CHAPTER 3

APPARATUS AND CALIBRATION

In order to measure the electrical resistivity of the soil samples, a resistivity box had to be fabricated. However, this apparatus will be useless unless it can be proved that it provides reliable results. As the experiment progressed, significant improvements made to the resistivity box resulted in more reliable data. The improved model will allow the apparatus to produce results that are closer to in situ electrical resistivity measurements.

Detailed description of each model of the resistivity box, including failed models, presented below, will allow future researchers to make further improvements.

3.1 Nine-electrode model resistivity box

The first model that used in the experiment was based on a very simple idea. Sediment packed in the box can be considering as an unknown resistor connected in series with two brass electrodes at both ends of the box. The measurements were made by applying the current to a soil sample through two current electrodes and then measuring the voltage drop across two potential electrodes. The resistance of soil sample can then be calculated from Ohm's law.

Unfortunately, not every soil or sediment is homogeneous; nine pairs of electrodes installed on every surface of the resistivity box helped avoid the effects of inhomogeneity. In this way, one can measure various parts of sample and average the results to arrive at a representative resistivity of the entire sample.

A cylindrical resistivity box was made from 0.925 cm thick PVC and an insulating plate was used as a cover. This box is 11.425 cm in diameter and 11.69 cm in height, and provides a sample volume of 1.2 liters. Each side of the cylindrical resistivity box is provided with three brass electrodes 12.5 mm in length and 2.5 mm in diameter. These electrodes were spaced at 30 mm center to center and can be

screwed into the compacted soil sample with an embedment length of 2.5 mm, as shown in Figure 3.1.
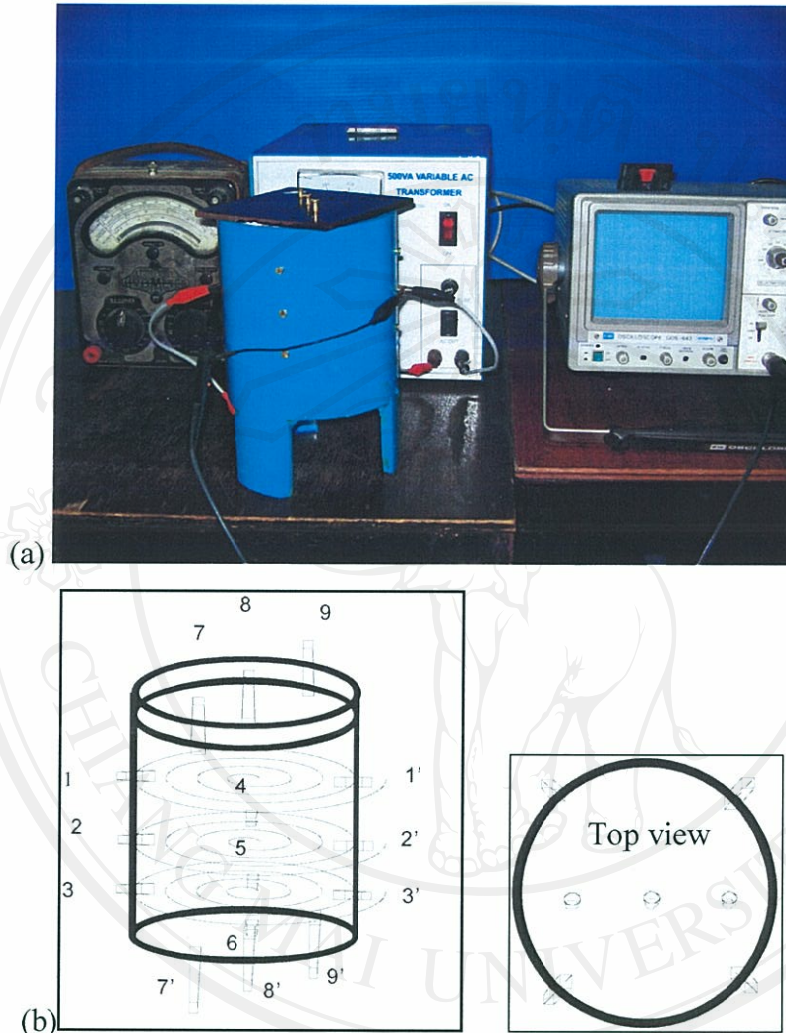

(a)

(b)

Figure 3.1 (a) The nine-electrode resistivity box.

(b) A schematic diagram showing the placement of electrodes in the nine-electrode resistivity box.

How much voltage needs to be applied to each sample? This question needs an answer before starting the experiment. Note that during the entire measurement and the calibration process, the resistivity values of whatever sample is inside the box can vary depending on the nature of the material. In order to make sure that the current flows through the entire sample, the voltage has to be sufficient. If the sample

is highly resistive, the necessarily high voltage may cause heat that result in measurement errors.

A small experiment helped determine what the proper voltage to supply to this apparatus is. This apparatus has 9 pairs of brass electrodes and each pair of electrodes may respond to various voltages and currents. The corresponding current (I) was obtained for each voltage in all pairs of electrodes using NaCl and KCl 0.1 molarity solutions. Using a constant AC power supply operating a single frequency source at 50 Hz and yielding an output voltage varying from 0 to 30 V, in step of 5 V. However, this frequency is the frequency used throughout Thailand and the nearby electrical appliances emit interference that produces a noisy signal. Such interference was observed many times during the experiment and occasionally caused difficulty when reading the oscilloscope. Unfortunately, this was the only power supply available in my laboratory.

