Calibration and Verification Practice for Dilatometers

CALIBRATION

An often asked question by user's of dilatometers is: What is the recommended frequency of calibration a dilatometer requires?

While it is impossible to give a categorical answer, this Technical Note will attempt to set forth the guidelines and recommendations for practical applications and to familiarize the reader with the concept of Verification Tests to ascertain proper operation.

Calibration, as a concept, means that something is out of adjustment and by this action, it is brought back to adjustment. A corollary question is: How long this state of "adjustedness" will remain with cycling, handling, and sometimes even abusive use? Theoretically, it should be present as long as none of the functions that were adjusted are actually altered or allowed to be altered. But how does one know when such alteration occurs in a system where a multitude of interactive functions makes it literally impossible to keep track of even a few key ones. Do we assume that the test following the calibration test is correct for sure, and each proceeding one is less and less correct? And to what extent? A drift, even if it is guaranteed to be present, but only amounts to 1/100th of 1% after each test is insignificant for almost a hundred tests when one strives for a 1% accuracy, while it is insignificant forever if one looks for ±5% data. Common sense dictates that it is more important to know the trend and magnitude of any such tendencies than to have an all out effort to eliminate them, simply because the latter one is often impossible to do.

In line with the previously set forth concepts, one should not recalibrate blindly in some arbitrary schedule unless there is a reason to believe that changes had occurred. In general, the following changes always require recalibration:

- replacement of pushrod
- replacement of dilatometer tube
- replacement of a sample T/C or pyrometer
- Aging of pushrod or dilatometer (buckling, cracks, discoloration)

Almost never require recalibration:

- replacement of dilatometer platform
- replacement of control T/C
- replacement of digital gage
- replacement of muffle tube

Other than such well defined occurrences, a sudden or gradual shift in performance is what may require recalibration.

VERIFICATION TESTS

To monitor performance however, one should perform Verification Tests. There are no specific guidelines for these other than the sample used for it must be certifiably stable by itself. This is an obvious requirement as using an unstable sample is the same as using a rubber yardstick. A mere belief on the part of the investigator or an organization that a material is stable simply because it is a metal, or shiny, or it is their product is improper and should be avoided. This is not to say that one may not use a grandfathered lot of material that has been studied extensively and proven to be usable for this purpose as an in-house reference. Nevertheless, the simplest choice is a recognized standard or reference material, often the same that is used for the calibration. The test cycle again should well approximate the actual usage cycle, or if it is not practical due to varied usage, any general purpose cycle may be selected. However, whatever cycle is selected it should be the same for all verifications.

The frequency of verification tests is highly dependant on the usage the instrument gets. A single user working on a particular program tends to need less frequent checks than a service laboratory where contamination by samples is more frequent, or in cases where multiple operators are present.

Reviewing a calibration set of 5 tests usually shows a tightly reproducible group of data points because the material variability is very small. Theoretically, this spread should be maintainable over long periods of time when all materials are operated within their set limits. In other words, a quartz dilatometer tube that is performing well as long as it is used below 900^{o}C will inevitably show a shift after being subjected to 1200^{o}C tests.

One must follow a statistical approach in predicting the occurrence (or absence) of a change based on previous patterns. This, however, will break down when singular or randomly occurring events introduce changes that the historical accumulation of data does not contain. For example, a device may have seen regular use for years by a group of investigators testing pretty much the same type of samples until one day a new hiree elects to test an experimental compound that fully but not visibly contaminates the pushrod. An infrequent verification program will not catch this until the next verification test and therefore it will not be known just how much of the prior data is suspect. In this instance, the frequency of verifications may simply be defined by the tolerance level of the user is to losing data, and how much is too much.

QUALITY ASSURANCE PROGRAMS

In hundreds of thousands of operating hours at our plant and at various customers monitoring our products' performance, we have noted certain patterns that can be used as guidelines in developing a verification program with a very high probability of success. There are several elements to this:

a.) Prior Review

In case of a single investigator, this is not an issue, but with several different ones, or in a testing laboratory environment, it may be a key factor. It is recommended that a committee (made up of the chief operator and one or two materials scientists) review each new material type before it is tested for possible interaction, corrosion, or contamination and safe temperature limits. This can prevent the random occurrence of significant changes that is so difficult to police. Such reviews should not be extended for every sample, only to a new material or composition.

b.) Utilization Log

This is an entry in a log book that is maintained over an extended time period and every test (successful or failed) is entered. This can be an invaluable tool in investigating malfunctions, breakages, and poor data.

c.) Calibration and Verification Log

This is a separate log of calibration and verification tests and the results, again, maintained over a long period of time. The verification results should also be plotted on a control chart which is usually a deviation chart. For example, the first group of calibration tests can serve as the ZERO line with a 5% range above and below. (It is necessary to select a representative single temperature only, usually the highest one.) One can determine the standard deviation for this group and use the ±2 std as the desirable band instead to stay within. If the values are below ±1%, then the latter should be used. On the x-axis, the time intervals are marked. At each date, only a single test used to be performed unless it falls outside of the band. Each verification test is plotted and after awhile trends, drifts (usually due to aging) become noticeable before their effect takes on significance.

It is also practical to post such a plot right above the instrument as it generates confidence in operators and visitors alike. Very often such a regular program satisfies in-house quality assurance requirements fully and presents a quality conscience image toward management.

The frequency of verification tests can be either based on time intervals or test intervals. In the first instance, it is done, let's say, on the second Friday of each month, while in the latter case, after each 20 tests. In either case, it is imperative to maintain the effort uniformly, In normal use, once a month or one per 20 tests is a good level unless experience dictates otherwise. It is not a good practice to verify or especially not to recalibrate after every two-three tests. Doing so one shows serious lack of confidence in the dilatometer as a reliable tool. It also leads to trying to resolve minor variations which are most likely random in nature. (Noise can not and should not be analyzed and attributed significance.)

SUMMARY

In summary, a calibration test is one when the operator determines the corrections needed to calculate a true and absolute value, while a verification test is one where the operator ascertains that the instrument operates in the same fashion as before by comparing specifically generated data, and does nothing as long as the results fall within pre set limits.

Example UTILIZATION LOG

DVD-1650 DILATOMETER

Page: _____________ Date: __________________

Investigator: ____

Department: ____

Project: ____

Conditions noted: None _________ As follows: _____________________________________

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Sample: ____

Test:	#____________	#____________	#____________
Program:	#____________	#____________	#____________
Delay:	____________	_____________	_____________
Atmosphere:	_____________	_____________	_____________
Prot tube (matl):	_____________	_____________	_____________
Prot plates (mat):	_____________	_____________	_____________

	Rate/Temp/Dwell	Rate/Temp/Dwell	Rate/Temp/Dwell
A	/ /	/ /	/ /
B	/ /	/ /	/ /
C	/ /	/ /	/ /
D	/ /	/ /	/ /
E	/ /	/ /	/ /
F	/ /	/ /	/ /
G	/ /	/ /	/ /
H	/ /	/ /	/ /
I	/ /	/ /	/ /
J	/ /	/ /	/ /
K	/ /	/ /	/ /
L	/ /	/ /	/ /
M	/ /	/ /	/ /
N	/ /	/ /	/ /

Problems:	_____________	_____________	_____________
Damages:	_____________	_____________	_____________
Discolorations:	_____________	_____________	_____________