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CALIBRATION OF TUBE TYPE DILATOMETERS

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The general method of calibrating tube type dilatometers is discussed in this report. For the most accurate results, calibration standards of known thermal expansion must be used. For less accurate results, the average thermal expansion for the tube material reported in the literature, or supplied by the tube manufacturer can be used. Accuracies of the order of ±1% can be attained by calibrating with a standard and ±4% by using average values for the tube material.

Secondary calibration standards are available from the U.S. Bureau of Standards. Copper and platinum standards are recommended by ASTM. For extremely high temperature, tungsten is about the best standard.

It is a common misconception that accurate thermal expansion results can be obtained with a vitreous Silica (quartz) tube dilatometer by correcting the data with handbook values for the thermal expansion of quartz. Accurate thermal expansion of quartz can vary over a wide range as shown in the data in Fig. 1. Even if one knows the precise thermal expansion of the quartz tube he is using, the most precise thermal expansion still cannot necessarily be measured because

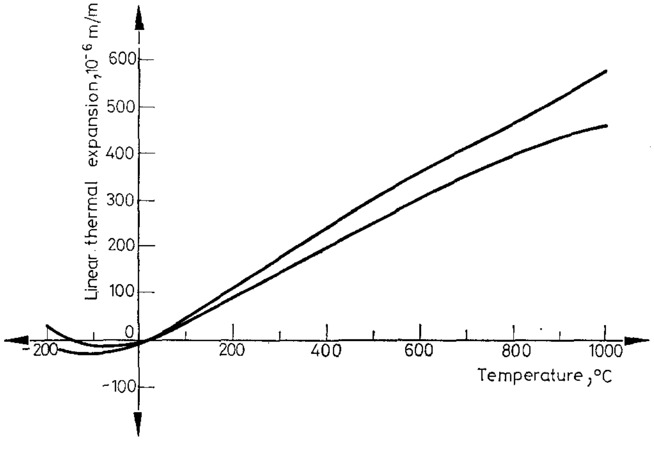


Fig. 1. Thermal expansion of vitreous silica

other system inaccuracies may exist. At high temperatures, the thermal expansion of quartz is affected by foreign materials like the oils in human hands or by reactions with the material being tested. The foreign materials react with the quartz and cause it to devitrify resulting in a change of its thermal expansion. When quartz is heated over about 1000°, devitrification takes place even though foreign substances are not present, changing its thermal expansion. Thermal variations over the dilatometer can cause greater errors than the variations in the thermal expansion of quartz. To obtain the most precise thermal expansion of a material the precise expansion of the quartz tube does not have to be known at the time of testing provided the system is calibrated with secondary standard. The thermal expansion of tubes that are used over and over again especially at higher temperatures changes so that repeated calibration is mandatory.

For optimum accuracy, the quartz tube should be handled with white gloves and periodic calibration should be performed with secondary standards.

Behavior of the tube dilatometer

Before proceeding into a discussion of calibrating a tube type dilatometer, there are several things that should be pointed out for a better understanding of the discussions to come. The tube type dilatometer utilizes the comparative method of testing. The thermal expansion of a specimen is compared to the thermal expansion of the tube material and after due consideration is given to the tube material, and the dilatometer, the thermal expansion of the specimen can be determined. Therefore, the thermal expansion of the tube material must be known along with system performance.

In a tube type dilatometer, the specimen is held at the bottom of tube during the measurement. A rod made from the same material as the tube contacts the free end of the specimen and as the specimen expands it pushes the rod. The movement of the rod is a measure of the thermal expansion of the material and is measured by a gage fixed between the tube and rod at the open end of the tube. This simple explanation assumes that tube and rod are at the same temperature so that the rod compensates for the expansion of the tube. At the bottom of the tube where the specimen is located there is no rod to compensate for the expansion of the tube and it is for this reason that a correction must be made in the experimental data to take into consideration the expansion of the tube. The tube material should have as low a coefficient of thermal expansion as possible so that the correction is as small as possible, and the tube material must have the capability to withstand repeated heating to the top temperature of the test. The tube material must also be thermally stable, so that the same thermal expansion occurs every time it is heated. It is desirable that the expansion be the same on heating and cooling so that the same correction values can be used during the heating and cooling cycle of a test.

Quartz is an excellent material from which to make a tube for thermal expansion testing. It can be used to test materials from absolute zero to 1000° in air over and over again and its thermal expansion is comparatively small, typically 0.5×10^{-6} m/m° the range *RT* to 1000°, the range in which most thermal expansion measurements are made. Above 1000° in air, alumina is the accepted material for the tube and rod to about 1600° and above 1600° one must test in a vacuum. For vacuum testing tungsten is the best tube and rod material although tantalum has been used extensively by the author principally because a tantalum tube was considerably less expensive than a tungsten tube. Other tube materials could be molybdenum or certain grades of graphite and sapphire.

As mentioned previously, the tube compensates for the thermal expansion of the rod, but this assumes that the tube and rod are at the same temperature. It is not an easy matter to keep the tube and rod at the same temperature during a test, but it can be done quite well and any deviation from the ideal can be compensated for in a correction factor for the system. To emphasize the point: suppose the tube were at a higher temperature than the rod when a measurement is made. This means that the change in tube length will be greater than the corresponding change in rod length and a lower specimen expansion will be measured. If on the other hand the rod is at a higher temperature than the tube a greater specimen expansion will be measured because the rod is longer than it should be. So, it is imperative that the tube, rod and specimen be at the same temperature when a reading is made.

