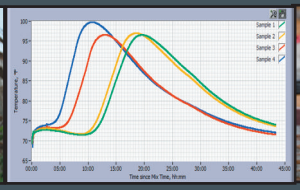
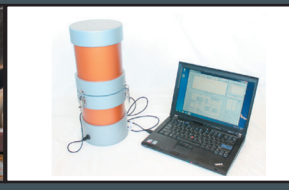
calmetrix

P-CAL 1000

User Manual





CALMETRIX INC.

P-CAL 1000 PORTABLE HIGH PERFORMANCE CALORIMETER FOR CONCRETE AND CEMENT

USER MANUAL

IMPORTANT SAFETY RULES

CAUTION: Always make sure to hold your P-Cal firmly with both hands when moving it, especially when it contains a sample, as it could fall on your feet and cause an injury.

DISCLAIMER

Calmetrix does not assume any liability as related, but not limited to, the accuracy, relevance or interpretation of results while using P-Cal.

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TABLE OF CONTENTS

A. BEFORE YOU START USING THE EQUIPMENT

What is in your box?

1. P-Cal 1000 equipment
2. USB Cable to connect the P-Cal to your computer's USB port
3. Quick Set-up guide
4. USB Memory Stick with Software and Manuals

CalCommander Software Installation.

1. Insert the USB Memory Stick in your compuetr's USB port and browse to the Software folder. Look up and double-click on the CalCommSetUp.exe file and follow the on-screen installation instructions
2. Restart your Computer
3. Connect the P-Cal to your computer using the USB cable that is provided with the equipment. When connecting your P-Cal for the first time your Windows Operating System will automatically install the necessary drivers. Click on the CalCommander Icon on your Desktop to open CalCommander.
4. Proceed to the Settings tab to find the Module ID numbers for software registration.

NOTE: Your P-Cal includes one license for each of the following software modules:

- F/P-Cal Logger to operate the equipment
- F/P-Cal Reports to retrieve past results and create reports
- F/P-Cal Set to infer thermal indicators of setting times
- P-Cal Strength to calibrate and predict strength for a given mix design

Register your software by clicking on the Registration link in the Support Section at http://www.calmetrix.com. Fill out the form or email your Module ID numbers to registration@calmetrix.com. We will email you your Registration numbers.

5. You can find videos, software updates and registration, user manuals and other support items in the Support Section on our website at http://www.calmetrix.com

B. GENERAL USE

P-Cal contains state of the art high efficiency insulation, enabling quasi-adiabatic conditions. The equipment can be used with cement paste, mortar of real concrete (including coarse aggregates). Please refer to Section F in this manual for recommendations of use with paste and mortar samples. The temperature curve measured by P-Cal provides a “fingerprint” of the chemical reactions in the sample that can be analyzed to infer specific reactions during early stages of hydration, usually 24 to 36 hours after batching/mixing. Because of the very limited heat loss, P-Cal can be used for strength prediction based on the maturity concept.

P-Cal can be used with a single sample container of 4”x8” (100 mm x 200mm) maximum size. Smaller containers can be used, but it is important to choose a standard container type and use the same for all tests, so as to be able to compare results from different test runs.

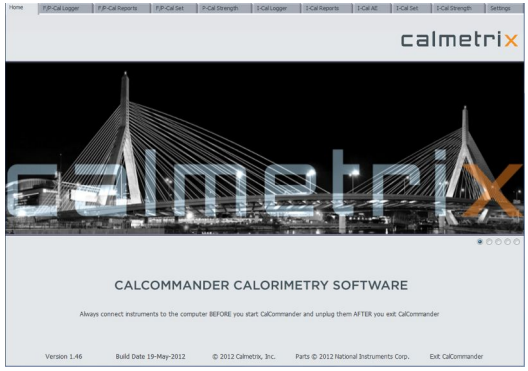
C. HANDLING INSTRUCTIONS

Sample containers must be clean on the outside before inserting them into the P-Cal. Cement or admixtures on the outside will cause the sample container to get stuck in the calorimeter, with expensive and time-consuming repair as a result. All containers must be closed tightly to avoid accidental spillage.

AVOID transporting the P-Cal with samples inside, as equipment might be damaged. If you have to move or transport the unit during a test with a sample loaded inside the unit, make sure to keep the P-Cal upright in the same position during the entire duration of the test, for consistency in thermal insulation.

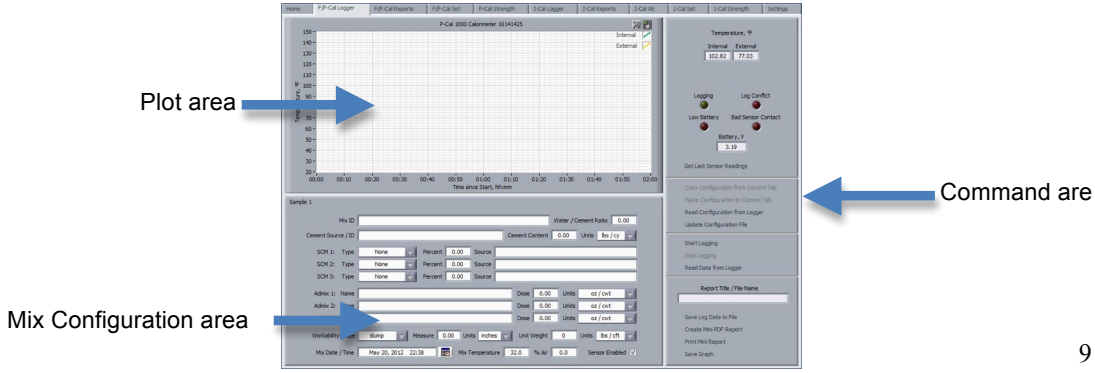
D. OPERATING THE EQUIPMENT – CALCOMMANDER SOFTWARE

Connect the P-Cal to your computer using the USB cable (provided). Open the CalCommander software. You will see the Welcome Screen.



Click on the F/P-Cal Logger tab on top.

