Determination of Moisture Content of Deicing Salt at Point of Delivery

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16. Abstract

The object of this research was to find or develop a simple, affordable method or device for quickly measuring the moisture content of road salt in the field. The test should be able to be carried out in about fifteen minutes and be capable of measuring moisture contents up to approximately 5% to an accuracy equal to +/- 0.5%. Of the several methods for moisture measurement, the infrared, nuclear and capacitive methods were eliminated because of high cost, measurement errors or ineffectiveness. The primary focus was electrical conductivity and microwave methods. The electrical conductivity/ resistivity properties of moist deicing salt was measured for six salt sources including both solar and mined salt. The electrical properties of a given salt is shown to vary directly with moisture content but is also effected by temperature, packing density and to a lesser degree by particle size. Yet, accounting for these variables, it is possible to predict water content using conductivity to +/- 0.4%. For all of the salts combined, the accuracy reduces to +/- 0.8%. Microwave drying is the most accurate method for measurement of salt moisture content. The reduction of data requires a bit of practice. The equipment is inexpensive but requires a power supply.

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SI* (MODERN METRIC) CONVERSION FACTORS

APPROXIMATE CONVERSIONS TO SI UNITS

LENGTH

Symbol	When You Know	Multiply By	To Find	Symbol
in	inches	25.4	millimetres	mm
ft	feet	0.305	metres	m
yd	yards	0.914	metres	m
mi	miles	1.61	kilometres	km

AREA

Symbol	When You Know	Multiply By	To Find	Symbol
in^2	square inches	645.2	millimetres squared	mm^2
ft^2	square feet	0.093	metres squared	m^2
yd^2	square yards	0.836	metres squared	m^2
ac	acres	0.405	hectares	ha
mi^2	square miles	2.59	kilometres squared	km^2

VOLUME

Symbol	When You Know	Multiply By	To Find	Symbol
fl oz	fluid ounces	29.57	mililitres	mL
gal	gallons	3.785	Litres	L
ft^3	cubic feet	0.028	metres cubed	m^3
yd^3	cubic yards	0.765	metres cubed	m^3

NOTE: Volumes greater than 1000 L shall be shown in m^3

MASS

Symbol	When You Know	Multiply By	To Find	Symbol
oz	ounces	28.35	grams	g
lb	pounds	0.454	kilograms	kg
T	short tons (2000 lb)	0.907	megagrams	Mg

TEMPERATURE (exact)

Symbol	When You Know	Multiply By	To Find	Symbol
^{0}F	Fahrenheit temperature	5(F-32)/9	Celcius temperature	^{0}C

APPROXIMATE CONVERSIONS TO SI UNITS

LENGTH

Symbol	When You Know	Multiply By	To Find	Symbol
mm	millimetres	0.039	inches	in
m	metres	3.28	feet	ft
m	metres	1.09	yards	yd
km	kilometres	0.621	miles	mi

AREA

Symbol	When You Know	Multiply By	To Find	Symbol
mm^2	millimetres squared	0.0016	square inches	in^2
m^2	metres squared	10.764	square feet	ft^2
ha	hectares	2.47	acres	ac
km^2	kilometres squared	0.386	square miles	mi^2

VOLUME

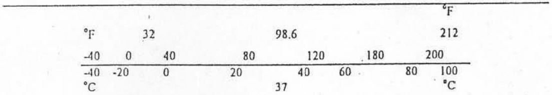
Symbol	When You Know	Multiply By	To Find	Symbol
mL	millilitres	0.034	fluid ounces	fl oz
L	litres	0.264	gallons	gal
m^3	metres cubed	35.315	cubic feet	ft^3
m^3	metres cubed	1.308	cubic yards	yd^3

MASS

Symbol	When You Know	Multiply By	To Find	Symbol
g	grams	0.035	ounces	oz
kg	kilograms	2.205	pounds	lb
Mg	megagrams	1.102	short tons (2000 lb)	T

TEMPERATURE (exact)

Symbol	When You Know	Multiply By	To Find	Symbol
$^{\circ}$C	Celcius temperature	1.8C+32	Fahrenheit temperature	$^{\circ}$F



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* SI is the symbol for the International System of Measurement

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1. Introduction

The Departments of Transportation of the New England States procure large quantities of deicing salt each year to maintain highways free of ice and snow during the winter months. Moisture content of salt in its bulk form can vary depending on conditions. The DOTs wish to pay only for salt. To insure that one pays only for salt, the moisture content of the delivered material must be determined and the weight of the delivered product adjusted for excess moisture. The gravimetric method is used most often, in which a sample of salt is weighed, dried in a conventional oven overnight and weighed again to determine the amount of moisture. A faster method is sought that maintains sufficient accuracy. However, the gravimetric method remains the standard to which other methods must be compared.

An ideal test would be capable of measuring moisture contents in the range of 2 to 7% of dry weight to an accuracy of +/- 0.5%, and require not more than about fifteen minutes to complete. The device for making these measurements should be relatively cheap so that one could be available at all points of delivery.

The research began with the request for a copy of the specifications from the Departments or Agencies of Transportation in New England and New York. Simultaneously a search on the internet began to find which instruments and techniques would provide the means to achieve the goals of this project. Meters that measure moisture in wood were mentioned in the request for proposal as a method that might work. These meters are based on the principle that the moisture content of wood affects the conductivity, and that these two properties are uniquely related. The search, however, was not limited to this type meter alone. In fact the search investigated meters that measure moisture in paper, soil, coffee beans, etc. Morton Salt Company was also contacted about methods for measuring moisture in salt. A typical letter used in this search and sent by email is shown in Appendix B

2. State Salt Specifications

The State salt specifications follow AASHTO M143-86 with some minor modifications by each state. As an example, the Connecticut salt specification is presented in Appendix A. This specification presents particle size distribution, chemical purity, etc. The States have followed the specification for particle size for mined rock salt with a few exceptions. Vermont and New Hampshire limit the amount passing the No. 30 sieve to10%. The other States allow 15% passing the No. 30 sieve.

One item not covered in the AASHTO Spec. is moisture content at delivery. As a result each State has developed specifications about moisture that fit their needs. Review of the specifications shows:

1. Vermont rejects all rock salt shipments with moisture content over 1-1/2%.
2. Massachusetts retains the option of rejecting a shipment with 2.0% moisture content or keeping it and reducing the payment by 10%.

3. Connecticut accepts up to 2.0% moisture content, but reduces the payment by the amount of moisture over 2.0%.
4. New York State defines moisture content on the basis of total weight of salt, and reduces payment for moisture contents over 2.0%
5. Maine basically has a progressive penalty formula for accepting rock salt shipments over 1.0% moisture and rejects all shipments over 2.5% moisture, except in emergencies, and with an additional penalty of weight reduction.
6. New Hampshire rejects all rock salt shipments above 3.0% moisture and has a weight penalty for shipments between 1.0% and 3.0%.

3. Moisture Measurement Methods: A Review

The conventional analytical and gravimetric methods require substantial time to complete. In the infrared method the material's particle size, particle shape, particle surface characteristics and color can cause moisture measurement errors. The microwave methods often require a large space and the portable devices tend to be expensive. This investigation concentrated on the most common methods for measuring moisture contents: gravimetric, conductivity, capacitance, microwaves, nuclear, and chemical.

The gravimetric is perhaps the widest used. Moisture evaporates faster from a solid at temperatures above ambient. To insure that all of the moisture is driven off the solid a temperature slightly greater than the boiling point of water is used whenever this temperature does not cause a change in the solid substrate. To determine the water content of a substance such as road salt, one must weigh the wet material, put it into an oven and dry it until a constant weight is reached. This normally required several weights to insure that all of the water has been driven off. The temperature of the oven must of course be high enough to drive off all the water. A temperature of about 105^oC is usually high enough to accomplish this for salt and soil without adversely affecting the basic material. A faster alternative is to heat the wet salt sample in an oven using microwaves and dry it to a constant weight. This could be done in the desired time limit of 15 minutes.

The meters that measure the moisture content of wood use the conductivity of wet wood to determine its moisture content. Normally these devices use a pair of probes to penetrate the wood to a depth of interest and use an alternating current to determine the electrical resistance to flow. The device measures the conductivity in the vicinity of the probe tips and there is normally a warning that the readings will be affected if the moisture contains salt. The salt makes the water more conductive. While this is a shortcoming if one wants to measure the moisture of some other substance, it should be an advantage when trying to measure the moisture of salt, since the moisture dissolves salt according to the amount present which in turn will affect the conductivity by making it more conductive. The measurement of the conductivity of salt may be affected by the presence of contaminants that precipitate on the surface of the salt crystals and dissolve first when rewetted. In these cases the conductivity of substances other than the salt is being measured.

Capacitance measures the dielectric constant of the substance between the electrodes. While pure water has a relatively high dielectric constant, the presence of salt makes the mixture too conductive to be handled in this way.

Microwaves can be used in two ways to measure the moisture content of salt. First, they can be used as the heat source to dry the material rapidly in a gravimetric method. Second, they can be used to measure moisture directly by measuring the amount of energy that is being absorbed by the water as the microwaves pass through the material. A portable meter to accomplish the second method costs $8800,which is too expensive for the application envisaged here.

Nuclear gages are used to measure the moisture and density of compacted soil by most transportation departments. Since this device is already part of the equipment that DOTs normally have available it was tried on salt but was not successful.

The chemical method is generally used for determining the moisture content of soils and uses a chemical that reacts with water to generate a pressure. The amount of pressure generated by the reaction is related in the original moisture content of the soil. Morton Co. reported that it uses this device but that they have not found it to be very accurate for salt.

4. Properties of Road Salt

There are two general sources of road salt: mined and solar. Perusal of State specifications for the two sources show some differences but the particle size distributions required by each State are similar and follow several standard specifications. The road salt must be at least 95% pure.

Six samples of road salt were collected throughout this study from different suppliers for laboratory determination of moisture content and correlation to other properties such as electrical resistance/resistivity. Each sample was placed in a five-gallon bucket which was a covered with plastic sheet and stored at room temperature until tested. The samples are summarized in the following Table 4.1.

Table 4.1 Information for Salt Samples used in Test Program.

