Measurements of Soil Redox Potential

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Soil redox potentials (Eh) are commonly measured and operationally defined using platinum (Pt) electrodes, but there are variations in the methodology and instrumentation for measuring the associated voltages that may lead to imprecise and inaccurate estimates of oxidation–reduction intensity in the soil environment. Most field measurements of Eh are either made manually using a volt meter or automatically with a recording device such as a data logger. A mesocosm experiment was conducted to evaluate the impact of these two measurement approaches on resulting Eh values in three different wetland soils. Noticeable differences were observed between data collected using a multimeter (simple volt meter) and those collected with a data logger. Because the multimeter permitted the observed voltages to drift while the Pt and reference electrodes equilibrated following closure of the circuit, it was initially suspected that these were the more accurate data. By adding a 10-Mohm resistor (standard component of commercially available multimeters) to a closed circuit containing the Pt and reference electrodes, data loggers could be modified to collect data similar to those obtained using a multimeter. Further testing, however, demonstrated that the voltage drift observed when using a closed 10-Mohm circuit was an artifact of alterations in the electrochemical soil environment surrounding the Pt electrode. By using an instrument with very high (200 Gohm) input resistance, the artifactual drift could be essentially eliminated. Our comparisons and experiments suggest that the most accurate measurements of soil Eh and soil redox status are obtained using volt meters with high input resistance (e.g., 200 Gohm). Accurate Eh values also are obtained using data loggers (20 Gohm resistance) in the standard configuration that maintains an open circuit except during the actual instantaneous voltage measurement.

Abbreviations: IRIS, Indicator of Reduction in Soil; PVC, polyvinylchloride; SHE, standard hydrogen electrode.

Anaerobic and reducing conditions in soils can affect myriad biological and biogeochemical processes. Therefore, it is important to be able to operationally define the intensity of the oxidation–reduction status of soils using a simple method that is both accurate and precise. Although several approaches are used for measuring reducing soil conditions, such as α,α dipyridyl which turns pink in the presence of Fe(II) (Childs, 1981) or IRIS (Indicator of Reduction In Soil) tubes, which are polyvinylchloride (PVC) tubing coated with an Fe(III) hydroxide paint (Rabenhorst and Castenson, 2005; Castenson and Rabenhorst, 2006; Jenkinson and Franzmeier, 2006; Rabenhorst and Burch 2006), the standard method is to measure the voltage difference between a Pt sensing electrode and a reference electrode (e.g., calomel or Ag/AgCl) inserted directly into the soil (Mueller et al., 1985). The voltage difference between the Pt and reference electrodes is then corrected for the

reference electrode's standard voltage relative to the standard hydrogen electrode (SHE, E = 0.0 V), and is recorded as the Eh.

One challenge that emerges when collecting and interpreting soil Eh data is the high degree of spatial heterogeneity observed over even very small distances within the soil (sometimes even over a few centimeters). Because of this, multiple electrodes are commonly used to provide better (more precise) estimates of soil Eh and the variability associated with the estimates. Depth functions or profiles of soil Eh can be obtained by placing electrodes at different depths within the soil, and the electrodes are often left in the soil to measure time-dependent Eh variation. Soil Eh measurements using Pt electrodes can either be collected manually or with a data logger. Manual measurements are typically made using a volt meter to create a circuit between Pt and reference electrodes. Significant instrument or electrode "drift" is usually observed during the voltage measurements, and although these values sometimes stabilize quickly, it can often take 5 min or more for the drift to become negligible (<1 mV/min).

In cases where soil Eh data need to be collected at a greater frequency a data logger can be used (although these can be costly). A salt bridge (Veneman and Pickering, 1983) can be used in conjunction with a data logger to maintain electrical contact of the reference electrode with the soil solution while ensuring that the reference electrode remains viable over an extended time period without drying out. During the programmed measurement cycles the data logger circuit is closed momentarily at the specified time of the Eh measurement (for example the Campbell 21X data logger requires approximately

3 to 5 ms to complete the measurement) and the circuit is otherwise left open.

Given the importance of Eh measurements for assessing the redox status of wetlands, marshes, swamps, and upland soils, accuracy and precision in measurement of this master variable are important but poorly understood issues. Therefore, the objective of this study was to compare and evaluate the common approaches to measuring redox potential in wetland soils using Pt electrodes.

