

Why Sample Preservation Matters in Shale Conductivity Experiments

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Abstract. A shale conductivity test begins before the specimen enters the loading cell. Storage, drying, cutting, and fluid conditioning define the material state subsequently measured. This note treats preservation as an experimental variable and proposes a paired design for separating handling effects from fluid damage. The contribution is a practical chain-of-custody and comparison framework. It does not assume that rewetting restores native shale or that a preserved sample automatically reproduces all reservoir conditions.

1. Preservation as an experimental boundary condition

Preservation studies demonstrate changes in shale properties following altered handling and hydration [1]. A fluid-screening protocol likewise combines mineralogy, water activity, swelling, and mechanical measurements [2]. These observations make the label "shale core" insufficient to define a conductivity-test specimen. A sample that has equilibrated with laboratory air has experienced an intervention, even if its mineral composition appears unchanged.

The distinction is relevant to both propped and unpropped tests. Propped conductivity depends partly on the supporting surface [3], whereas rough unpropped flow depends on aperture distribution and contact [4]. Changes introduced before loading can therefore enter the experiment through different mechanisms. A preservation record should document what was done rather than certify an unmeasured claim of perfect native-state fidelity.

Kakkar examined preserved reservoir shale while comparing stress and fluid sensitivity of propped and unpropped fractures [5]. Wu and coauthors explicitly emphasized preserved samples in the 2017 investigation [6], and the 2019 paper explains the concern that desiccation can alter mechanical properties [7]. The conference and journal records are closely related; their similar preservation rationale is methodological continuity, not independent proof of a universal preservation correction.

Earlier water-based-fluid work provides another relevant conductivity context [8]. Comparison with Barnett laboratory studies [9] should keep sample provenance visible rather than pool all formation labels into a single population. An outcrop specimen, a freshly preserved core, and a dried reservoir specimen may each be useful, but they answer different questions.

A four-stage sample-history record

Proposed record for a conductivity study.

- 1. Recovery and storage.** Record time, seal, temperature, and fluid contact to define pre-test exposure.
- 2. Preparation.** Record cutting fluid, orientation, and air exposure to identify changes introduced during preparation.
- 3. Conditioning.** Record humidity or fluid, duration, and mass change to define the tested state.
- 4. Test entry.** Record geometry, baseline properties, and specimen ID to connect the history to the result.

Preservation should begin with a statement of what the experiment intends to preserve. Candidate attributes include native fluid content, mechanical response, surface condition, and the absence of unintended chemical exposure. These attributes are not interchangeable. A sealed specimen may retain mass while its temperature or stress history changes; a specimen with unchanged bulk mineralogy may still have altered fracture surfaces. The target should therefore be defined in terms of the later measurement.

For a conductivity study, propose a record that follows the sample from recovery through storage, cutting, fracture creation, assembly, and test entry. At each transition, identify who handled it, when the transition occurred, what contacted the sample, and which observations were made. This is an experimental traceability proposal, not a claim that one storage method guarantees native-state fidelity. Preservation and shale-fluid protocol studies provide the motivation for documenting those steps [1, 2].

Where a step cannot be controlled, record the departure instead of assigning the specimen a binary preserved/not-preserved label. An unknown air-exposure duration should remain unknown. A reconstructed history can still be useful if its sources and gaps are clear. The resulting record allows later analysts to decide which comparisons remain defensible rather than applying an undocumented correction to every specimen.

2. Specimen allocation and handling controls

Experimental units and paired comparisons

A preservation comparison requires a clear distinction between the recovered core, the prepared specimen, and the tested fracture. Two halves cut from one interval share a geological origin, but they are not independent examples of an entire formation. Conversely, several hydraulic measurements on one prepared fracture describe a trajectory rather than several independent specimens. A proposed study should identify each level before assigning handling treatments, so that apparent replication does not depend on how often the apparatus records a reading.

