



APPLICATION NOTE 78-1 JULY 1966



Probe calibration area at Dymec. Probes are placed in ice-bath at left, or in one of row of variabletemperature baths. Data logging system includes probe scanner and quartz thermometer. Probe temperature data are recorded on printer and strip-chart recorder for direct examination, and also on punched cards for computer entry.

CALIBRATING THE QUARTZ THERMOMETER

INTRODUCTION

The advent of the Quartz Thermometer, with its capability of measuring temperature to a resolution of 1/10 millidegree, has naturally raised the question "how meaningful is the quartz thermometer reading, in terms of absolute accuracy as represented by the International Practical Temperature Scale?" The Application Note then describes the <u>factory</u> <u>calibration</u> procedure, which establishes accuracy specifications traceable to the National Bureau of Standards; the relatively simple <u>field calibration</u> required to maintain the quartz thermometer within its original specifications; and finally <u>special</u> <u>calibration</u> techniques which can be employed to secure maximum accuracy from the quartz thermometer under restricted measurement conditions.

Although the quartz thermometer is an electronic device, its accuracy is determined only secondarily by electronic instrumentation calibration techniques. Rather, it is treated as a thermometer, and calibrated by established thermometric methods. This Application Note describes some of the temperature standards in common use as thermometric references, and how the quartz thermometer may be calibrated against them. Some practical considerations in the use of the quartz thermometer are also discussed.

REFERENCES AND STANDARDS

Thermal standards are based on the International Practical Temperature Scale (1948) which defines two fundamental fixed points: the steam point and the triple point of pure water. The steam point is designated 100.000° Celsius. (Celsius and Centigrade are synonymous; the former is the preferred term.) The triple point is defined as the temperature of pure water, ice, and water vapor in thermal equilibrium, and has a designated value of $+0.01000^{\circ}$ C.



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By definition, the triple point of water, $+0.01^{\circ}C$, is correlated with the value of 273.16°K on the Kelvin scale. This correlation is the one fixed point common to both the Celsius and the Kelvin scales.

Fixed Thermometric Reference Points

Temperatures commonly used as fixed reference points in thermometry are:

a) Sublimation of carbon dioxide -78.51 $\pm .01^{\circ}C$ b) Freezing point of mercury $-38.863 \pm .005^{\circ}C$ c) Ice point d) Triple point of water e) Boiling point of water f) Freezing point of benzoic acid $122.362 \pm .005^{\circ}C$ g) Freezing point of tin $231.88 \pm .02^{\circ}C$

 $0.000 \pm .0003^{\circ}C$ $0.0100 \pm .0002$ °C $100.000 \pm .0005^{\circ}C$

of the water and of the ice, and with possible contaminants in the vessel and on the probes. The ice bath temperature is lowered by the presence of dissolved impurities; the correction factor is 1.86° C/gm-mole/kilogram water. De-ionized water usually exhibits a freezing point lowering of 0.0003 to $0.0004^{\circ}C$, depending on the type of molecules remaining. An ice bath reading must also be corrected for barometric pressure by a factor of +0.0001°C/cm of mercury above the normal pressure of 76.0 cm.

Because it offers satisfactory accuracy for most applications and is so easy to use, the ice bath is usually employed for calibrating the quartz thermometer. Instructions on how to make an ice bath are given in Appendix 1 to this Application Note.

Each of these points requires some special technique or apparatus to help in its utilization as a standard. The tolerances listed are those attainable with established techniques. Some practical aspects of utilizing these fixed points are given below.

a. Sublimation Point of Carbon Dioxide

The sublimation point of CO_2 is difficult to reproduce in practice. It is sensitive to barometric pressure and requires careful preparation to achieve a pure CO_2 gas atmosphere above the solid. Unless the air is displaced, only a partial pressure of CO₂ will exist above the solid, causing a reduction in temperature of $6^{\circ}C$ or more below the desired temperature.

