Electrical Property Measurements of the Meade Peak Phosphatic Shale Member of the Permian Phosporia Formation, Caribou County, Idaho

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Introduction

The U.S. Geological Survey (USGS) has studied the Permian Phosphoria Formation in southeastern Idaho and the Western U.S. Phosphate Field throughout much of the twentieth century. In response to a request by the U.S. Bureau of Land Management (BLM), a new series of resource and geoenvironmental studies was initiated by the USGS in 1998. These studies involved many scientific disciplines within the USGS and consist of: (1) integrated, multidisciplinary research directed toward resource and reserve estimations of phosphate in selected 7.5-minute quadrangles; (2) elemental residence, mineralogical and petrochemical characteristics; (3) mobilization and reaction pathways, transport, and disposition of potentially toxic trace elements associated with the occurrence, development, and use of phosphate rock; (4) geophysical signatures; and (5) improving the understanding of depositional origin.

This report presents the preliminary laboratory results of electrical property measurements made on samples from the Meade Peak phosphatic shale member of the Permian Phosporia Formation. The samples are from a more-weathered (section A) and less-weathered (section B) section of the phosphatic shale member, collected from the Enoch Valley mine located in Caribou County, Idaho. These sections are described in detail by Tysdal and others (1999). The electrical measurements were made to determine if samples from different units can be differentiated by their geophysical signatures.

For this laboratory study, the electrical properties were measured using the complex resistivity (CR) method, also known as spectral induced polarization. The instrumentation measures the sample's resistivity and phase as a function of frequency. Resistivity and phase are complex quantities, having both real and imaginary components, hence the method's name. CR measurements are made by transmitting electric current through the sample and measuring a received voltage. The current is transmitted as a sine wave and measurements are made over a wide band of discrete frequencies. CR data are plotted as resistivity and

phase spectra, which show the frequency dependency of the measured electrical properties.

Resistivity

Resistivity is a measure of a material's opposition to the flow of electric current. Resistivity is the inverse of electric conductivity, the ability to transport electric charge. Resistivity units are ohm meters (ohm-m). The resistivity of rock and sediment are dependent on several factors including the amount of water present, porosity, the amount of total dissolved solids (TDS) in pore water, and mineral composition. The amount and TDS of pore water are typically the dominant factors that determine a geologic sample's resistivity. All other factors being equal, dry rock is more resistive than saturated rock, and rock saturated with low TDS water is more resistive than rock saturated with high TDS water.

Phase

Phase is the angle of lag, or lead, of a sine wave with respect to a reference. Phase units are in milliradians (mR). Phase is a measure of charge storage or polarization. For these measurements the phase of the received voltage is measured using the current wave form as the reference. When electric current is transmitted through a material, a temporary induced polarization effect is generated. Induced polarization is a redistribution of charged particles, such as ions and electrons, from their equilibrium position due to the applied current. This redistribution or storage of charge is not an instantaneous process but requires a finite period to develop and to dissipate as the current varies. The time required to develop and dissipate the stored charge produces the phase lag observed between the received voltage and the transmitted current.

Frequency Dependency

Frequency dependency results from different mechanisms of charge transport and charge storage. Some of these mechanisms operate rapidly and are observed at relatively high frequencies, whereas slow mechanisms are

observed at lower frequencies. At low frequencies, the electrical properties of rocks and minerals are dominated by charge transport or conduction mechanisms, whereas at high frequencies charge storage or polarization mechanisms dominate.

A quantitative measure of frequency dependent resistivity, historically used to interpret induced polarization measurements, is the percent frequency effect (PFE) defined as:

PFE = $100(\rho_{low} - \rho_{high})/ \rho_{high}$

Where:

ρ_{low} = low frequency resistivity

ρ_{high} = high frequency resistivity

For this report, PFE values were calculated using data from 0.1Hz and 1.0Hz, which are given in table 1.

Instrumentation

The electrical properties presented here were measured using the non-linear complex resistivity (NLCR) impedance spectroscopy instrumentation in the USGS, Crustal Imaging and Characterization Team, Petrophysics Laboratory located on the Denver Federal Center. This NLCR instrumentation is a modification of the instrumentation designed by Olhoeft (1985) and described by Jones (1997). The NLCR instrumentation measures electrical properties over a wide band of frequencies ranging from 10^{-3} to 10^{6} Hz (millihertz to megahertz) with three discrete frequencies being measured per decade.

The NLCR instrumentation is limited in its ability to measure highly resistive material. As received, all of the Meade Peak samples were too resistive for the NLCR system to measure. In order to measure the samples, laboratory grade deionized water was added to lower their resistivity.

Samples were measured in a cylindrical sample holder made of white virgin Teflon. The sample occupies a space 1 inch in diameter and three inches long, having a volume of 2.36 cubic inches (38.62 cm^3). Electrical signals were

transmitted to, and received from, the sample through four equally spaced electrodes made from bright platinum mesh. Two electrodes at the ends of the sample holder were used to transmit current through the sample. The voltage response of the sample was measured across two electrodes spaced 1 inch apart in the center of the sample holder. Further description of the laboratory procedures and apparatus used to make these measurements can be found in Campbell and Horton (2000).

<u>Sample Preparation</u>

Six samples were measured for this report. Each sample was measured a number of times with different water weight percents. The measured samples are splits of larger samples that have been sub-sampled for mineral (Knudsen and others, 2000) and chemical (Herring and others, 1999) analysis. The samples consisted of broken rock fragments ranging in size from fine-powder to ~2cm^3. Samples as received were dry, no attempt was made to preserve the original moisture content. Typically, samples are measured in their "as received" condition. However, because the Meade Peak samples were too resistive, no as received measurements were completed. All measurements presented here were made after laboratory grade, deionized water was added to each sample to lower its' resistivity.

The samples were measured at three different water contents including, moist, 10% water by weight, and saturated. Percent water was determined by weight. For the moist measurement just enough water was added to uniformly wet the sample, typically 4 to 5 percent by weight. The mixture was thoroughly stirred then loaded into the sample holder for measurement. For the saturated measurement the sample holder was unloaded and water was stirred into the sample until saturated. Typically 13 to 14 percent water by weight was required to saturate a sample. In the following discussions the samples will be referred to as "moist" or "saturated". Water weight percent for each sample measurement is given in table 1.

The resistivity of rock and sediment samples varies as a function of water content. Therefore, it may be inappropriate to compare resistivity measurements made on samples having different water contents. For comparison purposes, a third set of measurements were made on a suite of samples each having 10 percent water by weight (table 2). For the 10 percent water samples, water was added to a dry split, thoroughly stirred then loaded into the sample holder for measurement.