MATERIALS AND METHODS
Experimental Design

Field soil conditions affecting Eh measurements are highly heterogeneous, so we used laboratory mesocosms containing thoroughly homogenized soil material to minimize substrate variability. The mesocosms (Fig. 1) were constructed from 20-L plastic containers filled with approximately 12 L of soil materials from A or Oe horizons from three different wetland soils selected to represent a range in properties related to redox processes (Table 1). We constructed Pt electrodes using 0.5-mm wire (99.998% Pt) sealed with epoxy (see Owens et al., 2005).

The Berryland and Indiantown A horizons (from freshwater wetlands) were air-dried and ground to pass a 4-mm sieve. The Oe horizon from the Transquaking soil (from a tidal marsh) was kneaded and homogenized in a saturated condition, to minimize sulfide oxidation that would occur during drying. Distilled water was added through a 5-cm diam. PVC tube open at the bottom placed in the center of each mesocosm. Water was added from the bottom-up in this manner to prevent entrapment of air during saturation. A 1-cm layer of water was maintained above the mesocosm surface for the duration of the experiment. The temperature of the mesocosms was maintained at approximately 24°C (the temperature of the laboratory).

Daily Electrode Measurements

In each mesocosm, 10 replicate Pt electrodes were installed at a depth of approximately 8 cm below the soil surface (Fig. 1). Five of the electrodes were used for manual Eh measurements using a digital multimeter and a calomel reference electrode that was temporarily placed at the surface of the saturated soil at the time of measurement (standard practice for manual Eh measurement). Voltages were recorded once daily for approximately 1 wk and then at 3-d intervals after that. Each time measurements were made, voltages were recorded every 1 s for 600 s and were collected on a laptop computer using an RS232 output port from the multimeter.

The other five Pt electrodes were connected to a Campbell model 21X data logger in conjunction with a calomel reference electrode that was permanently mounted through a salt bridge in each mesocosm (Veneman and Pickering, 1983). Values from the datalogger were recorded each time that manual measurements were made (1- to 3-d
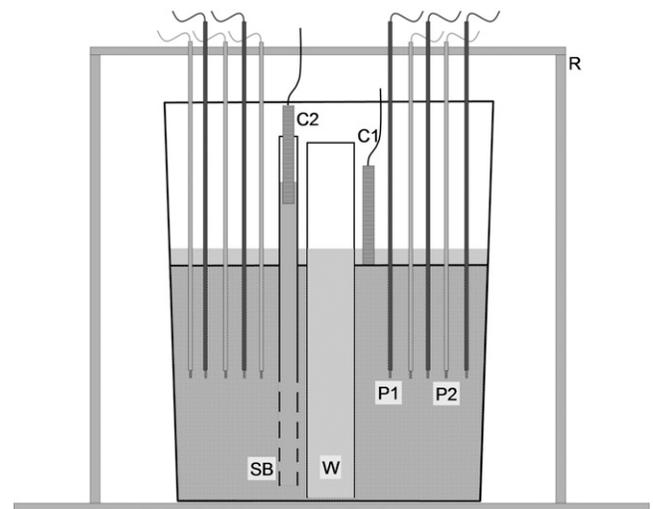

Fig. 1. Schematic of mesocosms filled with approximately 12 L of homogenized soil material. Five Pt electrodes (P1) that were secured to a wooden rack (R) were measured using a digital multimeter with a calomel reference electrode (C1) placed at the soil surface. Five other Pt electrodes (P2) were measured using a data logger and a calomel reference electrode (C2) attached to a salt bridge (SB). Water (W) was added through a 5-cm piezometer in the center of the mesocosm maintaining a 1-cm layer at the surface.

intervals from 19 May to 17 July 2006). Means of Eh measured manually and by the datalogger were compared for each measuring date. The electrodes were permanently installed in each mesocosm and thus temporal measurements were made using the same electrodes in the same locations (the same experimental unit). Because our interest is in whether the two methodologies yield different values on individual dates, the differences were compared for each date separately using a series of two-independent t test. Making numerous pairwise comparisons will increase the probability of a Type I error (i.e., declaring a false significant effect). Therefore, the calculated p values for the comparisons are reported.