Calibration at the triple point, on the other hand, requires a special cell, consisting essentially of an evacuated cylindrical container with a recessed tube into which the probe is inserted. Preparing a triple point cell for use is a tedious procedure, compared with the simplicity of an ice bath. Only one probe can be calibrated at a time in a triple point cell. An advantage of the triple point cell is that it is completely sealed, and therefore is not affected by impure water or by contaminants on the probe, nor by variations in atmospheric pressure. Dymec uses a triple point cell manufactured by Jarrett Instrument Co., Wheaton, Md., Model A-11.

b. Freezing Point of Mercury

The freezing point of mercury is an excellent fixed point. A practical design for a mercury freezing point cell is described in ISA Transactions Vol. 2, No. 2, October 1963. This information also appeared in Instruments and Control Systems, December 1963. We have made a cell of this type for use at Dymec that has proved to have a precision (repeatability) of .0002°C. The cell is commercially available from Avco Corp., Research and Advanced Development Division, 201 Lowell St., Wilmington, Mass.

As in all calibration techniques in which the probe is submersed in a liquid, the ice bath and the triple point cell are subject to a rise in freezing temperature due to the head of water over the sensitive element of the probe. This effect is for most purposes insignificant, amounting to only $+7 \times 10^{-6}$ °C/cm of immersion depth (in water).

e. Boiling Point of Water

The boiling point of water is difficult to reproduce without an extremely elaborate hypsometer, but it can be used as a transfer medium for calibration against a certified platinum resistance thermometer by careful adjustment of the boiling rate, and by positioning the two thermometers near each other in the steam, not in the liquid. The vessel should be tall enough to provide a steam space of 6 to 8 inches minimum, and should be of polished metal to provide radiation shielding.

c., d. Ice Point and Triple Point of Water

Various experiments at the National Bureau of Standards and at other laboratories have shown that both the ice point and the triple point are reproducible to about 0.0001°C and that the temperature difference is a consistent value of 0.0098°C.

Principal advantage of the ice point is that a satisfactory ice bath can easily be prepared by the user without special apparatus or procedures. Also, a number of probes can be conveniently immersed in the ice bath at the same time. A disadvantage is that the user must concern himself with the purity

f. Freezing Point of Benzoic Acid

The benzoic acid point can be used with apparatus available from James F. Scanlon Co., Whittier, Calif. Their Model MTS-100 appears suitable for this purpose.

g. Freezing Point of Tin

The freezing point of tin is available as a standard apparatus (Model FPCS-100) from Thermowells,

Inc., Downey, Calif. An extra deep well (extra recost) is recommended for the quartz thermometer we probe. Although the exact temperature varies the with different lots of tin, the reproducibility is 10 better than $.01^{\circ}$ C. The tin temperature is trace-temperature to NBS, since metal of known purity can be c

purchased from NBS. (This is true also for other metals, including mercury.)

Interpolating Between Fixed Reference Points

Reference temperatures between the fixed points are commonly obtained with a bath containing some type of liquid, which is held to a relatively fixed value by a thermostatically controlled servo loop. When the reference bath is stabilized, its temperature is measured by a thermometer of known accuracy, and appropriate comparisons are made. Usually the quartz thermometer will be compared against a certified platinum resistance thermometer used in conjunction with a Mueller bridge. The accuracy of this combination will normally be better than $.001^{\circ}$ C over the operating range of the quartz thermometer. room temperature and is placed in 10 grams of water near its boiling point, the temperature of the water will be lowered by about 5 percent. If 100 grams of water is used, the overall resultant temperature would be lowered less than 1/2 percent; if a liter of water is used, the resultant inaccuracies would be well outside the resolution capability of the instrument. The advantage of initializing the probe temperature for small volumes of liquid is obvious.

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Besides the effect of initial heat transfer, rate of heat loss down the probe cable must be considered. For the probes with non-armored cables, models 2850A, D, E, F, this amounts to less than 0.001 calories per second, per degree C difference in temperature between the measurand and the surrounding air. The probes with armored cables, models 2850 B, C, will create much higher heat loss rates and should not be used for small measurand volumes or in critical areas. They are intended for use in large tanks and pipes and other industrial applications.

The accuracy with which the calibration can be transferred to the quartz thermometer is limited by the short-term stability of the bath. A small bath that can be used with good results is made by Rosemount Engineering Co., Minneapolis, Minn., their Model 910A. It can be used with methanol down to -40° C, with water in the 30° C to 100° C region, and with silicone oil to 240° C. Transfer accuracy of a few millidegrees can be achieved in this manner, and as many calibration points as the application requires can be checked.