Results

The results of the electrical property measurements are presented as resistivity and phase spectra given in figures 1 through 17 and summarized in tables one and two. The horizontal axis of each figure is logarithmic scaled frequency. Each figure has two traces, with resistivity plotted as square symbols (left legend, linear scale) and phase plotted as triangle symbols (right legend, logarithmic scale). Phase values are normally negative and are plotted as a solid triangle. However, if the phase is positive, unusual but possible, the sign of the value is changed and plotted as an open triangle.

Table 1 give a summary of properties from moist and saturated measurements, including water weight percent, 0.002, 0.1 and 1.0Hz resistivities; PFE, phase peak magnitude in milliradians (mR) and phase peak position in frequency (Hz). Table 2 gives the electrical properties of the samples measured at 10 percent water content, for comparison purposes. Table 3 gives the XRD mineral analysis of the Meade Peak samples (from Knudsen and others, 2000).

Discussion

In general, the samples all have similar shaped resistivity and phase spectra, showing a resistivity decrease with increasing frequency; and a low-frequency phase dispersion typically centered at ~2 Hz. The higher frequency

data, typically greater than 10^4, were very noisy and have been deleted from the spectra.

Resistivity

The low frequency resistivity of the Meade Peak samples varies from 28 to about 2550 ohm-m, depending on water weight percent. For the moist measurements resistivities ranged from 277.4 to 2551.85 ohm-m. For the saturated measurements the resistivities drop an order of magnitude, ranging from 28.6 to 240.6 ohm-m.

As expected, the addition of water lowers the resistivity of samples. In most cases, the resistivity decreases as the water content increase. However, the resistivity of sample wpsb139 increased from 659 to 760 ohm-m when the water content was increased from 5.3 to 10 percent. The cause of this anomalous behavior is uncertain; however, it is probably due to sample heterogeneity. Because the sample was unloaded, mixed, and reloaded for each measurement, the actual measured material may be slightly different. If a sample was not sufficiently homogeneous, it is possible that different sample splits had different mineral compositions and or a different size fraction resulting in a different observed resistivity.

There are some notable correlations between sample mineralogy and sample resistivity. The most conductive sample, moist or saturated, was wpsb157. This sample does contain the highest total amount of clay minerals (muscovite, illite and kaolinite), which may produce the low observed resistivity. The most resistive sample, moist or saturated, was wspa040. This sample differs from the others in that it is from the more-weathered "A" section, however, its bulk mineralogy is not significantly different than some samples from the less-weathered "A" section. Interestingly, sample wspa040 does have a relatively high total clay content, about 8 percent. Being from the more-weathered section, the high resistivity may be due to a loss of soluble ions, such as Ca and Na, which would reduce ionic conduction resulting in a relatively high resistivity.

Phase

The phase magnitude of the Meade Peak samples varies from –2.49 to –232mR; whereas phase peak positions range from 0.02 to 50Hz. For the moist measurements phase magnitude ranged from –50.55 to –141.66mR with peak positions ranging from 1.0 to 20Hz. For the saturated measurements phase magnitude ranged from –2.49 to –95.62mR with positions ranging from 0.02 to 50Hz. Samples typically exhibit single phase peaks; however, sample wpsb059 when saturated shows two low-magnitude peaks at relatively low frequencies. Phase magnitude always decreases between the moist and saturated measurements; however, 10 percent water weight phase values are generally higher than moist measurements.

As with the resistivity data, there are some notable correlations between the observed phase magnitude and peak position with sample mineralogy. Sample wpsb157 has the highest phase peak position (20-50mR) of all the samples. The high peak position may be related to the samples high clay content. Clay minerals typically have a small grain size resulting in a high surface area available for surface polarization processes. Smaller grain size may increase the frequency of polarization responses (Bertin and Loeb, 1976; Sumner, 1976).

Sample wpsb059 has the lowest overall peak magnitude (-2.49 to –78.34mR) and lowest peak positions (0.02 to 1.0Hz). This sample also has the highest carbonate fraction, about 23 percent. however, it is unclear if the low phase response in due to the relatively high carbonate content. Unmineralized carbonate rocks have been observed to have a low polarizability (Bertin and Loeb, 1976).

Frequency dependency

PFE values provide a quantitative measure of frequency dependency. PFE values vary considerably as a function of water weight percent. In general, the PFE values decrease between the moist and saturated measurements, with the exception of sample wpsb117. However, for two samples, wpsa040 and

wpsb139, the PFE increases at 10 percent water then decrease at higher saturations. The cause of this intermediate increase may be due to the heterogeneous nature of the samples and the fact that a different split was used for the 10 percent water weight measurements.

10 Percent water weight comparison

For the 10 percent water weight samples, wpsa040 has the highest resistivity, 1055 ohm-m. This is the only sample from section A, the more-weathered section, and may be more resistive due to weathering. Sample wpsb157 has the lowest resistivity of all samples and the highest clay content. The low resistivity probably results from the high clay content.

PFE values range from 3.85 to 34.68 percent for the 10 percent water weight percent samples (table 2). Sample wpsb139 had the highest PFE, whereas sample wpsb157 had the lowest. Interestingly, sample wpsb139 does have the highest apatite content and the lowest total clay, whereas sample wpsb157 has the lowest apatite content and the highest total clay.

The electrical properties of the 10percent water weight samples have some notable correlations with sample mineralogy. The sample with the highest phosphate (apatite) content, wpsb139, has the highest PFE, 34.68 percent, and largest magnitude phase peak, -232 mR. Of the samples from section B, wpsb139 also has the highest resistivity, more than twice that of other section B samples. This suggests that phosphate-rich parts of the section may be differentiated from non-phosphate bearing rock by their higher resistivity and frequency dependent electrical properties.

The sample with the highest clay content, wpsb157, has the lowest resistivity, lowest PFE and the highest frequency phase peak. This suggests that waste units may be differentiated from phosphate bearing rock by their lower resistivity and frequency dependent electrical properties.

Summary

Electrical property measurements were made to characterize the electrical geophysical properties of samples from the Meade Peak phosphatic shale member of the Phosphoria Formation. The electrical properties vary primarily as a function of water content but also as a function of sample mineralogy. As received, the Meade Peak samples were too resistive to measure. The addition of water lowered the resistivity of samples. Moist samples are an order of magnitude more resistive than saturated samples. In general, the PFE values decrease between the moist and saturated measurements,

The most resistive sample was from section A, the more-weathered section. Of the section B samples, the sample with the highest phosphate content also had the highest PFE, largest magnitude phase peak, and the highest resistivity. The sample with the lowest phosphate content has the highest clay content, the lowest resistivity, lowest PFE and the highest frequency phase peak.

These preliminary laboratory observations suggest that frequency dependent electrical property measurements have some potential in differentiating barren from ore-bearing shale members. However, it is important to note that only six samples were measured for this study. Additional measurements, on a larger phosphatic shale sample suite, are warranted.