Consider a design with six adjacent core pairs. Within each pair, assign one specimen to the reference handling procedure and one to a deliberately altered procedure. The six within-pair differences are the primary comparisons. Plot those differences individually before reporting their average. If one pair has a large response and the others do not, preserve that information rather than presenting a smooth pooled curve that conceals it. Pairing helps address local variability but cannot guarantee identical initial fractures.

The design should also define when the fracture is created. Creating fractures before the handling intervention asks how existing surfaces respond to storage. Creating them afterward asks how altered rock produces and supports a new fracture. These are distinct experiments, even if the subsequent conductivity test is identical. Preservation and fluid-screening studies motivate careful control of initial state [1, 2]; the paired design proposed here makes that state part of the causal question rather than an undocumented preparatory detail.

Prepare neighboring specimens from a documented interval and assign them to preserved, deliberately dried, and dried-then-reconditioned groups. Record the imposed drying temperature, duration, and humidity; "dry" is not an adequate treatment specification. Retain an untreated control wherever material permits. For each group, measure initial mass, geometry, and a selected mechanical response before conducting the same conductivity schedule.

The central comparison is a ratio between treatment groups at matched stress and measurement conditions. For illustration, if preserved and dried specimens yield 0.20 and 0.10 mD-ft, the observed group ratio is two. It is not a reusable correction factor. If reconditioned material yields 0.15 mD-ft, it falls between the groups but does not demonstrate restoration. Replicate specimens and uncertainty intervals are required to decide whether these differences exceed geological scatter.

Fluid-damage measurement procedures provide a basis for consistent flow testing [10]. Mechanical-property studies motivate recording rock characteristics independently of conductivity [11]. Where resources permit, local indentation can help characterize exposure-sensitive surface properties [12], while longer-duration coupled exposure tests motivate recording conditioning time and temperature explicitly [13]. None of these auxiliary measurements should substitute for the hydraulic comparison itself.

A proposed handling experiment can use blocks of neighboring material, with each block supplying one specimen to each conditioning group. Analyze differences within blocks before pooling across the entire core. This structure reduces the chance that a treatment contrast simply reflects sampling different intervals. It does not make neighboring pieces identical, so record their initial dimensions, mineralogy, and visible fracture characteristics.

Measure absolute initial conductivity before normalizing the loading response. Suppose two synthetic groups begin at 0.20 and 0.10 mD-ft and both retain 50% under loading. Their normalized curves coincide, yet their loaded conductivities are 0.10 and 0.05 mD-ft. The normalization correctly describes proportional loss but hides the initial group difference. A preservation comparison therefore needs both the starting state and the subsequent trajectory.

If conductivity cannot be measured before conditioning without disturbing the specimen, say so and rely on randomized group assignment and independent baseline characterization. Do not create an unmeasured pre-treatment value by assuming that neighboring specimens were identical. The proposed design gains credibility from transparent limitations, especially when sample availability prevents a fully paired hydraulic measurement.

Exposure timing and laboratory batches

A specimen may spend months sealed and only minutes exposed during cutting, transfer, and assembly. Reporting total storage time alone combines periods with different environments. A proposed exposure clock records each interval separately: sealed storage, open preparation, contact with cutting fluid, conditioning, assembly, and time under load before flow. The clock should retain temperature and fluid identity where known. Missing intervals should remain marked as unknown rather than being assigned a nominal zero exposure.

The most useful summary depends on the comparison. If the hypothesis concerns air exposure, the cumulative open duration is relevant, but its distribution may also matter. Ten brief transfers and one continuous exposure have the same summed duration yet different sequences. The study can retain both the sum and the event list without presuming that they are physically equivalent. An initial screening experiment could compare those two histories directly while holding the later conductivity procedure fixed.

This approach also clarifies delays caused by equipment availability. A specimen waiting in its intended sealed condition is different from one waiting after fracture preparation. Record actual events rather than replacing them with the planned schedule. Where timing uncertainty is appreciable, give an interval for the exposure duration. Such records make it possible to ask whether a conductivity difference follows the assigned handling treatment or an unplanned delay shared by only one group.