Familiarize yourself with the main window of the P-Cal Logger software module:

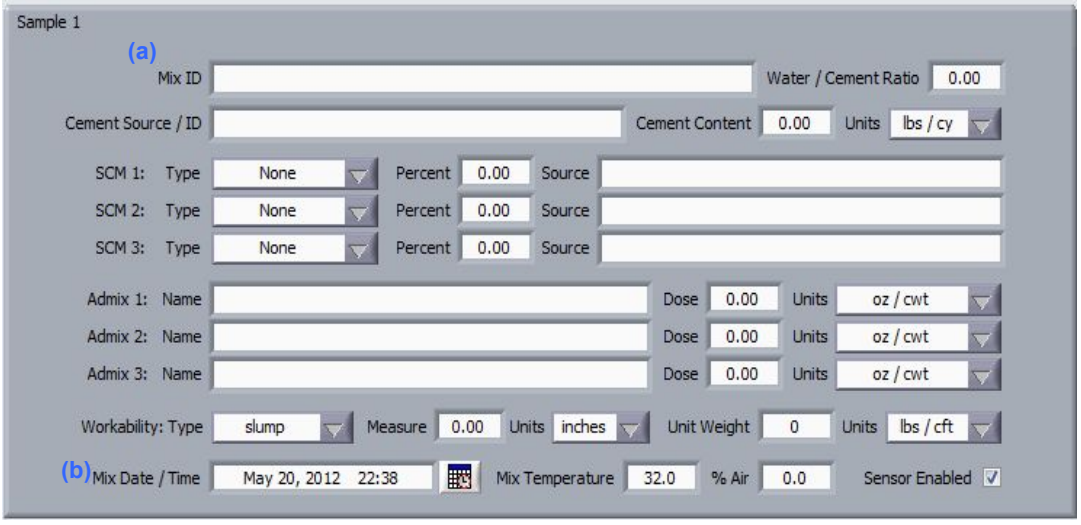


Mix Configuration area:

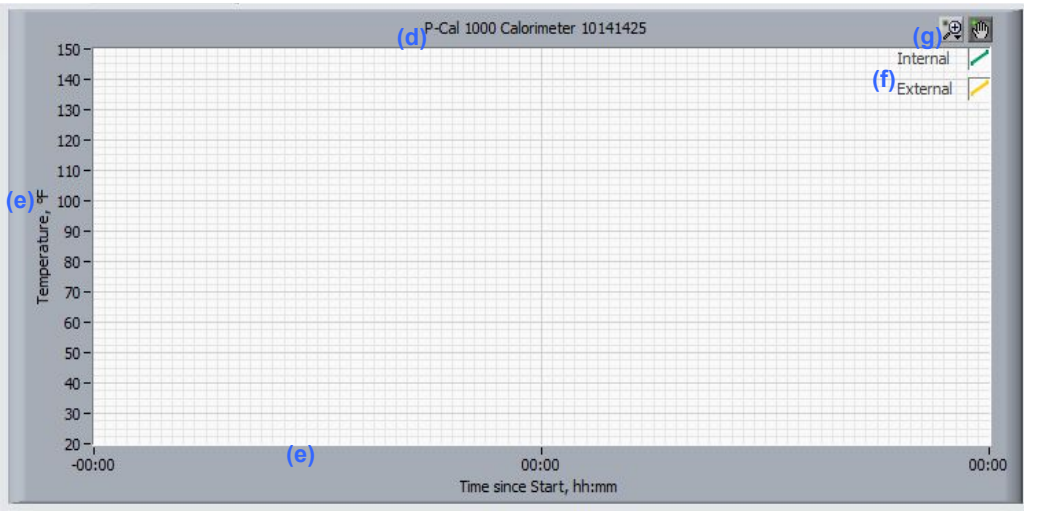
This part of the screen is used to capture all mix and sample related data.

(a) Enter the Mix data for each sample in the corresponding fields. This data will be stored along with the sample test data and will appear when you visualize curves in the F/P-Cal Reports or other software modules.

(b) Make sure you enter the mixing time (when the water meets the cement) in the "Mix Date/Time" field. This time will be used as the point of origin so as to make side-by-side comparisons of curves from tests performed at different times.



Plot area.



(d) The type and serial number of the equipment currently connected is displayed on top of the chart area.

(e) Right-click on the x or y-axis to change scaling from Automatic to Manual. In Manual mode, you can adjust the min and max using the cursor.

(f) Click on a graph legend/color on the right of the screen to change the appearance (color, line type, etc.)

(g) Click on the Zoom and Motion tools on top (f) to zoom in/out or move the graph.

Command area.

This area is used to operate the P-Cal's logging functions

Displays current temperature as measured both inside the P-Cal and for the external sensor located underneath the instrument (used for Compressive Strength estomates). To change units from °F to °C, click on the SETTINGS tab on top, select the Measurement Units field. Click on « Apply New Settings » on the bottom of the SETTINGS tab to validate your changes.

The Logging light turns green when your P-Cal is logging data.
Log Conflict lights up when more than one P-Cal is connected to your computer and the software is unable to determine proper start times for each equipment.
Low Battery will light up when the battery needs to be replaced. Refer to Section H – Troubleshooting.
Bad Sensor Contact indicates a bad connection between the sensor and the logger located within your P-Cal equipment. Refer to Section H in this User Manual to troubleshoot bad connections.
Click on "Get Latest Sensor Readings" to update the temperature and battery fileds.

Copy/Paste is not accessible on single channel instruments like P-Cal 1000.
When disconnecting your P-Cal, your configuration data will not appear automatically upon reconnection. Click on "Read Configuration from Logger" to display Configuration data.
Click on "Update Configuration Data" to save changes made to the Configuration Data after logging has started.

Click on "Start Logging" to begin logging temperatures . The sensor in your P-Cal will start logging. Note: it is not necessary to keep your computer connected after logging has been activated. All data will be stored in the P-Cal's internal memory. Reconnect your computer at a later time to retrieve data.
"Stop Logging" at the end of your test. After "Stop Logging" has been selected, you cannot continue logging data on your current test. Data will be saved automatically in your default Logs folder.
"Read Data from Logger" to update the graph at any time during testing.

Create or Print PDF reports, or Save the current graph as an image file.

E. OPERATING YOUR UNIT FOR THE FIRST TIME

Calibration and Connections.

Your P-Cal uses thermistors for temperature measurements, and does not require any re-calibration over time.

To verify that internal connections have not been compromised during shipping, a test run should be conducted by loading the unit with a sample of hot water. Verify that the sensor is logging the correct water temperature. After thirty minutes, the temperature reading should stabilize and should be within 2°F of the actual water température measured before inserting the sample into the calorimeter

Check for error signals – LOW BATTERY or BAD SENSOR CONTACT - on the upper right hand side of the P-Cal Logger screen. Refer to Section J Troubleshooting to address any issues.

F. SAMPLE PREPARATION AND RECOMMENDED TEST PROCEDURE

IMPORTANT NOTE: Testing should be conducted with a standard container of your choice. You can use standard 4”x8” (100 mm x 200 mm) cylinders, or any type of container of any size that fits in the instrument, made of glass, metal or plastic and that can be tightly closed. However, it is important to use the same container for all your testing if you want to be able to compare data from tests conducted at different times.