References

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Table 1. Water weight percent, 0.002, 0.1 and 1.0Hz resistivity measurements, PFE, phase peak magnitude (in milliradians) and position (in frequency) for the Meade Peak samples.

Sample	Water %	resistivity 0.002Hz	resistivity 0.1Hz	resistivity 1.0Hz	PFE	Phase mR	Peak Hz
wpsa040.1	5.41	1272.49	1227.16	1155.27	6.22	-63	5
wpsa040.3	14.43	190.11	186.84	180.21	3.68	-59.96	5
wpsb047.1	5.15	2551.85	2360.42	2133.38	10.64	-102	5
wpsb047.2	11.29	240.57	234.08	218	7.38	-80.49	5
wpsb059.1	5.38	1272.39	1161.44	1036.24	12.08	-78.34	1
wpsb059.4	17.02	75.26	75.51	75.02	0.65	-5.84	1
						-2.49	0.02
wpsb117.1	4.84	736.76	595.87	592.59	0.55	-141.66	2
wpsb117.3	13.86	134.29	132.27	123.69	6.94	-95.62	2
wpsb139.1	5.29	659.78	642.32	600.85	6.90	-59.97	5
wpsb139.4	14.15	163.23	161.9	157.59	2.73	-19.91	1
wpsb157.1	5.04	277.4	264.25	253.31	4.32	-50.55	20
wpsb157.3	13.82	28.58	28.38	28.17	0.75	-36.82	50

Table 2. Comparison of resistivity measurements made at 0.002, 0.1 and 1.0Hz, PFE, phase peak magnitude in milliradians and phase peak position (in frequency) for the Meade Peak samples at 10% water weight percent.

Sample	Resistivity 0.002Hz	Resistivity 0.1Hz	Resistivity 1.0Hz	PFE	Phase mR	Peak Hz
wpsa040.2	1055.49	960.82	829.15	15.88	-143.16	2
wpsb059.2	346.44	321.02	298.00	7.72	-48.66	0.2
wpsb059.3	386.61	353.89	323.59	9.36	-59.33	0.2
wpsb117.2	255.32	268.00	254.21	5.42	-80.22	2
wpsb139.2	760.42	668.65	498.40	34.16	-226.50	1
wpsb139.3	748.45	661.48	491.14	34.68	-232.59	1
wpsb157.2	62.31	63.35	61.00	3.85	-139.52	50

Table 3. Mineral analysis in percent of the Meade Peak samples obtained by X-ray diffraction (from Knudsen and others, 2000). Total clay is the sum of muscovite, illite and kaolinite.