It is also important to keep the measuring gage at the free end of the tube at the same temperature (generally at room temperature) during the test. Care must be exercised not to heat the measuring gage by the heat from the furnace which would again introduce error into the expansion measurement.

Experience has shown that it is economically impossible to heat the entire tube and rod to the same temperature at which the reading is made. It is generally satisfactory to have the tube and rod at nearly the same temperatures along their lengths. In other words a temperature gradient can exist over the length of the tube, but this gradient should be about the same on both the tube and rod. Any slight mismatch can be compensated for in the calibration of the system. Experience has shown, that is why the system must be calibrated and that is why the average thermal expansion of the tube material is not satisfactory as a correction factor to obtain the most accurate results.

It was stated earlier that quartz is the best tube and rod material because of its low thermal expansion and good thermal stability to about 1000°. It would be desirable to have tube and rod materials having similar thermal stability and low thermal expansion for use above 1000° in air. Unfortunately such a material does not exist and one must be willing to give up either or both of these properties to conduct tests above 1000° in air. Of course, tests could be conducted using optics to make the thermal expansion measurements above 1000°. However, here we are dealing with tube type dilatometers exclusively.

Alumina can be thermally stabilized and used in air to over 1600° but since the maximum limit of a platinum resistance furnace is about 1600° it is convenient to construct a dilatometer that is usable for testing to 1600°. Sapphire could be used, but is not available in long enough lengths and since alumina is less expensive it has become accepted as the standard tube and rod material to 1600°. The thermal expansion of alumina is about $10 \cdot 16 \times 10^{-6}$ m/m° in the temperature range 25° to 1600° which is about 20 times that of quartz and this is undesirable. As a result of this large thermal expansion, special care must be exercised with an alumina tube dilatometer to obtain accurate data.

Above 1600° thermal expansion testing must be conducted in vacuum and tantalum or tungsten tubes used to hold the specimen.

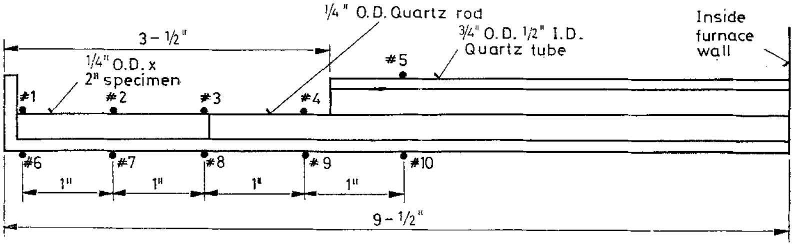
Tantalum tubes and rods, which have been used by the author because they are less expensive than tungsten tubes and rods, have a coefficient of thermal expansion of 7.8×10^{-6} m/m° from 25° to 2200°. Since the thermal expansion of tantalum is also high, special care must be taken to obtain accurate results with the tantalum tube dilatometer which will be discussed in the section on the tantalum tube dilatometer. The merits of tungsten over tantalum will also be discussed.

Furnace

A resistance furnace which is normally used to heat the specimen must heat the specimen uniformly so that the temperature over the specimen length is the same when the thermal expansion measurement is made. This is best accomplished by using a multi-tap furnace or one that has several zones that can be independently controlled. The multi-tap type furnace made by Marshall and others are suitable.

To make certain that the furnace will heat the specimen uniformly, a temperature profile of the furnace must be made using thermocouples as identified in Fig. 2. The parallel thermocouples on the specimen and tube or rod and tube should read the same temperature within about two degrees centigrade under steady state conditions although the temperature can vary axially somewhat from one end of the tube towards the other end. This axial variation should be kept as small as possible. The primary consideration is that the quartz tube and rod must be at the same temperature along their length so that the expansion of the tube is compensated by the expansion of the rod resulting in zero change at the measuring transducer except for that length of tube which holds the specimen. It is for this length that the quartz correction is needed because the specimen is taking the place of the rod. It is therefore necessary to get the tube and rod to the same temperature when the thermal expansion reading is made. A uniform temperature distribution is attained by placing external resistances between the taps on the outside of the multi-tap furnace between which the internal temperature is too high, and this condition is determined under steady state conditions. The external taps are electrically wired to the ends of each zone of the furnace. Ultimately, when the specimen is tested, it will be heated at a uniform rate and it is assumed that the steady state temperature distribution holds for the uniform rate of heating condition. From experience, it has been found that a good steady state temperature distribution will be satisfactory for the uniform heating rate tests. Any deviations or errors will be small and will be included in the correction when the system is calibrated with a secondary standard.

In Fig. 2, thermocouples were placed at one-inch intervals over a four inch length, measured from the end of the tube because in this example it had been shown from previous measurements, not given here, that the remainder of the tube-rod assembly was being heated uniformly. The data obtained in Fig. 2 were a check in the immediate specimen area only. With a new furnace thermocouples would be placed over the entire 9 – 1/2″ length at one inch intervals. The center of the specimen is located at the center of the furnace.