1. Ensure that the mixing procedure is sufficient to disperse binder properly.

2. If the water/cement (w/c) ratio is pre-defined, perform a pre-test mix with blank cement paste or mortar (no admixtures) and another pre-test with the highest desired dosage of admixtures. Adjust w/c ratio if necessary.

3. If the water demand of the cement – admixture combination to be tested is unknown, perform a pre-test to determine required water to binder ratio (binder includes cement and any supplementary material used as a cement replacement)

Preparation

4. We recommend you start logging data in your P-Cal at least one hour before inserting the first sample. This allows the unit to reach a stable baseline temperature.

5. Pre-dilute admixtures to ensure accurate weighing or use high accuracy micro-pipettes for accurate volumetric dosing.

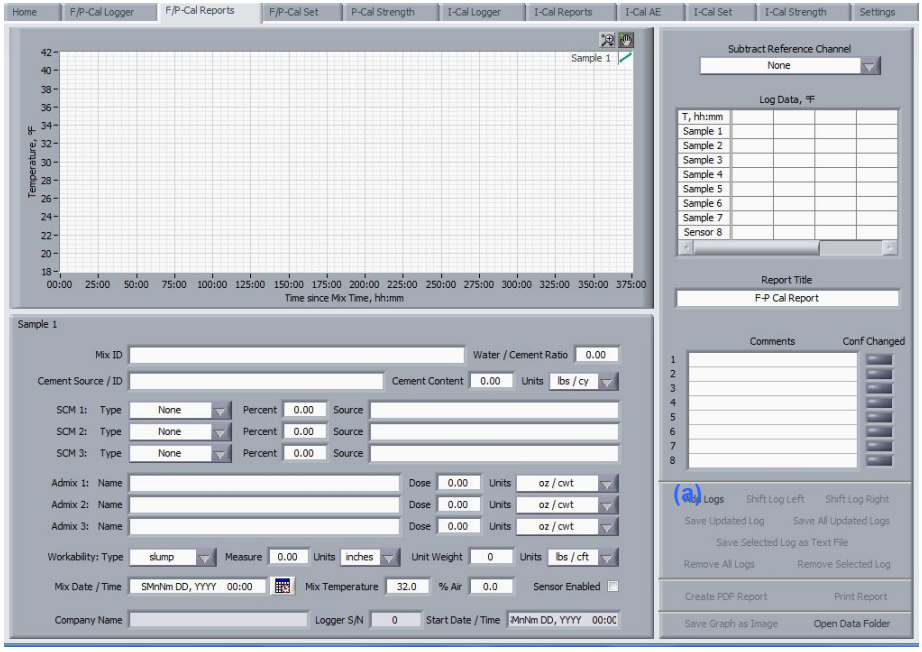
6. Use the same mixing sequences and times as far as possible within a test series

7. Prepare concrete according to standard or sample concrete directly from production (in batch plant mixer or ready mix truck). Be sure to enter mix data in the Mix Configuration area of the P-Cal Logger tab for future reference.

8. Fill your 100mm x 200mm or other standard sample container completely. Make sure the container is properly closed to avoid any spillage. Insert the container into the P-Cal and close it by securing the three latches.

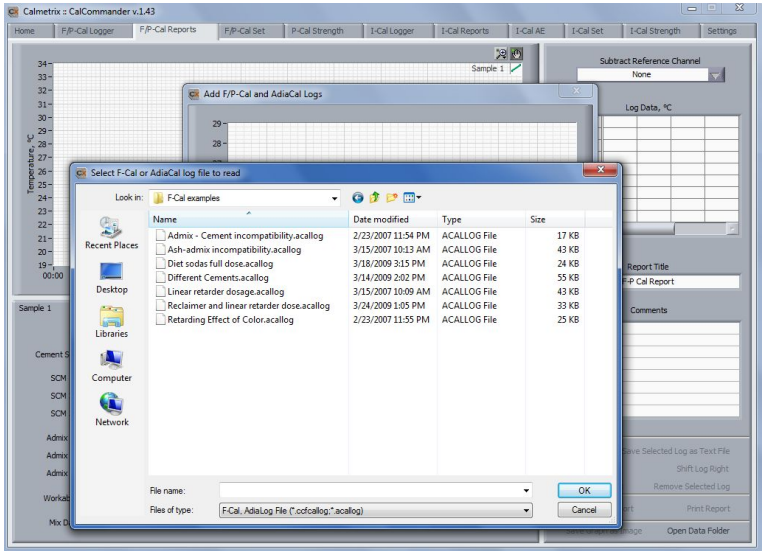
G. VISUALIZING RESULTS – F/P-CAL REPORT GENERATOR

With CalCommander open on your computer, click on the F/P-Cal Reports Tab on top. A new window will appear, as in the picture below.



(a) - Add Logs to plot – select C:\Users\Public\Documents\Calmetrix\CalCommander\Logs (default storage of completed log files) or any other location where you store completed log files. Select the log file you would like to display

A new window will appear.



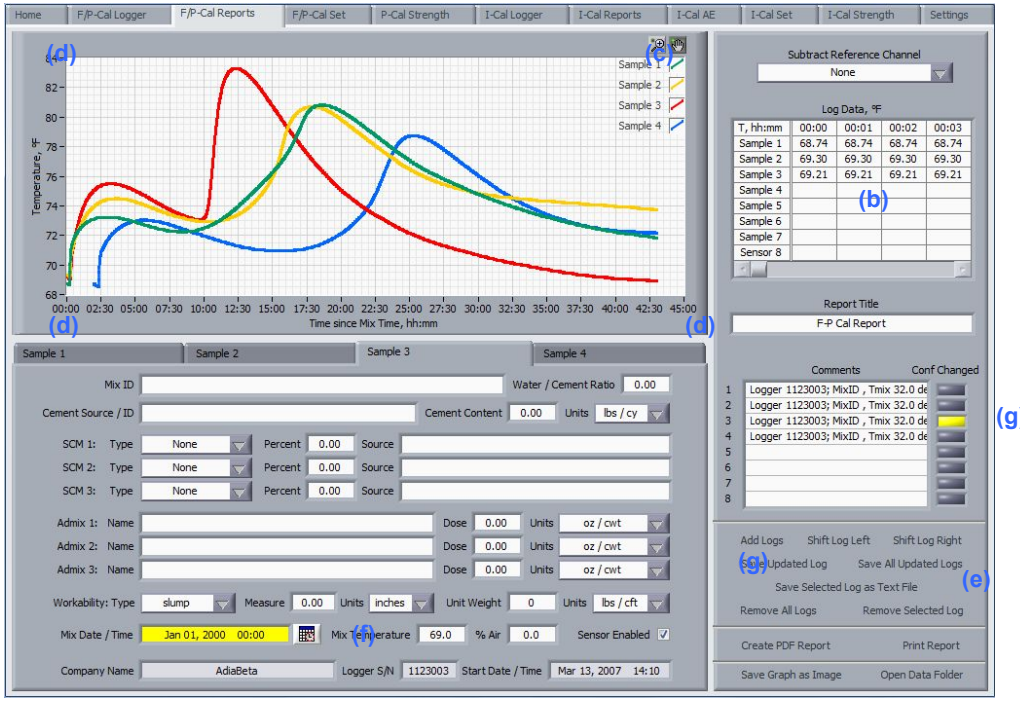
Select the log file you would like to load and click OK.