Sample Number	Date Collected	Supplier/Location	Salt Type
S-1	9/6/02	Morton/Bahamas	Solar
S-2	1/7/03	Morton/Mexico?	Solar
S-3	4/17/03	Morton/Bahamas	Solar
MS-1	6/3/03	Morton/Mexico	Solar
IR-1	6/3/03	International/Chile	Mined
AR-1	6/3/03	American/New York	Mined

There was concern that the physical and chemical properties of salt may vary by source, location or treatment. One such treatment is the addition of yellow prussiate of soda (YPS) for anti-caking, which probably is held at the surface of the particles. Impurities on the surface of the sodium chloride crystal can have a significant effect on the conductivity. Therefore some physical and chemical tests were performed to examine property variations.

4.1 Physical

The primary physical property used in highway specifications is particle size distribution. However, road salt, which is comprised of the mineral halite (NaCl), is a soft material with a Mohs hardness of 2 ½ on a scale of 10. Ten is the hardness of diamond. Thus, these particles are easily fractured during handling and preparation for testing. In addition salt is highly soluble in water and the process of drying tends to re-precipitate salt at the particle contacts (due to capillary forces) which results in a lightly cemented mass of salt. This cemented mass of salt must be disaggregated to determine particle size distribution by sieving.

Figure 4.1 shows gradation curves for one batch of salt that was processed several different ways to examine the variability of electrical properties at a constant moisture content of 3%. The dark lines on this figure show the coarse and fine limits from the DOT salt specifications for reference. A large quantity of this material (about 10 pounds) was oven dried and disaggregated by light hand crushing. A 2000 g sample of material was removed, moistened to 3% water content and stored in a plastic bag prior to testing. The remaining material was sieved to prepare 2000 g each of material at the coarse limit and the fine limit. Each of these samples were also moistened to 3% moisture and placed in a plastic bag. These three materials were divided into 4 sub-samples with each subjected to electrical measurements followed by additional oven drying and sieving. The data in Figure 4.1 show significant particle size variations under controlled conditions. Samples A-D, Fine E-H and Coarse I-L should each be represented by a single gradation curve under ideal conditions with the latter two sets of data coinciding with the coarse and fine limit curves. However, there is significant variation in Samples A-D and Fine E-H, which have smaller particles that appear to be affected by material wetting, drying and processing operations. The Coarse I-L samples have little variation and are affected less than the finer samples. The quantity of fines will have a significant influence on the packing density and the specific surface, which are in turn affected by the moisture film on the salt.

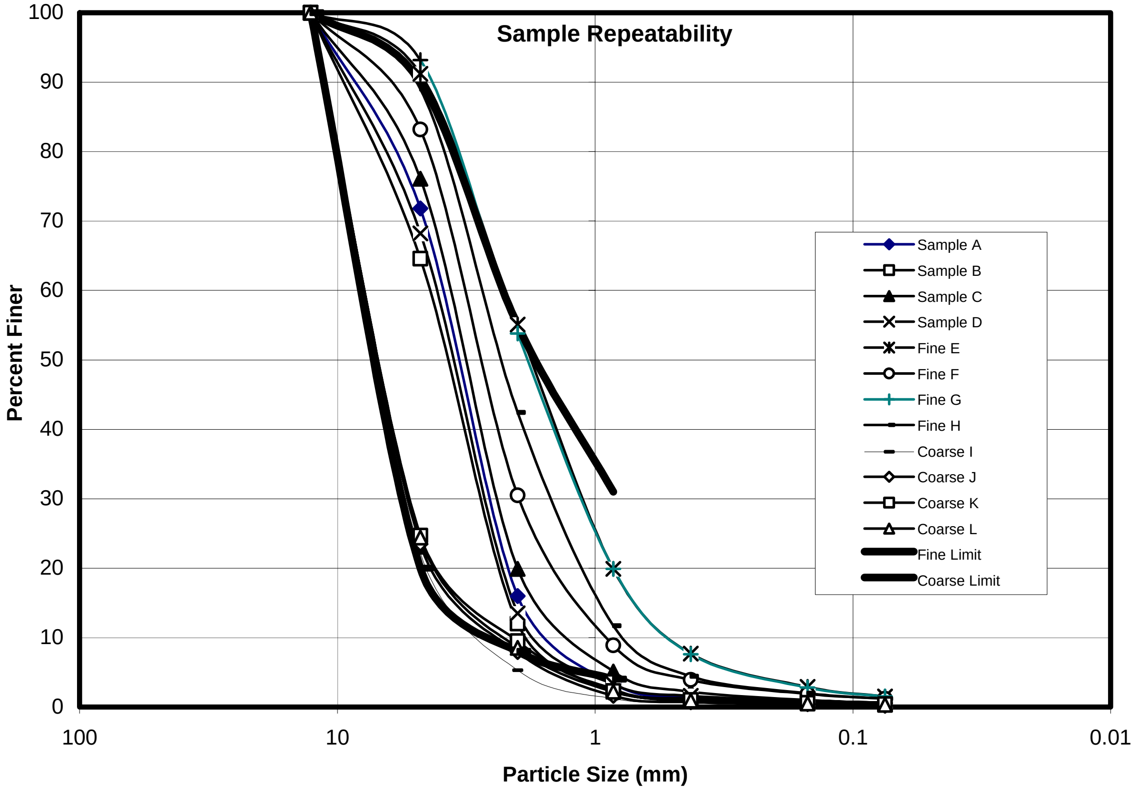

Figure 4.1 Gradation Curves for Sample S-2 in Repeatability Measurements

About 2000 g each of American Rock Salt, AR-1, International Chile, IR-1, and Morton Solar Salt, MS-1, was used to determine the gradation analysis for comparison with DOT specifications. Figure 4.2 shows the gradation curves of these salts along with the DOT gradation limits. It is seen that the Morton and American Rock salts fall within the coarse and fine limits set by DOT's, but International Chile Salt obtained from the source was found to contain more fine particles that the specifications allow.

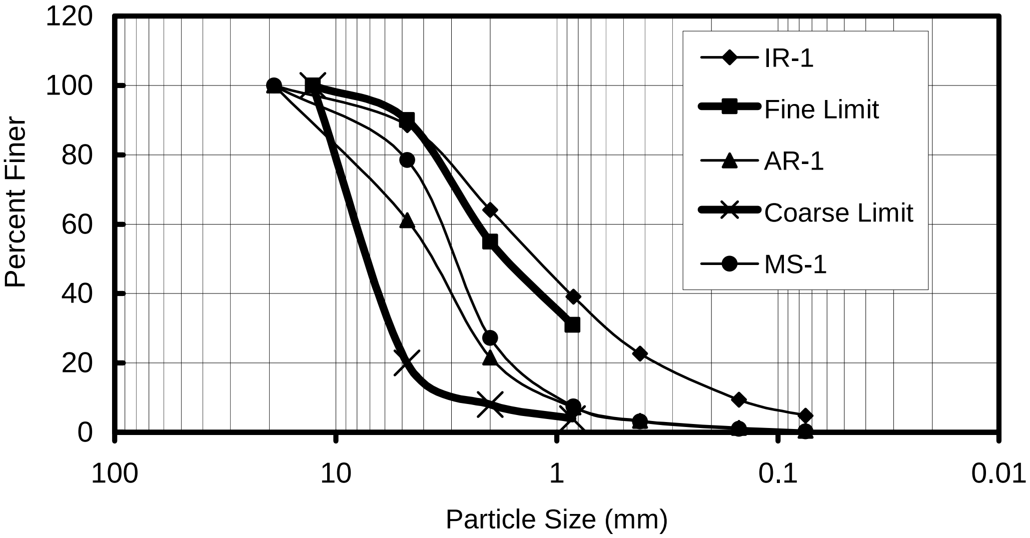

Figure 4.2 Gradation Analysis of Morton Solar, International Chile and American Rock Salts with Coarse and Fine Limits

4.2 Chemical

The DOT salt specifications refer to the purity of salt but there are no requirements for the chemical (cation) content, which may vary by source. The mineral halite is one of the commonest water-soluble minerals on earth and is formed by evaporation of enclosed bodies of seawater or inland lakes such as the Great Salt Lake. These deposits vary in age from the present to the Paleozoic (about 40 mybp). The natural salt layers may be mixed with other non-salt minerals such as clay, limestone and gypsum and the cations that combine with chloride, while predominantly sodium, may include calcium Ca, potassium K and magnesium Mg. Test results for two sub-samples of the same salt source (S-2 A and B) are summarized in Table 4.2 which shows that sodium is the dominant cation with trace quantities of calcium, magnesium, potassium and copper present. Since the two samples are from the same source, the results agree favorably with little variation.

Two samples of solar salt from different locations (Mexico S-2and Bahamas S-3) were also tested for cation content. Each sample was placed in a sterilized bottle and dissolved with de-ionized water. The saltwater analyses are presented in Table 4.3. While the results are not directly comparable to the results in Table 4.2, they again show the abundance of sodium compared to other metals but there is significantly more variation in the concentrations of the other soluble components. For example, magnesium has a four-fold difference between samples with smaller differences for the other metals. These soluble components create the conductive water layer that surrounds salt particles. These data suggest that salt chemistry and impurity variations will vary by source and processing to create unique material.

Table 4.2 Summary of Metals Variability for Road Salt

Environmental Research Institute
Metals Analyses
Date Rec'd: 4/29/03 Matrix: Solid
Date Prepped: 4/30/03 Prep. Method: 3050B
Date Analyzed: 5/1/03 Analysis Method: 6010B

Sample	Lab ID#	Metal Concentration*: ug/g (or ppm) by Dry Weight				
		Cu	K	Ca	Mg	Na
S- 2A	03167-01	1.41	365	426	171	233000
S- 2B	03167-02	ND	320	410	134	239000
Average Detect. Limit		1.01	20	20	20	101

ND= Not Detected
*ICP-OES= Inductively Coupled Plasma- Optical Emission Spectroscopy

Table 4.3 Metals Analyses for Two Different Salts S-2 and S-3

Environmental Research Institute
Metals Analyses
Date Rec'd: 5/6/03 Matrix: Water
Date Prepped: 5/13/03 Prep. Method: 200.7
Date Analyzed: 5/14/03 Analysis Method: 200.7

Sample	Lab ID#	Metal Concentration*: ug/l (ppb)				
		Cu	K	Ca	Mg	Na
S- 2	03178-01	287	34200	95500	18900	31300000
S- 3	03178-02	53	79300	119000	80100	28300000
Average Detect. Limit		5	100	100	100	10000

*ICP-OES= Inductively Coupled Plasma- Optical Emission Spectroscopy

5. Experimental Measurements

5.1 General

After reviewing the various methods of measuring the moisture content of salt, it was decided to investigate the conductance method known also as the resistivity /conductivity method. The property measured in this technique is the electrical resistance of the materials, usually with low frequency electricity. From the resistance and the geometry of the sample the resistivity can be computed. The conductivity is the reciprocal of the resistivity. The terms and relations used in this report are as follows:

$$R= (V/I)= \rho(L/A) \tag{1}$$

Where: R= measured resistance in ohms
V= measured electrical potential drop in volts
I = measured current in amps
ρ= specific resistivity in ohm-cm
L= length of path over which measurement was made in cm.
A= cross-sectional area of path in sq. cm

$$\lambda= (1/\rho) \tag{2}$$

Where: λ= conductivity in mhos/cm, and
mho = (1/ohm)

These two equations show that there is a simple relation among resistance, resistivity and conductivity. All results will be shown in one of these variables. In some of the preliminary tests, we worked with the relation V/I which equals resistance.