Sample	apatite	quartz	muscovite	illite	albite	orthoclase	buddingtonite	kaolinite	dolomite	calcite	Total Clay
wpsa040	55	25.3	6.7	0.1	0.1	2.8	9.1	1.1	0	0	7.9
wpsb047	56	22.3	4	0.1	2.3	1.6	4	0.5	8.4	0	4.6
wpsb059	50	10.9	5	0.1	0.6	0.1	9.2	0.6	21.7	1.6	5.7
wpsb117	21.7	33.7	11	0.1	8.4	1.6	10.9	0.4	6.6	0.2	11.5
wpsb139	93	3.8	2.4	0.1	0.1	0.1	0.1	0	0	0	2.5
wpsb157	10.2	49	17	5.4	4.7	1.4	2.5	0.3	2	0.4	22.7
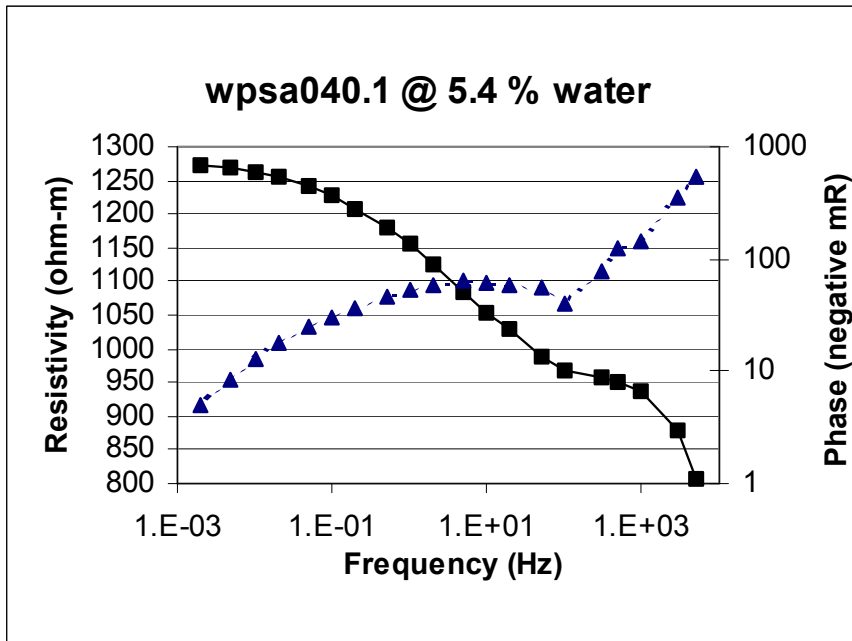
Figure 1. Sample wpsa040.1 at 5.4 water weight percent.
