1.0 Purpose and Scope

This procedure prescribes the Sandia National Laboratories (SNL) Waste Isolation Pilot Plant (WIPP) process for the calibration of dilatometers and a spirometer used to determine the volumetric change of a sample under testing conditions. Dilatometers and the spirometer used by SNL Geomechanic Test Lab are hydraulic and air cylinders respectively that incorporate displacement transducers to indicate the position of the cylinders’ piston. The use of two separate dilatometers and a spirometer is anticipated for hydrostatic compaction testing of simulated waste.

One dilatometer, hereafter referred to as the 22K (Kip) dilatometer, consists of a commercial cylinder with a piston inside. The cylinder is mounted in an MTS test frame and the piston is driven into or out of the cylinder by the frame’s system actuator (Figure 1). The second unit, hereafter referred to as the 220K (Kip) dilatometer, is the primary pressure intensifier for the 220K test frame (Figure 2). An intensifier is an integral pair of hydraulic cylinders, used to produce pressure via the movement of fluid. The moving piston of the 220K dilatometer is instrumented with a displacement transducer. The third unit, referred to as the spirometer, is a cylindrical air chamber with a rolling seal designed to measure air volume (Figure 3). While these three units differ in appearance, operation is similar. The dilatometers will use calibration data consisting of quantity of expelled fluid being compared to a corresponding voltage output from the displacement transducer. The spirometer will use quantity of injected air compared to a corresponding voltage output from the displacement transducer. Notes are made in the calibration instructions (section 2.7) where operational differences exist between the three units.

Dilatometer calibration will require a 50 ml graduated burette and bench balance/ scale (calibrated scale). First, a mass to volume relationship will be established for the test fluid (Isopar H) using the burette and calibrated scale. Next, the filled dilatometer will be operated to incrementally expel fluid into a beaker placed on the calibrated scale. Fluid weight versus displacement transducer output will be recorded. This data will be processed to determine a final sensitivity in milliliters per volt.

This document is not meant to substitute the manufacturer’s instruction manuals recommendations for the dilatometer system being calibrated. The user is responsible for reading and understanding the appropriate manuals for the items being calibrated and the equipment used.

Acronyms and definitions for terms used in this procedure may be found in the Glossary located at the SNL WIPP Online Documents web site.
2.0 Implementation Actions

2.1 Safety

The activities described in this SP shall conform to SNL Environmental Safety and Health Programs (ES&H). All activities described in this SP are subject to ES&H requirements governed by the WIPP Industrial Safety Program and the WIPP Industrial Hygiene Program.

Hazards for this activity consist of moving equipment/pinch-points, potential high pressure and low (24vdc) voltage. These routine hazards are addressed in Operating Procedure for General Laboratory Activities Requiring Mechanical and Thermal Loading, Pressurization and Elevated Voltage.

For more detail on hazards and hazard mitigation for the 22K frame, refer to Data Package for 22 KIP Frame in Building 849 and, specifically, the addendum to the data package detailing the addition of the auxiliary intensifier. Additionally, refer to JSA-22K_2.0 for 22K frame operation.

For details on operation of the 220K frame/intensifier, refer to JSA-220K_2.0, Operating Procedure for the 1MN (220 KIP) Triaxial Testing System.

2.2 Responsibilities

The Principal Investigator (PI) and the Principal User, whose activities warrant the use of this procedure, shall be responsible for implementing the requirements of this procedure.

The Principal User is also responsible for assuring that the test system, calibration standards and meter are in good working order.

The staff member performing the calibration is responsible for following the requirements of this procedure and shall verify that the latest revision of this procedure is being implemented.

If this procedure cannot be worked as written, the user has the responsibility to stop work and resolve all concerns with either the PI, Principal User or QA representative, as appropriate, prior to proceeding with the work.