Repeat the steps above to add more files from other log files. You can add up to 8 files on any Report Generator graph.

Click Accept and the files you selected will appear in the Report Generator window.

The F/P-Cal Report Generator can read files that were generated by the following equipment: Calmetrix P-Cal 1000, F-Cal 4000, F-Cal 8000 and Grace AdiaCal™.

Once you selected and loaded all the curves you would like to visualize, the following window will show.



(b) - Actual log data is displayed in this area. Temperatures are measured and logged every four minutes.
(c) - Use Graphing tools to Zoom In/Out, click on the legend to change colors or style of graphs.

(d) - Click on the min/max values on the x or y-axis to change the min and max to modify the range and scale.
(e) - “Save Selected Log as Text File” saves the information, including all temperature values from the currently selected Sample tab in a .txt file. Data from .txt files can easily be copied and pasted in an application such as Microsoft Excel for spreadsheet calculations on plotted data.
(f) If the Mix Time or Mix Temperature fields are highlighted in yellow, you should verify that the data captured in these fields is correct.
(g) If you edit data in any field, a yellow light will appear until you permanently save the change by clicking on “Save Updated Log”

H. INFERRING SETTING TIMES – F/P-CAL SET

F/P-Cal Set has two ways to infer setting tines:

Method 1: Post-testing analysis of calorimetric curves to infer setting times in lieu of physical testing such as with a penetrometer needle. This feature is useful if you have multiple samples in a large test matrix and need approximations of setting times without incurring the cist and time for physical testing.

Method 2: Live analysis while conducting a calorimetry test to infer setting times, or saw cut times and get notified when a mix reaches its initial of final setting time or saw cut time.This method can be applied to get an indicator of when to start performing finishing work

How to use Method 1 (Post-testing analysis).

F/P-Cal Set can help you infer initial and final setting times within close approximation by applying the "Fractions Method" and generate Thermal Indicators of Set (TIS). This method is based on the assumption that for a given mix design there is a correlation between the heat evolution and setting times. Initial set is reached when the temperature level in a sample placed in the P-Cal reaches a certain fraction of the peak. Each mix has its very own fraction values corresponding to initial and final set. Once the fraction values are determined for a given mix design, they can be used to predict setting times when the same mix is used again in the future.

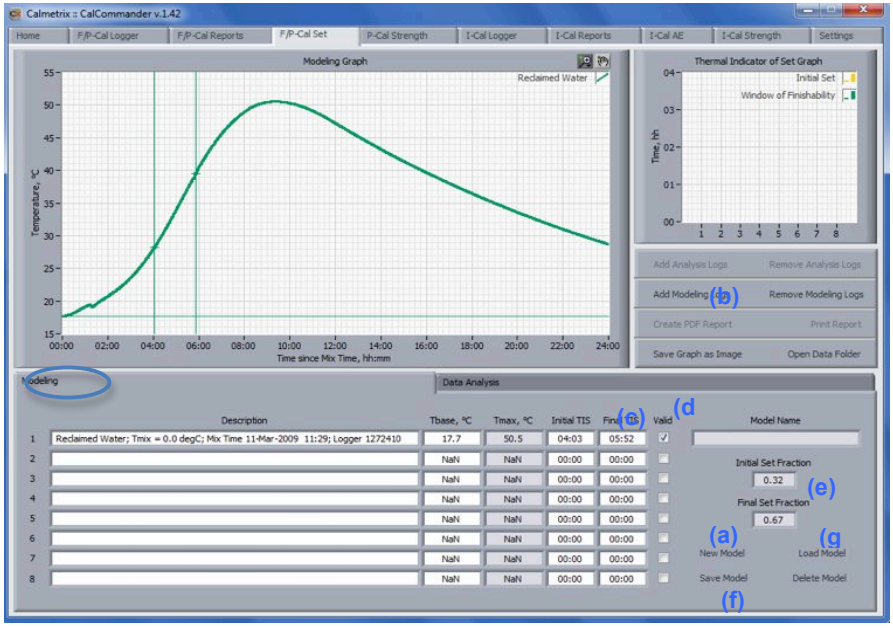
To be able to predict setting times for a given mix design, you will first have to create a model which establishes the relationship between the temperature curve and initial and final set, as measured by a standard method, for example ASTM C403.

<u>First, build a model</u>

To build a new Model for a given mixture, proceed as follows:

1. Go to F/P-Cal Set and select the "Modeling" tab. Click on New Model (a)

2. Click on “Add Modeling Log” (b). A new window will pop up, letting you browse to the file containing the calorimetry curve for the mix for which you would like to build a model. You can load up to 8 curves for the same mix.
3. Select one or several curves for this mix and click on “Accept”. The selected curve(s) will appear on the screen, as shown below.
4. For each curve, enter the measured Initial Setting time in the Initial TIS field and the Final Setting time in the Final TIS field (c). These values should be measured using the standard you normally use to determine setting times, e.g. ASTM C403. Now select the curves that you want to use to build your Fractions Model for this mix by checking or un-checking the “Valid” box (d). The average fraction values of all “Valid” mixes will display in the Initial Set Fraction and Final Set Fraction fields to the right (e).
5. Save your model by clicking on “Save” (f). You will be asked to provide a name for your model. Once saved, your model will be stored in the Calibration folder and can later be selected when predicting setting times for the same mix design using the Data Analysis tab.