This method appeared promising, since the effect of the water on the salt is to produce a solution of ions that can conduct electricity, and the more water present the more ions in solution, the lower the resistance and the greater the conductivity. There are some physical properties of the sample that affect the measurement, even if the overall dimensions of the sample are held constant. The two most important properties are temperature and density. The conductivity increases with increasing temperatures and with increasing density.

Road Salt is a granular material and like all granular materials can exist at various densities without breaking the particles. The moisture contents of interest in this research are at such a low level that most of the water will be held in layers around the particles. As a result, as the sample is made denser, more solution will be present between the electrodes to decrease the resistance and increase the conductivity. Samples can be compacted to about the same density by tamping the particles lightly into the sample cell.

The second problem is temperature. The resistance of a solution decreases as the temperature increases, because the energy of the ions increases with increasing temperature. The lowering of the temperature makes the solution more resistive. Charts can be worked out to correct for the effects of temperature at the time of measurement and reducing the resistivity and therefore the moisture content to some reference temperature.

The third item that may affect the electrical resistivity is the chemical composition of the salt. The present specifications allow for up to 5% impurities in road salt. The resistivity of the salt may be affected by these impurities by an amount to be determined. It is anticipated that salt from various sources may have differing impurities in both amount and type. The quantitative effects of these impurities on the measured resistivity must be determined.

5.2 Handling of the Salt Samples

Salt samples to date have been obtained at various times from the maintenance garage near the intersection of Routes 32 and 74 in Willington, Connecticut. The samples were obtained at random from the storage pile. Laboratory handling of the samples was investigated using several methods to vary the moisture content. The first sample obtained was oven dried at $105^{o}C$, before sieving to determine particle size and adding water to vary moisture content. Subsequent samples were allowed to dry at room temperature before sieving or adding water.

Some samples were tested with the particle sizes as received. For other tests the various sized particles of salt were separated by sieving and recombined to produce samples that complied with the limits of the salt specifications. All sieved samples appeared to have been reduced to their individual particle sizes, i.e. the small particles had not adhered to the large particles.

The resistance of the road salt was measured in plexi-glass boxes of two sizes. In the experiments using the conductivity bridge, only end electrodes were used, but polarization of the electrodes made reproducibility of the results difficult. The measurements were then made with a four-electrode system, which produced the appropriate reproducibility. In experiments using four electrodes, current was applied through end electrodes and two other electrodes measured potential drop along the specimen of salt in the cell.

5.3 Electrical Resistivity/ Conductivity Method

Testing was done with the Tramex wood moisture meter device, a Barnstead Conductivity Bridge and two laboratory arrangements of the four-pole system. Photos of these devices and test set-ups are shown in Appendix D (Figures D-1 to D-5).

5.3.1 The Tramex Test Results. The first conductivity tests on moist salt were done with a Tramex Professional Moisture Meter for wood. At this stage of the testing not all of the parameters of the tests were understood. An understanding of the behavior of the salt in conductivity measurements developed as testing proceeded. Measurements with the Tramex device were also done in connection with other methods to compare this device with the other methods.

The results of the first test series with the Tramex device are shown in Figure 5.1. As can be seen from Figure 5.1 the device may be sufficiently sensitive to accurately measure moisture content up to about 1-1/2 to 2%. Above a moisture content of 2% the curve is too flat to distinguish the various water contents. These measurements were made with the S-1 sample.

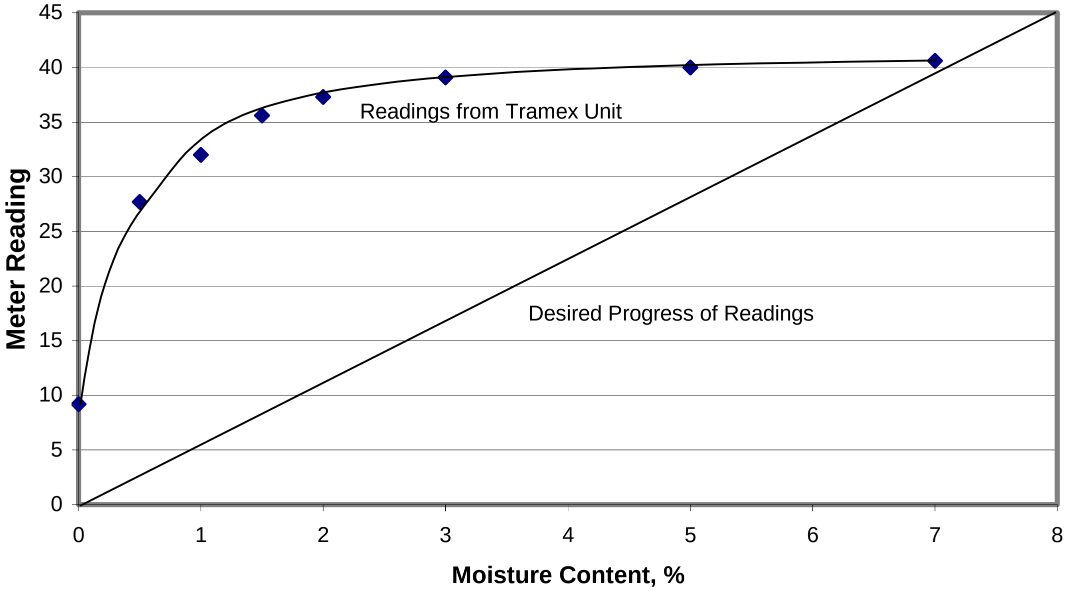

Figure 5.1 Plot of Moisture Content of Salt vs Meter Readings using Tramex

The Tramex device would work fine for the purposes of determining the moisture content of road salt if the meter readings followed a straight line such as the one shown in the middle of the graph. The company was contacted and queried about the possibility of modifying the measuring circuitry to make the reading follow a more linear plot. The company after some consideration told us it could not be done.

We then turned to investigate making the conductivity measurements more sensitive using methods and techniques found useful in other applications.

5.3.2 Tests Using the Conductivity Bridge. The Conductivity Bridge is a standard laboratory device normally used to measure the resistance between two electrodes in a conductivity cell. For solutions normally found in a laboratory, cells with platinum-platinum black electrodes are used. The platinum black coatings reduce the polarization at the electrodes occurring during the measuring process.

The standard cells are too small and the electrodes are too close together to measure the resistance of a sample of road salt. In addition platinum black electrodes are not appropriate for a field device, since they must be renewed regularly. An appropriate cell was constructed from acrylic plastic that was 50mm wide, 50mm high and 100mm long. This cell has stainless steel electrodes at both ends of the 100mm length. The 50mm dimensions in the other directions insure that it can easily accommodate the sizes in the road salt.

These tests showed that the resistivity of the salt changed significantly with packing-density of the salt. As the density of the packing increased, so did the conductivity, i.e. the resistance decreased. They also showed that the moisture of the salt migrates with movement, time and vibration. This effect is most noticeable for moisture contents above 3%, but it occurs at all of the moisture contents.

It became apparent during the tests to check on the reproducibility of the measurements that there was a problem with polarization of the electrodes. As a result we switched to the four-pin method of measuring the resistivity/conductivity.

5.3.3 The Four-Pin Method of Analysis. This is the method to measure the resistivity /conductivity of soil and other granular material. The polarization effect on the measurement was eliminated by applying and measuring current across the end electrodes and measuring the potential drop across two electrodes separated by a known distance along the center of the sample. Some tests were affected by a migration of moisture to the bottom half of the cell. A new cell was constructed with a removable base so that the excess moisture that migrated toward the bottom of the sample could be removed before the measurement was taken, yielding a more accurate resistance for the measured moisture content.

The tests were conducted in the following manner:

1. The cell and electrodes were washed and rinsed with de-mineralized water then dried.
2. The end electrodes were placed in the cell.
3. The salt was placed in three layers and tamped lightly with a pestle 30 times on each layer. The electrodes that measured the potential were placed with the second layer.
4. When the cell was filled the top was fitted in place and the cell turned over, the bottom removed and dried and replaced.
5. The filled cell was then weighed.
6. Alternating current was applied through the end electrodes
7. Potential Gradient was measured midway along the cell

Current was originally applied to each sample at five levels. An example of the results is shown in Figure 5.2. As can be seen from Figure 5.2 the applied current versus the measured potential gradient form a straight line, the slope of which represents the resistance of the salt between the measuring electrodes. This figure shows a typical pattern for the measurements. The measurements shown were all taken at 2% moisture.

After many measurements using 5 different levels of current always resulting in a straight-line plot, the number of current levels was reduced to three.

A series of resistivity measurements were made at various water contents, which showed an interesting trend. This trend is shown in Figure 5.3. The trend shown in Figure 5.3 is slightly curved. Over a shorter span of moisture contents the curve can be approximated with a straight line.