2.3 Standards

Fluid weight measurements will be measured using a bench balance/scale. Air volume measurements will be measured with a 3.0 liter calibrated syringe. Ambient environmental conditions during calibration will be monitored using a temperature/humidity sensor. Displacement transducer voltage output will be measured using a high resolution digital multi-meter. Scale(s), temperature sensor and multi-meter used during the performance of this procedure shall be calibrated and traceable to NIST through the Sandia Primary Standards laboratory in Albuquerque, NM or an approved supplier. The serial number and expiration date will be recorded in the applicable scientific notebook or scientific notebook supplement (supplement) and the resulting calibration data sheet. The burette and syringe used to determine fluid density and air volume respectively are precision bore units which are certified by the manufacturer; no additional calibration of these items are required. A copy of these certifications shall be maintained in the supplement. No M&TE shall be used in the performance of this procedure if they are past their expiration date without prior approval of the PI or Principal User.
2.4 Frequency

The dilatometer and spirometer shall be calibrated at a frequency consistent with manufacturer’s specifications or based on its performance history. If no baseline exists and the manufacturer has no specification, it will be calibrated annually (on twelve month intervals).

The PI or Principal User may elect to lengthen or shorten the calibration interval based on the results of previous calibrations and the stability of the equipment. Any deviations from the established calibration interval will be justified in the applicable scientific notebook/supplement.

2.5 Acceptance Criteria

The accuracy of the dilatometer and spirometer will be based on the requirements of the PI or Principal User. It is anticipated that the linearity error of each dilatometer and spirometer will be no more than +/- 1.0% of full scale. If the calibration of the dilatometer or spirometer deviates from the PI’s specifications the calibration will be identified as ‘Limited’. All calibrations shall be performed with the ambient temperature between 20-30ºC. The temperature shall also be stable, within +/-2ºC during the calibration. If the temperature fluctuates beyond these values, abort the calibration and repeat when the temperature has stabilized.

2.6 Corrective Action

If the dilatometer or spirometer cannot be brought into tolerance during the performance of this procedure or if the equipment becomes damaged, it shall be tagged and taken out of service until repaired. If during the performance of an As-Found calibration the dilatometer or spirometer is found to be out of tolerance, a Corrective Action Request (CAR) will be issued to document the results and impacts related to the change in the dilatometer performance. Corrective action could include evaluation and adjustment to any data collected by this channel, re-evaluation of the calibration interval, or an assessment of the process in which the instrument is being utilized. Results of all activities related to the out-of-tolerance condition will be summarized in the CAR.

2.7 Dilatometer/Spirometer As-Found Calibration / Verification

The calibration process includes the following general steps: (1) For dilatometer, weigh a known volume of fluid to determine fluid mass to volume relationship, (2) Perform an As-Found Calibration at multiple data points, (3) Calculate calibration uncertainty using the error between the expelled volume (dilatometer) or injected air (spirometer) and the measured output signal from the dilatometer’s or spirometer’s displacement transducer using a best straight line fit routine, (4) Compare the resulting errors with the established acceptance criteria (tolerance) to determine the adequacy of the calibration, and (5) Validate the calibration calculations per NP 9-1.

Note: All dilatometer and spirometer calibrations, including the initial calibration, will be considered as-found. No changes to MTS system calibration settings (other than start point ‘zeroing’) are permitted.

Note: See Figures 1, 2 and 3 for general dilatometer and spirometer configurations.

Note: Any differences in procedure for the two dilatometer and spirometer configurations will be detailed in the applicable step(s) below.
2.7.1 Fluid Density Measurement (Dilatometer Only)

2.7.1.1 Turn on the equipment to be used in the performance of this procedure, allow the equipment to warm-up a minimum of 20 minutes before starting the calibration.

2.7.1.2 Connect the burette to the fluid reservoir of the 220K intensifier using rubber hose.

2.7.1.3 Connect a low-pressure air supply to the reservoir. Building/shop air or regulated bottle air may be used. Set supply pressure at 10 to 20 psig.

2.7.1.4 Open the reservoir valve to slowly fill the burette. Note: the burette has a precision vent tube at the top allowing for an accurate "zero" (start) reading. Observe the burette for bubbles. The burette may be tapped to dislodge bubbles. Additional fluid may be added to assure bubbles have been removed from the burette. Note: Excess fluid will be expelled from the vent tube, retaining a precise "zero" reading.

2.7.1.5 Place a suitably sized cup or beaker on the calibrated scale and tare the scale reading to remove cup/beaker weight.