The response of the 0.1 M KCl solution with the various currents is presented, in Figure 3.2.
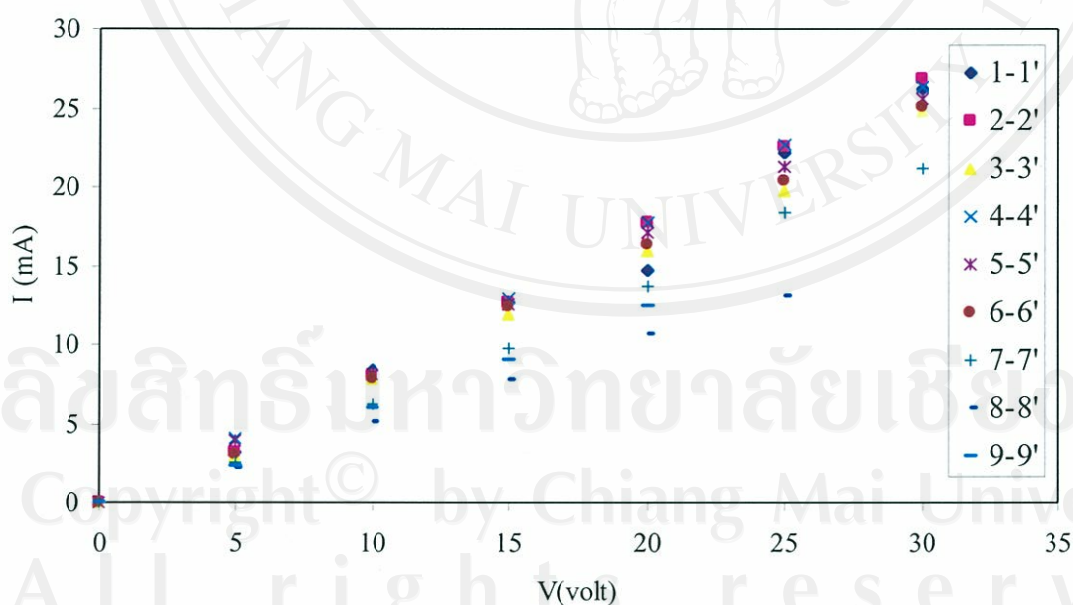

Figure 3.2 Applied voltages versus measured current response of nine pair electrodes for 0.1M KCl solution.

The results suggest that the proper voltage that should be applied to the apparatus is between 1 to 10 V because, within this range, the current remains relatively constant among the electrode pairs but at higher voltages, the currents vary significantly between the electrodes. This graph served as a reference for all the succeeding experiments. Mostly experiments used 5 V, but in some of the experiments, where the sample being tested had a high resistivity 10V proved a better choice.

The next step was to begin the calibration of the nine-electrode resistivity box. In geologic materials placed in the box, any pore fluid will drain toward the bottom of the box due gravitational and capillary forces; therefore, each electrode pair will register a different value. If each pair is measured one by one and the measurements then averaged using the rule of parallel resistors, which requires the inverse of the sum of each obtained value (Equation3.1)

The total resistance of parallel resistors $R_{total} = (R_1 + R_2 + R_3)^{-1}$ (3.1)

Alternatively, three electrodes can be connected in a parallel network. Therefore, the design used two types of circuits as shown in Figure 3.3. Ideally, assuming the sample is homogenous; these two circuits should result in identical measurements.
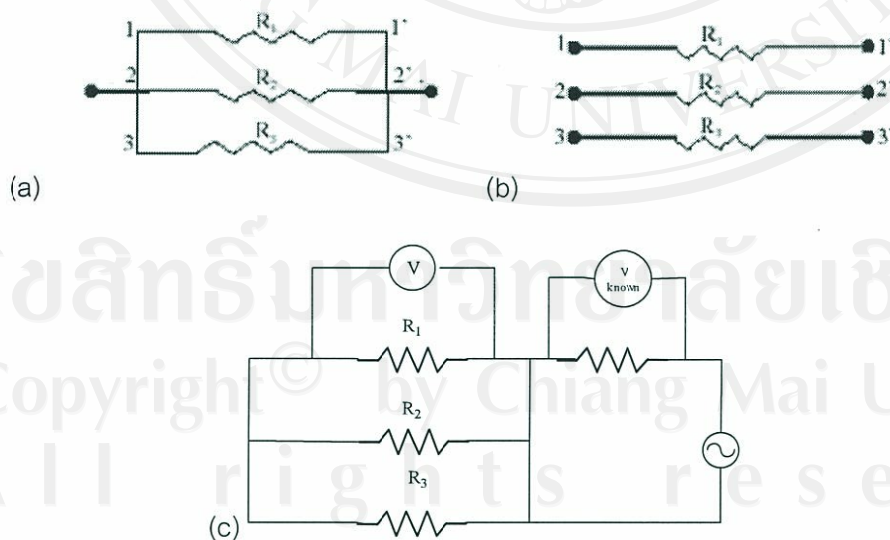

Figure 3.3 (a) Three-electrode resistance schematic (b) Individual electrode resistance schematic. (c) Schematic for the nine-electrode model resistivity box.

Unfortunately, the real calibration did not match the theory. Calibration using a homogenous standard 0.1 M NaCl solution, gave unacceptably different results for the two types of networks as shown in Table 3.1

Table 3.1 Comparison of three-resistor and individual resistor models using 0.1 M NaCl solution (at 10 V applied voltage).