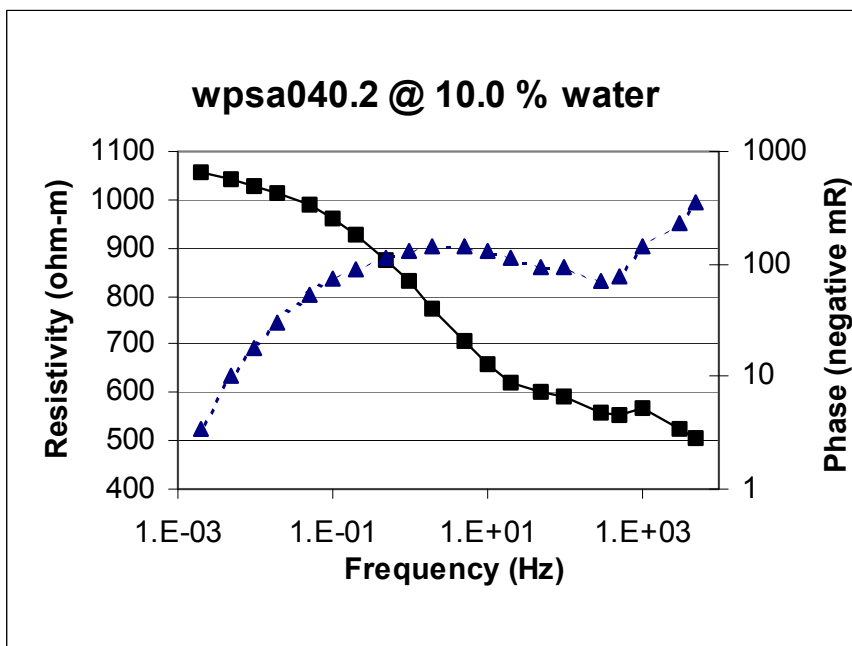
Figure 2. Sample wpsa040.2 at 10 water weight percent.
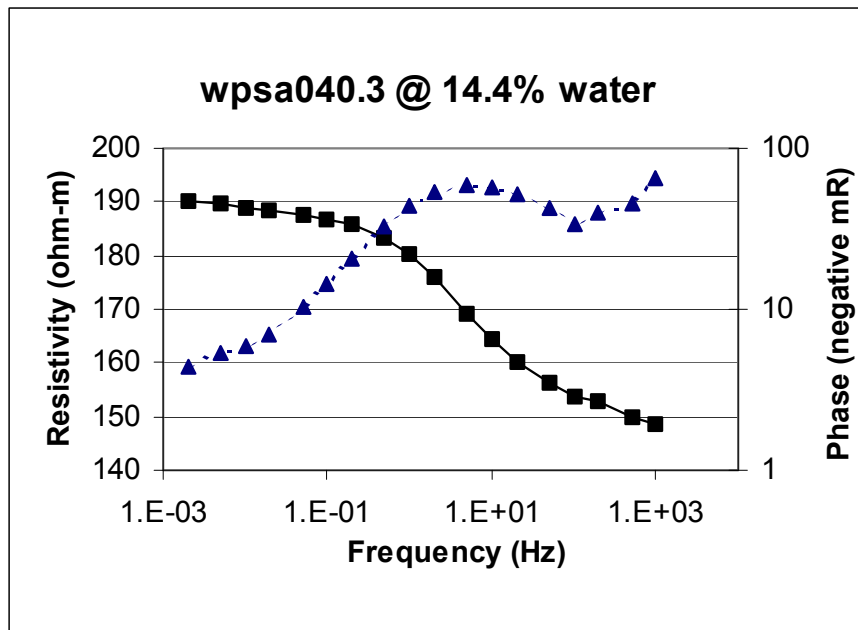
Figure 3. Sample wpsa040.3 saturated at 14.4 water weight percent.
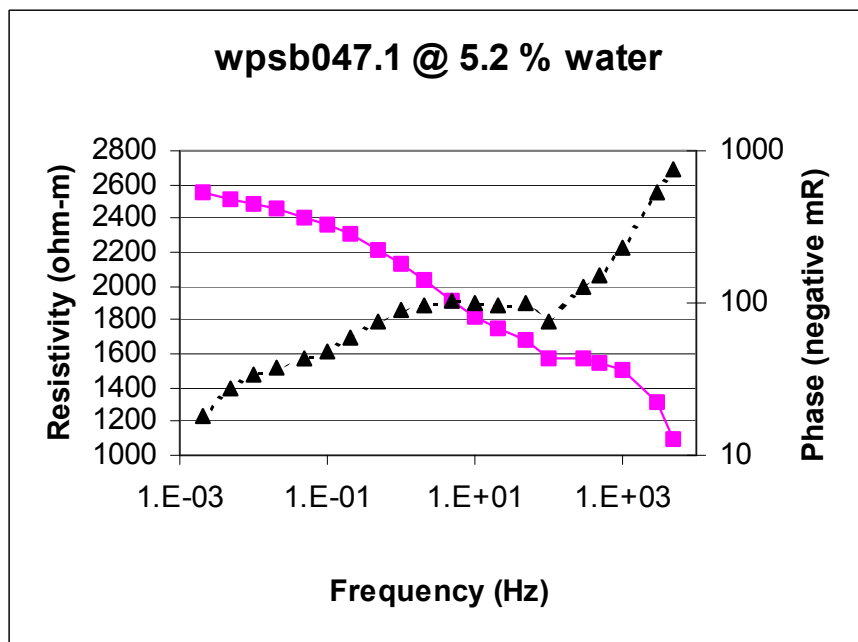
Figure 4. Sample wpsb047.1 at 5.2 water weight percent.
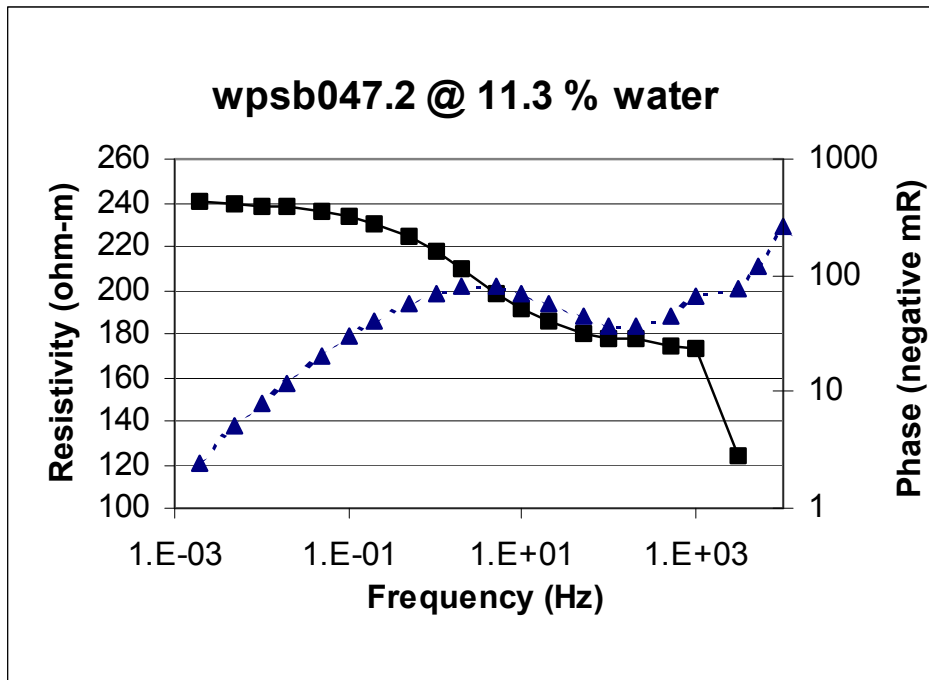
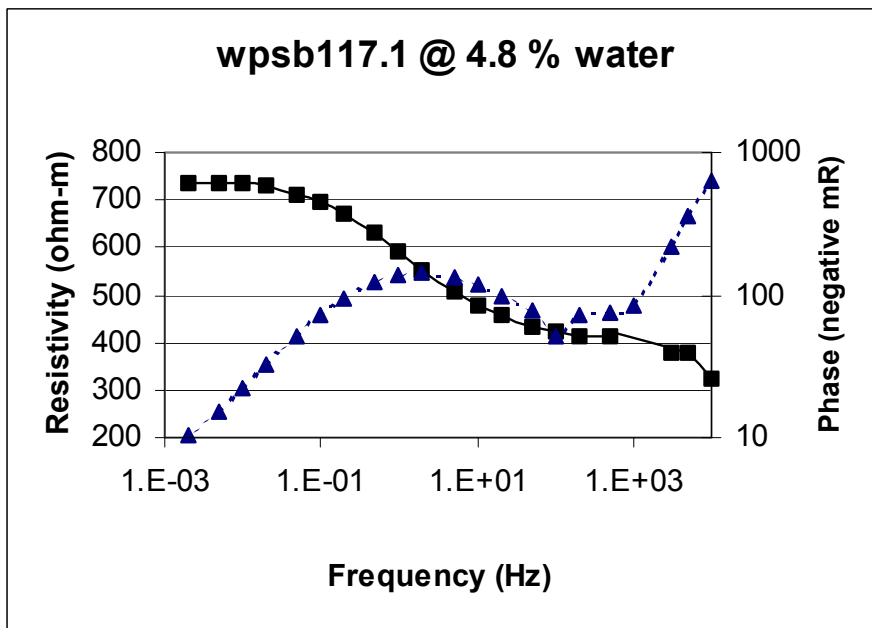Figure 5. Sample wpsb047.2 saturated at 11.3 water weight percent.
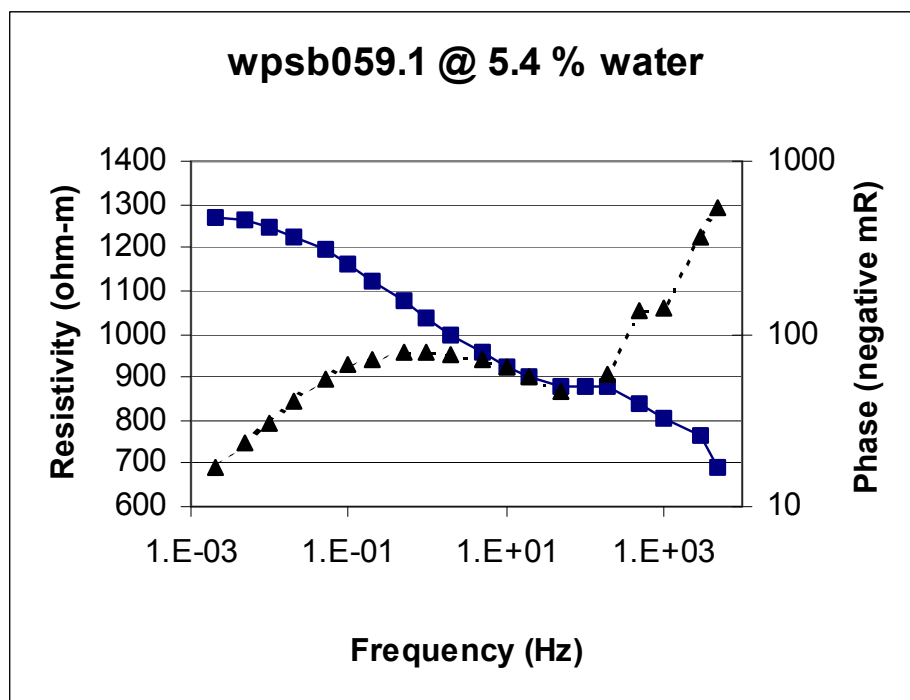
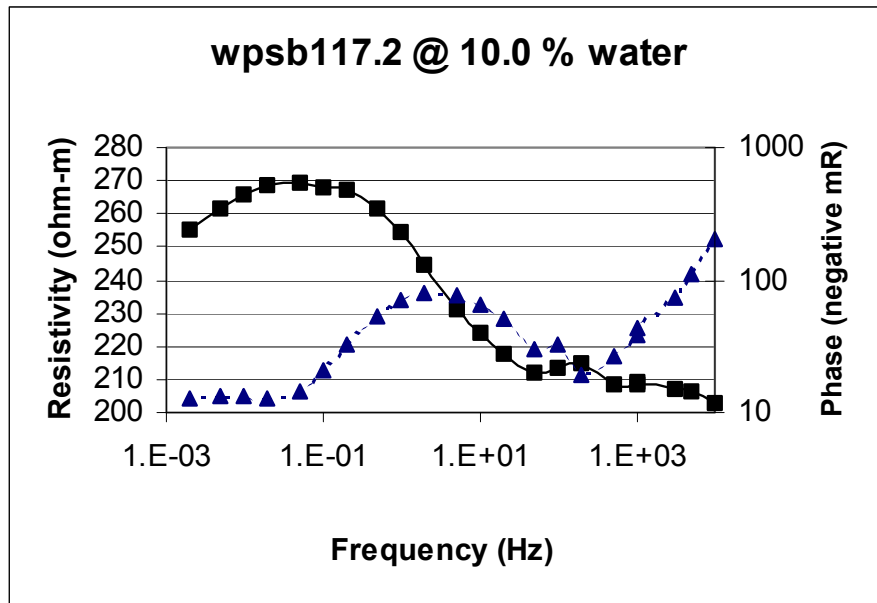