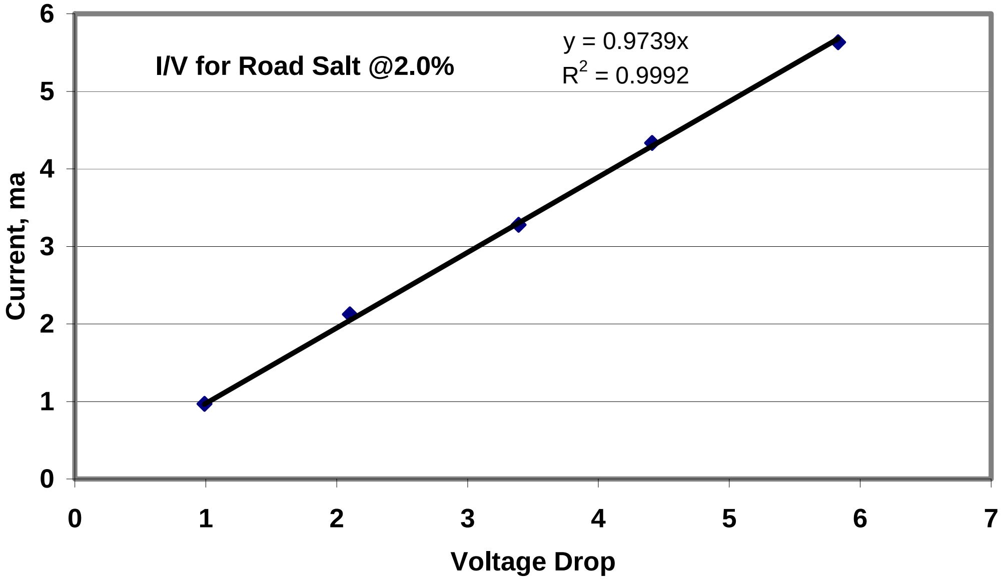

Figure 5.2 Plot of current vs voltage drop @ 2.0% moisture

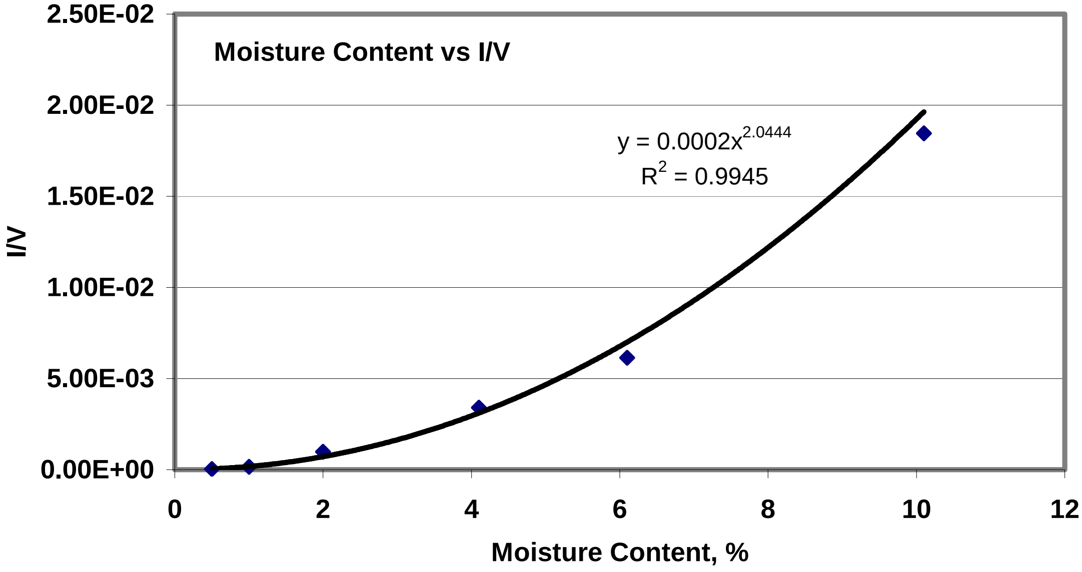

Figure 5.3 Plot of V/I vs Moisture Content

The first part of the investigation examined the effect of various factors. A series of tests were run at 17-18°C. It was hoped that one curve of resistivity vs moisture content could be developed for all salt samples. It was noted that a second sample of salt (S-2) obtained from the same maintenance garage yielded greater values of conductivity than the first. The comparisons of the conductivities of samples S-1 and S-2 are shown in Figure 5.4. The upper line in Figure 5.4 is S-2 and the lower line is S-1. Both samples were measured at 17°C. The difference was thought due to the manner in which it was dried. The first sample had been dried in the oven at 105°C.

A part of sample S-2 was dried at room temperature and another portion was dried in the oven at only 30°C. Testing showed that these two specimens of salt have the same conductivity at each of the tested water contents. The results of the conductivity tests on these samples are shown in Figure 5.5. From this plot it is clear that the conductivities of the two specimens of salt sample S-2 follow the same curve.

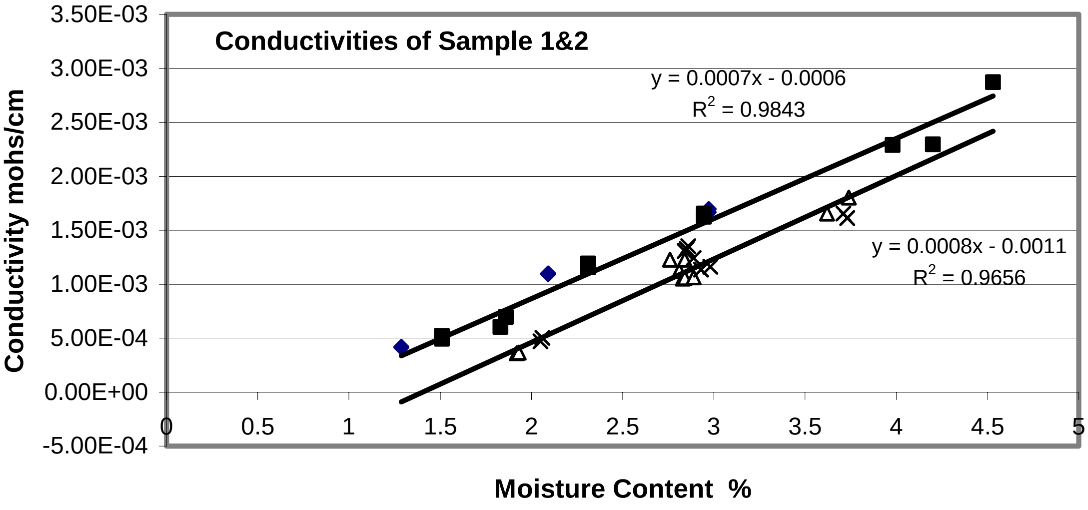

Figure 5.4 Conductivities of S-1 and S-2

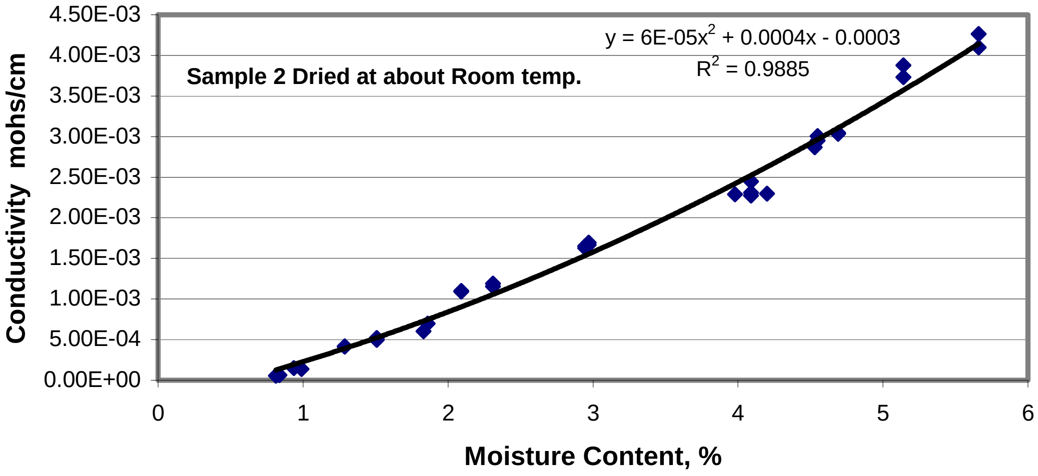

Figure 5.5 Conductivity for Sample S-2 Dried at Room Temperature

S-2 was also tested at three temperatures: 4, 17 and 30°C. The 17°C test results were obtained from tests at room temperature. The 4°C results were obtained from tests conducted in a walk in refrigerator, and the 30°C results were from tests conducted in an oven set at that temperature. The results are shown in Figure 5.6. The upper line was obtained from the results at 30°C and the lower at 4°C. The middle line was at room temp (17°C)

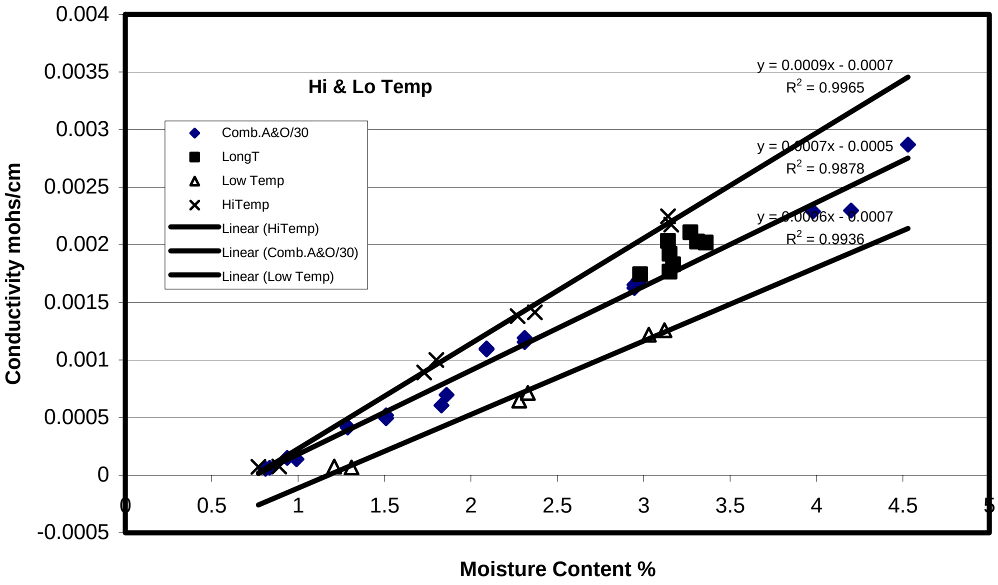

Figure 5.6 Affect of Temperature on Conductivity

As can be seen in Figure 5.5 the conductivities change at each water content with temperature, however, this is a correction that could be accounted for.