Steady state temperature in °C at thermocouples

1	2	3	4	5	6	7	8	9	10
452	452	452	444	430	452	452	450	448	430
735	737	738	728	706	734	735	735	727	711
1036	1037	1037	1023	1000	1034	1039	1043	1024	1000
1060	1060	1057	1049	1028	1056	1056	1061	1050	1022
1162	1161	1162	1152	1126	1159	1158	1165	1152	1122

Fig. 2. Typical temperature distribution over specimen, quartz tube, and quartz rod

The data in Fig. 2 show that the temperature over the specimen length varies by a maximum of 3° at all of the temperature levels investigated. In general, the temperature variation is within about 5° at all thermocouples with a few exceptions. This is considered a very good temperature distribution; typically the temperature distribution is poorer than that depicted in Fig. 2. In general, a minimum variation between parallel thermocouples is desirable because the correction determined with the secondary standard, which will be explained later, will be minimized.

Transducer and recorder

The transducer used to measure the thermal expansion must be calibrated for deflection as a function of its voltage output or more particularly deflection as a function of chart divisions the pen moves on the *x*-*y* recorder used for the measurement. The pen movement is directly proportional to the voltage output of the transducer. A typical recorder is a Moseley Autograf Model 7001A made by Hewlett Packard. The *x-y* recorders are generally used since the thermal expansion can be recorded on one axis and the temperature on the other axis. The detail of calibrating each dilatometer will be presented in the section covering each dilatometer. For the immediate purpose it is sufficient to say that the transducer must be calibrated in order to obtain accurate results.

The *x-y* recorder must also be calibrated before each test. This is done electrically using a Model 8662 precision L&N potentiometer or an equivalent. The millivolt output of the potentiometer is fed to the *x* and *y* axis of the *x-y* recorder and the deflection of the pen noted. If, for example, the *x-y* recorder is set for 15 mV full scale, the pen should deflect full scale (10 inches on the Model 7001A *x-y* recorder) when 15 mV is set on the potentiometer. The full scale or range will be determined by the expected thermal expansion which will be explained later. The instructions for the use of the potentiometer for these calibrations is described in the instruction manual for the potentiometer. In the case of the Model 8662 potentiometer the standard cell normally set at room temperature for the thermocouple being used is set at zero and the main dial is set at the proper millivolt setting for which the calibration is desired. The output of the potentiometer is the difference between the dial setting and the standard cell setting. When the standard cell is set at zero, the potentiometer output is that set on the dial. If the standard cell is set at some value, say 1.00 mV, this value would have to be subtracted from all main dial settings to obtain the correct potentiometer output.

For the temperature axis, the test temperature range and the thermocouple output will determine the recorder range that will be used and calibrated.

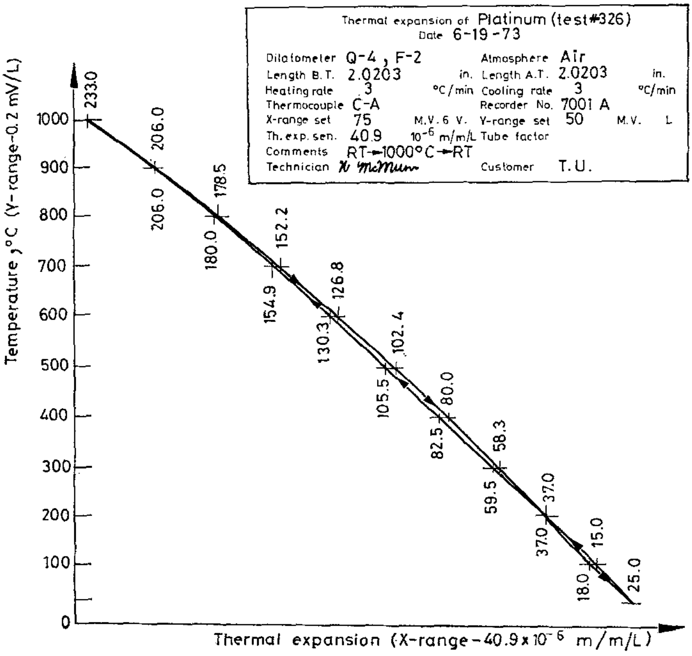
During a test, it is advisable to check the temperature indicated by the *x-y* recorder with the potentiometer. The measuring thermocouple on the specimen should be wired to a two pole knife switch so that the technician can switch from the recorder to the potentiometer rapidly. Often electrical interference will affect the *x-y* recorder or the thermocouple resulting in an erroneous temperature measurement.

Calibration procedure

Now that the furnace temperature distribution has been optimized and the transducer and *x-y* recorder calibrated the thermal expansion of chemically pure platinum, a secondary standard, can be measured to obtain a system calibration. This will permit correction for the inaccuracies in our individual calibrations and any other system excentricities that would otherwise result in errors such as thermal expansion variations between the quartz tube and rod.

A typical thermal expansion calibration curve using pure platinum is shown in Fig. 3. The temperature axis is labelled with a template corresponding to a chromel-alumel thermocouple output as shown in Fig. 4. Since the same thermocouple is used in routine tests to 1000°, it was convenient to make a template from plexiglas so that the temperature axis can be easily and uniformly identified every 100° from test to test. Similar templates could be cut for other thermocouples. The laboratory in which these tests are made is temperature and humidity controlled so that the starting temperature is always 25 °.