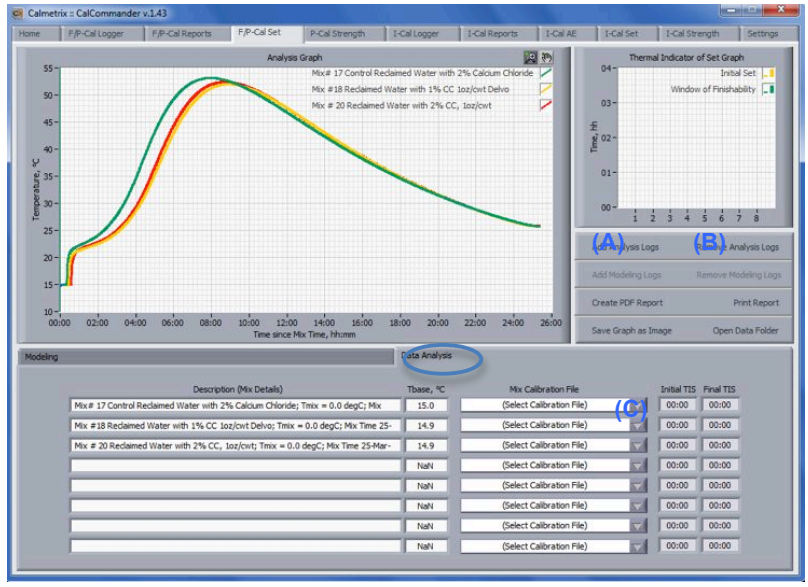


6. You can later open and edit an existing model by clicking on Load Model (g)

Now you're ready to use the setting time prediction tool

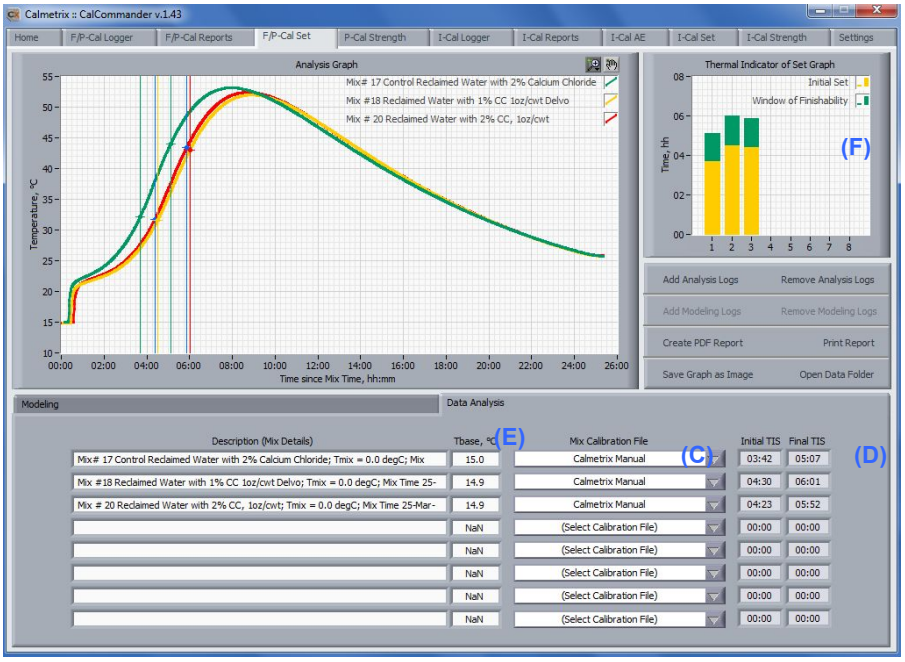
Once a model has been established for a given mixture, it can be used to predict early and late times of set of the same mixture by using only a calorimetry curve. To use the prediction tool, proceed as follows:

1. Click on the "Data Analysis" tab.
2. Click on "Add Analysis Logs" (A) to select the calorimetry data for which you would like to make a setting time prediction and click OK. You can add up to 8 calorimetry curves.



3. You can remove one or several logs by clicking on Remove Analysis Logs (B).
4. Once you have loaded all curves you would like to analyze, select the calibration field to use for each by clicking on the drop-down menu in (C).

5. The software will calculate and display the Thermal Indicators of Set (D).

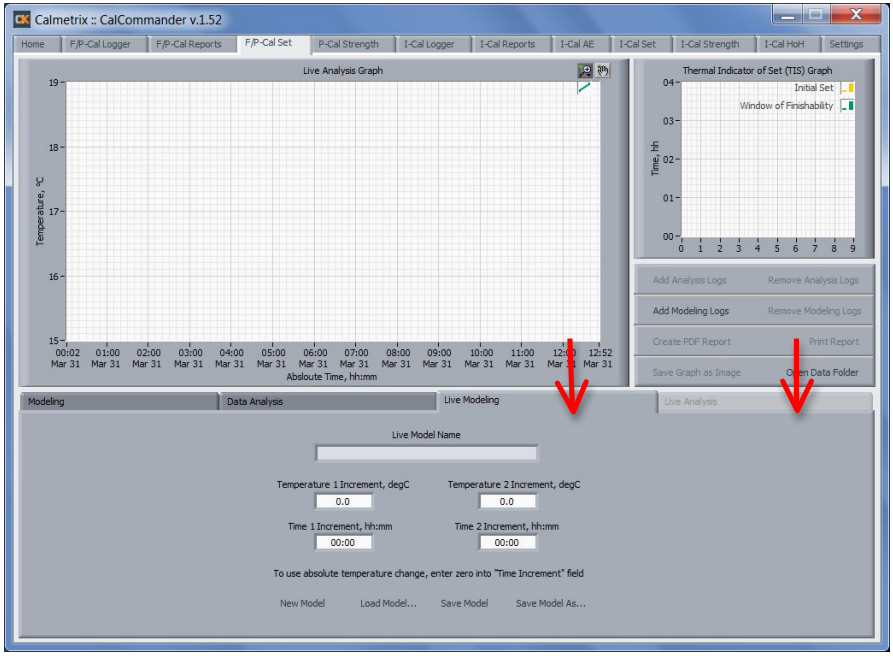


6. Tbase (E) is the temperature in the mix at the beginning of hydration. F/P-Cal Set pre-fills this field with the initial temperature when the test was started. You can modify this temperature manually, e.g. if you know the real mix temperature, to fine tune.
7. The upper right hand side graph (F) displays the calculated initial set, final set and the Finishability time window.

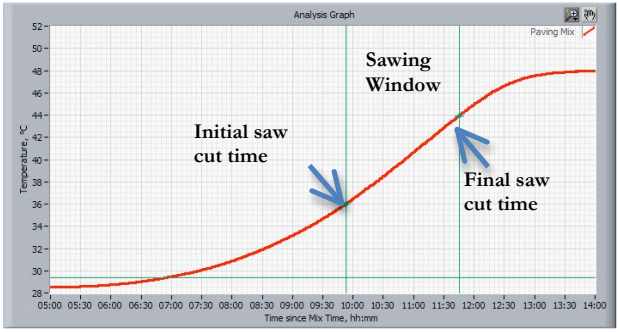
How to use Method 2 (Live Analysis).

This method is often used to determine saw cutting times, but it can also be applied to the live determination of Setting times. Just like in Method 1, you will have to create a model for a given mix to link its saw cut times or setting times measured in physical testing to its calorimetric curves.