A third sample (S-3) was obtained from the Willington site but from a different pile of salt. S-3 showed the same conductivities at room temperature as S-1. Conductivities of the two samples are shown in Figure 5.7. As can be seem from the Figure the conductivities fall along the same line.

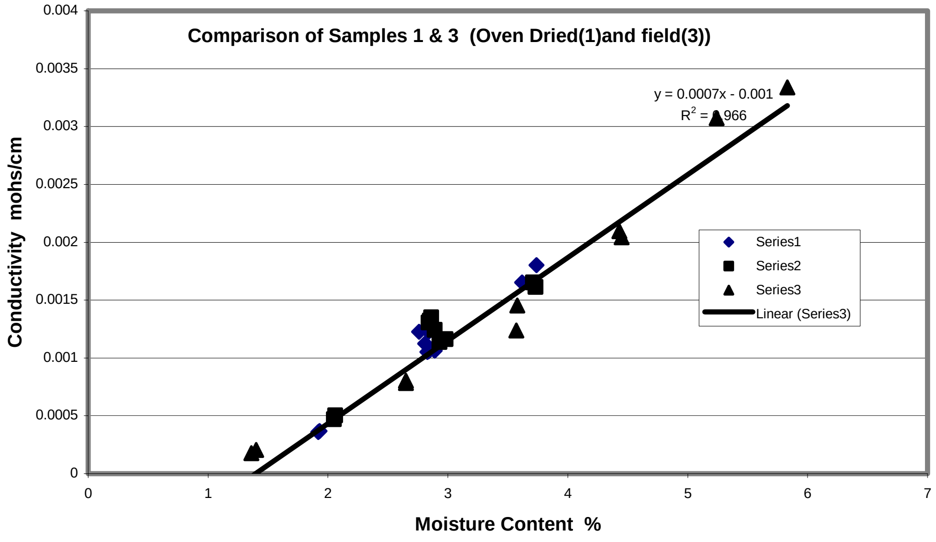

Figure 5.7 Comparison of Oven and Air Dried Samples

A comparison of Sample 2 with Sample 3 is shown in Figure 5.8

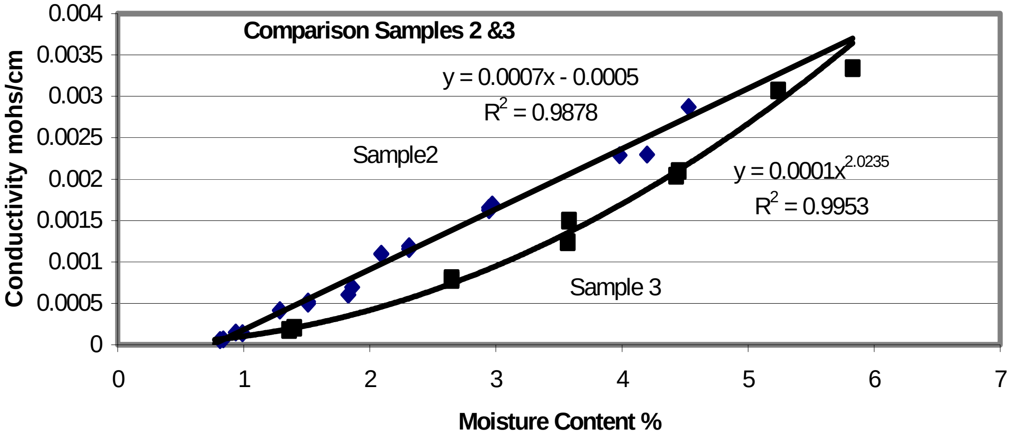

Figure 5.8 Conductivity Curves for Two Different Samples S-2 and S-3

The upper straight line follows the results from Sample 2 and the lower curve follows the data from Sample 3.

The objective of this research is to have a technique for measuring the water content to an accuracy of +/- 0.5%. An examination of the data shows that this limit can be obtained for our samples from the same source. For example the data from samples S-1 and S-3 are shown in Figure 5.9

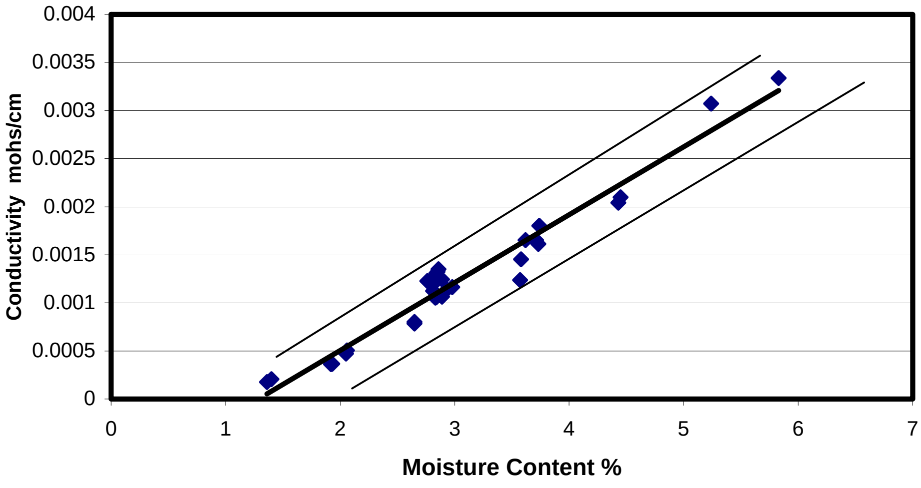

Figure 5.9 Data from Samples S-1 and S-3 Showing Confidence Limits

A statistical analysis of the data shown in Figure 5.9 indicated that at the 95% level of confidence the water content can be measured by conductivity to a limit of +/- 0.4%. This is within the desired range of +/-0.5% sought in this study.

A similar analysis of the data from sample S-2 is shown in Figure 5.10. The data from sample S-2 also yielded a suitable limit to using the conductivity to measure the moisture content. The figure shows the confidence limits to be +/- 0.4% at the 95% level.

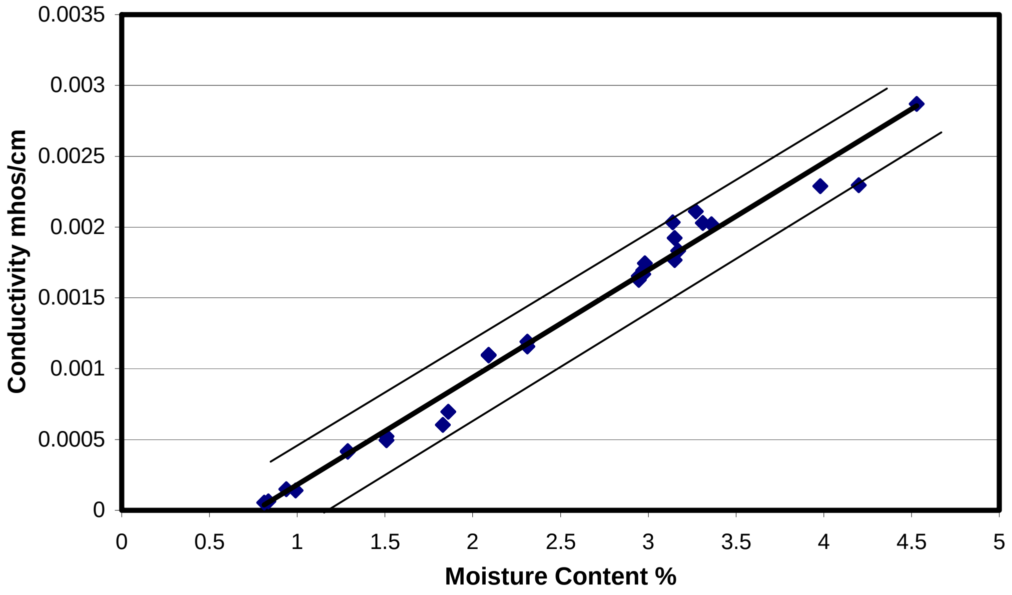

Figure 5.10 Plot of Data for Sample S-2 Showing 95% Confidence Limits

If, however, we combine the data from samples S-1, S-2, and S-3, the conductivity can only measure the water content to a limit of +/- 0.8% at the 95% confidence level. The plot of the combined data is shown in Figure 5.11

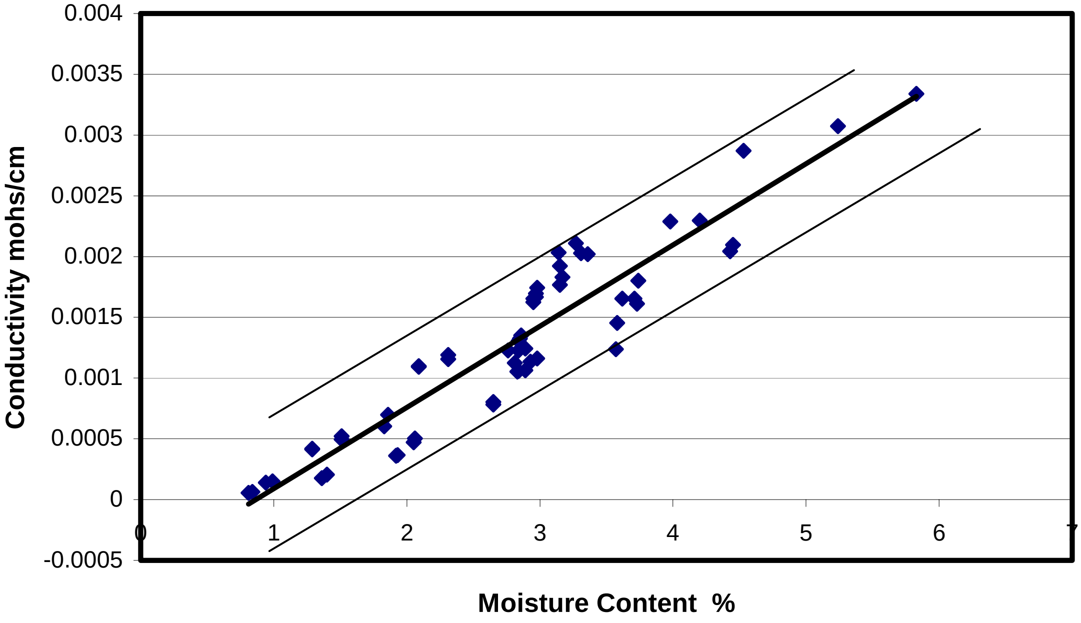

Figure 5.11 Confidence Limits for Combined Data from Samples S-1, S-2 & S-3

The previous data had been obtained with the “short box” fabricated in our shop. In looking for a commercially available meter to do the job we decided to try the Nilsson Meter, which is a device for measuring the conductivity of moist soil. Its configuration is similar to the “short box” and there is only a slight difference in the cell constants (1.0 to 0.95), which does not affect the results when the proper cell constant is used in the calculations.