Electrodes in the soils from the two freshwater wetlands (Berryland and Indiantown) functioned normally and reliably throughout the 9 wk of the experiment. All electrodes in the tidal marsh (Transquaking) mesocosms initially functioned normally, but eventually failed, apparently due to the effect of harsh soil conditions on electrode components. The Transquaking soil had much higher organic C content, was brackish (with a porewater salinity of approximately 7 ppt), and had abundant sulfides present—conditions which may have contributed to deterioration of the epoxy seal. Failure of the Pt electrodes may also be the result of PtS or $Pt(OH)_2$ coatings that can develop on the metal surface, which should be bright (polished) Pt for accurate measurement (James and Bartlett, 2000). Of the five electrodes attached to the datalogger, two failed by the end of the second week, and the

Table 1. Characteristics of soils used in this study.

| Series Name	Classification	Pedo-Geomorphic Setting	Horizon	Organic C	Texture	Sampling Location
				g kg^{-1}		
Berryland	Sandy, siliceous, mesic Typic Alaquods	Late Pleistocene dunes	A	55	Loamy Sand	38°12′7″N 75°30′2″W
Transquaking	Euic, mesic Typic Sulfihemists	Brackish tidal marsh near Chesapeake Bay	Oe	370	Mucky Peat	38°52′38″N 76°32′39″W
Indiantown	Coarse-loamy, siliceous, active, acid, mesic Cumulic Humaquepts	Flood plain of the inner Coastal Plain	A	67	Silt Loam	39°1′43″N 76°50′30″W
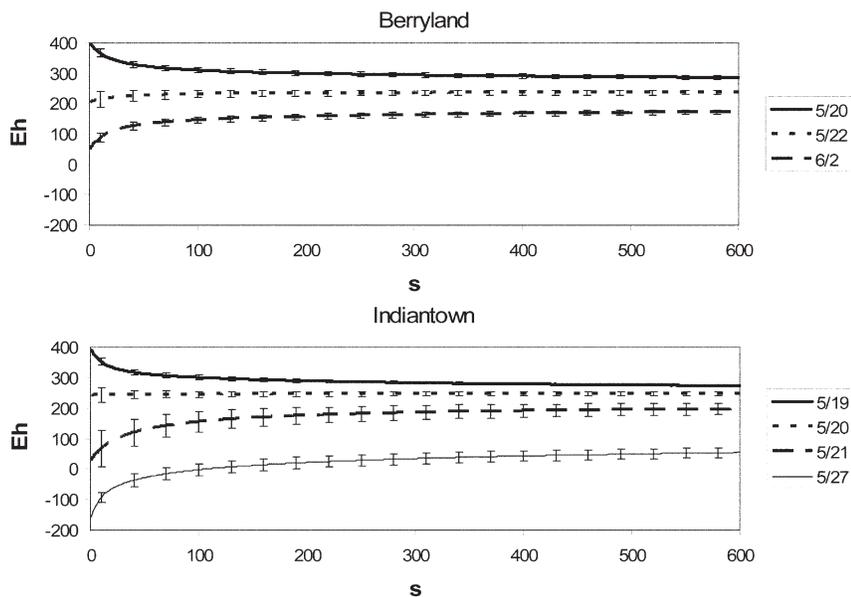
Fig. 2. The Eh measurements made using a digital multimeter and a calomel reference electrode in two soils on several different dates illustrating the drift and stabilization of readings over a period of 600 s. Lines and bars represent means and standard errors for five replicate electrodes.

remaining three electrodes failed after 5, 7, and 9 wk, respectively. All five of the electrodes used for making manual measurements failed between 7 and 9 wk. The failure of individual electrodes was easy to identify as Eh readings dropped precipitously to unrealistic values of –400 to –500 mV. As electrodes in the marsh soil degraded, the data from the failed electrodes were excluded from analysis.

Evaluation of Resistor Effects

To address possible issues related to input resistance of instruments used to measure Eh, we made voltage measurements with identical Pt and reference electrodes in the mesocosms, measuring first with the data logger and then manually with the multimeter. The data logger was programmed to record the data from each of the 15 electrodes (five in each mesocosm) after periods of 0, 10, 20, 30, 60, 120, 300, and 600 s in its normal configuration (thus requiring 2.5 h to complete one cycle with 15 electrodes). In this "normal" configuration, the circuit between the Pt and reference electrodes was left open except for momentary closure when the data logger recorded the voltage measurements.

Similar measurements were also made with the data logger in an alternate configuration in which a 10-Mohm resistor was added to the circuit. This was accomplished using a pair of relays to close the circuit between the Pt and reference electrode with the resistor in line, open it momentarily during the time that the datalogger made the voltage measurement (3–5 ms), and then reclose the circuit until the next measurement was made. Using this alternate configuration for the data logger (using relays and in-line resistors), voltage measurements were made in a standard Fe(II)–Fe(III) solution (Light, 1972) and in the soil mesocosms using resistors of different magnitude (0.1, 1, 10, and 50 Mohm).

