INITIAL REPORT OF THE PETROPHYSICS LABORATORY --

1977-1979 ADDENDUM

by

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From the time the author-copy of Circular 789 (Hunt and others, 1979) was prepared until it appeared in print, significant changes occurred in the Petrophysics Laboratory. To update that report, this open-file report will briefly summarize more recent acquisitions and facilities changes.

ELECTRICAL PROPERTIES

The facilities to measure and investigate electrical properties have been augmented by the acquisition of:

1) another computer with higher speed data acquisition and processing capabilities, including extended graphics and a removable 20 MByte disk.

2) modification of the chamber in Figure 15 of Circular 789 for use as a microwave anechoic vacuum chamber. (See Olhoeft and others, 1979a.)

3) more digital instrumentation, including spectrum analyzers, time domain reflectometers, 0.5-1300 MHz network analyzer, 0-200 amu residual gas analyzer (added to chamber in Figure 14 of Circular 789), digital 4-electrode, return-current shield LCR meters, and other instruments. The frequency range of measurement is now 10^{-6} to 10^7 Hz by four-terminal, driven-shield instrumentation fully automatically, by two-terminal semi-automatically to 1.3×10^9 Hz, and manually to 4×10^{10} Hz.

4) a variety of new and redesigned sample holders, including:

   a) brine sample holder to measure electrical resistivity on brines up to salt saturation as a function of frequency, temperature to 600°C, pressure to 30 MPa hydrostatic, and salt concentration. (See Ucok and others, 1979a,b; Ucok, 1979.) Materials contacting the sample are limited to 3:2 Mullite, platinum, and Inconel.

   b) core sample holder to measure simultaneously and independently the electrical resistivity of brine-saturated rocks and of the saturating fluids under the same conditions as the brine sample holder except with a temperature limit of 400°C. (See Figure 1 and add Kalrez to the materials list.)

   c) electrochemical corrosion cell to study the details of physical chemistry reactions and kinetics involved in nonlinear complex resistivity (Olhoeft, 1979a).

     d) combination cell to simultaneously measure streaming potential, nonlinear complex resistivity, and hydraulic permeability in solution-saturated rocks under confining pressure, and differential pore pressure across the length of the sample, at various frequencies for temperatures up to 400°C. (See Figure 2.)

5) a 100-MHz impulse radar system for CDP (common depth point) determinations of in situ dielectric and scattering properties as well as depth profile surveys. (See Olhoeft, 1978e, 1979b, and Olhoeft and others, 1979b.)

6) nonlinear complex resistivity borehole logging system for in situ electrical property and geochemical investigations (Olhoeft and Scott, 1980).

7) the added capability to measure seismic velocities and attenuations and/or thermal properties by using the mathematical formulations of Circular 789 equations 9 through 13 (pages 6 and 7), but after changing the data acquisition transducers from electrodes to piezoelectric lithium niobate transducers or to thermisters and/or thermocouples.

Four-terminal, driven-shield sample holders are required for the measurement of wet-sample electrical properties to eliminate the effects of electrode polarization and other sources of error. Figure 3 illustrates the typical geometry of the electrical measurement on a core sample. Current flows through the sample and the electrical properties are determined by measuring the electric field response, E, to the applied current density, J, and computing the resultant transfer function. Error sources include effects due to electrochemical charge transfer processes at the electrode-sample boundaries, the effects of current flow around the sample due to sample surface conduction and/or sample holder leakage paths, and capacitative or inductive coupling between various wires used to attach the sample to the measurement apparatus. There may also be instrument and apparatus errors such as sample holder resonances, bridge transformer nonlinearity, sample loading by insufficient input impedance, and others, but these will be ignored in this discussion.

Figure 4 illustrates an example of a measurement geometry in which these errors are minimized. The most significant error due to electrode effects is minimized by using separate current injection and electric field monitoring electrodes (four terminals). At the current injection electrodes, +J and -J,