To use Method 2, you will use the "Live Modeling" and "Live Analysis" in the F/P-Cal Set software tab. The Live Analysis tab can only be used when the calorimeter is connected to the computer and is logging.

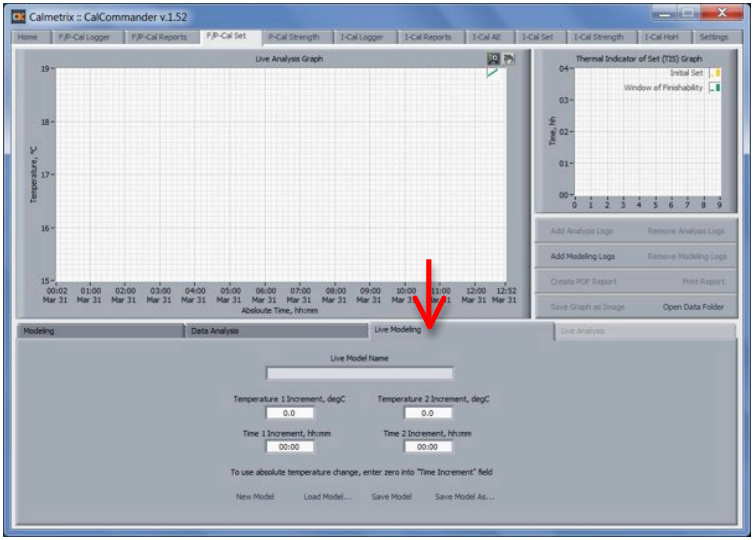


Each mix has specific hydration characteristics that influence the initial and final saw curing times, as per the curve below.



In order to adjust to these mix-specific characteristics, users can create a simple model for each mix and store it in a database so that the saved model can be called up every time the same mix is used.

To create a model, proceed to the Live Modeling Section.



1. Click on New Model to create a model for a new mix, or click on Load Model to modify an existing model.

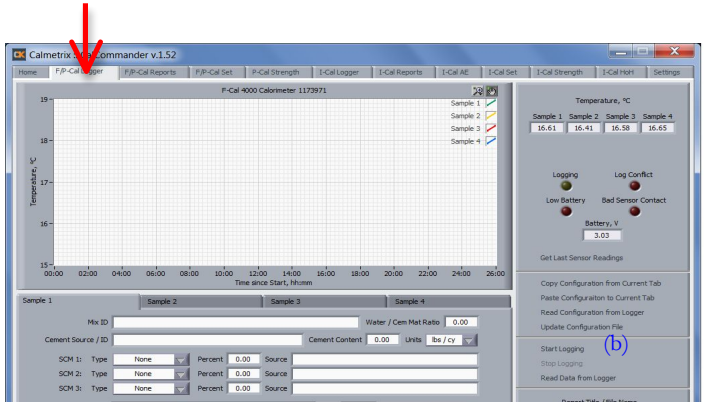
2. Input parameters for the initial saw cut time in section A. The parameters are defined as the minimum temperature increment (A1) in a given time period (A2). For example, if you input 1.5°C in A1 and 00:10 (10 minutes) in A2, initial saw cut is reached at the time t defined by the first instance when:

   $T_t - T_{t-10} \geq 1.5°C$, where T_t is the temperature reading at time t

   If you input "0" in the Time Increment field, the model is such that the initial saw cut time is reached as soon as the temperature increment from the start of the experiment reaches the value specified in A1.

3. Proceed as above for the final saw cut time parameters in section B. When finished, click on Save Model or Save Model As… to store your model in the database for future use.

<u>Now you can use your calorimeter for live determination of saw cutting times</u>

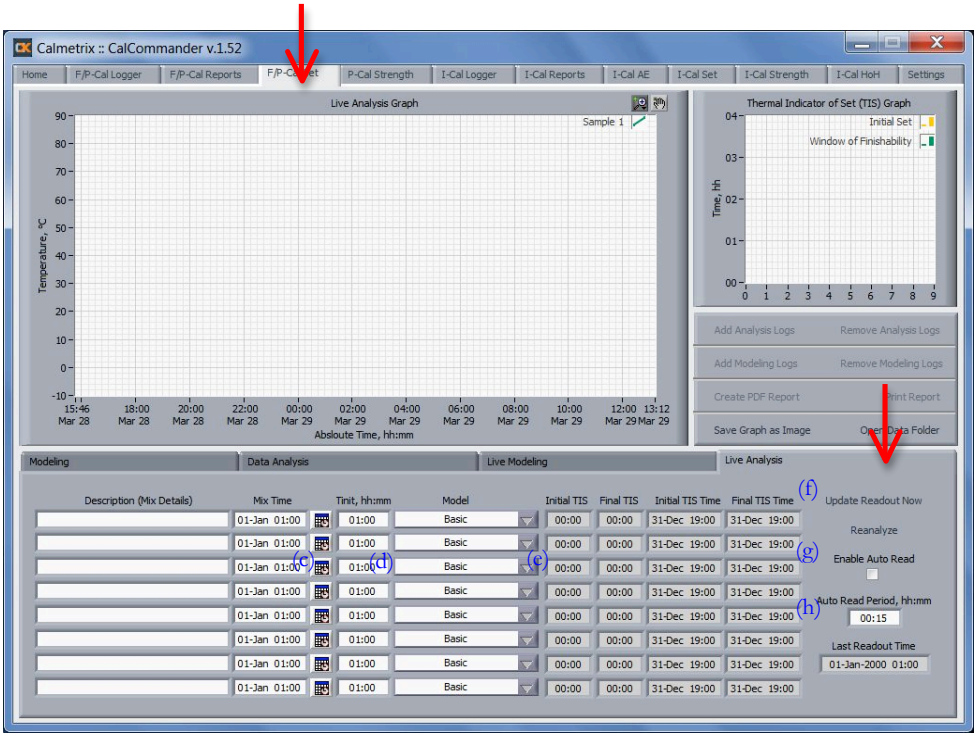


1. Before the concrete mix is prepared, connect your calorimeter to the computer (wired USB connection).

2. Open CalCommander on the computer and proceed to the F/P-Cal Logger tab. Click on Start Logging (b).

3. When preparing a mix, take a sample in the sample container you are usually using with your calorimeter. Write down the mix time and introduce the capped cylinder in your F-Cal.

4. Go to the F/P-Cal Set tab and proceed to the Live Analysis section.



5. Introduce the Mix Time in (c) and the approximate time the equipment takes to stabilize before actual hydration measurements begin in (d). This is the time it takes to absorb any temperature shocks coming from the difference in temperature of the materials used in the mix, and the temperature in the calorimeter before starting the experiment. Unless conditions are extreme (very hot materials or very cold outside temperature), the default value of 1 hour should be a good estimate.

