taken to avoid over-pressuring sample prior to each velocity test to avoid complications from hysteresis.
- Measure shear wave speed in NS orientation

Measure velocities in the following sequence of effective confining pressure:

5, 10, 30, 60, 100, 30, 60, 100, 30, 60, 100 MPa
5) Electrical Conductivity Tests

Prepare 3 samples of each rock type (cored parallel to up/down direction). For now, I propose testing of only 1 sample for each rock type. Nominal sample dimensions: right cylinders, 1.969 inches long x 1.0 inch diameter. [This is our standard dimension. If this presents problems for you, go ahead and use your own sample dimensions.]

5.1) Crab Orchard, Berea, Wilkeson, Sierra granite: (use 0.1 molar KCl$^{\text{aqueous}}$)

Procedure:
- Fully saturate samples: Vacuum dry for 24 hr at 60°C (48 hr at room temperature).
- Introduce brine into sample while still in vacuum chamber. [There are different ways to do this. The point is to avoid trapping air in the sample; especially in low-porosity samples.]
- Assemble sample column and place in pressure chamber.
- Evacuate to remove stray air bubbles and fill pore pressure system with brine.
- Flush equivalent of 1 pore volume of brine through sample.

Comments on test conditions:
- Confining and pore pressure only; no differential stress.
- Pore pressure lines can be vented to atmosphere during tests.
- Care should be taken to avoid over-pressuring sample prior to test at each confining pressure to avoid complications from hysteresis.
- Wait 1 hour at each pressure before measurement to allow sample to relax.
- Run test at 100 Hz to avoid electrode polarization.
- RECORD SAMPLE TEMPERATURE FOR EACH TEST
- Report results as conductivity and formation factor

Measure conductivity in the following sequence of effective confining pressure:

5, 10, 30, 60, 100, 30, 60, 100, 30, 60, 100 MPa
6) Thermal Conductivity Tests

Prepare 3 samples of each rock type (cored parallel to up/down direction). For now, I propose testing of only 1 sample for each rock type. Nominal sample dimensions: right cylinders, 1.0 inch long x 1.0 inch diameter. [You should use sample dimensions that are appropriate to your test configuration.]

6.1) Crab Orchard, Berea, Wilkeson, Sierra granite: WET tests (use distilled water)

Procedure:
- Fully saturate samples as described:
- Vacuum dry for 24 hr at 60°C (48 hr at room temperature).
- Introduce water into sample while still in vacuum chamber. [There are different ways to do this. The point is to avoid trapping air in the sample; especially in low-porosity samples.]
- Perform bench-top test if appropriate
- Assemble sample column and place in pressure chamber.
- Evacuate if needed to remove stray air bubbles and fill pore pressure system with water.

Comments on test conditions:
- Confining and pore pressure only; no differential stress.
- Pore pressure lines can be vented to atmosphere during tests.
- Care should be taken to avoid over-pressuring sample prior to test at each confining pressure to avoid complications from hysteresis.
- Wait 1 hour at each pressure before measurement to allow sample to relax.

Measure conductivity in the following sequence of effective confining pressure:

10, 40, 100, 10, 40, 100 MPa