For each of these scenarios, measurements were also made manually with a multimeter using the identical Pt and reference electrodes used by the logger. These manual data were recorded from each of the 15 electrodes continuously for a period of 600 s with the circuit remaining closed throughout this time. Additionally, voltage measurements were made in new mesocosms made from the Berryland and

Indiantown soils using both a lab grade pH/Eh meter with high input resistance (200 Gohm) and the multimeter.

RESULTS AND DISCUSSION
Daily Electrode Measurements

When the soils were initially saturated and Eh values still reflected oxidized conditions, the observed drift in manually measured voltages over the 600-s measurement period was negative with readings stabilizing approximately 100 mV below the starting point (Fig. 2). Within a few days, however, as the Eh values became more reducing, the observed drift over the measurement period became positive, with readings stabilizing up to 150 mV higher than the starting point.

Comparison of the redox data collected manually (after an equilibration period of 600 s) with those data recorded by the logger (during an instantaneous [3–5 ms] circuit closure) demonstrate a systematic difference between the two methods (Fig. 3). After the first few days, the data collected using the equilibrated multimeter were consistently higher than those recorded by the data loggers, with differences ranging between 30 and 250 mV. The p values calculated for pairwise comparisons of Eh on individual dates for the Berryland and Indiantown soils, were less than 0.05 for almost the entire experimental period, and for the Transquaking soil, the p values were <0.05 for the first few days and then again for the period following Week 3. Making a large number of pairwise comparisons results in an increased probability of making a Type I error (i.e., declaring a false significant effect). Thus, the calculated p values for the comparisons are included in Fig. 3. However, even if one or more Type I errors did occur, the large number of comparisons with $p < 0.05$ and the systematic difference between the two methodologies clearly demonstrate that there are fundamental differences between values measured manually or with the loggers.

The large and systematic differences between the data sets were disconcerting and immediately posed the question of which were the more accurate data. As we considered the manner in which the data were collected, it occurred to us that the data loggers were recording data without any equilibration period, and therefore might be suspect.

This question was addressed by comparing voltages measured with identical electrodes both using the data logger (programmed to collect data at intervals of 0, 10, 20, 30, 60, 120, 300, and 600 s) and using a digitial multimeter (recording continually for 600 s) (Fig. 4). The values recorded by the multimeter showed a positive drift toward equilibrium consistent with the curves observed in Fig. 2. The values recorded by the data logger for each Pt electrode, however, were relatively constant over the 600-s period and were similar to the values recorded using the multimeter at the very beginning of the Eh drift. Because these data were collected after the initial 9-wk experiment, the electrodes in the Transquaking soil (electrodes 6–10) give erroneously low values, although comparisons between the two measurement methods show the same trends.

Evaluation of Resistor Effects

Based on our observations, something was clearly amiss with one of the approaches used to measure redox. van Bochove et al. (2002) proposed that a 10-Mohm resistor should be placed into the circuitry of the data logger and that a closed circuit (including the 10-Mohm resistor) should be maintained between the Pt and reference electrode during the periods between measurements recorded by the data logger. This approach was apparently intended to allow the voltage to drift and stabilize before the measurements were taken. The 10-Mohm value for the resistor was apparently selected because most multimeters have an input resistance of approximately 10 Mohms (the input resistance of the mulitmeters we routinely used ranged between 10 and 11 Mohms). When measurements were made again with the same instrumentation (data logger and multimeter on identical Pt electrodes) but after adding a 10-Mohm resistor to the data logger circuit in the manner described in van Bochove et al. (2002), measurements by the data logger closely followed the pattern of drift and equilibrium produced when recording measurements with the multimeter (Fig. 5).

To test whether the magnitude of the input resistance had any significant impact on measured voltages, several different resistors (0.1, 1, 10, and 50 Mohm) were inserted into the data logger circuitry and voltages were measured in a redox standard solution (Light, 1972) and within the soil mesocosms. In the standard Fe(II)–Fe(III) solution, when resistors of 50 and 10 Mohm were included in the circuit, the readings were highly stable and were identical (Fig. 6). Readings also remained stable after reducing the resistance to 1 Mohm. Only when the resistance was decreased to 0.1 Mohm did the measurement show any drift, and the equilibrated reading was approximately 10 mV lower than the others. When similar measurements were made in soils, however, the results were dramatically different (Fig. 7). The results are illustrated with data from a single electrode each from the Berryland soil (Electrode 1) and from the Indiantown soil (Electrode 11); similar results were observed for all electrodes. The magnitude of the observed drift in Eh measurements was strongly related to the input resistance of the system. The difference between observed Eh drift in the standard solution (low degree of drift) vs. the soil environment (high degree of drift) can be attributed to the stability of the Fe(II)–Fe(III) solution, which is resistant to current effects because it contains only one balanced redox couple. The Pt electrode