Heat Transference to the Quartz Thermometer Probe

Response of the quartz thermometer probes is specified in relation to water moving at a certain speed. This specification is broad enough to apply to most situations in which the probe is placed in a liquid and there is some relative movement between the probe and the liquid, either as a result of natural flow or deliberate agitation. However, this is not true of gaseous measurands, and the quartz thermometer is not recommended for measuring gas temperatures unless the thermodynamics of heat transfer for a particular application are carefully investigated.

PRACTICAL MEASUREMENT CONSIDERATIONS

Thermal Effects of the Quartz Thermometer Probe

Usually the quartz thermometer probe will not be at the same temperature as the liquid measurand at the start of a measurement, and therefore when the probe is first immersed it will absorb heat from, or transfer heat to, the measurand, with resultant change in measurand temperature. Subsequently, heat flow along the probe and its cable may influence measurand temperature. Another corollary of heat transfer, not so obvious, is in measurement situations where the quartz thermometer probe is not in direct contact with the measurand, and there may actually be a high resistance heat path between the two. Examples are the triple point cell and the tin cell. Measurement accuracy can suffer because of heat leakage through the probe, such that the probe is, in effect, placed at some unknown point in a temperature gradient between the measurand and the outside

The immersion effect of the probe will normally be insignificant when the volume and/or the heat capacity of the liquid is relatively large, and the initial temperature of the probe is close to that of the measurand. However, when the measurand volume is <u>not</u> great, and when the probe is <u>not</u> preheated (or precooled) to be near the temperature of the measurand, measurement inaccuracies become significant.

The smallest of the quartz thermometer probes, model 2850A, has a thermal mass equivalent to 0.5 gram of water. If this probe is initially at environment.

FACTORY CALIBRATION OF THE QUARTZ THER MOMETER

Calibration of the quartz thermometer instrument is independent of the probe and follows conventional electronic practice. The probes, on the other hand, are calibrated against a reference thermometer in a variable-temperature bath, as follows.

Probes are calibrated in batches: up to fifty probes at a time are placed in large, well-stirred temperature-controlled liquid baths. Use of stirred liquid as the calibrating medium and the large volume eliminate the heat transfer effects discussed earlier. The liquids used are: alcohol at low temperatures, water at medium temperatures, and oil at high temperatures.

Each probe is connected to a separate oscillator, and the oscillator outputs are scanned (by a 2918A Scanner) and measured by a 2801A Quartz Thermometer. The probe temperature measurements are printed sequentially on an $\bigoplus 562A$ printer at each calibration temperature. A strip-chart recorder is used to verify stabilization of the baths at each temperature setting.

SPECIAL CALIBRATION TECHNIQUES

The accuracy specifications published for the quartz thermometer and the calibration data furnished with each probe are intended for a broad range of measurement conditions, and are conservatively stated. Higher accuracies are achievable when the actual measurement conditions are known, and the quartz thermometer can be calibrated for a specific application.

The accuracy of any special calibration will depend upon:

Nine calibration temperatures are used, ranging from -80° C to $+240^{\circ}$ C, in 40° steps. The calibration baths are monitored by a transfer standard calibrated against an NBS-certified platinum resistance thermometer and a Mueller bridge. The transfer standard is regularly checked for drift in a triple point cell, in a certified tin freezing-point standard, and against the freezing point of tripledistilled mercury. If excessive drift is noted, the transfer standard can be recalibrated against the certified thermometer. These checks, plus the excellent short-term stability of the temperaturecontrolled baths, achieve a calibration accuracy of 0.02° C, relative to NBS, at all nine points.

The crystals are individually classified into groups on the basis of the zero-degree frequency and the average slope. The grouping permits matching of probes for use with the dual-channel 2801A instrument.

- 1. Accuracy of the reference thermometer.
- 2. Stability of the calibration environment (i.e. constant temperature bath, triple point cell, etc.)
- 3. Hysteresis of the quartz thermometer. Control over (1) and (2) is in the hands of the calibrator; hysteresis is a relatively repeatable phenomenon of which we can say the following:

a. Hysteresis is a characteristic of the quartz resonator itself. It is exhibited as a frequency offset when the probe is returned to a given temperature after a temperature excursion. The resultant error at 0° C is specified to be less than $\pm 0.05^{\circ}$ C when the probe is used anywhere within its operating range

