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Dielectric measurement of rock samples

The Petrophysics Laboratory at CSIRO offers a range of dielectric measurement options in the frequency range of millihertz (mHz) to gigahertz (GHz) for solid and drill cutting samples.

Dielectric properties of rock are indicators of wettability, porosity, mineralogy, pore structure and pore geometry. In industry, dielectric logging is simple and more rapid than other logging techniques. Dielectric logging may offer satisfactory determination of moisture content and mechanical properties where other techniques such as nuclear magnetic resonance (NMR) and laboratory analysis of core samples are slower and less cost effective. It also provides useful information for resistivity logging, ground probing radar, borehole radar and SP logging.

The Laboratory conducts dielectric spectrographic analysis of rock samples including core samples provided by industry and other research organisations, as well as powdered drill cuttings samples. A combination of instruments is used to achieve complete frequency coverage from 1 mHz to 3 GHz and operation at overburden pressure.

4-terminal apparatus

The Petrophysics Laboratory has developed a 4-terminal dielectric/ resistivity cell to handle cylindrical samples; either cut cores or packed material. Rock samples can be subjected to independent confining and fluid pressures of up to 30 MPa. The cell is connected to an Autolab FRA30 potentiostat (EcoChemie, Netherlands), which enables single or multi-sine measurements over swept frequencies from 10 µHz to 10 MHz, using an



4-terminal dielectric/resistivity measurement is used in the frequency range from 1 mHz up to 10 KHz. An Autolab FRA30 potentiostat is connected to the in-house developed 4-terminal cell.

autobalancing impedance bridge circuit. The preferred frequency range for the entire apparatus is 1 mHz up to 10 KHz.

Parallel plate cell

Parallel plate capacitance measurement is the most commonly recognised dielectric spectroscopy method and typically achieves its best performance from 1 KHz to 10 MHz with a large volume of investigation. The CSIRO Petrophysics Laboratory has developed a 3-terminal capacitor dielectric cell connected to an Agilent 4294A impedance analyser, to offer improved measurement over the common 2-terminal capacitor. The cell requires disc-shaped samples that are dimensionally stable with faces milled to be both smooth and parallel, to avoid the creation of an air gap. Parallel plate dielectric measurement is possible from 100 Hz to 100 MHz, however, the preferred range is limited to 1 KHz to 10 MHz.



The parallel plate cell is used for thin discshaped samples in the range from 1 KHz to 10 MHz using an impedance analyser.

Miniature parallel plate cell

The miniature parallel plate cell has been designed for small chips and rock fragments created during borehole drilling. Despite their size, rock fragments obtained during drilling more accurately represent in-situ formation geology than pressed/reconstituted drill cuttings. The miniature parallel plate cell is designed to allow the in-situ dielectric characteristics of the rock to be more properly captured. The cell requires a large rock drilling fragment, machined to fit inside the cell, and is analysed with an Agilent 4294A impedance analyser.



Drawings of the miniature parallel plate cell developed for small rock fragment samples.

Small loaded coaxial transmission line

Coaxial transmission line dielectric cells are suitable for frequencies exceeding the range achieved with parallel plate cells, whilst retaining a large volume of investigation. The small loaded coaxial transmission line cell is designed to analyse synthetic standard reference materials and small quantities (~15 g sample) of drill cuttings. It can also be used for any sample (including competent rocks) that can be machined to fit. The small loaded coaxial transmission line offers high precision in the range of 1 MHz to 3 GHz when used in conjunction with an Agilent E5070B network analyser.



Using a coaxial transmission line cell the sample (white) is loaded between the inner and outer conductors.

Large loaded coaxial transmission line

The large loaded coaxial transmission line cell has been designed to fit the standard 1.5 inch core sample and to be operated at overburden pressures of up to 35 MPa. The samples are cut from core and a hole is drilled through the centre. The apparatus can then be externally, axially loaded to achieve a plain strain condition and recreate effective overburden pressure. The cell is then attached to an Agilent E5070B network analyser and offers high precision measurement of dielectric properties from 1 MHz to 3 GHz.



Overburden pressure dielectric analysis is achieved using the cell depicted above. The sample is loaded into the coaxial cell for dielectric analysis and a purpose-built loading rig is applied to simulate in-situ overburden pressure.

End loaded transmission lines

End loaded transmission lines are often favoured in dielectric spectroscopy for their simplicity and rapid acquisition of data. Although they require a prepared flat face, they are still generally the fastest of all the dielectric measurement procedures and they are useful from ~1 MHz to 3 GHz. The CSIRO Petrophysics Laboratory employs three different probe diameters, a commercially obtained 2.5 mm (Agilent 8E5070B) and two additional custom-made probes of 5 mm and 10 mm. These are used in conjunction with an Agilent E5070B network analyser. Although they are rapid and simple to operate, analysis in the laboratory has demonstrated that reliability and repeatability are questionable and caution must be used. Measurement redundancy is always used to achieve confidence in

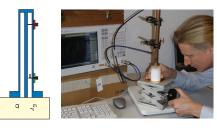
the repeatability, and the sample must be carefully placed against the probe to avoid an air gap.



The end loaded transmission line probe is simply placed against the sample. A network analyser is used to complete the measurement.

End loaded cavity perturbation probes

End loaded cavity perturbation probes are discrete frequency, high precision devices, and a number of these are used to achieve an adequate set of data points in the frequency range from 100 MHz to 1 GHz. An Agilent E5070B network analyser is used to determine the frequency shift of the resonant cavity for both standards and unknowns. The range of frequencies achieved in the laboratory is 100 MHz to 1 GHz, and diameters of the probes designed are above 10 mm so that a reasonable volume of investigation is achieved.



The resonant cavity probe is simply placed against the sample. A network analyser is used to complete the measurement.

Synthetic standards

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It is common in dielectric spectroscopy to require a range of standards to which results can be compared. For geological dielectric spectroscopy, lossy standards are also required to emulate the results observed for shales. We have developed a number of in-house dielectric synthetics which we can machine to fit any of the above cells, to cross-correlate the performance of the various logging systems and to gain more confidence in our results.

Additional petrophysics and rock strength services

Dielectric properties are often related to porosity, mineralogy and microstructure. The CSIRO Petrophysics Laboratory also uses x-ray tomography, fluid porisimetery/permiametery, NMR, seismic propagation, triaxial testing and other techniques to offer a complete range of services and information about rock samples provided by our clients.

Submitting samples

CSIRO is able to process a wide range of samples common in petrophysics research and the extractive industries. The Laboratory has been designed to cope with solid rock samples including cores and solid chunks, and crushed rock samples, particularly drill cuttings, as well as liquid samples which are associated with drilling and pore fluids.

Dielectric and other measurement services are customised to each client's individual requirements. CSIRO can suggest what kind of samples a client should provide and how the samples should be stored immediately after recovery to optimise the quality of the measurement procedure. The Laboratory can facilitate measurement of single dielectric samples as well as batches of hundreds of samples. Please contact us to discuss your individual requirements.

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