equilibrates rapidly with the Fe(II) and Fe(III) components of the solution through the transfer of a miniscule quantity of electrons between the ions and the Pt, so that the energy level of the electrode matches the solution electron potential
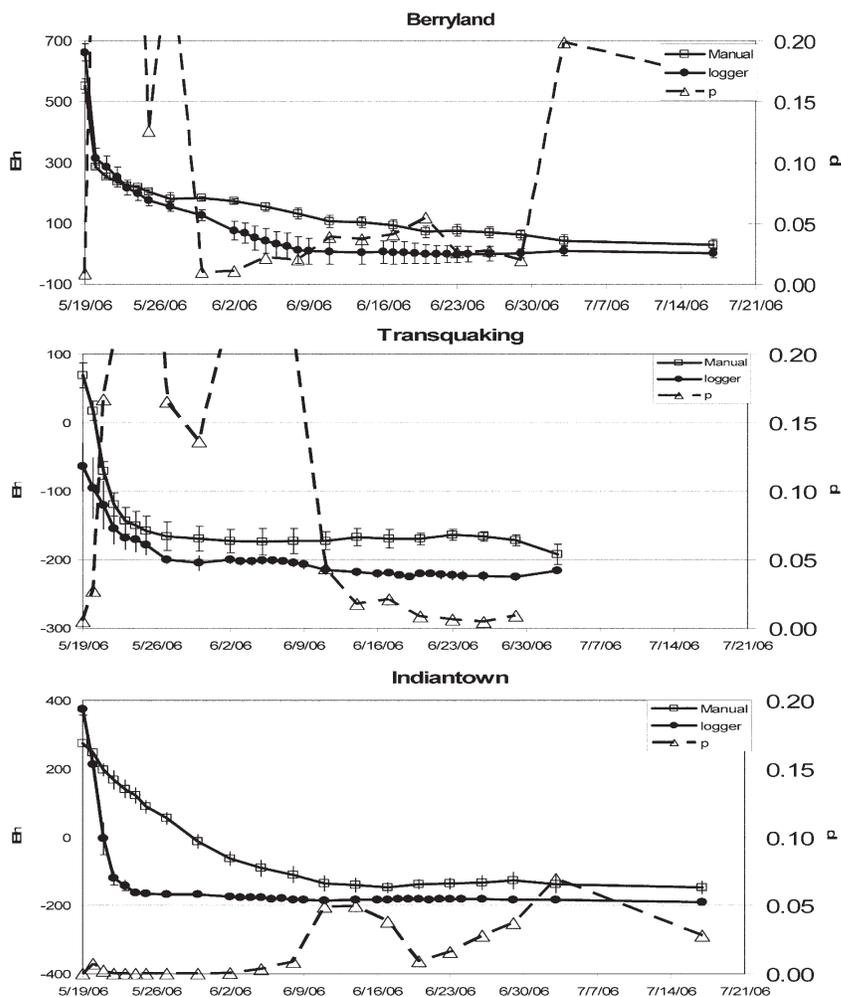

Fig. 3. Redox data for the three mesocosms collected manually with a digital multimeter (after a drift of 600 s) and using a data logger. Lines and bars represent means and standard errors for replicate measurements. Note the systematically lower values recorded by the data loggers. Values for p that were calculated for pairwise comparisons of the two methods on individual dates are shown on the second Y axis.
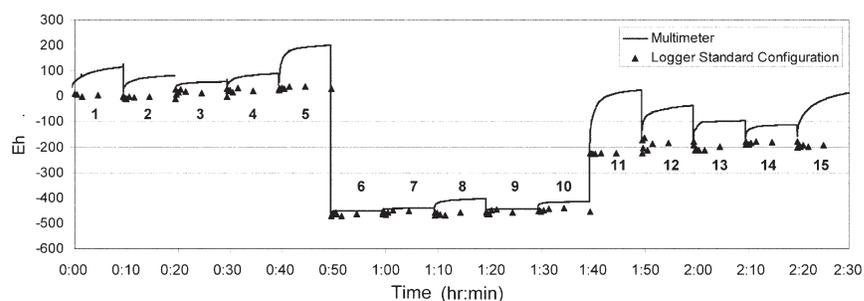