By the time the Nilsson meter had been procured, the ambient temperature in the laboratory had risen to 26 to 27^{o}C. Also, three additional samples of road salt had been obtained: MS-1, IR-1, and AR-1. The conductivities of these three samples at room temperature are shown in Figure 5.12. As can be seen from this figure the conductivities behave similar to samples S-1, S-2 and S-3.

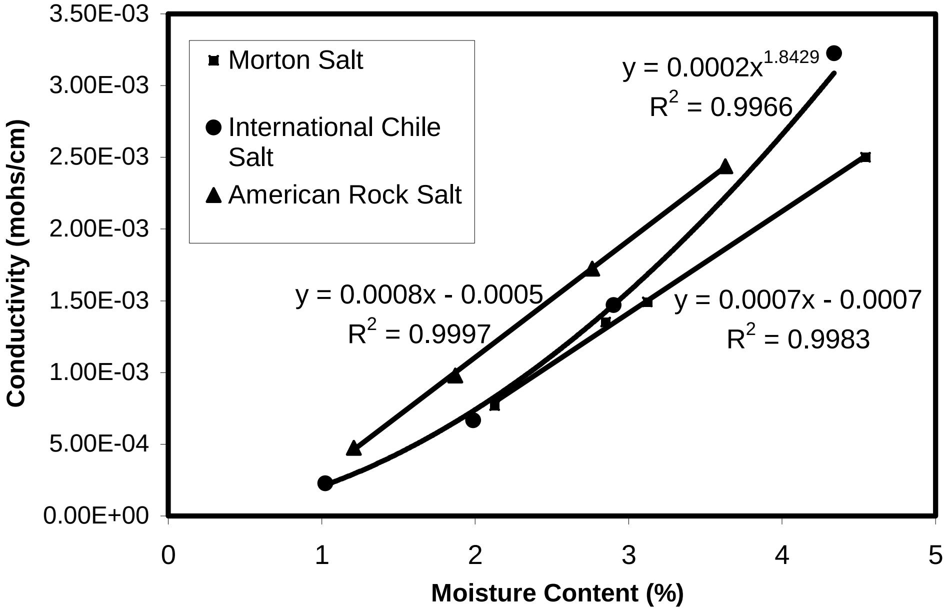

Figure 5.12 Conductivities for Samples MS-1, IR-1 and AR-1

It was decided to compare and analyze all data available at a temperature of 30°C. The conductivities of samples S-3, MS-1, IR-1, and AR-1 are shown in Figure 5.13. The confidence limits on the data at the 95% level are shown in Figure 5.14. At the 95% level the conductivity can measure the water content to +/- 0.7%.

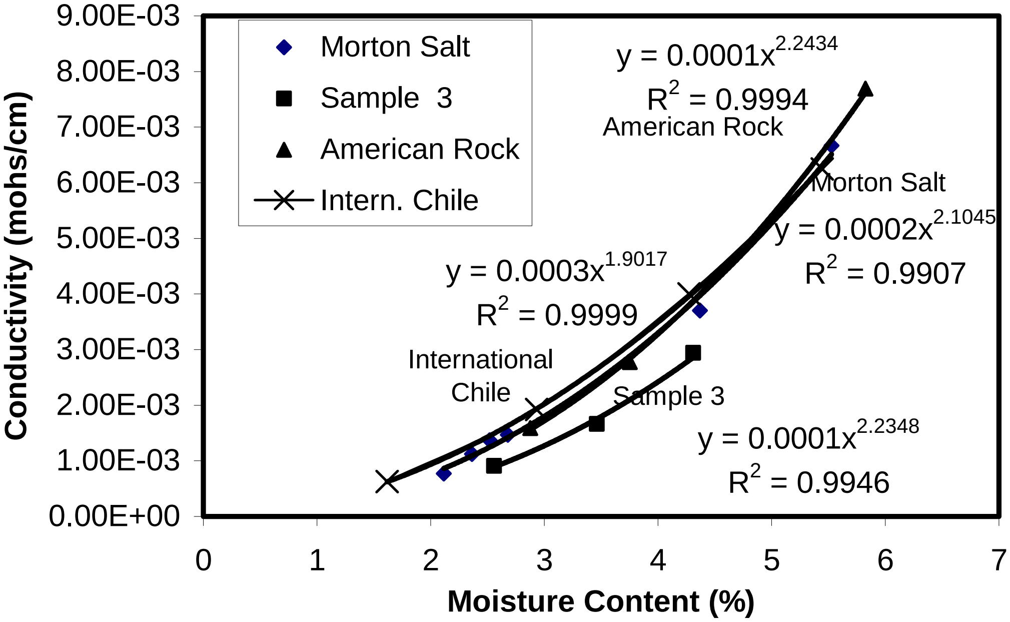

Figure 5.13 Conductivities of Samples MR-1, IR-1, AR-1 and S-3

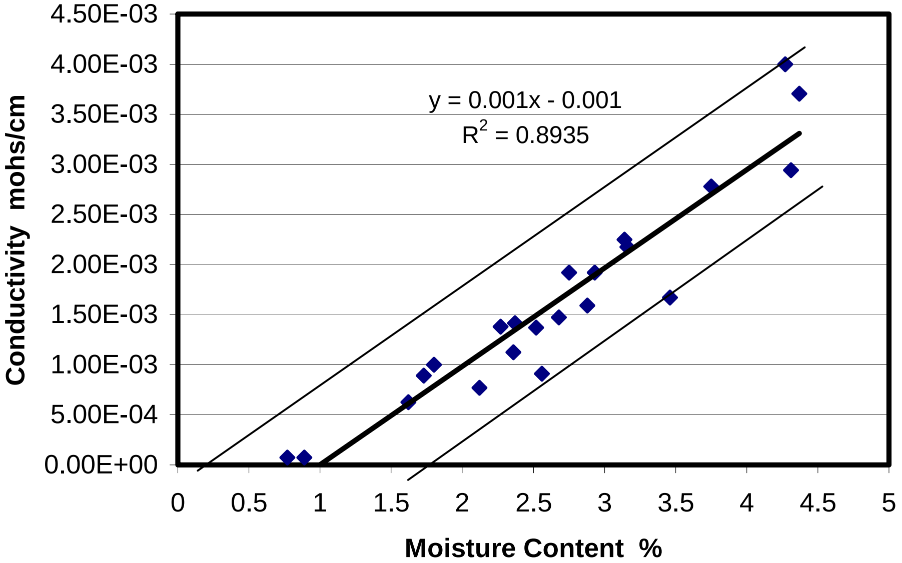

Figure 5.14 Plot Showing Confidence Limits of Road Salts Tested at 30^{o}C

To test the possible source of the data scatter observed in the tests on road salt, it was decided to try testing salt that had been prepared for specific uses. Three samples were obtained: Fisher Reagent NaCl, USP/FCC; Morton table salt; and Diamond Crystal table salt. Each of these has been purified for a specific use, but is NaCl with just a small amount of other materials included. They are purer than road salts from various sources. Morton salt contains a small amount of calcium silicate, and the Diamond Crystal salt contains silicon dioxide and tri-calcium phosphate. No mention of other chemicals appears on the label for the Fisher Reagent NaCl. The particle size distribution of these three salts is approximately the same.

All three samples were tested at about the same density at various water contents. The results are shown in Figure 5.15. As can be seen from this figure the Fisher salt and the Diamond Crystal salt have about the same conductivity over the range of water contents tested, but the Morton salt has a conductivity about three times greater at all water contents. Apparently the conductivity of salt is very sensitive to the presence of small amounts of other chemicals.

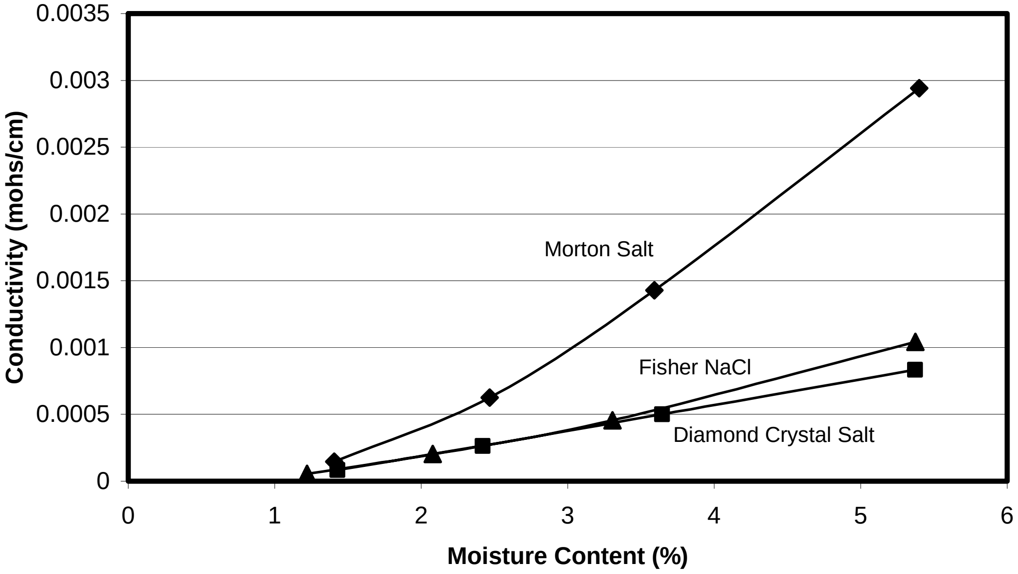

Figure 5.15 Conductivity vs Moisture Content Morton and Diamond Crystal Tables Salts and Fisher Reagent NaCl

5.4 Microwave Gravimetric Method

The uncertainties with the conductivity method caused us to take another look at the gravimetric method, except we decided to use a microwave oven instead of the conventional oven that requires overnight drying. In using a microwave oven certain precautions are required. The microwave oven can be damaged if the energy has nothing to energize. One must always be sure that there is some moisture to be heated in the oven, hence a container of water must always be present, since the salt itself will be moisture free during the last part of the test.