6. Choose the model your would like to use in (e). Please note that the software comes with a Base Model, which can be used until a specific model has been determined for each mix.

   Click on Update Readout Now (f) to initiate the live reading.

   Check the Enable Auto Read box (g) for the software to retrieve data automatically. Input the desired Auto-Read time period in (h).

   Click on Reanalyze to recalculate data if you switch to a different model during the experiment.

7. The software will automatically display the Initial and Final saw cutting times in the Initial TIS (Time of Saw or Time of Set) and Final TIS fields when reaching the temperature increments as defined in the model.

I. PREDICTING COMPRESSIVE STRENGTH – P-CAL STRENGTH

P-Cal 1000 comes with P-Cal Strength, CalCommander's software module for strength prediction.

P-Cal Strength can be used to infer compressive strength for multiple curing ages or to predict future compressive strength gain from 4-5 physical test data points collected within the first 96 hours of testing.

Users will have to perform physical strength testing at 5 or more curing ages, and can then use a calorimetry curve to estimate compressive strength at additional curing ages.

To be able to estimate compressive strength for a given mix, you will first need to:

- Generate compressive strength data from physical tests, and a calorimetry curve (see Data Generation step-by-step below)
- Create the correlation model between compressive strength and degree of hydration (see Modeling step-by-step below)

Data Generation:

Step 1: Place your P-Cal unit(s) close to your mixing station, and connect the first one to a computer with the CalCommander software installed, then start CalCommander and select the "F/P Cal Logger" tab.

Step 2: Type in all relevant mix information [see section D – Operating your Equipment in this manual for details]

Step 3: Mix concrete and prepare samples for compressive strength testing a minimum of two samples per age at a minimum of four to five ages.

Step 4: Make sure you entered the Mix Time in the corresponding fields in the F/P Cal-Logger tab and click on "Start Logging", then close CalCommander and disconnect the USB cable from the computer.

Step 5: Unfold and uncoil the external temperature probes (see fig. A) from underneath each of the P-Cal's bottom and place them in the vicinity of the samples for compressive strength testing, protected from direct sunshine and wind.

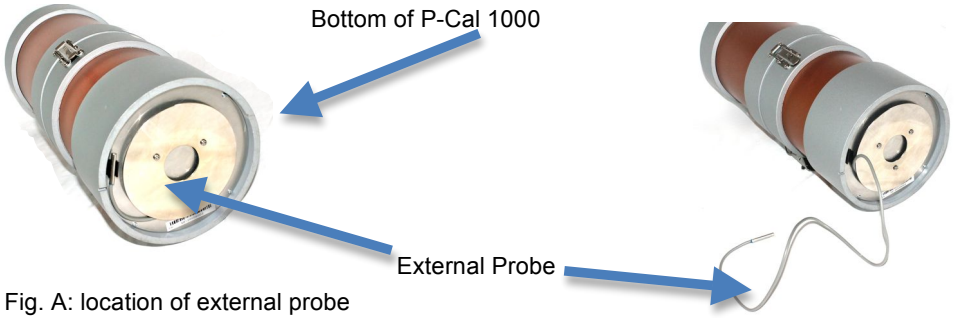


Fig. A: location of external probe

If compressive strength samples are customarily moved to a curing tank / fog room, insert the external temperature probe into the curing tank or fog room. The external probe should at all time measure the temperature of the curing location.

IMPORTANT NOTE: Do not submerge the P-Cal unit (other than the external probe) in water, and do not place the P-Cal (other than the external probe) in a fog room, as the equipment may suffer irreparable damage.

Step 6: Measure setting time of concrete or optionally use F/P-Cal Set to estimate the initial setting time of the concrete tested for compressive strength.

Step 7: Test the samples prepared for compressive strength measurement, which are in the curing bath, the fog room or curing at ambient temperature, and make sure to record the curing time for each measurement (for example measure compressive strength at 16 h, 1 day, 2 days, 4 days, 7 days, 28 days).

Modeling and Strength Prediction

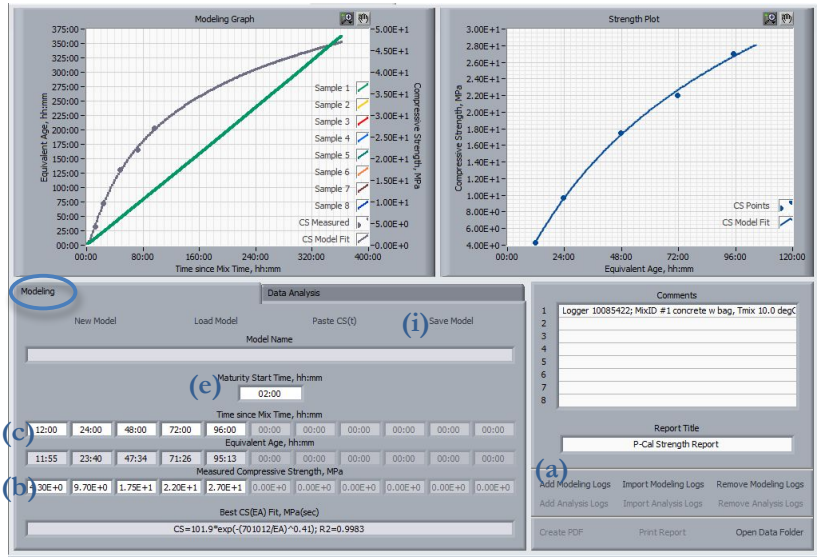
In this section, you will build a model that correlates degree of hydration as measured by the calorimetry curve to compressive strength development.

IMPORTANT NOTE: We recommend that you have at least 96 hours of calorimetry data, and at least four physical measurement of compressive strength before building a model.

Step 1: Connect the computer to your P-Cal unit. Start CalCommander and select the “F/P-Cal Logger” tab. Click on “Read data from logger” and wait for the data to load on the screen. When finished, click on “Save log data to file” and save to your location of choice. click on “P-Cal Strength” and select the “Modeling” tab.