Figure 6. Sample wpsb059.1 at 5.4 water weight percent.
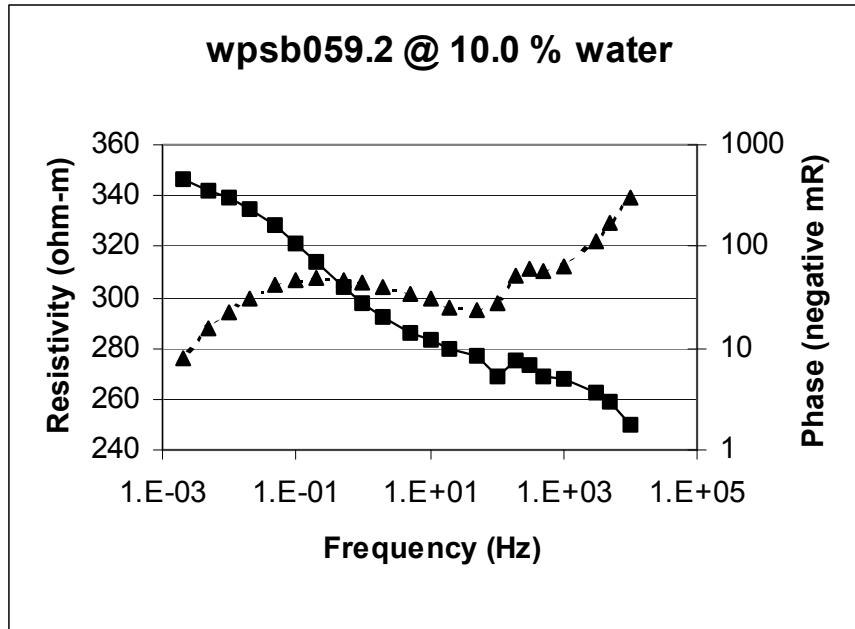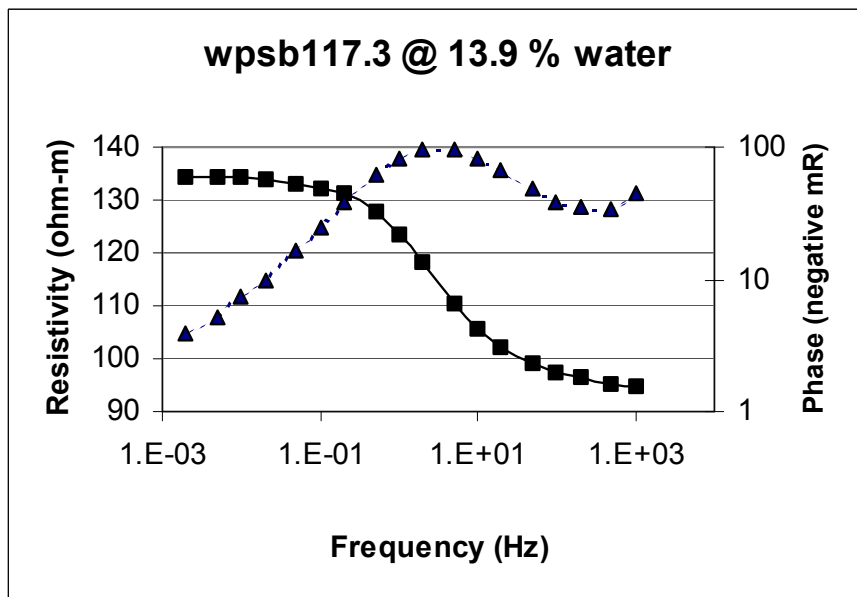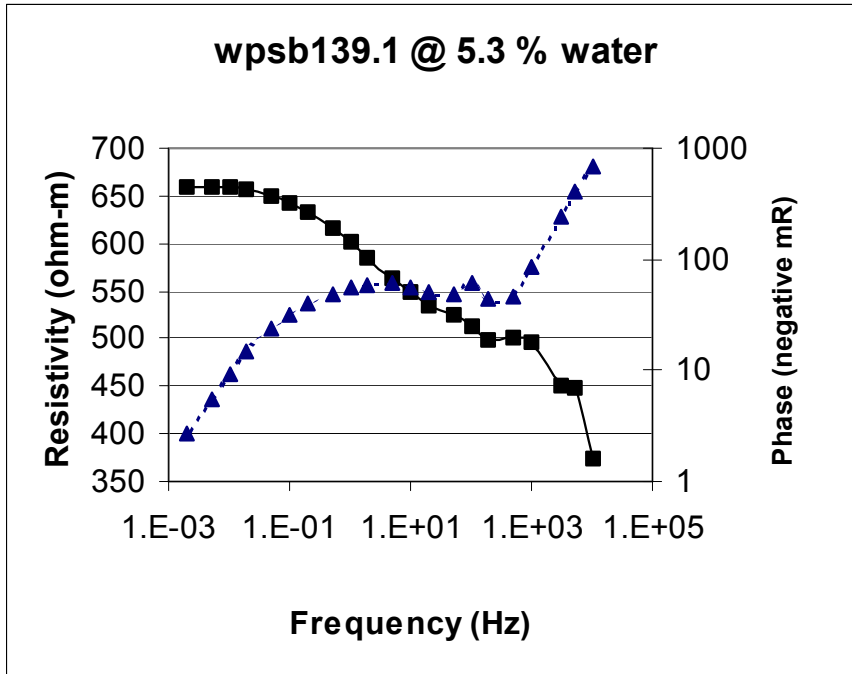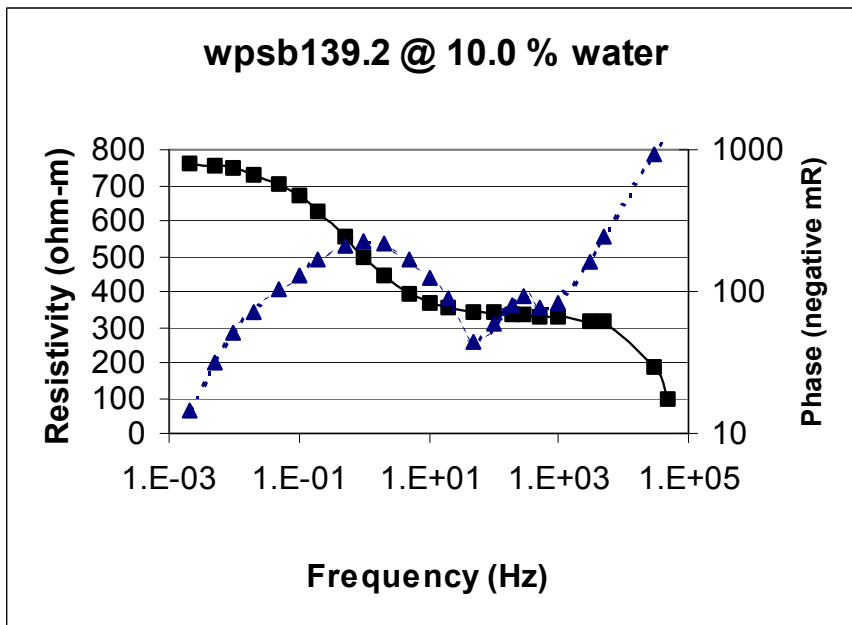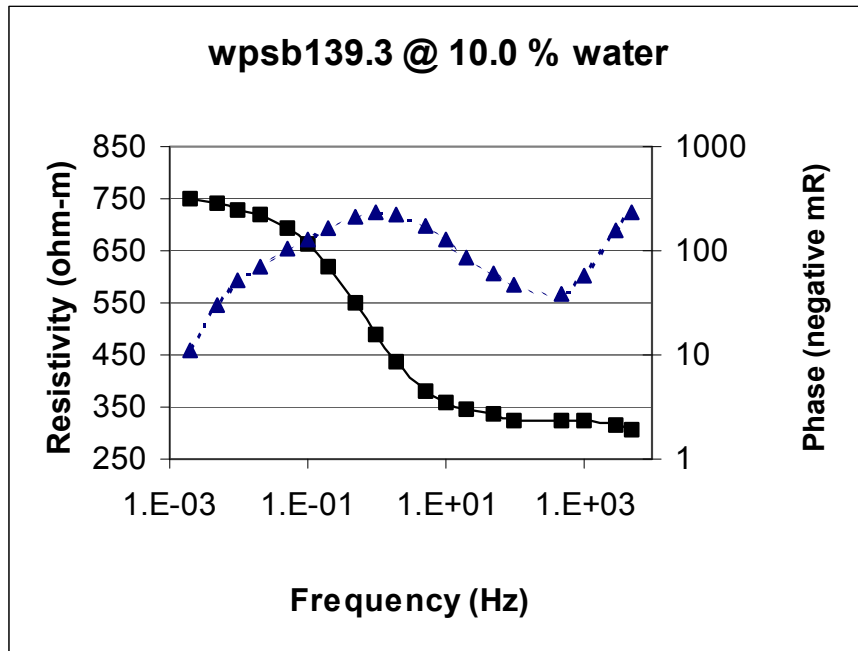