Table 5.1 Moisture Content from Oven at 105°C and Microwave

Moisture from Microwave in %	Moisture from Drying at 105°C in %
2.47	2.34
2.53	2.39
4.74	4.76

As can be seen from Table 5.1 the Microwave results are gives comparable to conventional oven drying, and are within acceptable limits.

6. Test Observations

Although it may not be important at the moisture contents in the range acceptable to the State DOTs, a significant amount of moisture migration was noted in handling samples at moisture contents at 3% and higher. It was observed during the tests at these moisture contents that significant moisture migrated from the salt in the top of the cell into the salt in the bottom of the cell, enough to make a measurable difference between the moisture in the top of the cell and that in the bottom.

These observations must be kept in mind when making measurements in the field. Assuming that the trucks will be weighed at the point of loading, i.e. weigh the truck empty, weigh the truck full, and the difference is the weight of the moist salt., certain difficulties must be addressed if the moisture content is to be measured at the point of delivery. These difficulties will exist whichever method is used to measure moisture content.

As the truck carrying the moist salt moves along the road the vibration of the truck and the time of transit will cause the water to migrate downward in the pile. Therefore how to sample the salt at the point of delivery becomes a problem, if an accurate value of water content for the entire load is obtained.

One approach is to sample entire height of the salt pile with a thin walled tube, then measure the moisture content of the salt at the bottom, middle and top of the tube and average the values. A method such as this is very important for those states that reject a shipment if it exceeds a certain amount of moisture.

7. Summary and Conclusions

Conductivity of six samples of moist road salt was tested at several water contents. Each sample, from different suppliers/sources, has a unique relation of conductivity vs moisture, which yields a relation that can predict the water content from the conductivity to within +/-0.4%. However, when data for all road salts from various sources are used, the water contents cannot be measured by conductivity closer than +/- 0.8%. Therefore:

1. The primary goal of this study to develop an accurate, quick, low cost system for predicting water content of road salt has been unsuccessful. Electrical conductivity was considered the most promising method of satisfying these goals but the results of this study show that it is not suitable as a general method because of salt chemistry and purity effects.

2. Conductivity might be developed as a method for road salt from a specific source so long as calibration checks are run frequently.

3. The most accurate method of measuring the water content of road salt is the gravimetric method, whose testing time can be reduced to 15 minutes with the use of a microwave oven.

4. Care must be taken with extracting a representative sample from a pile of moist road salt that has been subjected to vibration for a period of time. One must expect that the moisture content toward the bottom of the pile is greater that that near the top.

8. References

ASTM (1995), Annual Book of Standards, Volume 03-02 on Corrosion of Metals, “G 57-95a Standard Test Method for Field Measurement of Soil Resistivity Using the Wenner Four Electrode Method”, West Conshohocken, PA

Birch, F., J. F. Schairer and H. C. Spicer (1942) Handbook of Physical Constants, Geological Society of America Special Paper 36, January, 325 p.

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Mason, B. and L. G. Berry (1968) Elements of Mineralogy, W. H. Freemen Co., San Francisco 550 p.

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Salt Institute (2003) Web Site, www.saltinstitute.org/

Appendices

Appendix A. Example of DOT Salt Specifications

Connecticut Department of Transportation
Bureau of Engineering and Highway Operations

Specification for Road Salt – Sodium Chloride

REFERENCE FILE NO. 139R
Issued July 7, 1955
Revised June 1, 1998

Scope: This specification prescribes the composition, storage, inspection, acceptance and delivery of road salt obtained from (natural deposits/artificially produced) which is to be used for snow & ice control on highways and bridges.

Requirements: All road salt shall conform to AASHTO M 143 (ASTM D-632) Type 1, with the exceptions and additions stated herein. When material is not in conformance as stated herein, and the state formally agrees to accept such material, payment reduction shall apply and will be the sum of the individual reductions based on the bid price.

Inspection & Testing: At the vendor's location the stockpile shall be covered as required and the road salt shall be tested by Division of Materials Testing. The Bureau of Finance and Administration shall accept the material prior to any shipment to the State. Salt from different origins (natural deposits/artificially produced) shall be stockpiled separately. If at any time, the purity of road salt is less than 95 percent sodium chloride, the vendor shall maintain this material in a physically separated stockpile. Once the stockpile has been accepted, material shall not be added to the stockpile without prior notification to and additional testing by the State. Failure to properly control these stockpiles may result in revocation of the award.

Material Acceptance:

PURITY: The road salt requirements for material acceptance shall be as stated in AASHTO M-143 (ASTM D-632) Type 1, except sections 9.1.2 and 11.2 will not apply. It is intended that only products meeting the specified sodium chloride content (95.0 percent or greater) will be accepted; however, at the sole discretion of the Department of Transportation, salt having a purity of less than 95.0 percent sodium chloride content may be accepted with an adjustment in payment in accordance with Table A-1.

TABLE A-1: Adjustment in Payment for Purity of Sodium Chloride

Percent of Sodium Chloride	Percent Payment of Unit Bid Price
95.0% to 100%	100
93.0% to 94.9%	95
91.0% to 92.9%	90
90.9% & below	75

Grading: The gradation requirements for material acceptance shall be as stated below. Failure to conform to these requirements may result in rejection of the stockpile. If nonconforming material is accepted, a reduction in payment of 2 percent per screen shall be assessed for deviations in the gradation.

Sieve Size	Percent Passing by Weight
12.5 mm (1/2 in.)	100
9.5 mm (3/8 in.)	95 to 100
4.75 mm (No. 4)	20 to 90
2.36 mm (No. 8)	10 to 60
600 µm (No. 30)	0 to 15

Moisture: Full payment will apply to the road salt when its moisture content does not exceed two (2.0) percent. Road salt with a moisture content greater than (2.0) percent may be accepted at the discretion of the Department, with an adjustment in weight for moisture content over 2.0 percent.

Anticaking Agent: Salt furnished under this contract shall be free flowing and granular. All bulk salt shall be treated with an approved conditioner, such as sodium ferrocyanide, to prevent caking while in storage. This treatment shall be prior to shipping product from the origin (natural deposits/artificially produced). This conditioner shall be visible and introduced uniformly throughout the salt at a maximum rate of 50 parts per million or 0.0050 percent.

Appendix B. Letter of Inquiry Concerning Moisture Meters

Dear Ladies and Gentlemen:

We are looking for a portable meter to measure the moisture of road salt. The instrument we need must be capable of measuring the moisture to +/-0.5 percent between moisture contents of 0 to 10 percent.

Does Rodex make such a meter and what is its cost?

Richard P. Long, Prof. Emeritus
School of Engineering
University of Connecticut
261 Glenbrook Rd
Storrs, CT 06269-2037

TEL: (860) 486-2074
FAX: (860) 486-2298

long@uconnvm.uconn.edu

Appendix C. Nilsson Conductivity Meter and Probe Geometry

The resistivity of salt at various moisture contents were measured using Soil Resistivity Kit developed by Miller Co. It uses a soil box as shown in Figure C-1. Inside of its dimensions are approximately 2.5-inch wide x 9 inches long x 1.5 inch deep (4 cm x 22 cm x 3 cm). The multiplication factor for this box in calculating the resistivity is one.

Figure C-1 Miller Soil Resistivity Box

This box is connected to Nilsson Model 400 (provided along with the kit) as shown in Figure C-2 to measure the resistance between inner electrodes when an alternating current is applied between the end/outer electrodes.