Electrode pair	Resistance (ohm)	Resistance(ohm)		Percentage error (%)
		computed	experimental	
1-1	144.3586			
2-2	153.7459	50.63142	52.76873	4.13405
3-3	158.2492			
4-4	159.041			
5-5	144.6345	51.3598	55.13394	7.08801
6-6	159.5197			
7-7	147.8387			
8-8	145.7364	47.28758	51.53991	8.60556
9-9	132.9545			

The reasons for this discrepancy are that, 1) each pair of electrode was used for two functions - both as a current electrode and also as a potential electrode, thus, the resistances values given by this method are not the real values, but rather they are the resistance values of both the sample and the electrodes themselves, and 2) the electrodes are too close too each other, perhaps nine electrodes are too many to fit in such a small resistivity box. The measurements are sensitive, it is better not to move the electrodes closer together. From these reasons newly designed models replaced this original model.

3.2 Four-electrode model resistivity box

The next design was based on a four electrode configuration that is used in resistivity exploration techniques. The box is made from 8 mm thick cylindrical PVC. There are two brass potential electrodes on the top cover of the box which penetrate 10 mm into the sample and another two current electrodes were placed at each side of the container wall (Figure 3.4). After packing the sample inside the box, the cover lid is replaced and the four electrodes will be arranged in line as in a Wenner configuration. The Wenner configuration is one of the most commonly and popularly electrodes system. But it is the simpler in that the current and potential electrodes are maintained at an equal spacing (Kearey and Brooks, 1991).
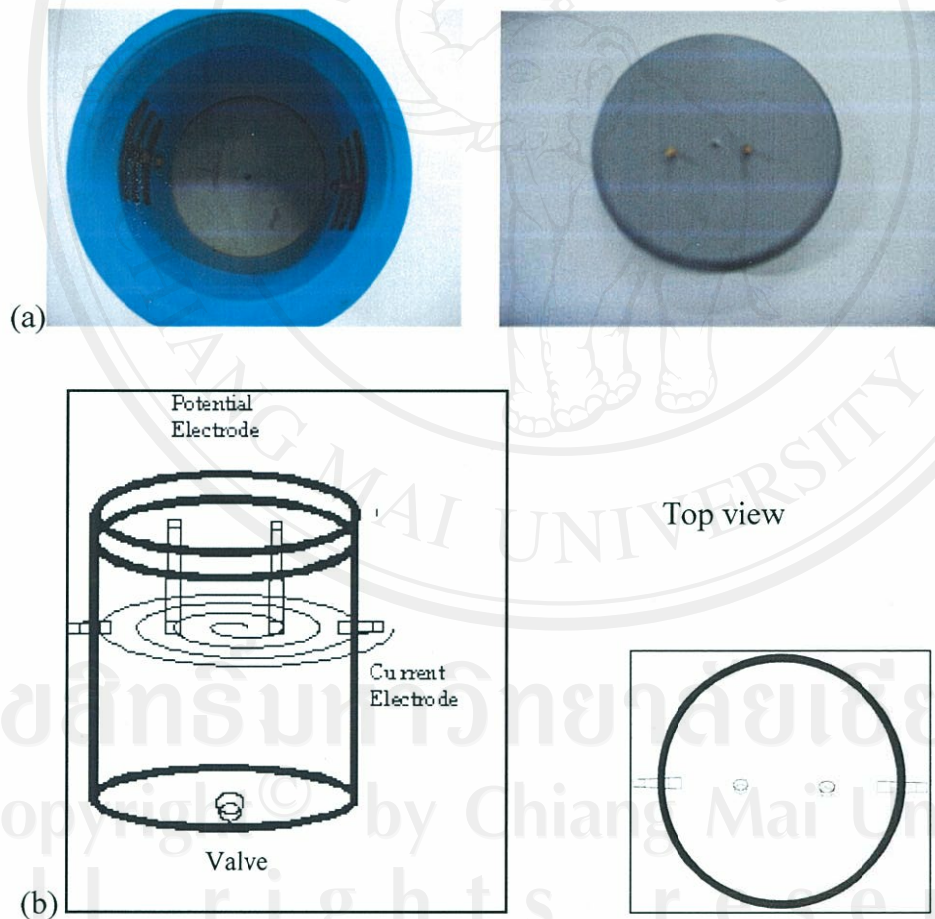

Figure 3.4　(a)　The four-electrode model resistivity box.

(b)　The schematics of electrodes adjustment of the four-electrode model resistivity box.

Thus, the four electrodes are arranged in a Wenner configuration with the separation between the two current electrodes equal to inside diameter of the box. The separation between the two potential electrodes is one third of the box's diameter. The electrode configuration is shown in Figure 2.6. In a four-electrode configuration measured over homogeneous material, the electrical resistivity can be expressed as Equation (2.4)

To calculate the electrical resistivity from the measured voltage from this four-electrode model resistivity box, all dimensions R must be replaced in Equation (2.4). According to Wenner configuration, all R shall be equal but small imprecision in constructing the box caused $R_1 \neq R_2$ and $R_3 \neq R_4$. Specifically, this resistivity box has

$$
\begin{aligned}
R_1 &= 36.50 - (3.05 / 2) - 5.00 \\
R_2 &= 34.05 - (3.05 / 2) - 5.00 \\
R_3 &= 66.45 - (3.05 / 2) - 5.00 \\
R_2 &= 64.00 - (3.05 / 2) - 5.00
\end{aligned}
\tag{3.1}
$$

Where 3.05 mm is the diameter of screw and 5 mm is the length of screw penetrating. After replaced the actual length of all R's in Equation (2.4), the geometry constant of this resistivity box can be calculated as shown below (Table3.2).