Figure 6. Sample wpsb059.2 at 10 water weight percent.
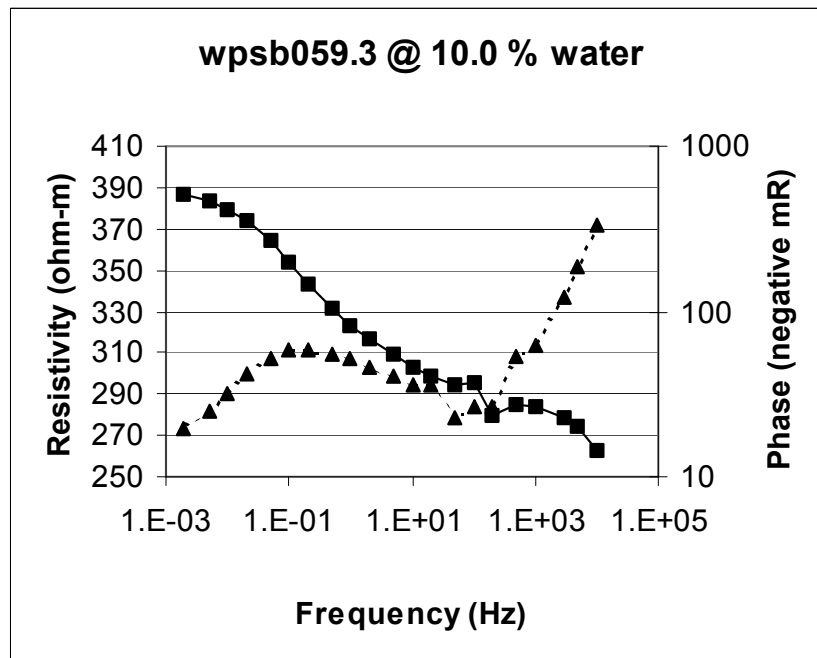
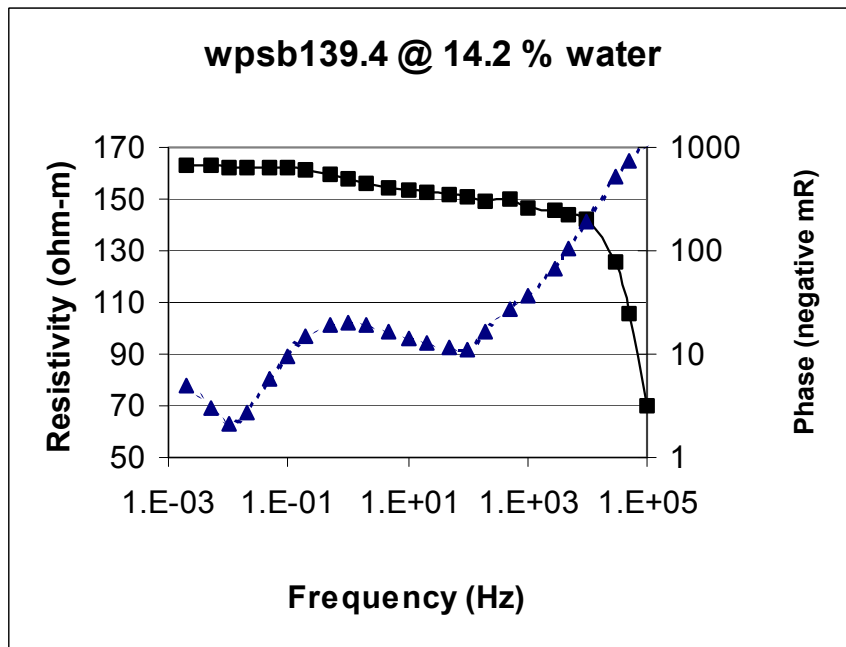Figure 7. Sample wpsb059.3 at 10 water weight percent, repeat.
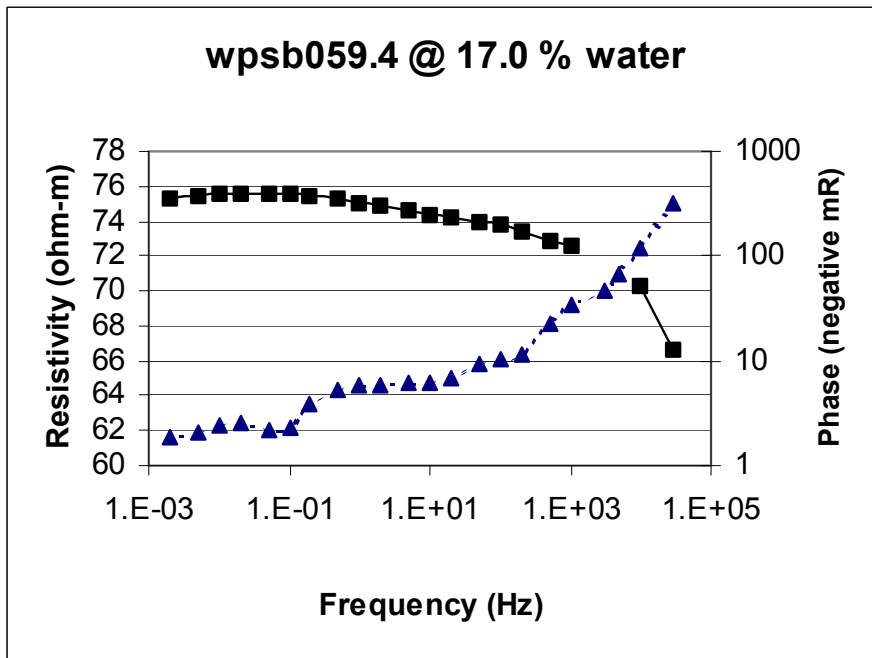
Figure 8. Sample wpsb059.4 saturated at 17.0 water weight percent.