If every reference specimen is tested during one week and every altered specimen during another, the treatment contrast also includes any change in apparatus condition or operator practice. A proposed schedule interleaves treatments within practical constraints and records maintenance, sensor replacement, and calibration events. When complete randomization is impossible, describe the blocking arrangement. The purpose is to prevent a convenient laboratory schedule from becoming an unrecognized explanation for the observed handling effect.

Reference checks before and after a batch can reveal instrument drift, but they cannot reconstruct an unobserved specimen history. Keep apparatus checks and rock controls as separate records. A stable reference flow path supports confidence in the measurement system; a stable preserved-rock control supports a different claim about specimen behavior. Neither alone establishes that all treatment groups experienced identical stress, temperature, and timing throughout their tests.

A simple analysis can report within-batch treatment differences alongside the overall difference. If the sign changes across batches, inspect the event records before fitting a more complicated preservation model. Report whether the discrepancy tracks specimen provenance, handling duration, or apparatus intervention. This procedure makes a negative or inconsistent result useful: it identifies the conditions under which the handling comparison remains unresolved and guides the next experiment toward the source of that uncertainty.

3. Indicators of specimen change

Mass and mechanical observations

A mass change can help identify that a specimen experienced a change in state, but it does not uniquely locate the change at hydraulically important contacts. Surface liquid, retained fluid, lost particles, and weighing conditions can affect the observation. Specify the weighing procedure and its timing relative to handling. A reproducible mass record is useful even when its physical interpretation remains incomplete.

Similarly, local hardness or modulus measurements should be linked to location and loading protocol. A set of measurements away from the eventual fracture surface may characterize the

rock but provide only indirect evidence about the contacts controlling flow. The mechanical and indentation studies cited here motivate auxiliary measurements [11, 12]; they do not justify converting a percentage modulus change directly into the same percentage conductivity change.

A proposed interpretation sequence is to first establish that handling changed a measured property, then ask whether the hydraulic response changed under matched conditions, and finally examine a mechanism connecting them. If only one link is observed, preserve that distinction. This avoids treating a correlation among mass, hardness, and conductivity as proof that one variable completely mediates the effects of preservation.

Repeated weighing can provide a useful handling indicator if the weighing procedure itself is standardized. Define whether surface liquid is retained, drained, or removed by a specified procedure. Record container tare, instrument resolution, elapsed time outside storage, and the point in the preparation sequence. A change in total mass is an observation; assigning that change exclusively to loss of native pore water requires additional evidence about fluids, dissolved material, and loose solids.

For a hypothetical specimen initially weighing 50.00 g, a later reading of 49.50 g represents a 1.0% decrease relative to initial total mass. It does not establish that water saturation fell by one percentage point. That interpretation would require pore volume and a defensible fluid inventory. If the comparison instead uses dry-rock mass as its denominator, state that denominator explicitly and explain how it was obtained without changing the specimen used for conductivity testing.

A mass trend can trigger closer examination without serving as an automatic exclusion rule. Predefine a handling tolerance if the experiment requires one, and retain the data from specimens that fall outside it with their status documented. Mechanical measurements and hydraulic baselines provide complementary observations [10, 11]. Agreement among several indicators is more informative than treating a single unchanged mass reading as proof that the specimen's mechanical or surface state has remained unchanged.

A restoration challenge

A useful extension of a preservation study is a restoration challenge: compare a reference specimen, an intentionally altered specimen, and an altered specimen subsequently subjected to a specified reconditioning procedure. The last group tests whether selected observables return toward the reference. It should not be labeled restored to reservoir condition merely because its mass or visible appearance resembles the reference. State the reconditioning fluid, duration, temperature, and the observable used to define an apparent return.