there is still a charge transfer process, but as the current is
everywhere the same throughout the system, this electrode process does
not contribute to the measured potential drop across the sample and thus
does not influence the measurement. The electrochemical charge transfer
process at the electric field (or potential) monitoring electrodes, +E and
-E, is minimized by using extremely high input impedance amplifiers (above
10^{11} ohms) to reduce the current drawn through this loop to a charge-
tunneling level below the levels required to initiate Faradaic charge
transfer processes. For many rocks and minerals, this high-input-impedance
separated-electrode assembly is sufficient to reduce electrode effects from
an order of magnitude error to less than a fraction of a percent. In many
minerals however, the metal of the electrodes will directly react with the
material of the sample if they are in direct contact. To minimize this effect,
the platinum field monitoring electrodes are separated from the sample by a
very thin layer of fumed silica paste (made from 2:1 volume ratio of fumed
silica and 0.1 molar KCl solution), which removes the contact polarization
reactions and introduces an additional error of less than 0.3 percent in
resistance magnitude, less than 2.3 milliradians phase, and less than 0.1
percent THD (total harmonic distortion). Porous teflon membranes work nearly
as well as fumed silica, but "filter paper", plastic sponges, fritted glass,
many porous ceramics, and related papers are unsuitable (either because of
much larger errors or instabilities with time).

To reduce leakage currents and the effects of surface conduction, the
sample may be potted as shown in Figures 10 and 11 of Circular 789 or
electronic circuitry may be employed to cancel the surface current. In the
three-terminal dielectric measurements of Figures 3 to 7 in Circular 789,
the guard electrode circuitry accomplishes this removal of leakage and surface
currents while also cancelling the coupling of the wires. In the four-terminal
arrangement, driven-shield guarding may be employed as in Figure 4. The
driven shields of the first-stage isolation amplifiers (gain = 1), G, are
connected to a third pair of electrodes on the surface of the sample, +D and
-D. These guard electrodes are driven by the circuitry to the same potential
as their counterpart guarded electrodes, +E and -E. As the E and D electrode
counterparts are driven to the same potential, no current may flow along the
surface of the sample. If the physical separation of the E and D electrodes
is very small, the current is driven into the interior of the sample to

become totally volume current flow. The driven shield circuits also increase the effective input impedance of the shield monitor and reduce capacitative coupling. A driven common-mode shield should be added to reduce inductive coupling effects if necessary.

MAGNETIC PROPERTIES

A large-sample magnetic susceptibility bridge has been constructed from specially-wound coils and a standard lock-in amplifier. A cryogenic-detector vibrating-sample magnetometer has been constructed for use with very small samples and investigations of the magnetic properties of single grains.

SPECTROSCOPIC PROPERTIES

Instrumental measurement capabilities have been extended by the addition of:

1) a modern UV-visible-NIR spectrophotometer with much more flexibility than the one described in Circular 789. The wavelength range is from 190-800 nm with better than 0.5 nm resolution and from 800-3000 nm with better than 0.3 nm resolution. The instrument now has a variable scan rate and intensity expansion, and it can operate in transmission, absorption, and first or second derivative modes with data recorded on chart paper and magnetic tape. Computer programs have been written to store and manipulate the data in a variety of ways, including differentiation, addition, subtraction, and averaging.

2) an integrating sphere has been attached to the spectrophotometer to improve the signal to noise ratio when measuring the reflection and transmission data of geological materials and vegetation.

3) an excitation flourescence attachment for the spectrophotometer.

4) a manual pellet press for producing samples of fine particulates mixed with "Halon".

TEXTURAL PROPERTIES

In addition to the hydraulic permeability described above under electrical properties, the statistical properties of 16 rock types have been compiled to study the statistical distribution of dry bulk density and grain density. From the statistical dependence of these properties, the statistical distribution of other properties may be inferred through

the known relations between dielectric permittivity, seismic velocity, thermal conductivity, and others to dry bulk density (see Olhoeft, 1979e).

Lastly, the Petrophysics Laboratory can characterize the porosity to acquire helium (or other inert gas) -accessible porosity, mercury-accessible porosity, water-accessible porosity, occluded porosity (by crushing the sample), and total porosity. These numbers may differ from each other by as much as factor of two in limestones and basalts (Johnson, 1980). Also, the pore size distribution may now be characterized by direct measurement with mercury penetration porosimetry and by inference from isothermal gravimetric adsorption of various gases.