Figure 9a. Sample wpsb117.1 at 4.8 water weight percent.

Figure 9b. Sample wpsb117.2 at 10 water weight percent.

Figure 10. Sample wpsb117.3 saturated at 13.9 water weight percent.

Figure 11. Sample wpsb139.1 at 5.3 water weight percent.

Figure 12. Sample wpsb139.2 at 10 water weight percent.

Figure 13. Sample wpsb139.3 at 10 water weight percent, repeat.

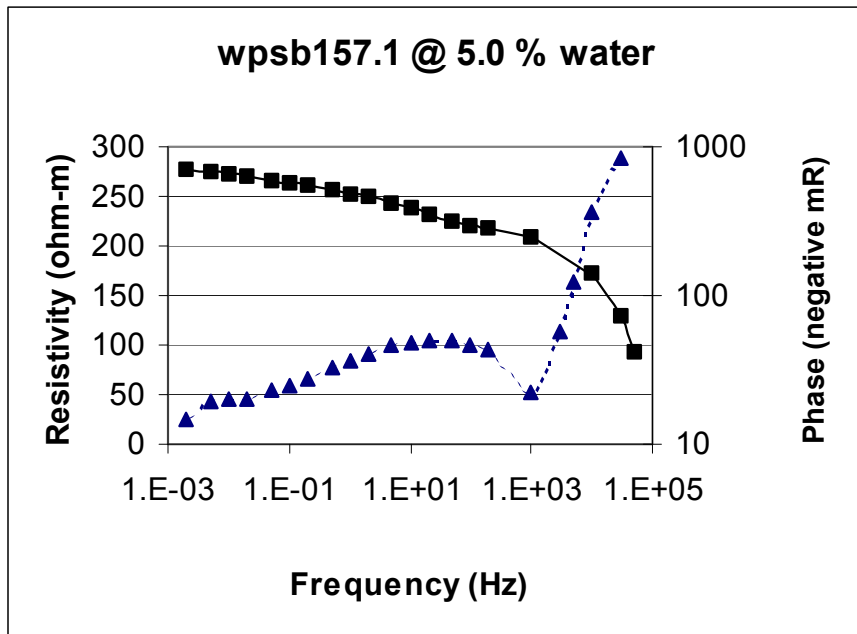Figure 14. Sample wpsb139.4 saturated at 14.2 water weight percent.

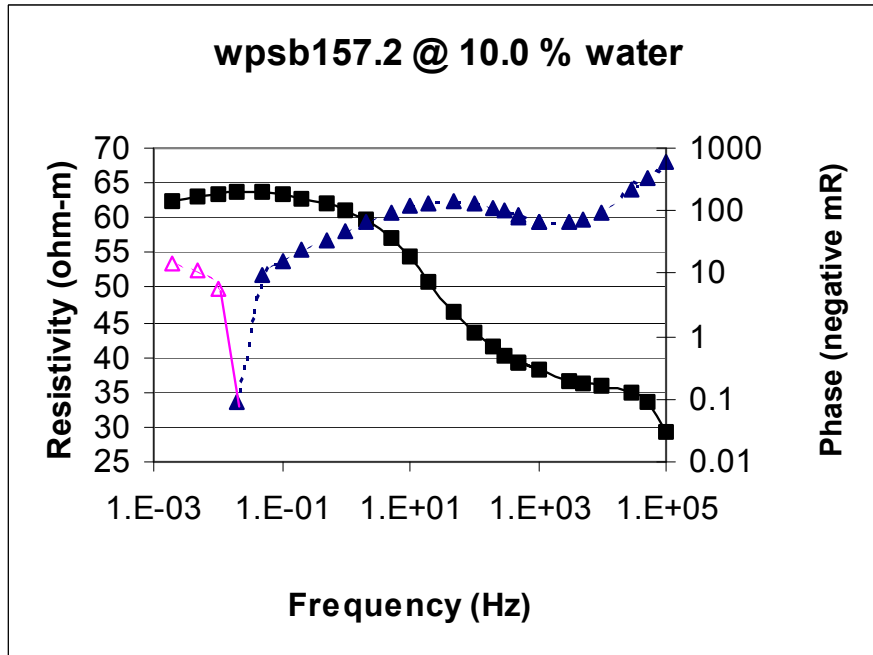Figure 15. Sample wpsb157.1 at 5.0 water weight percent.

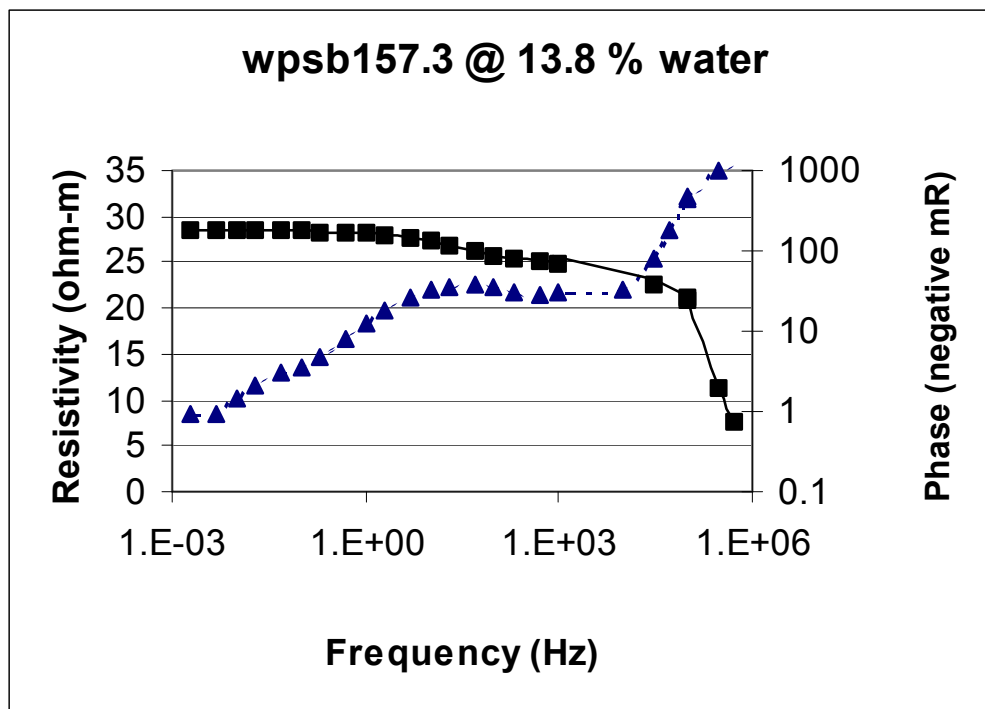Figure 16. Sample wpsb157.2 at 10 water weight percent.

Figure 17. Sample wpsb157.3 saturated at 13.8 water weight percent.