Table 3.2 The geometry constant of the four-electrode resistivity box, calculated from Equation 2.4, by using the configuration's dimensions.

R_1 (mm)	R_2 (mm)	R_3 (mm)	R_4 (mm)	Geometry constant (mm)	Geometry constant (m)
29.975	27.525	59.925	57.475	176.4675	0.1765

After obtaining the geometry factor by calculating from theory, it should also be measured in the laboratory for comparison purposes before calibrating the resistivity box. According to Ohm's law (Chapter 2), the resistance R of sample in the apparatus can be expressed as R = V/I.

The value of R can be correlated with electrical resistivity, ρ, as shown by Equation (2.4). The term representing the geometry factor can be made more complicated by the geometry of the box and electrode arrangement itself, therefore, in the experiment that term reduced to a factor "a" as expressed by:

$$\rho = a \times R \qquad (3.2)$$

If the geometry of the box does not cause any boundary effect, the geometry factor obtained from the calibration experiment should be equal to the calculated geometry factor.

The calibration measurement was done by applying a current of known voltage, 5 V, to the current-electrode pairs. The AC current cannot be measured directly by an ammeter, the current (I) was measured via a series connecting a known resistor to one of the current electrodes, then measure the voltage (V_{known}) passing through the known resistor using an oscilloscope. Then by using the Ohm's law, the current of this circuit can be calculated.

The reading of resistance of the material consists of a voltage (V_p) measured by a oscilloscope between potential electrodes, and the current (I) as measured above. The resistance of unknown material in the box can then be calculated. In this calibration, the material is the various concentrations of standard NaCl, KCl and $MgCl_2$ solutions which have known electrical resistivity, ρ. The electrical resistivity of these solutions can be found very easily by using a conductivity meter. The conductivity of the solution can then be converted to resistivity by this relationship $1 Sm^{-1} = 1\Omega^{-1}m^{-1}$. The conductivity and resistivity values of the solutions used in the calibration are shown in Appendix A. The measurement's schematic is shown in Figure 3.5.
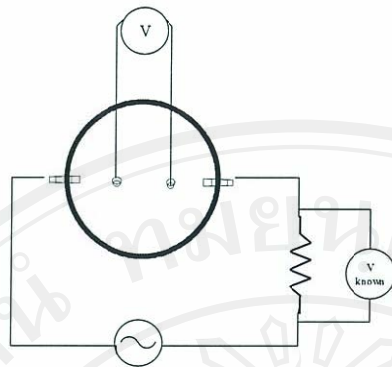
Figure 3.5 The measurement's schematic of the four-electrode resistivity box.

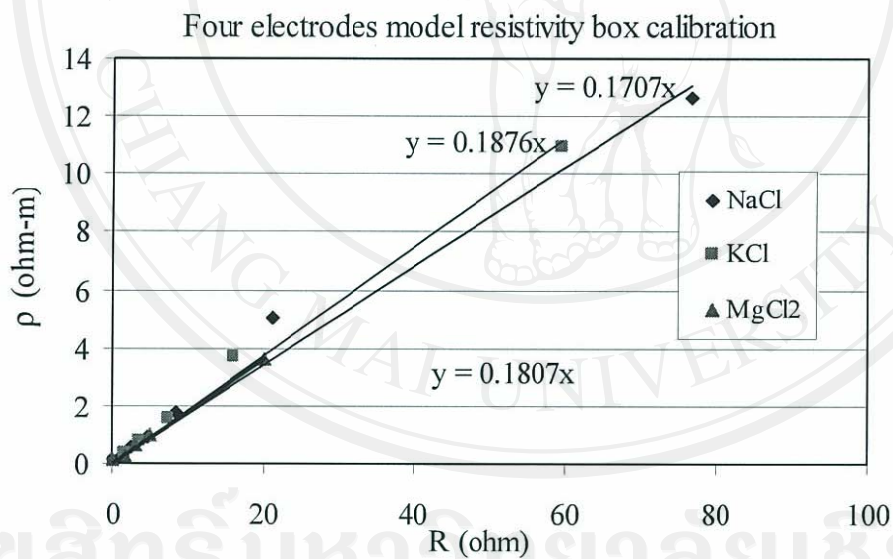Parameter 'a' is obtained by plotting ρ against the resistance, R as shown in Figure 3.6.

Figure 3.6 Graph of electrical resistivity (ρ) against the measured resistance (R) obtained from the four-electrode resistivity box calibration.

From Figure 3.6, for NaCl, KCl and $MgCl_2$ solution, the slope of ρ versus R is 0.1707, 0.1876 and 0.1807, respectively. Hence the average value of the parameter a, was adopted as 0.1797.

Comparing the geometry constant obtained by calibration (0.1797), with the geometry constant calculated from Equation (2.4) (0.1765), shows that percentage error between these two geometry constants is approximately 1.8%. This small error suggests that the boundary effect can be ignored. When the dimensions of the box is large enough compared with the size of grains in the sample.