From the calibration of the transducer, on the 75 mV range of the x-axis of the x-y Moseley 7001A recorder, it is known that one division is equal to 40.9×10^{-6} m/m expansion. Actually, the thermal expansion of a two inch specimen was measured but the calibration coefficient is given in inches per inch. At each 100° increment on the heating and cooling cycles, the divisions of expansion on the papergraph are counted with 25° as the zero expansion point. Other pertinent information is given in the legend of each graph as shown in Fig. 3. The number of divisions expansion at each temperature is tabulated as shown in columns (1) and (2) in Table 1. The number of divisions expansion of each temperature level is multiplied by the coefficient 40.9×10^{-6} m/m/div to yield the total uncorrected thermal expansion from 25° to each temperature in 10^{-6} m/m as given in column (3). The values in column (3) are the expansion of platinum from 25° to the respective temperatures in 10^{-6} m/m of specimen without quartz corrections or any other corrections that would introduce errors. Column (4) lists the true thermal expansion of platinum as reported in the ASTM standards. The values in column (3) are compared to the values in column (4) and their difference given in column (5) which are the corrections for the system at each temperature.



Thermal expansion of Platinum (test#326)
Date 6-19-73

Dilatometer Q-4 , F-2
Atmosphere Air
Length B.T. 2.0203 in.
Length A.T. 2.0203 in.
Heating rate 3 °C/min
Cooling rate 3 °C/min
Thermocouple C-A
Recorder No. 7001 A
X-range set 75 M.V. 6 V.
Y-range set 50 M.V. L
Th. exp. sen. 40.9 10^{-6} m/m/L
Tube factor
Comments RT→1000°C→RT
Technician W McMunn
Customer T. U.

Fig. 3. Typical x—y recorder curve of temperature *vs* linear expansion

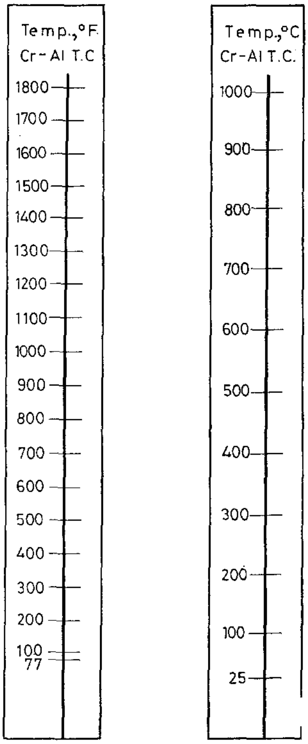


Fig. 4. Plexiglass chromel-alumel thermocouple for temperature axis of x—y recorder curve

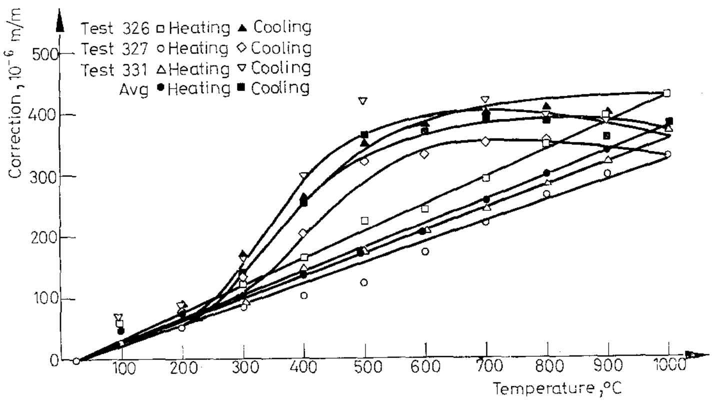


Fig. 5. Calibration of dilatometer containing Q-4 fused silica tube and rod using chemically pure platinum standard

Another way of looking at this is to say, what must be added to the values in column (3) to make them the same as column (4) and these are the values given in column (5). The corrections (column 5) are next plotted as a function of temperature (column 1) as shown in Fig. 5 as Test 326. Since it is known through experience that the correction curve as described to this point is variable, additional calibration tests must be conducted to determine the magnitude of the variability and to determine an average correction for the system. Two such additional tests are presented in Fig. 5 along with the average of the three tests which was used as the correction curve for the system.

New quartz tube and rod

As is obvious from the data in Fig. 5, the corrections on the heating and cooling cycles are different so that different corrections must be used for specimens tested on heating and cooling. The maximum variability occurs at 1000° and is about 100×10^{-6} m/m. By using an average correction this variability reduces to about $\pm 50 \times 10^{-6}$ and if a material having a total thermal expansion of 10.000×10^{-6} m/m was tested, the error would amount to 50/10.000 or 0.5% which is quite low. The correction in Fig. 4 is for a particular quartz tube quartz rod combination (Q4) and only for this combination these corrections were obtained from tests conducted during three consecutive days and give no information as to the change in corrections when the tube and rod combination are used daily in tests to 1000° testing glasses, metals, and other materials that may contaminate the quartz changing its thermal expansion properties. Devitrification starts at 1000° without contamination, and is a factor. Oil from the technicians hands also introduces contamination which accelerates devitrification. It has been stated by quartz manufacturers that quartz should be handled with white gloves to minimize the effect of contamination from the operators hands. The use of white gloves by the technician is not a practical procedure so that frequent calibrations are needed to monitor the changes in the thermal expansion of the quartz tube and rod combination.