2.7.1.6 Open the burette valve to slowly drain fluid from burette into the weighing cup. Dispense approximately ~ 45 to 48 ml. Close valve and wait several seconds for any residual fluid to run from burette walls. Carefully reopen the valve to obtain an even increment reading on the burette in the 46 to 50 ml range.

2.7.1.7 Record the dispensed volume and measured weight in the scientific notebook (or supplement).

2.7.2 Dilatometer Calibration Procedure

Warning – 22 KIP dilatometer: Verify that a burst disk of less than 2300 psi is present in the assembly before starting calibration tasks. This will protect system components in the event that the isolation valve is inadvertently left in the closed position during frame operation. Visually inspect the burst disk for dents or damage and replace if such degradation is identified.

2.7.2.1 Turn on the equipment to be used in the performance of this procedure, allow the equipment to warm-up a minimum of 20 minutes before starting the calibration.

2.7.2.2 For 22K Dilatometer - Position the dilatometer assembly in the 22K frame such that the frame actuator is within 1/8-inch of its center-of-travel when the dilatometer piston is also within 1/8-inch of its' center-of-travel. This will permit the full range/capacity of the 22K dilatometer to be used. Electrically zero the frame’s displacement transducer at this time.

Note: No special positioning is required for the 220K dilatometer.

2.7.2.3 Close dilatometer line valve (if open). Open the dilatometer vent/drain valve. Operate the dilatometer to completely empty fluid via the vent/drain. This will also remove any trapped air. Close vent valve.

2.7.2.4 Apply building/shop air supply to reservoir at 40 to 60 psig.

2.7.2.5 Open dilatometer line valve; this will apply pressurized fluid to the dilatometer. Momentarily open vent valve to purge any trapped air from dilatometer or lines. Close vent valve.
2.7.2.6 Operate dilatometer to slowly fill cylinder with fluid. The full stroke should take at least 60 seconds; filling at an excessive rate can introduce bubbles into the system.

2.7.2.7 Again momentarily open vent valve to purge any trapped air from dilatometer or lines. Close vent and line valves. Note: if any air is noted at this step, the dilatometer should be drained and refilled following steps 2.7.2.3 through 2.7.2.7.

2.7.2.8 Place a large cup or beaker (~ 1000 ml capacity) on the calibrated scale.

2.7.2.9 Connect a transparent, small diameter hose (<= 1/8 inch) to the dilatometer output line. Position this hose above the weighing cup/beaker. Secure in place to prevent hose touching the beaker.

2.7.2.10 Momentarily open the dilatometer line valve to fill the dispensing hose. Close valve and check hose for bubbles. Manipulate hose and/or open line valve again to remove trapped air in hose. Close valve.

2.7.2.11 Remove the air pressure supply from reservoir. Open line valve again to vent pressure from dilatometer. A small amount of fluid might be expelled from the hose. Leave line valve open.

2.7.2.12 Determine the desired calibration points for the dilatometer being calibrated. Recommended points/steps are as follows:

- 30 gram increments for the 22K dilatometer (approx 40 ml)
- 15 gram increments for the 220K dilatometer (approx 20 ml)

These points/increments are based on the following: A minimum of ten, approximately evenly spaced points across the dilatometers range/capacity is required. The 22K dilatometer has a capacity of ~ 750 ml. The 220K dilatometer has a capacity of ~310 ml. The Isopar H fluid has a density of approximately 0.75 grams/ml. The above recommended increments will produce approximately 15 calibration points.

2.7.2.13 Empty the cup/beaker if required. Replace cup/beaker on scale. Tare the scale reading to remove cup/beaker weight.

2.7.2.14 Record the initial voltage of displacement transducer and the initial weight.

Note: Weight should be zero at this point. Displacement transducer voltage will be approximately negative 9 to 10 volts dc, as each dilatometer has a voltage output range of -10 volts to +10 volts.

2.7.2.15 Operate the dilatometer to slowly expel the desired weight of fluid into the cup/beaker based on the criteria of step 2.7.2.12. Record the indicated weight and voltage values.