After obtaining the good calibration result, it seemed satisfactory to use this model in the experiment. However, based on advise from Dr. Christoph Grissemann, a Senior Researcher in the Federal Institute for Geosciences and natural resources of the Federal Republic of Germany, and co-advisor on this thesis, a new resistivity box was constructed with an improved design. The new design used electrodes made from stainless steel instead of brass, because stainless steel does not cause electrolysis. Unfortunately, only a very low quality stainless steel is available locally. Although the four electrodes with a Wenner configuration seems to work fine, using only four electrodes may still not good enough to cover the heterogeneity that occurs geologic materials, hence the new design uses six electrodes .

3.3 Six-electrode resistivity box

In this version of the resistivity box, instead of having one normal set of electrodes in the Wenner configuration with two current electrodes on opposite sides of the box and two potential electrodes on the lid of the box, the box has another four electrodes on the each side along the box's curvature and lacks the two electrode from the top lid of the box. The six electrodes can be arranged into four sets with Wenner configurations by moving the potential-electrode pair and current-electrode pair. .

During the calibration using mostly solutions, it did not seem necessary to make the box's lid solid and stable, it only seemed necessary to make sure that there was not any leakage. But when using real geologic material, especially a clay-rich sample, the wet clays expand; therefore lid must be tight enough to prevent the sample from swelling and deforming the box. If the sample is allowed to swell or shrink the change to sample compaction will affect the resistivity values. Therefore the lid was thickened and more threads were added to the fitting. Small holes were also drilled through the lid so that any pore vapor could escape.

A decision had to be made about the best way to pack the soil samples and soak the samples in the box. Clay–rich materials are tough to deal with because they have very low hydraulic conductivity and therefore take a long time before they are soaked with the test fluid. In some German laboratories, CO_2 is used as a convection gas. The streaming of CO_2 gas replaces the air in the pore spaces. Because the concentrated CO_2 has a much higher partial pressure than in the atmosphere (and thus pore air) in the resulting partial pressure-gradient increases the rate of fluid infiltration.

Although this method is conservative, and time-efficient, it is necessary to have a two-way pressure gauge valve, which unfortunately was unavailable. Instead, a vacuum pump at the bottom of the resistivity box allowed the testing fluid to drain from the top of the box. This method has the disadvantage that the sample may shrink from the strong pressure of the vacuum pump.

To implement this method, a hole was drilled at the bottom of the box and a small valve was attached. This valve was use for draining standard solution during the calibration, and it was also used as the connected path with the vacuum pump in order to accelerate the percolation rate of the testing fluid as it soaked through the sample, from the top to the bottom. The final version of the six-electrode resistivity box is shown in Figure 3.7.
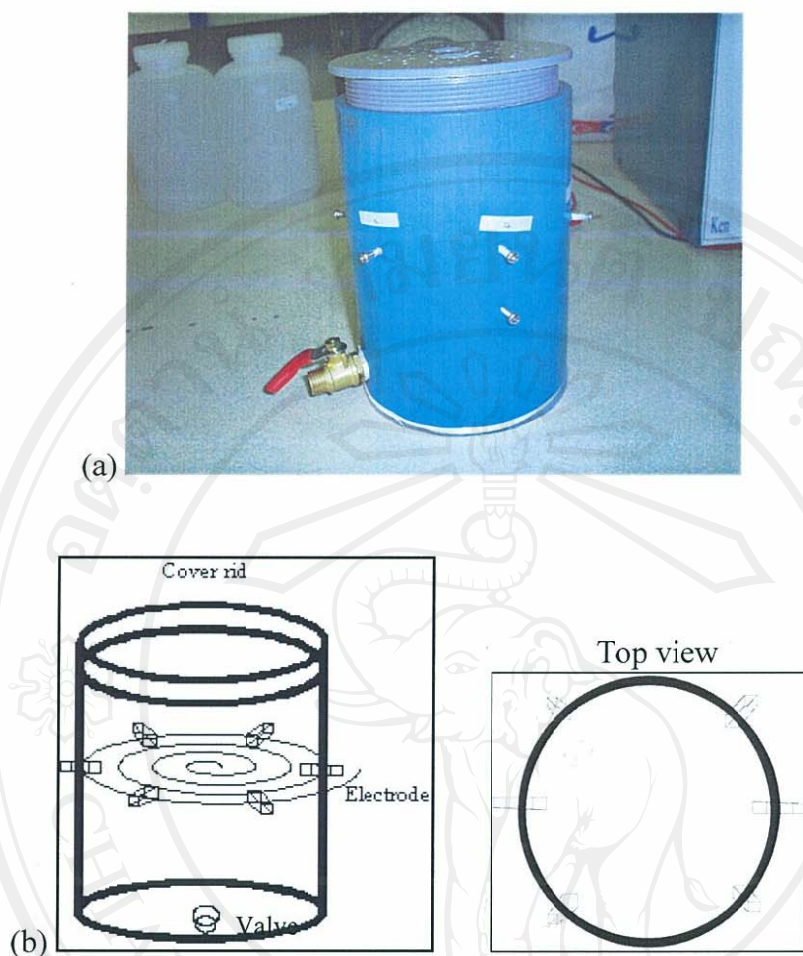
Figure 3.7 (a) The six electrodes model resistivity box.

(b) Schematic of the electrode arrangements of the six-electrode resistivity box.