Effect of repeatedly cycling quartz to high temperature

During the course of daily thermal expansion testing to temperature as high as 1000° and using the same quartz tube and rod, it was of interest to know how the quartz expansion and hence the dilatometer corrections changed with time. Records of the corrections were kept and Fig. 6 shows the correction curves obtained using the same tube and rod combination with the same dilatometer over a period of approximately seven months. As shown in Fig. 6, the average correction appears to have increased progressively from the test conducted 6 – 27 – 73 to the test conducted 4 – 17 – 74, with the exception of the test conducted on 4 – 14 – 74. During the entire period 6 – 17 – 73 to 4 – 17 – 74, the tube and rod were used daily in tests on glass to about 700° maximum, plastics to 150° maximum, and metals and ceramics to about 1000°. Other quartz tube–rod combinations not reported here were investigated but the same trend in increasing corrections was always present but broken by a test, during the period of investigation. It seems hardly possible that the thermal expansion of the quartz tube and rod are not stable and increase or decrease with usage. Perhaps the tube and rod were used to higher temperatures and under more contaminating conditions during the period which the corrections reverse in trend. The rate of heating the platinum specimen could be the cause of the reversal, however, every effect is made to conduct all tests at the same heating rate using a program controller. Since experience has shown that the quartz corrections can change as discussed above, frequent calibration of the dilatometer is required for the best accuracy. If one is going to conduct a very critical thermal expansion test on a material, it would be best to conduct a calibration test before and after specimen is tested to obtain the most accurate data. It would also be wise to run each test several times to reduce variability.

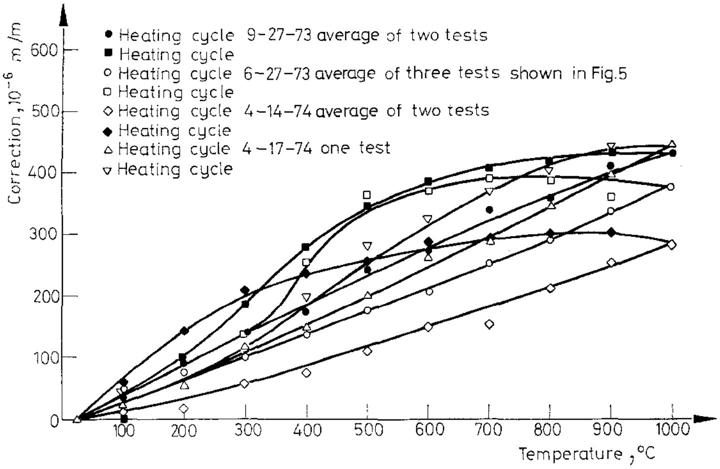


Fig. 6. Calibration of dilatometer containing Q-4 fused silica tube and rod using chemically pure platinum standard

Average quartz corrections versus calibration corrections

The average accepted coefficient of thermal expansion of quartz from 25° to 1000° is 0.5×10^{-6} m/m°. If corrections based on this coefficient are compared to the data shown in Fig. 5, it is evident that in general all corrections on the heating cycle lie below the average quartz corrections. It must be remembered that the data in Fig. 5 in most cases is the average obtained from several tests conducted around those dates so that the individual tests that were used to obtain these averages deviated more than the curves in Fig. 5 indicate.

On the cooling cycle the data in Fig. 5 shows that the measured corrections are, in general, higher than the accepted values of quartz. This is not the fault of the quartz; it expands and contracts at the same rate. This is probably the result of the temperature gradient over the quartz tube and rod system. The temperature distribution, as explained in the section on the furnace, is set under static conditions where the temperature is allowed to stabilize before a reading is taken. The data obtained in Fig. 5 were obtained while the specimen was heated at 3° per minute and this appears to be the reason the cooling cycle date is higher than the generally accepted quartz thermal expansion values. The difference cited is not serious as long as the corrections are repeatable from test to test. One could use the average accepted values and still obtain data that will solve problems. If highly accurate data are required calibrations should be conducted before and after the specimens are tested.

High expansion standard versus low expansion standards

As one goes about his daily task of measuring the thermal expansion of solids and calibrating the apparatus, a question arises. What should be the thermal expansion of a secondary standard suitable for calibrating equipment used in daily thermal expansion testing? The ASTM standard E 228–71 requires the use of chemically pure platinum from –195° to 1000° and OFHC copper from –195° to 100°. The average thermal expansion coefficient of platinum is $10 \cdot 19 \times 10^{-6}$ m/m° in the range 0° to 1000° 8.00×10^{-6} m/m° in the range –195° to 0° and copper 16.99×10^{-6} m/m° from 0° to 100° and 13.83×10^{-6} m/m° from –195° to 0°. These are high coefficients of thermal expansion and these secondary standards are suitable for calibrating dilatometers used for testing materials with similar thermal expansions. However, if one is testing low expanding glasses or quartz, platinum and copper are not the best secondary standards to use in calibrating the dilatometer. Experience has shown the author that the calibration values obtained with high expanding secondary standards are not suitable to correct the thermal expansion data measured with low expanding materials such as quartz. If one were to use the correction data obtained with platinum and apply it to data obtained by testing quartz, one would find his results could be in error by as much as a factor of two. One just cannot use corrections generated with platinum or copper on data obtained with a low expanding materials like quartz because the thermal expansion of the two materials are radically different. One must use a standard that has about the same expansion as the material he is going to test to obtain the optimum accuracy for the dilatometer. The platinum has a higher heat capacity and better thermal conductivity than quartz in addition to the radical difference in thermal expansion that contribute to the error. But, the biggest error comes from the dilatometer itself. No dilatometer will give perfect results; each has an inherent lack of accuracy and this inaccuracy is a larger percentage of the thermal expansion when low expanding materials are being tested.