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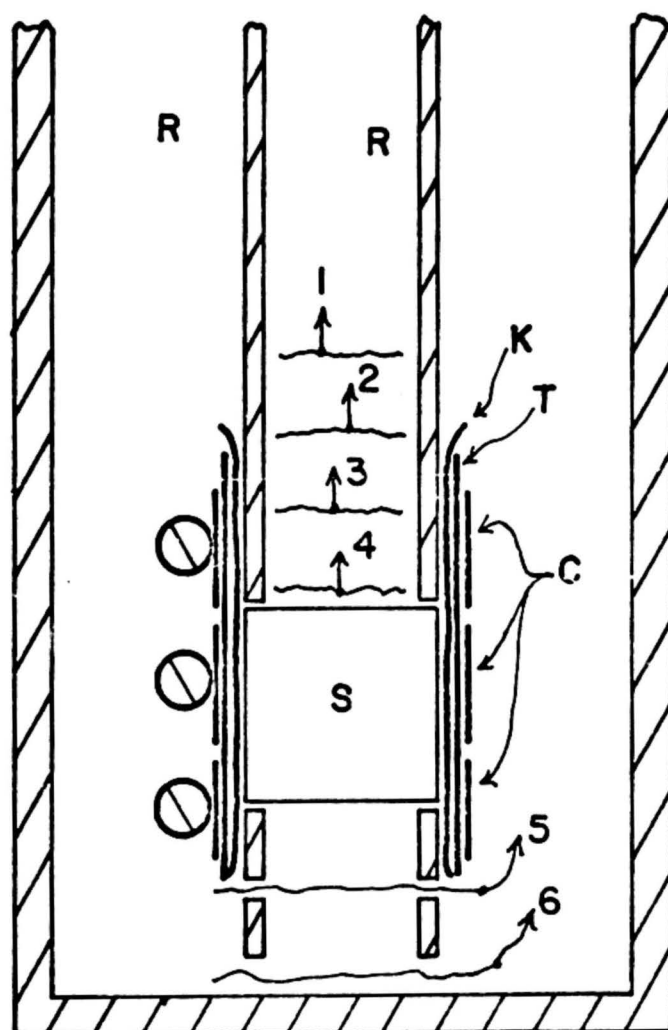
Figure 1 - The geothermal sample holder for measuring independently the electrical resistivity of a brine and a brine-saturated sample. S is the sample, K is the Kalrez per-fluoroelastomer sleeve, T is a stainless steel sheet clamped by stainless steel hose clamps (C) around the sample, R denotes the reservoirs of brine. Electrodes 1-2-3-4 are the +J, +E, -E, and -J electrodes, respectively, for measuring the resistivity of the brine. Electrodes 3-4-5-6 are the +J, +E, -E, and -J electrodes for measuring the resistivity of the brine-saturated sample. All electrodes are platinum mesh. The remaining unlabelled pieces are 3:2 mullite ceramic. The whole assembly is inserted into an Inconel pressure vessel. A type-S thermocouple is inserted in the outer, lower brine reservoir near the sample at the level of the middle stainless steel clamp.
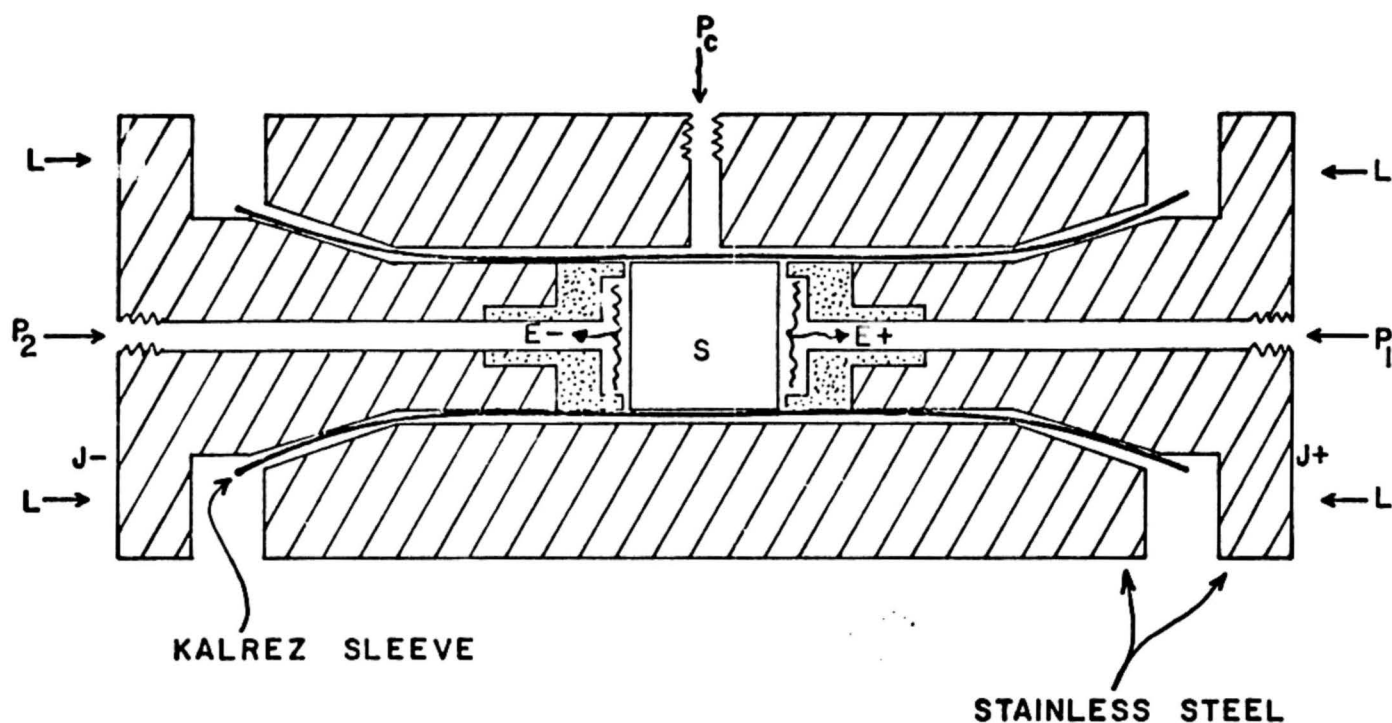