Note: An exact weight increment is not critical; weight increments within +/- 2 grams of target are acceptable.

2.7.2.16 Continue operating the dilatometer in increments, pausing at each desired point to record weigh and voltage data, until approximately 15 calibrations points are recorded.

2.7.2.17 Close all valves. Open drain valve to vent any excess pressure from dilatometer. Shut off power and secure dilatometer system.

2.7.2.18 Record identification information for all instruments used.
2.7.3 Spirometer Calibration Procedure

2.7.3.1 Turn on the equipment to be used in the performance of this procedure and allow the equipment to warm-up a minimum of 20 minutes before starting the calibration.

2.7.3.2 Position the spirometer so that it is fully retracted and position the calibrated syringe so that it is fully extended.

2.7.3.3 Connect the spirometer to the syringe via a flexible plastic tube.

2.7.3.4 Position the LVDT on the spirometer so that it is electrically near one end of its calibrated range. Note that the LVDT may go out of range in the middle of the spirometer stroke. For example, a 2 inch range LVDT will measure the equivalent of approximately 3.5 liters of spirometer volume. If possible, the LVDT should be reset coinciding with resetting of the syringe position. For the aforementioned 2 inch range LVDT, resetting at every 3 liters of spirometer volume and therefore the full stroke of the 3.0 liter syringe would provide sufficient electrical overlap of the LVDT.

2.7.3.5 There is a three way valve on the end of the spirometer. Position the three way valve so that air can transfer from the syringe to the spirometer.

2.7.3.6 Operate the 3.0 liter syringe slowly and transfer air to the spirometer stopping approximately 8 times (approximately every 375 mL) to record syringe position and spirometer LVDT output. The total volume of the spirometer is over 8 liters. Eight data points for every 3 liters will give approximately 21 total data points.

2.7.3.7 When the syringe is fully compressed, position the three way valve on the spirometer so that the spirometer position will remain fixed and the syringe can fully extend. If necessary, reset the position of the LVDT. Turn the three way valve so that the syringe can transfer air to the spirometer. Repeat steps 2.7.3.4 and 2.7.3.5 until the spirometer is fully extended.

2.7.3.8 Disconnect the syringe from the spirometer and shut off power to appropriate devices.

2.7.3.9 Record identification information for all instruments used.

2.7.4 Calibration Calculations

2.7.4.1 Calculate the sensitivity and error of the system using a product like Excel’s Regression Analysis Tool to develop a best straight line fit calculation for the calibration results.

Transfer all manually recorded data to the spreadsheet for processing. Both the fluid density and the dilatometer linearity will be determined in this file. An example processed calibration sheet is shown in Appendix B and C. Calculations in this calibration data will be validated per NP 9-1.

2.8 References

- MTS Flex Test GT - Operator Manual
- Calibrated Scale - Operator Manual
- Operating Procedure for the 0.1MN (22 KIP) MTS Load Frame, OP-6315-22
- Operating Procedure for the 1MN (220 KIP) Triaxial Testing System, OP-6315-220
- Data Package for 22 KIP Frame in Building 849 (with auxiliary intensifier addendum)
- Operating Procedure for General Laboratory Activities Requiring Mechanical and Thermal Loading, Pressurization and Elevated Voltage. OP-6315-GLA
- ES&H100 Corporate Policies
3.0 Records

The following records, generated through implementation of this procedure, shall be prepared and submitted to the WIPP Records Center in accordance with NP 17-1 (Records):

**QA Record**
- Corrective Action Request (CAR), Form NP 16-1, if required
- Scientific Notebook or Scientific Notebook Supplement
- Dilatometer Calibration Data Sheet (See Appendix B)
- Spirometer Calibration Data Sheet (See Appendix C)

4.0 Appendices

Appendix A: General Dilatometer/Spirometer Configurations (Figures 1, 2 and 3)
Appendix B: Example Dilatometer Calibration Data Sheet
Appendix C: Example Spirometer Calibration Data Sheet
Appendix A
General Dilatometer/Spirometer Configurations (Figures 1, 2 and 3)

Figure 1
22 Kip Dilatometer
Figure 2
220 Kip Dilatometer
Figure 3
Spirometer
**Appendix B**

**Example Dilatometer Calibration Data Sheet**

### Calibration Data

**1000 ml Dilatometer**

For: Geomech Lab 6117  
Date: 6/3/2008  
s/n MTS 30k-4432  
By: DRB  

**Comments:** System uses LVDT s/n 3477 paired with conditioner s/n 807. Weight and analog output signals manually recorded.