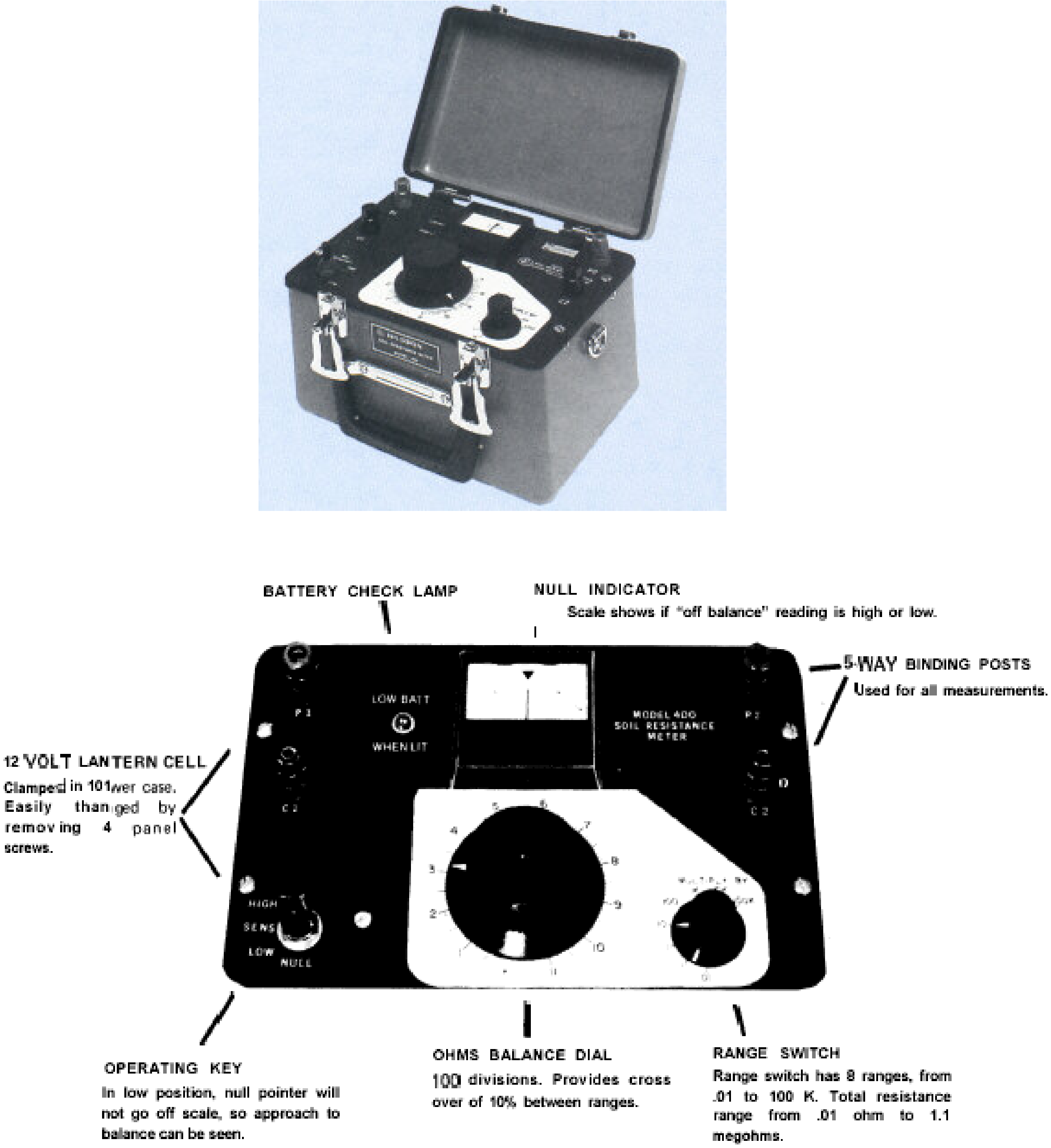

Figure C-2 Nilsson Model 400 4- Pin Soil Resistance Meter

Procedure for conducting the test

1. The Miller Box is rinsed with de-mineralized water and then dried.
2. The salt was placed in three layers and tamped lightly with a pestle 30 times on each layer. After three layers of slight tamping, the salt is placed to fill the remaining portion of the cell and flushed with a spatula.
3. The leads are used to connect the Miller Box to Nilsson Model as shown in Fig. C-3.
4. The unit generates a low voltage 97 Hz square wave current between C1 and C2 binding posts.
5. The detector senses the voltage drop between the P1 and P2 binding posts and indicates a difference on the null detector.
6. When the null detector is balanced using the range switch and the dial, the resistance in ohms between P1 and P2 was obtained by multiplying the dial reading by the range switch position.
7. The sample for moisture content determination is taken from the center of Miller box.

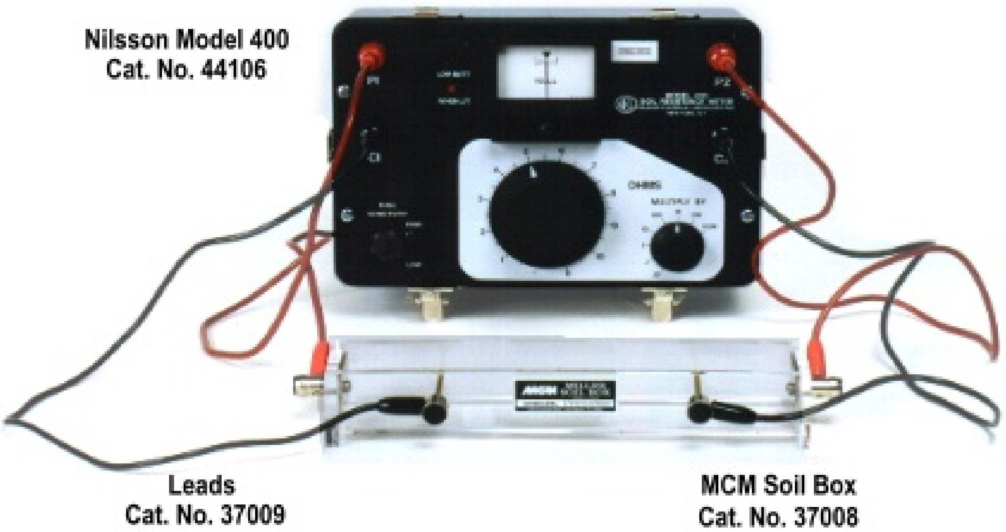

Figure C-3 Soil Box and Nilsson Meter Configuration

Appendix D. Photos of Test Apparatus

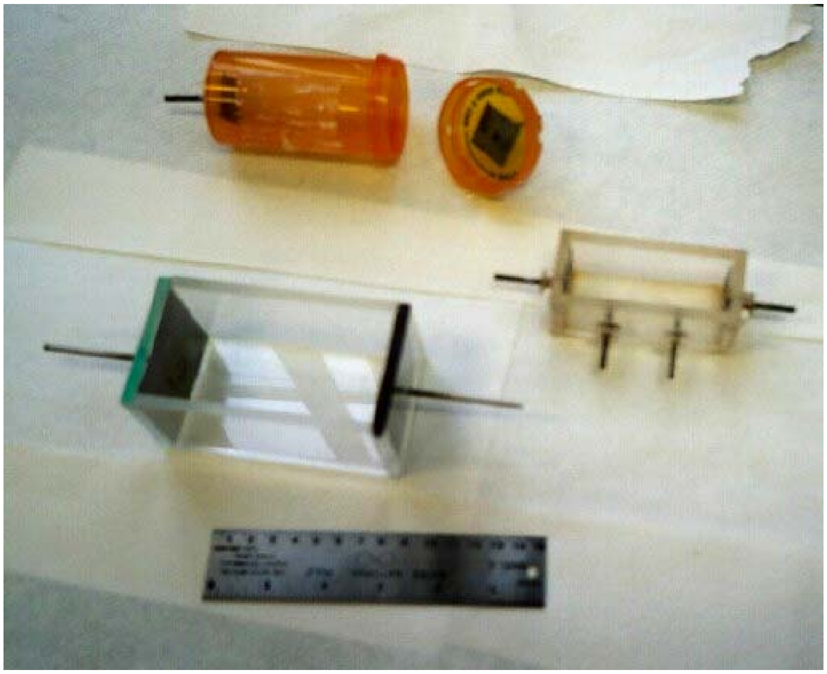

Figure D-1 Three Conductivity Cells Used in Laboratory Tests

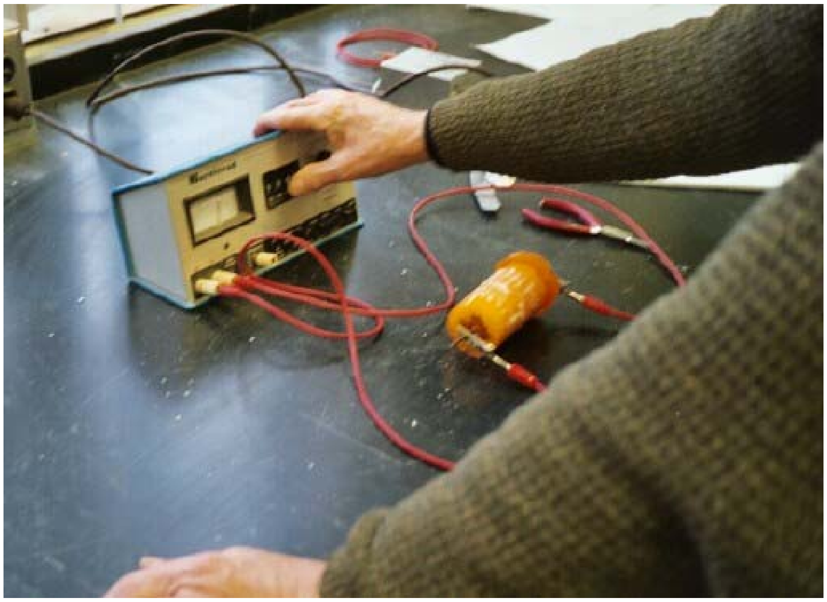

Figure D-2 Conductivity Bridge used to Determine Moisture in Salt

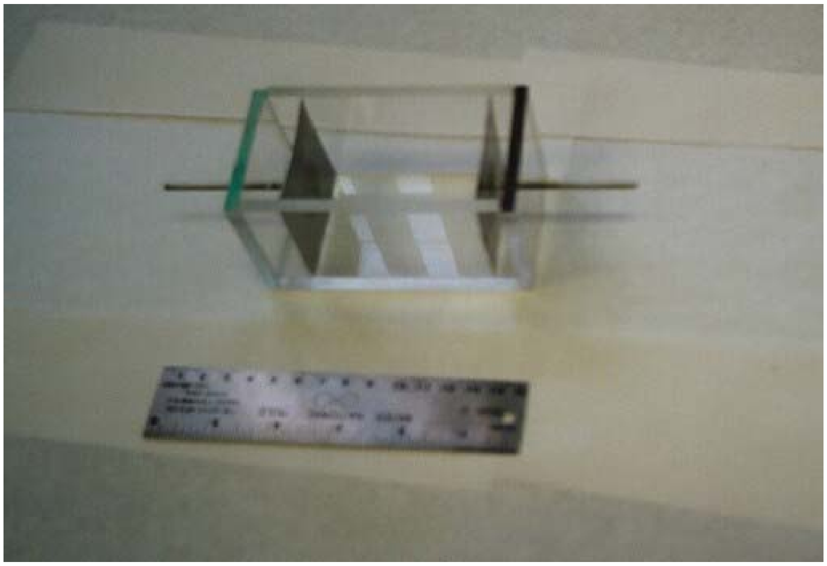

Figure D-3 Large Conductivity Cell

Figure D-4 Tramex Wood Moisture Meter

Figure D-5 Road Salt Retained on #10 Sieve

Appendix E. Conductivity vs Water Content at Room Temperature

Morton Salt (25°C)

Resistance (ohms)	Conductivity (mohs/cm)	Water Content (%)	Tramex
1300	0.000769231	2.126	38.8
740	0.001351351	2.851	39.5
670	0.001492537	3.122	39.7
400	0.0025	4.543	40.2

International Chile Salt (27°C)

Resistance (ohms)	Conducitivity(mohs/cm)	Actual Water Content	Tramex
4400	0.000227273	1.024	35.8
1500	0.000666667	1.987	38.6
680	0.001470588	2.903	39.6
310	0.003225806	4.338	40.2

American Rock Salt (27°C)

Resistance (ohms)	Conductivity (mohs/cm)	Water Content (%)	Tramex
2100	0.00047619	1.210	38.2
1020	0.000980392	1.870	39.3
580	0.001724138	2.763	40
410	0.002439024	3.631	40.2