Fig. 4. Redox measurements made sequentially from five electrodes in each of the three mesocosms using the data logger (standard configuration) programmed to record data after periods of 0, 10, 20, 30, 60, 120, 300, and 600 s (solid triangles) and also made continuously using a multimeter for 600 s on each of the same electrodes (continuous line). Electrodes 1 through 5 were in the Berryland mesocosm, Electrodes 6 through 10 were in the Transquaking mesocosm, and Electrodes11 through 15 were in the Indiantown mesocosm.
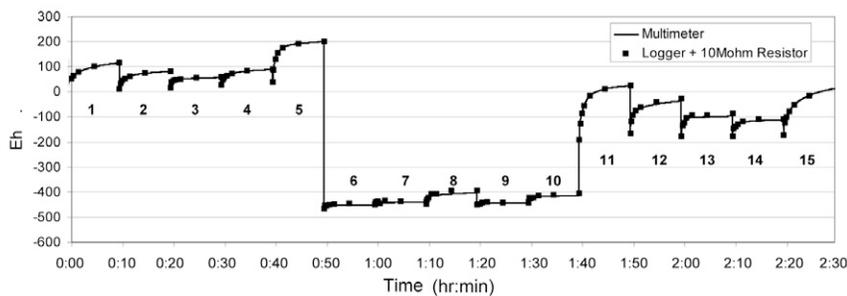
Fig. 5. Redox measurements made sequentially from five electrodes in each of the three mesocosms using the data logger configured to include a 10-Mohm resistor and programmed to record data after periods of 0, 10, 20, 30, 60, 120, 300, and 600 s (solid squares). Measurements were also made continuously using a multimeter for 600 s on each of the same electrodes (continuous line). Electrodes 1 through 5 were in the Berryland mesocosm, Electrodes 6 through 10 were in the Transquaking mesocosm, and Electrodes 11 through 15 were placed in the Indiantown mesocosms.

(Compton and Sanders, 1996). In contrast, in soils with multiple redox couples contributing to measured redox potentials, drift may be expected in response to the magnitude of current flow. For example, redox couples containing dissolved and solid phase organic C and sulfides in gaseous, soluble, and solid phases may react slowly with the Pt electrode before a stable reading is obtained. Current flow in the circuit at low resistances can thereby change the ratios of oxidants and reductants in the soil environment surrounding the Pt electrode, resulting in different measured Eh values.

Ohms law states that $E = I \times R$, where E = voltage (volts), I = current (amps), and R = resistance (ohms). When measuring voltage (E), R must be very high to keep the current flow (I) at a minimum so as not to disrupt or alter the system being measured. Theoretically then, the ideal input resistance for making voltage measurements would be a value approaching infinity. Typically, laboratory-grade pH (volt)meters (which are much more expensive than multimeters) have input resistance of approximately 200 Gohms (over four orders of magnitude greater than multimeters). When measurements were made
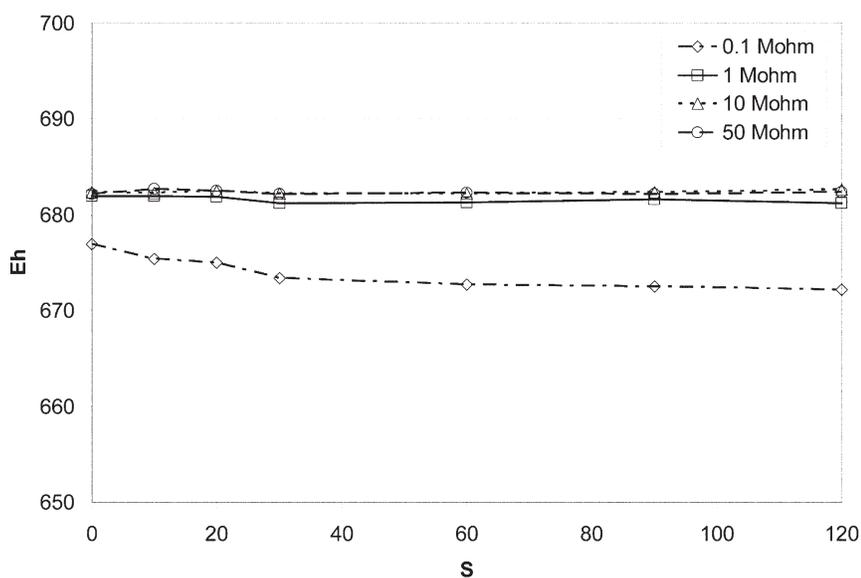
using both a multimeter (10 Mohm input reistance) and using a lab grade pH/Eh meter (200 Gohm input resistance), the voltages measured using the high-resistance instrument showed essentially no drift, and the values were very close to the instantaneous initial measurements recorded by the multimeter before drift began (Fig. 8). The data for replicate electrodes in both Berryland and Indiantown soils showed similar patterns.