An illustrative result could show normalized mass returning from 0.99 to 1.00 while conductivity at a specified stress returns only from 0.40 to 0.55 of the reference value. Those selected values would indicate recovery of one measured quantity without recovery of the other. They would not identify the cause of the remaining hydraulic difference. Initial geometry, mechanical response, and exposure chemistry would still need examination before attributing that difference to irreversible damage.

The preserved-shale evidence emphasizes that loading and fluid histories can leave persistent conductivity changes [5, 6, 7]. It therefore supports testing reversibility rather than assuming it. A restoration challenge is particularly useful when researchers wish to compare legacy dried material with newly preserved samples. It can define which measurements are comparable after the proposed conditioning, while keeping any remaining differences visible instead of applying an unsupported universal correction factor.

4. Interpretation and transferability

Avoiding an unsupported correction

Avoid normalizing every curve by its own initial value before first inspecting absolute conductivity. Such normalization can hide a preparation-induced difference present before loading. Report absolute and normalized curves together, along with the sample-history record. Water-damage experiments also show why the final gas-flow measurement must retain the preceding exposure history in its description [14].

A preservation-sensitive result may reflect altered aperture, mechanical support, surface chemistry, or several of these together. A difference in hardness does not uniquely quantify a difference in conductivity. The proposed experiment therefore uses auxiliary measurements to test explanations, not to impose a predetermined causal assignment. If a treatment changes initial geometry, report that change directly rather than absorbing it into a fluid-damage coefficient.

Departures and data reuse

An article should describe the actual sample history alongside the intended history. Include storage duration, known fluid contact, preparation conditions, and the interval between conditioning and testing. Where available, report an image or measurement before and after preparation. These records help distinguish a treatment effect from an assembly change, particularly when a fracture has been split and reassembled.

The findings should be transferred only to an explicitly defined population. A comparison among three handling states from one interval does not establish a universal multiplier for an entire formation. Different initial surface geometries and support conditions can alter the

hydraulic response [3, 4]. A useful transfer statement identifies the sample class, the handling departure tested, and the conditions under which its effect was measured.

The most informative follow-up is often a replication of the handling contrast in another block or interval, rather than a longer sequence of readings on the same specimen. Repeat readings refine measurement precision; new specimens test variability. Keeping these roles separate makes preservation research useful to later conductivity studies without suggesting that any laboratory history fully reconstructs the reservoir.

A reusable dataset should end with a short transfer statement describing the population and state actually tested. For example, a study might support comparisons among neighboring preserved specimens subjected to the same cutting and assembly protocol, while leaving comparisons with outcrop material untested. This statement is more precise than declaring the data representative of shale generally. It tells a modeler which differences were controlled and which additional assumptions are needed to apply the results elsewhere.

The transfer statement can distinguish measured, documented, and inferred conditions. Fracture orientation may have been measured; storage temperature may have been documented as a range; native fluid retention may remain inferred from the handling protocol. Keeping those categories separate prevents an administrative record from being mistaken for direct characterization. It also allows a later user to revise one uncertain assumption without discarding the entire conductivity dataset.

For a cross-laboratory comparison, exchange the event histories and endpoint definitions before comparing fitted coefficients. Agree on a common comparison stress and exposure state, then show both absolute conductivity and relative change. If the records do not support an equivalent starting state, present the datasets as complementary experiments. Their difference may still be scientifically valuable, but it cannot be uniquely assigned to mineralogy, fluid chemistry, or preservation without a design that separates those factors.

5. Conclusions

Preservation should be treated as a documented boundary condition and, where possible, a tested factor. A defensible report states the native-state target, the actual handling history, and the departures that could not be avoided. Paired experiments can reveal sensitivity to those departures. They cannot make a specimen identical to the reservoir, but they can make its limitations explicit and comparisons across laboratories substantially more interpretable.

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Numbered references correspond to the citations in the text. Links lead to DOI records or the identified source record.

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