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<th>MTS settings</th>
<th>Range</th>
<th>Total Gain</th>
<th>Exc volts</th>
<th>Phase</th>
<th>Temp</th>
<th>Humidity</th>
</tr>
</thead>
<tbody>
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<td>+/- 3.004 in</td>
<td>2.004</td>
<td>9.000</td>
<td>90.2</td>
<td></td>
<td>22.6 C</td>
<td>22%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Measured Volume -ml</th>
<th>Measured Weight - gm</th>
<th>Calculated Density gm/ml</th>
<th>Sensitivity: 49.257 ml / volt</th>
<th>Offset: -9.810 volt</th>
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</thead>
<tbody>
<tr>
<td>50</td>
<td>37.910</td>
<td>0.7582</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>measured weight - gm</th>
<th>measured output -vdc</th>
<th>calculated volume - ml</th>
<th>calculated output - ml</th>
<th>Deviation: % reading</th>
<th>Deviation: % full scale</th>
</tr>
</thead>
<tbody>
<tr>
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<td>-9.792</td>
<td>0.00</td>
<td>0.90</td>
<td>#DIV/0!</td>
<td>0.07</td>
</tr>
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<td>75.21</td>
<td>-8.647</td>
<td>57.02</td>
<td>57.30</td>
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<td>0.02</td>
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<td>114.00</td>
<td>114.79</td>
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<td>0.06</td>
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<td>-0.03</td>
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<td>675.66</td>
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<td>962.98</td>
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<td>0.10</td>
</tr>
</tbody>
</table>

**RESULTS:** PASS; meets linearity reqmt of +/- 1% of full scale reading.

**Standards used:**  
- Scale, Mettler PM 4600, s/n 123009, exp 2/30/09  
- Multi-meter, Keithley 2000, s/n 0691969, exp 5/10/09  
- Burette, 50 ml, s/n 345678, exp na  
- Temp/Humidity, Hydroclip, s/n 60189152, exp 9/10/08
## Appendix C

### Example Spirometer Calibration Data Sheet

#### Calibration Data

**8000 ml Spirometer**

<table>
<thead>
<tr>
<th>For: Geomech Lab 6914</th>
<th>Date: 9/3/2014</th>
</tr>
</thead>
<tbody>
<tr>
<td>s/n 000340</td>
<td>By: STB</td>
</tr>
</tbody>
</table>

Comments: Spirometer uses LVDT s/n 706. Calibration syringe is 3 liters.

### Conditioner settings

<table>
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<tr>
<th>Range</th>
<th>Total Gain</th>
<th>Exc volts</th>
<th>Exc. Freg</th>
<th>Temp</th>
<th>Humidity</th>
</tr>
</thead>
<tbody>
<tr>
<td>+/- 1 in</td>
<td>0.77 mV/V/0.001”</td>
<td>3.0</td>
<td>10k</td>
<td>22.6 C</td>
<td>22%</td>
</tr>
</tbody>
</table>

Sensitivity: **600.00** ml / volt

Offset: -5.000 volt

### Standard measured Calculated

<table>
<thead>
<tr>
<th>Standard Output - mL</th>
<th>measured output -vdc</th>
<th>Calculated Output – mL</th>
<th>Deviation: % reading</th>
<th>Deviation: % full scale</th>
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</thead>
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<td>6000</td>
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<td>0.10</td>
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</table>

**RESULTS:** PASS; meets linearity reqmt of +/- 1% of full scale reading.

**standards used:**

- Multi-meter, Keithley 2000, s/n 0691969 exp 5/10/15
- 3 Liter syringe, 3000 ml, series 5530 exp na
- Temp/Humidity, Hydroclip, s/n 60189152 exp 9/10/15
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