Figure 2 - The combination sample holder for measuring streaming potential, electrical resistivity, and hydraulic permeability with independently controlled pore pressure (P1 and P2) and confining pressure (Pc). The sample, S, is contained within a Kalrez sleeve and clamped between the stainless steel end pieces and outer cylinder by load, L. The long, tapered clamping area is to prevent the Kalrez sleeve from tearing. The stainless steel end pieces are the current electrodes, +J and -J, for the resistivity measurement, while platinum mesh electrodes, E+ and E-, participate in both resistivity and streaming potential measurement. The dotted region around the E-electrodes is a highly porous ceramic insulator. The wires from the E electrodes are brought to the outside down the P1 and P2 channels through ceramic insulators and then pressure feedthroughs.
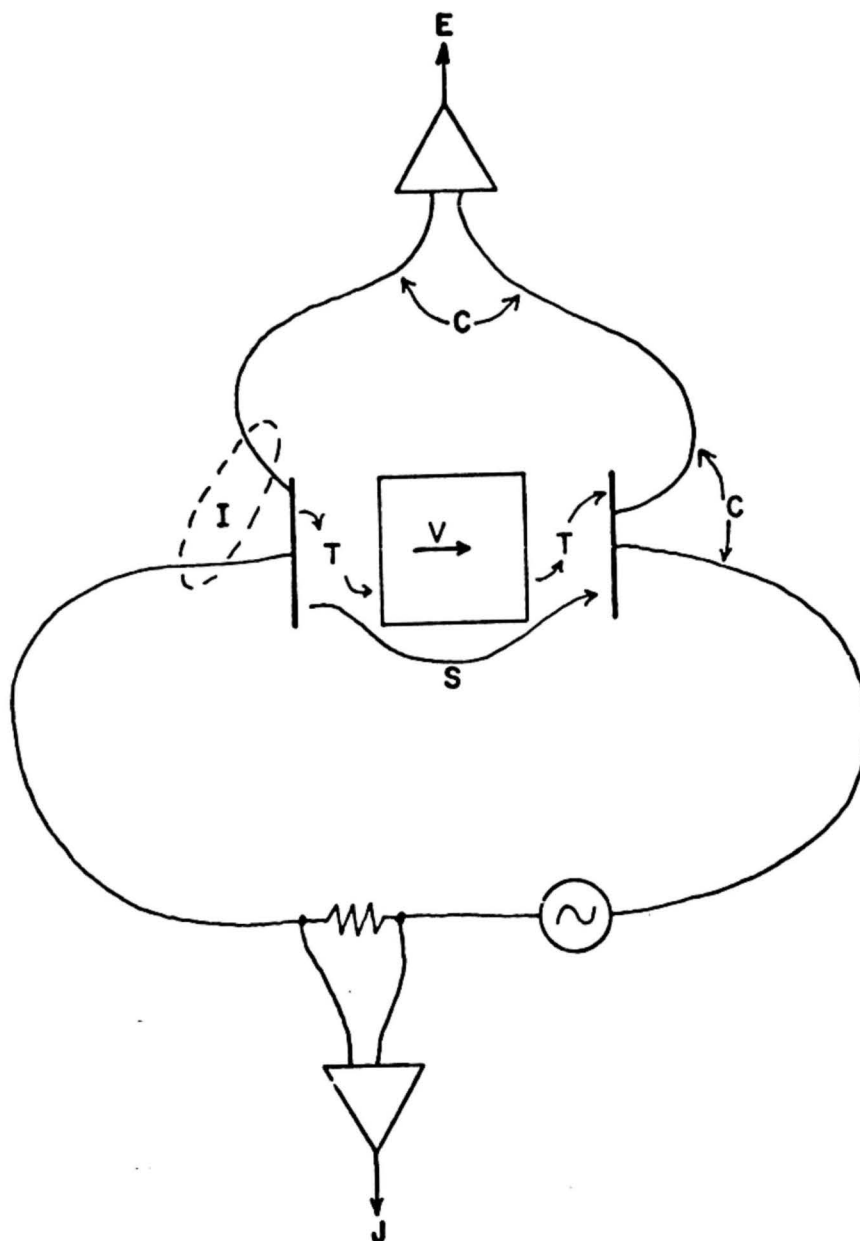
Figure 3 - Schematic illustration of several sources of error in electrical properties measurements on wet samples. V is the volume current conduction through the sample. S is the surface