For years, it has been accepted practice to purchase a secondary standard from the U.S. Bureau of Standards with an expansion coefficient in the range 10×10^{-6} m/m° and to use it for calibrating dilatometers which are used to test materials with higher, equivalent, and lower expanding coefficients. In recent years it has been realized that this practice is not good. For this reason the Bureau of Standards has adopted the policy of supplying secondary standards with high, low, and intermediate coefficients of thermal expansion so that the experimenter can choose a secondary standard that has a thermal expansion coefficient near the material he is testing. If a laboratory is testing all kinds of materials having high, intermediate, and low thermal expansion coefficients it should have a corresponding variety of secondary standards and separate correction data for dilatometer using each standard.

Heating rate effects

The rate at which a specimen is heated should be slow enough to insure that the specimen, rod, and tube are at the same temperature at any instant. Enough time must be allowed for heat transfer to take place from the heating elements of the furnace to the tube, rod, and the specimen. The heat transfer will take place by conduction, convection, and radiation. If the heating rate is too slow, high accuracy will be maintained but it will merely take more time to run the test, and the testing cost will increase. If on the other hand the heating rate is too fast, inaccuracies will result and will be somewhat proportional to the heating rate. When the heating rate is too fast, the tube will expand faster than the rod and specimen and in effect the tube will be longer, hiding or reducing the thermal expansion.

Over the years heating rates of the order of 1° every three minutes have been advocated but this is too slow, resulting in a time consuming expensive test which is frowned upon by industry. It has been the author's experience that a heating rate 3° per minute for a quartz tube dilatometer is satisfactory. With this heating rate a technician can set up a specimen in the morning, measure its thermal expansion to 1000° and cool it near the starting temperature in one shift. The specimen does not cool to room temperature by the end of his shift due to the mass of the resistance wound furnace which holds the heat, but the cooling curve is close enough to the starting temperature (room temperature) that it can be very accurately extrapolated to room temperature.

For tests to 1600° using the alumina tube dilatometer the heating and cooling of a specimen is accomplished over two shifts or about 16 hours which means that the specimen is heated and cooled at a rate of 3.5° per minute, which is an acceptable rate. However, it would be better to heat the specimen slower than 3.5° per minute because of the large coefficient of thermal expansion and contraction of alumina. Again a high mass platinum resistance wound furnace is used to heat the specimen and the rates are reduced as room temperature is approached, on the cooling cycle, requiring some extrapolation of the data.

For tests to 2200° using the tantalum tube dilatometer, the heating and cooling rates are about 4.5° per minutes because it is desirable to complete the test in two shifts. Since the tube and rod are metals, good heat conduction takes place and a faster heating rate can be used. This dilatometer has a low mass and the heating rate can be controlled over the entire heating and cooling cycles.

Secondary standards

The best way to calibrate a dilatometer is by using a primary standard. However, primary standards are so expensive that an alternate standard must be used. The alternate standard is a secondary standard which is comparatively inexpensive. Secondary standards are supplied by the U.S. Bureau of Standards. As of this writing the Bureau of Standards can supply the following standard reference materials (SRM).

Thermal expansion standards

These SRM's cover the temperature range from 20 to 1900 K having coefficients of thermal expansion over the range of 0.5 to 25×10^{-6}/K

SRM	Type	Temperature range, K	Diameter, mm	Length, mm
731-L1	Borosilicate Glass	80–680	6.4	51
731-L2	Borosilicate Glass	80–680	6.4	102
731-L3	Borosilicate Glass	80–680	6.4	152
732-L1	Sapphire	IN PREP		
732-L2	Sapphire	IN PREP		
732-L3	Sapphire	IN PREP		
736-L1	Copper	20–800	6.4	51
736-L2	Copper	20–800	6.4	102
736-L3	Copper	20–800	6.4	152
737-L1	Tungsten	IN PREP		
737-L2	Tungsten	IN PREP		
737-L3	Tungsten	IN PREP		
739-L1	Fused Silica	80–1000	6.4	51
739-L2	Fused Silica	80–1000	6.4	102
739-L3	Fused Silica	80–1000	6.4	152

The secondary standards are prepared by testing a number of specimens from a given heat of material and concluding that the thermal expansion of the tested specimen (primary standards) is representative of the whole heat. The individual specimens for sale are then cut from the bar stock and the thermal expansion measured with the primary piece assigned to them. Extreme care is exercised in testing the material. Basically, the thermal expansion is measured optically and the specimen is heated with a resistance wound furnace. The method of holding the specimen at a predetermined temperature until thermal equilibrium is established is used. A continuous curve as advocated by the author is not used. The method used by the Bureau of Standards is very accurate and considerable effort is expended to attain thermal equilibrium. Such effort is too expensive for industry.