After the assembly was completed, the box still needed to be calibrated. The same methodology was used to calibrate the box, as outlined above, by using the known resistivity values of NaCl, KCl and $MgCl_2$ standard solutions. The modification changed the four-electrode model into a six-electrode model with four sets of Wenner configurations on each side of the box. Each set of electrodes obtained unequal geometric constants for the following reasons:

1) Small construction errors in the dimensions of the box, such as a very small misplacement of an electrode or a very small error on the curvature of the box can cause differences in the geometric constants.

2) The electrodes may not be perfectly perpendicular to the box.

Therefore, in the calibration process, four sets of electrodes need measuring and their geometric constants need to be calculated; this step is very important to ensure that the resistivity value measured with this box are reliable.

The fluid suction ability of the soil is heterogenous, not only because of the effects of gravitational force, capillary force and the bonding action of adsorbed, aligned water molecules between two clay-mineral surfaces but now the vacuum pump also have a very strong influence to this heterogenous. The pressure from the vacuum pump was not equally, so the inhomogeneities of the degree of saturation and conductance occurred throughout the box. There is no perfect measuring method to get the representative values of an entire sample. The six-electrode model is a compromise between eliminating the errors from inhomogeneities, and still providing information about the sediment from the varying resistivity values from each set of electrodes. The calibration results for each set of electrode configurations are shown in Figures 3.8, 3.9, 3.10, and 3.11. Also the final geometry constants of the box are summarized in Table 3.3.
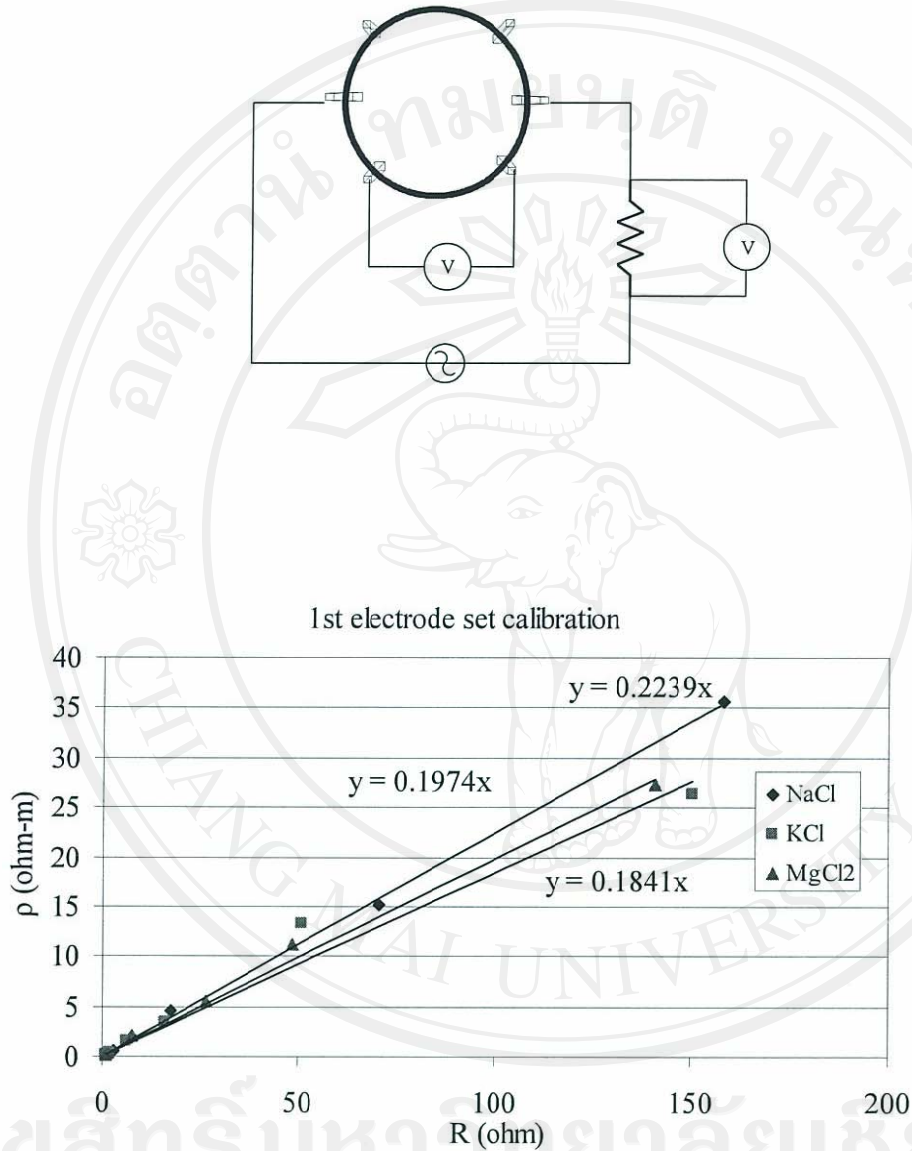
Figure 3.8 The first set measurement's schematic of the six-electrode model
resistivity box and resulting graph of of electrical resistivity (ρ) against the
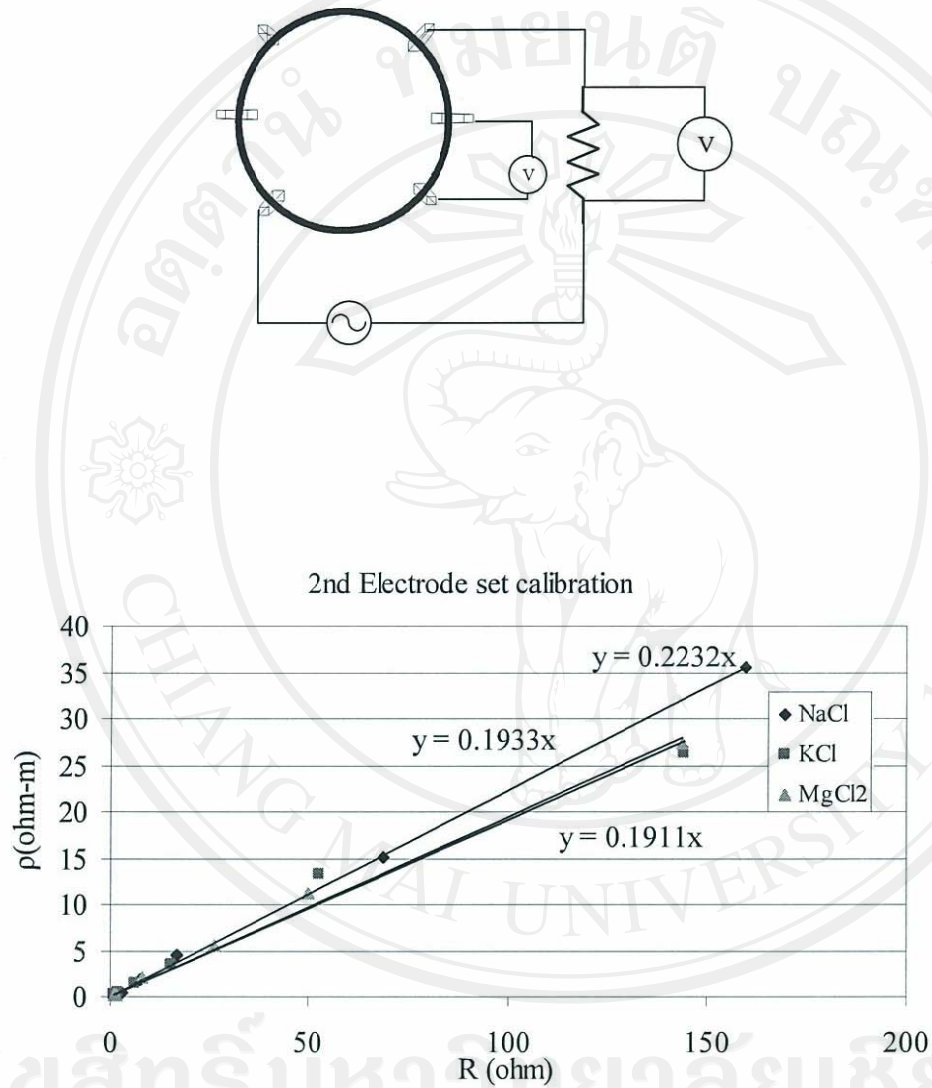measured resistance (R).