conduction and leakage paths around the sample. T represents the charge transfer impedances between the sample and the electrodes. I is the inductive coupling between the current and potential leads (J and E). C is the mutual or cross capacitance between wires connecting the sample holder to measurement instruments. Instrumental errors are not shown.
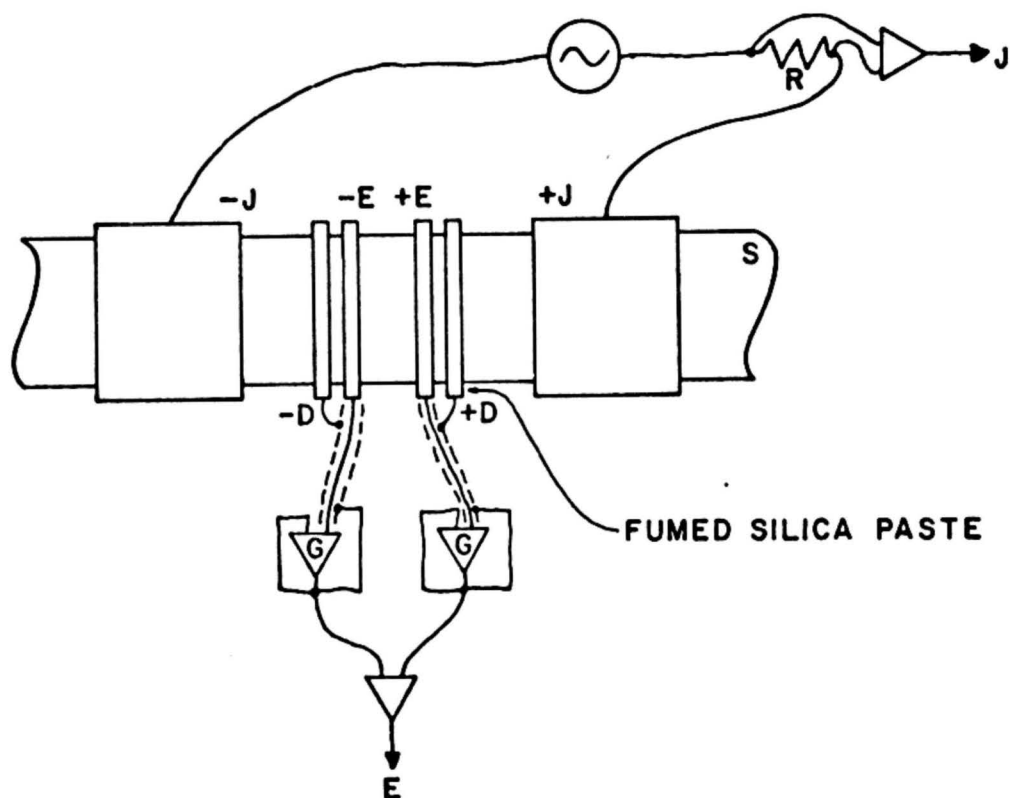
Figure 4 - Schematic illustration of a four-electrode sample holder which avoids or minimizes the errors of Figure 3. +J and -J are platinum current electrides connected to the core sample, S. +E and -E are platinum potential electrodes connected to the core through a thin layer of fumed silica paste between the electrodes and the sample. +D and -D are driven-shield guard electrodes (driven by preamps G to the same potential as seen by E) to prevent surface conduction and leakage paths around the core and also to minimize capacitative coupling effects. Inductive coupling may be minimized by use of common mode driven shielding (not shown).