For industry, it is best to use the secondary standards supplied by the Bureau of Standards and employ the technique of obtaining a continuous curve of temperature versus thermal expansion. The Bureau of Standards data are probably accurate to ± 0.5%. This order of accuracy is always desirable but not practical because of cost. Results obtained by the continuous recording of thermal expansion and temperature can be as good as ± 2% on a routine daily basis and this degree of accuracy is more than enough to solve most problems. If one is interested in accuracy of the order of ± 0.5% it will cost more.

Table 1

Calibration of dilatometer containing *Q*-4 Silica tube and rod using chemically pure platinum standard (Test 326)

	Measured thermal expansion			
Temperature, °C	Lines deflection on *y*-Axis	10^{-6} m/m at rate 40.9×10^{-6}m/m/line on *y*-axis	ASTM thermal expansion of pure platinum in 10^{-6}m/m	Correction factor for quartz tube in 10^{-6} m/m
25	0	0	0	0
100	15.0	614	675	61
200	37.0	1513	1604	91
300	59.5	2434	2558	124
400	82.5	3374	3537	163
500	105.5	4315	4541	226
600	130.3	5329	5572	243
700	154.9	6335	6629	294
800	180.0	7362	7713	351
900	206.0	8425	8823	398
1000	233.0	9530	9961	431
900	206.0	8425	8823	398
800	178.5	7301	7713	412
700	152.2	6225	6629	404
600	126.8	5186	5572	386
500	102.4	4188	4541	353
400	80.0	3272	3537	265
300	59.3	2384	2558	174
200	37.0	1513	1604	91
100	15.0	614	675	61
25	0	0	0	0

One could generate his own secondary standards by the same technique used by the Bureau of Standards. This is not advisable because the Bureau of Standards can do the job cheaper and better. Also, if all laboratories are using the same standards, as from the Bureau of Standards, there will be a better uniformity of data generated among laboratories. It is recommended by the author that the secondary standards supplied by the Bureau of Standards be used to calibrate all dilatometers.

None of the secondary standards available to date are suitable for calibrations to 1600° in air. For these ranges it is best to use chemically pure platinum which is available from a number of suppliers .To date there are not enough secondary standards to cover very many temperature ranges. The best one can do for the present is to purchase all of the secondary standards available and use the one closest to the thermal expansion of the material being tested.

Table 2

A typical certificate of analysis for a standard reference material from the U. S. Bureau of Standards. Thermal expansion as a function of temperature

T	Expansion $\Delta L/L_{293}$	Expansivity α	T	Expansion $\Delta L/L_{293}$	Expansivity α
80 K	-1×10^{-6}	-0.70×10^{-6}/K	320K	$13._5\times10^{-6}$	$+0.53\times10^{-6}$/K
90	$-7._5$	-0.61	340	$24._5$	0.56
100	-13	-0.53	360	36	0.58
110	-18	-0.46	380	$47._5$	0.60
120	$-22._5$	-0.38	400	$59._5$	0.61
130	-26	-0.31	420	72	0.62
140	$-28._5$	-0.24	440	85	0.63
150	$-30._5$	-0.17	460	97	0.63
160	-32	-0.10	480	110	0.63
170	$-32._5$	-0.04	500	122	0.63
180	$-32._5$	$+0.02$	520	135	0.62
190	-32	0.08	560	159	0.61
200	-31	0.13	600	183	0.59
210	$-29._5$	0.19	640	206	0.56
220	$-27._5$	0.23	680	228	0.54
230	-25	0.28	720	249	0.51
240	-22	0.32	760	269	0.49
250	$-18._5$	0.36	800	288	0.47
260	$-14._5$	0.39	840	307	0.44
273	-9	0.43	880	324	0.42
280	-6	0.45	920	340	0.40
293	0	0.48	960	356	0.38
298	$+2._5$	0.49	1000	371	0.37

A typical certificate of analysis for a fused quartz secondary standard is given in Table 2. From this table, the reader can see how the Bureau of Standards presents its calibration standards data. The overall coordination and evalution of data leading to certification was performed by R. K. Kirby and T. A. Hahn.

References

1. NBS Special Publication 260, "Standard Reference Materials Price and Availability List." U.S. Department of Commerce, National Bureau of Standards.