Figure 3.9 The second set measurement's schematic of the six-electrode model resistivity box and the resulting graph of electrical resistivity (ρ) against the measured resistance (R).
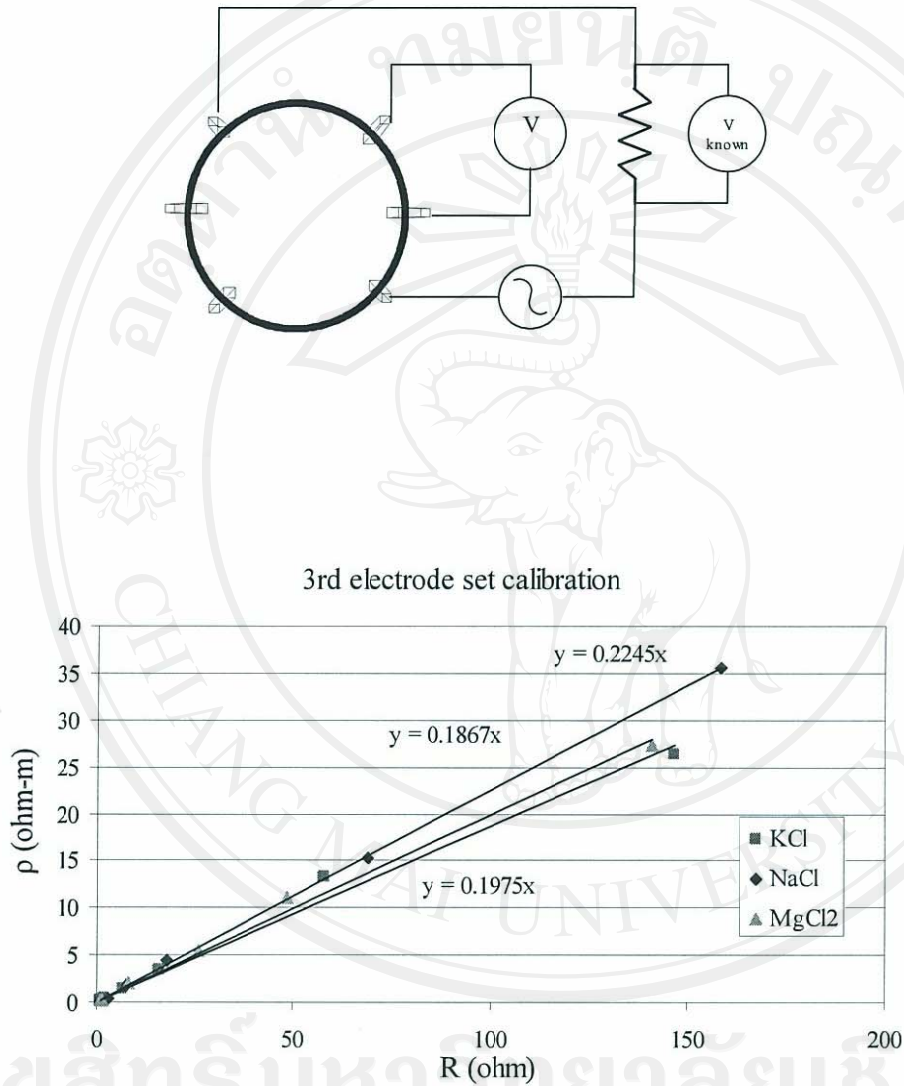
3rd electrode set calibration