When voltages are measured in common electrical systems, the 10-Mohm input resistance incorporated into commercial multimeters is apparently sufficient to obtain reliable measurements. However, when microvoltages are measured in soils using instruments with an input resistance of 10 or even 50 Mohms, the measurements drift over time and then equilibrate at different values depending on the input resistance, indicating that the input resistance is too low to provide accurate measurements without affecting the soil electrochemical environment. Before the circuit is closed to initiate the measurements, the resistance is effectively infinite (an open circuit). Therefore, when the voltages are measured using a low resistance device such as a multimeter the most accurate datum is the very first one observed, since current flow immediately begins to change the electrochemistry of the system being measured. Because these measured values change most rapidly within the first few seconds, the reliability or usefulness of trying to collect these data with multimeters is questionable, suggesting that a high resistance device should be utilized.

In a sense, the operational definition of Eh is dependent on the input resistance, and the act of making the measurement can alter the final voltage "created" by the current flow by influencing the activities of electroactive species in the soil (indeed, this is Heisenberg's Uncertainty Principle in action for electrons in colloidal soil environments). Without a very high input resistance, we are not measuring an intrinsic property of the soil, but are changing it until the electrode and soil come into a new equilibrium, based on the "applied" current. It seems reasonable to assume that the change due to current flow will vary depending on the initial (pre-observation) ratios of oxidants and reductants in the system. This explains the initial negative trend under oxidized conditions (Fig. 2) in comparison with the later positive trend under reduced conditions. The phenomenon that we observed highlights the high degree of variability in electrochemical behavior of soils, in comparison with solutions. It may, in fact, be possible to exploit the observed changes in measured voltages following the application of current to distinguish the active components of the soil redox complex by correlating observed voltage drift with the electrical activation energy associated with

Fig. 6. The Eh of a standard Fe(II)–Fe(III) solution measured using a data logger configured to include resistors of various strength into a circuit that was closed except for momentary periods when the voltage was measured.

the various oxidation–reduction couples present in soils.

A perusal of the literature identifies some workers who have recognized the importance of using instruments with high input resistance. In his early paper, Bohn (1971) suggests that currents as low as 1 uA could induce electrochemical alterations at the electrode site. If one were measuring potentials of approximately 100 mV, then a 1 uA current could be generated if the input resistance was 0.1 MOhms, which as illustrated in Fig. 6 and 7, will likely cause dramatic alteration and significant electrode drift. Bohn (1971) goes on to suggest that instruments used for measuring redox potential should draw "negligible current" which he defines as <1 nA (which would correspond to an input resistance of approximately 100 Mohm). In describing how redox potentials should be measured, Eshel and Banin (2002) recently stated that a "high input-impedance potentiometer" should be used. Perhaps assuming that researchers understood the importance of using high resistance instruments, others, however, have simply prescribed the use of voltmeters, without specifying that they be high resistance instruments (Patrick et al., 1996; Faulkner et al., 1989; Austin and Huddleston, 1999). This has invariably led to the use, by some, of volt meters with lower input resistance.

CONCLUSIONS

This study, using soil mesocosms to minimize soil heterogeneity, identified important effects of instrumentation on measurement of redox potential (Eh). The use of data loggers to record soil Eh (the voltage differential between platinum (Pt) and reference electrodes) will produce accurate and reliable measurements, as long as the configuration preserves an open circuit until the moment when the measurement is made and the input resistance of the data logger is sufficiently high (e.g., the 20-Gohm input resistance of the Campbell 21X data logger is sufficiently high). Adding a 10-Mohm resistor in a closed circuit between the Pt and reference electrodes is effective in causing the data logger to mimic the results provided by a low-resistance multimeter, but this produces erroneous data, presumably by causing electrochemical alterations in the soil environment near the surface of the Pt electrode. When Eh data are collected manually, a standard multimeter with low (10 Mohm) input resistance should not be used, because the voltage measurement itself causes current
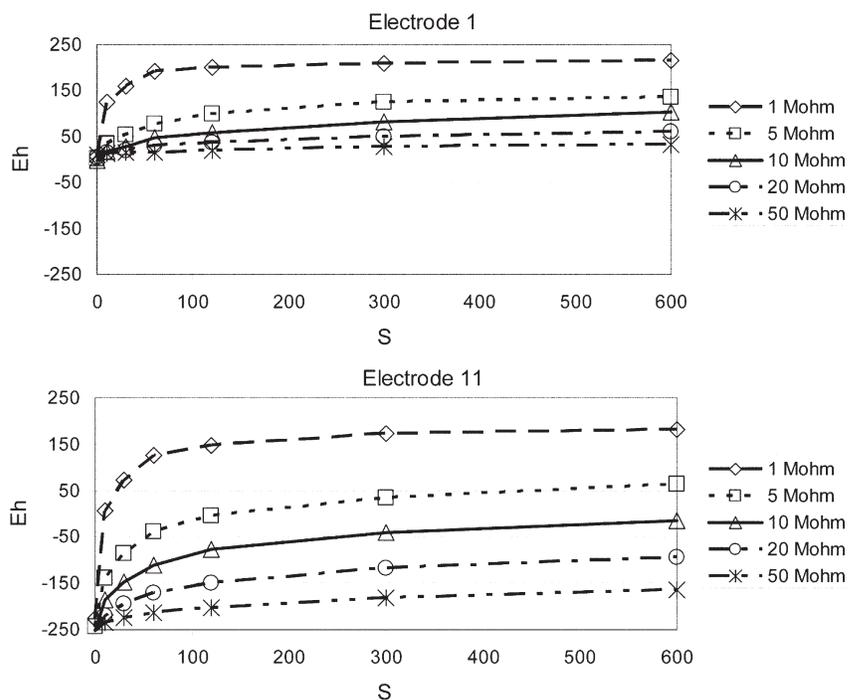