References not cited

1. J. VALENTICH, New Values for Thermal Coefficients, Product Engineering, July 19, 1965.
2. J. VALENTICH, Thermal Expansion Measurement, Instrument and Control Systems, Vol. 42, October 1969.
3. J. B. CONWAY, Properties of Some Refractory Metals, General Electric Atomic Products Division, GEMP-375, June 29, 1965.
4. J. L. WHITE, The Suitability of Platinum, Molybdenum, Tantalum and Tungsten as High-Temperature Length Standards, NRL Report 5159, Naval Research Lab., Washington, D.C., June 17, 1958.
5. I. E. CAMPBELL, High-Temperature Technology, John Wiley and Sons, Inc., New York, 1956.
6. R. R. MATHESON G. E.'s Lamp Research Leads to Unique Ceramic Material, Ceramic Age, June, 1963.
7. R. E. TAYLOR and G. L. DENMAN, Thermal Expansion — 1973, AIP Conference Proceedings No. 17, American Institute of Physics, New York, 1974.
8. M. G. GRAHAM and H. E. HAGY, Thermal Expansion — 1971, AIP Conference Proceedings No. 3, American Institute of Physics, New York, 1972.
9. Symposium on the Thermal Expansion of Solids, J. Appl. Phys., 41, 13, (1970) 5043—5154.
10. J. B. AUSTIN, A Vacuum Apparatus for Measuring Thermal Expansion at Elevated Temperatures with Measurements on Platinum, Gold, Magnesium, and Zinc, Physics, 3 (1932) 240—267.
11. F. C. NIX and D. MCNAIR, Phys. Rev., 61 (1942) 74—78.
12. F. C. NIX and D. MCNAIR, Phys. Rev., 60 (1941) 597.
13. T. H. RUBIN, W. ALTMAN and H. L. JOHNSTON, J. Am. Chem. Soc., 76 (1954) 5289.
14. T. A. HAHN, Thermal Expansion of Copper from 20 to 800 K, J. Appl. Phys., 41, 13 (1970).
15. R. C. ROSSI, Thermal Behavior of Ceramic Composites, Air Force Report No. SAMSO-TR-69-176, Customer Services, Clearinghouse, U.S. Dept. of Commerce, Springfield, Va. 22151.
16. D. J. MICHEL, Bibliography on Thermal Expansion in Intermetallic Compounds, Atomic Energy Commission Report No. NYO-3560-17, Clearinghouse, U.S. Dept. of Commerce, Springfield, Va. 22151.
17. N. S. RASOR, Thermal Properties of Materials, WADC Technical Report 56—400 Part I, ASTIA Document No. AD 118144, 1957.
18. R. H. KEITH, A Kinematic Dilatometer, Technical Report 65—107, The University of Dayton Research Institute, Dayton, Ohio 45409.
19. C. P. KEMPTER, J. Chem. Phys., 43, 5 (1965) 1739—1742.
20. G. K. WHITE, Measurement of Thermal Expansion of Low Temperatures, Cryogenics, March. 1961.
21. S. F. JACOBS, Ultraprecise Measurement of Thermal Coefficients of Expansion, Appl. Opt., 9, 11 (1970).
22. R. A. SCHAPERY, Thermal Expansion Coefficients of Composite Materials Based on Energy Principles, J. Composite Materials, 2, 3 (1968).
23. W. SOUDER, Measurements on the Thermal Expansion of Fused Silica, Scientific Papers of the U.S. Bureau of Standards, Vol. 21, S524.
24. L. T. LOYD, Recording Quartz Differential Dilatometer, Argone National Lab-S372, 1959.
25. S. C. FULKESON, Apparatus for Determining Linear Thermal Expansion of Materials in Vacuum or Controlled Atmosphere, Oak Ridge National Lab-2856, 1960.
26. P. S. GAAL, Quartz Pedestal Dilatometer, Temperature Measurement Society, Sixth Conference and Exhibit, April 1969.

ZUSAMMENFASSUNG — Die allgemeine Methode der Eichung von Rohrdilatometern wird erörtert. Um genaue Ergebnisse zu erzielen müssen Eichstandards bekannter thermischer Ausdehnung verwendet werden Für weniger genaue Ergebnisse können Literaturangaben bezüglich der durchschnittlichen thermischen Ausdehnung des Rohrmaterials, oder die von der Herstellerfirma der Rohre angegebenen Korrekturwerte verwendet werden. Genauigkeiten der Grössenordnung von ± 1% können bei der Eichung mittels Standards und ± 4% unter Verwendung von Durchschnittsdaten des Rohrmaterials erzielt werden.

Sekundäre Eichstandards können von dem U. S. Bureau of Standards bezogen werden. Kupfer- und Platinstandards werden von ASTM empfohlen. Für extrem hohe Temperaturen wird Wolfram als bestes Standardmaterial empfohlen.

RÉSUMÉ — La méthode générale d'étalonnage des dilatomètres à tige est discutée. Les résultats les plus exacts sont obtenus par l'emploi d'étalons dont le coefficient de dilatation est connu. La valeur moyenne de la dilatation communiquée par la littérature ou fournie par le fabricant de tige peut aussi être utilisée mais les résultats sont moins exacts. Une exactitude de l'ordre de ± 1% peut être atteinte en réalisant l'étalonnage avec un étalon et de ± 4% en prenant les valeurs moyennes du matériau.

Des étalons secondaires sont disponibles auprès de l'U. S. National Bureau of Standards. Le cuivre et le platine sont recommandés par l'ASTM. Aux températures très élevées, le tungstène est à peu près le meilleur étalon.

Резюме — В настоящем сообщении обсужден общий метод калибрования дилатометров трубового типа. Для получения наиболее точных результатов должны быть использованы калибровочные стандарты с известным термическим расширением. Для менее точных результатов может быть использовано среднее термическое расширение материала трубки, сообщенное в литературе или в приложении данным производителем трубки. При калибровке со стандартом точность может быть достигнута порядка ±1% и ±4% — при использовании средних значений для материала трубки. Вторичные калибровочные стандарты доступны из Бюро Стандартов США. Медные и платинотвые стандарты рекомендованы ASTM. Для экстремально высоких температур наилучшим стандартом является вольфрам.