Figure 3.10 The third set measurement's schematic of the six-electrode model resistivity box and the resulting graph of electrical resistivity (ρ) against the measured resistance (R).
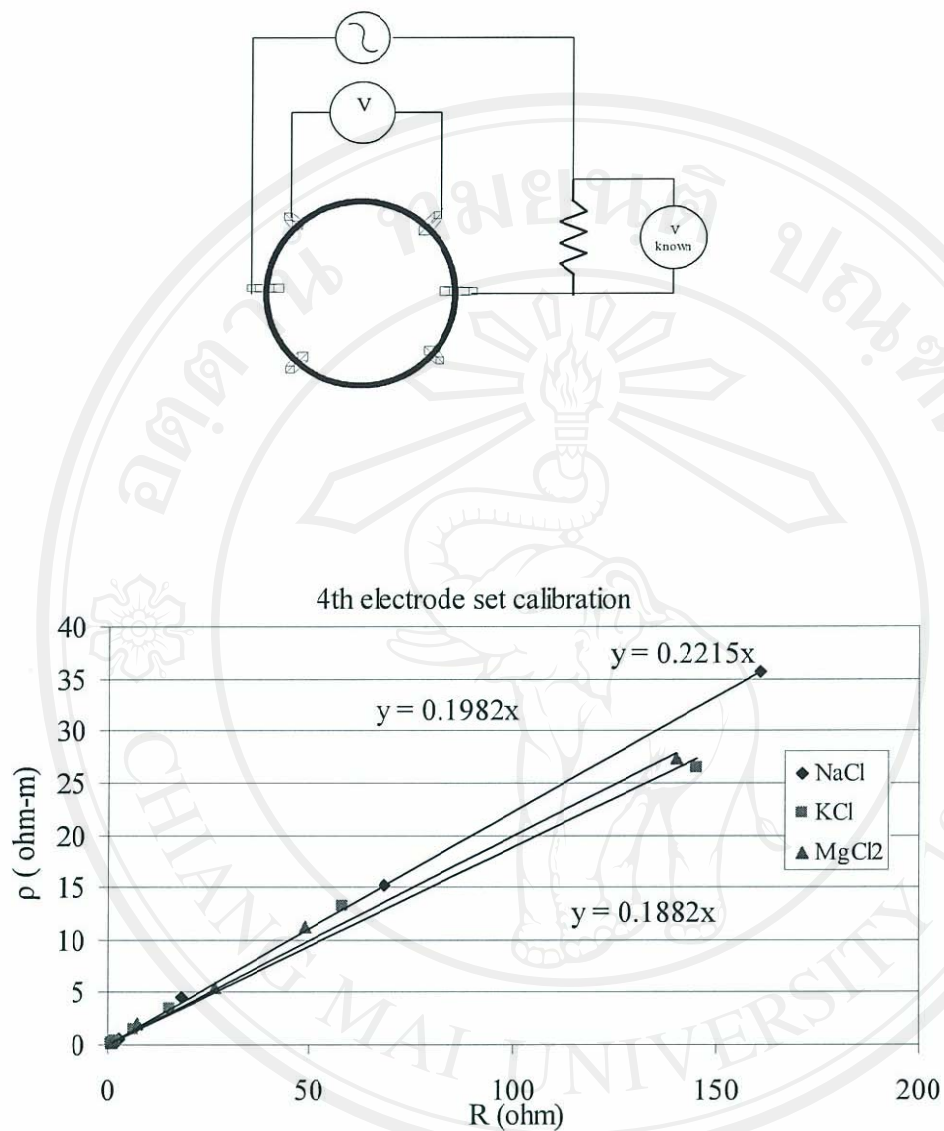
Figure 3.11 The fourth set measurement's schematic of the six-electrode model resistivity box and the resulting graph of electrical resistivity (ρ) against the measured resistance (R).

Table 3.3 The geometry factors of the four sets of electrode configurations obtained from the calibration of the six-electrode model resistivity box.

Electrode set	Constant geometry factor			Geometry factor	% error
	NaCl	KCl	$MgCl_2$		
1st	0.2239	0.1841	0.1974	0.2018	-0.32927
2nd	0.2232	0.1933	0.1911	0.2025	0.032927
3rd	0.2245	0.1975	0.1867	0.2029	0.214027
4th	0.2215	0.1982	0.1882	0.2026	0.082318

The small error between each geometry factor can be considered that the box construction is standardized and the curvature of the cylindrical do not cause any affected to the measurement.