Fig. 7. The Eh measurements in two soils (Electrode 1–Berryland; Electrode 11–Indiantown) using a data logger configured to include resistors of various strength into a circuit that was closed except for momentary periods when the voltage was measured.
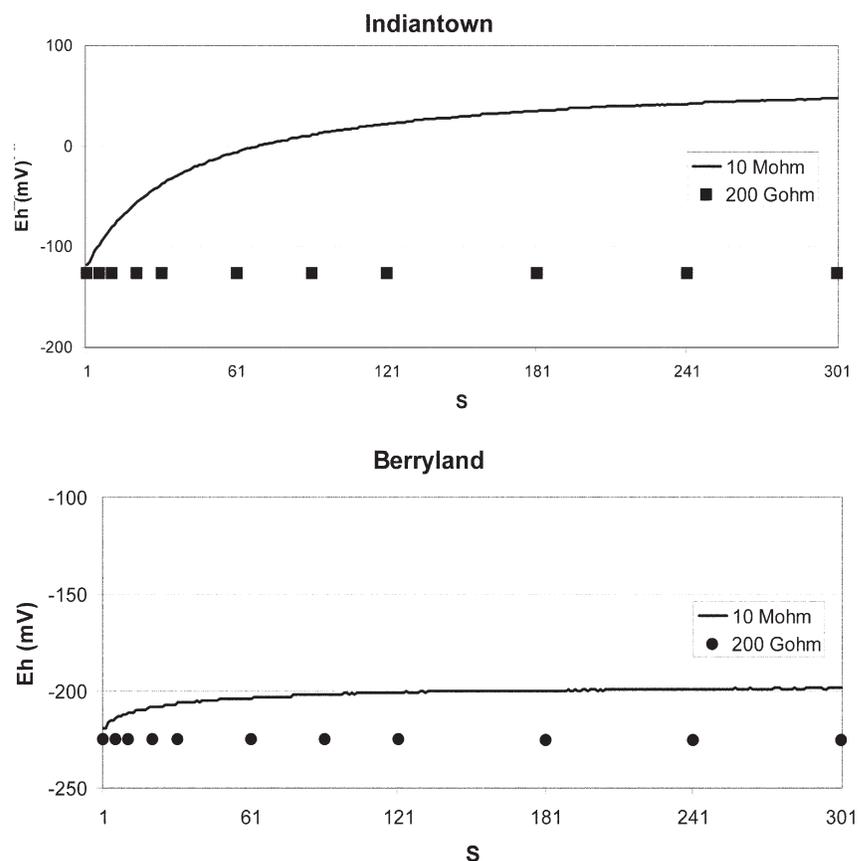

Fig. 8. The Eh measured in Indiantown and Berryland soils using a multimeter (10 Mohm resistance) and a lab grade pH/Eh meter (200 Gohm resistance) using the same Pt and reference electrodes. While this figure depicts the results from a single electrode selected at random, similar trends were observed with replicate electrodes.

flow in the soil, resulting in voltage drift that is unrepresentative of true soil-Eh conditions. Rather, a meter with adequately high input resistance should be used, such as a laboratory grade pH meter (usually of 200 Gohm resistance). Although some early work identified the importance of this matter (Bohn, 1971), its significance has not always been recognized.

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