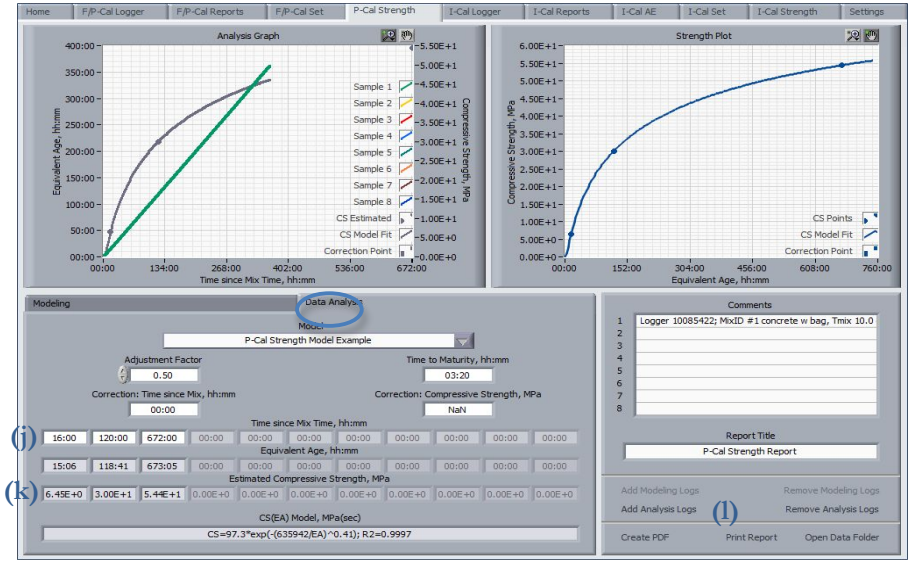
Step 2: Go to the P-Cal Strength tab and click on the Modeling tab. Click on “Add Modeling Logs” (a), then browse for and select the calorimetry data previously recorded and saved in Step 1 above.

Step 3: Enter values of compressive strength (b) and the corresponding curing time (c) from the physical test data performed in the Data Generation part. Also enter the Initial Setting Time for the mix in the Maturity Start Time field (e).



Step 4: Press “Save model” (i) and give it a descriptive name. This model is now stored on your computer and you can use it to predict strength for the same mix.

Step 5: Go to the “Data Analysis” tab. Click on “Add Analysis logs” (l) to upload the calorimetry data for the mix you just saved in Step 1 above. Select the Model you saved in Step 5. Now you can enter curing ages in the corresponding fields (j) and get a strength estimate / prediction in the (k) fields below.



IMPORTANT NOTE: P-Cal Strength automatically extrapolates the curves for curing ages that are greater than the total duration of the calorimetry log period. This allows you to make compressive strength predictions. However, a minimum log time of 96 hours is recommended for predictions.

Examples of applications:

Example 1: you ran a calorimetry curve for 15 days. You would like to get estimates of compressive strength at 1, 3, 7 and 28 days. For 1-, 3 and 7-day strength, the software will use actual values from the calorimetry curve. For 28 days, the software will use an extrapolated value.

Example 2: you ran a calorimetry curve for 72 hours (3 days), and you would like to know an estimate of compressive strength at 18 hours, 36 hours and 5 days. You can easily estimate compressive strength

for 18 hours and 36 hours, but the extrapolated value for 5 days would not be reliable. A minimum log time of 96 hours (4 days) is required for strength prediction.

The following are useful papers we recommend as a resource for data interpretation and examples of uses for your P-Cal field calorimeter and Calorimetry in general.

1. Sandberg, P., and Liberman, S., "Monitoring and Evaluation of Cement Hydration by Semi-Adiabatic Field Calorimetry," *Concrete Heat Development: Monitoring, Prediction, and Management,* ACI SP-241-2, Atlanta, GA, April, 2007, pp. 13-24.
2. Riding, K.A., Poole J.L., Juenger, M.C.G., Schindler, A.K., and Folliard K.J., "Calorimetry Performed On-Site: Methods and Uses", ACI SP-241.
3. Poole, J.L., "Hydration Study of Cementitious Materials Using Semi-Adiabatic Calorimetry," PhD Dissertation, The University of Texas at Austin, Austin, TX, 2007.
4. Wang, K., Ge, Z., Grove, J., Ruiz, M., Rasmussen, R., and Ferragut, T., "Developing a Simple and Rapid Test for Monitoring the Heat Evolution of Concrete Mixtures for Both Laboratory and Field Applications", National Concrete Pavement Technology Center, Ames, IA, January 2007, 58 pp.
5. Cost, V. T., and Gardiner, A., "Practical Concrete Mixture Evaluation via Semi-Adiabatic Calorimetry," *2009 Concrete Technology Forum – Focus on Performance Prediction*, Cincinnati, OH, May, 2009, 21 pp.
6. Cost, T., "Thermal Measurements of Hydration Concrete Mixtures: A Usefule Quality Control Tool for Concrete Producers", NRMCA Publication 2PE004.

J. TROUBLESHOOTING

P-Cal is a very robust equipment and any issues can usually be solved easily without lengthy troubleshooting or repair times. Most common problems can be identified on the F/P-Cal Logger module screen of your CalCommander software.

If the "Low Battery" light is lit: You will have to change the battery as soon as possible. The logger requires one 3-Volt CR-2032 lithium battery. Press Stop logging when possible. You will not be able to continue the current test run after you changed the battery. Disconnect the equipment from your computer, carefully unscrew the bottom metal plate of your P-Cal and locate the logger (a small rectangular box). Unplug the gray sensor cables and the black USB cable from the logger. Make sure you mark the gray sensor cables to remember to which of the two plugs they were connected. Remove the logger, then unscrew the logger case. Lift the circuit board and carefully push the battery out with a small blunt instrument, or pull it out with your fingernail. Insert a new battery, positive side facing up. Carefully realign the logger case and re-fasten the screws. Reconnect the temperature sensors and USB cable making sure the connectors are pushed all the way in. Place the logger back and reassemble the unit.

If "Bad Sensor Contact" is lit: Stop logging, withdraw the sample and proceed to unscrew the bottom part of the equipment as described in the "Low Battery" section above. Locate the logger and ensure that the sensor connector is pushed in all the way. If the problem persists, call Calmetrix Customer Support at +1 (617) 203-2090 or email support@calmetrix.com.

If erratic or clearly erroneous values appear in the temperature readings: Insert a sample cylinder filled with water, measure the water temperature and start logging. Start logging and wait for 30 minutes. Check that the temperature readings are within a 2°F (1°C) range of the actual water temperature. If the sensor indicates a temperature out of that range, proceed as with the "Bad Sensor Contact" section above. If the problem persists, contact Calmetrix customer support.

ENJOY YOUR P-CAL 1000 EQUIPMENT

Questions? - Find us at:

+1 617 203-2090
support @calmetrix.com
http://www.calmetrix.com

The Calmetrix Team

v. 100274 – 1.53

Calmetrix Inc.
PO Box 70
Arlington, MA 02476-0001
USA

T: +1 617 203-2090
e: info@calmetrix.com
w: http://www.calmetrix.com