The data for each sensor are applied to a computer program which generates preset numbers for the best average slope, both for the 0° to 100°C range and for the full operating range of -80° to +250°C. (These numbers are later entered on thumbwheel switches on the 2800A or 2801A instrument to be used with a given probe.) The computer also prints a table of deviations in 10° steps for each range, so that the user can apply corrections for residual nonlinearity to the displayed readings, in critical applications. The maximum deviations permitted are 0.02°C for the 0° to 100°C range and 0.15°C from -40° to +250°C, referred to the best-fit straight line through 0°C. $(-80^{\circ}C \text{ to } +250^{\circ}C)$, after one temperature cycle or after repeated cycles.

b. Hysteresis can be minimized by restricting the range over which the probe is used.

c. Hysteresis offset will be reduced or eliminated by soaking at a different temperature; the greater the temperature difference, the faster the effect. To illustrate: the zero shift introduced by an excursion from 0° to -80° C is more likely to be removed by a few minutes at $+200^{\circ}$ C than by an hour at $+80^{\circ}$ C.

d. Hysteresis will always occur to some extent, and should be determined for each probe for each temperature range over which a special calibration is desired.

NORMAL FIELD CALIBRATION PROCEDURE FOR THE QUARTZ THERMOMETER

Since the quartz thermometer's slope linearity is a function only of the crystal cut, it can be regarded as constant, and therefore periodic calibration at one known temperature is all that is required to maintain its accuracy within the specifications published for the instrument and the individual calibration data supplied with each probe. For most purposes an ice bath, prepared as described in Appendix 1, is satisfactory. However, the quartz thermometer can be calibrated at any reference temperature within its operating range. These hysteresis considerations lead to the following basic rule: <u>SPECIAL QUARTZ THER-</u> <u>MOMETER CALIBRATIONS SHOULD ALWAYS BE</u> <u>CARRIED OUT IN A LOOP</u>. This requires that each calibration point, with the exception of one or both end points, be repeated, once for increasing temperature and once for decreasing temperature.

Calibration Procedure

Actual details depend on the nature of the equipment available to the calibrator. Important considerations, however, are: 1. Coupling of the probes to the constanttemperature reference environment. Both heat transfer path resistances and probe stem conduction must be minimized.

2. True frequency should be recorded during a calibration. This requires that the preset switches on the back of the 2801A be set to 8150, which corresponds to a sensitivity of 1kc/°C.

The first and last points on a calibration should be an easily repeated fixed point to which future work with the calibrated probe can be referred. The ice point is most convenient; once a calibration is made, future measurements are generally preceded and followed by a check of the ice point to determine hysteresis – and the resulting measurement uncertainty – during the measurement.

3. Linearity Offsets

In addition to slope and preset, it may be desirable to have a table of linearity offsets similar to that provided with the factory calibration. This requires that the experimental data be fitted with a second or higher order curve; the difference between this curve and the best straight line (of slope S) is the non-linearity.

4. Hysteresis

The experimental uncertainty due to hysteresis generally requires a calculation of hysteresis at each repeated point in the calibration loop. Consider the case of such a repeated point, where the first measurement was at temperature T_1 and 2801A output was f_1 , and the second measurement at T_2 gave output f_2 . Now if T_1 is exactly equal to T_2 , as in the case of a fixed point, then hysteresis (H) is correctly calculated by

Typical calibrations might include the following:

Exa	ample 1	Exam
Rai	nge: -80° to 120° erval: 40°	C Range Interv
Or	der of Calibration:	Orde
a. b.	0°C. -40	a. b. 1
c.	-80	c. 2
d.	-40	d. 3
~		

xample 2

Range: 0° to 40° Interval: 10° Order of Calibration: a. 0° C. b. 10c. 20d. 30 $H_{1-2} = f_2 - f_1$

But if T_2 differs slightly from T_1 , as it normally does in practice, then obviously this formula will not apply; the normal slope frequency difference corresponding to the temperature differential will have to be subtracted out, giving:

 $H_{1-2} = (f_2 - f_1) - (f_{T_2} - f_{T_1})$



e. 40
f. 30
g. 20
h. 10
i. 0

Data Reduction

One could use any number of techniques to reduce the data from such a calibration. The basic quantities needed, however, are the following:

1. Slope

This is the average slope of the frequency vs. temperature plot for each probe, over the range of interest, expressed in kilocycles/degree C. where f_{T_1} and f_{T_2} are the theoretical frequencies at T_1 and T_2 , calculated from the known slope of the crystal. (This is the average slope listed on the factory calibration chart supplied with the probe, for example, 0.9884 kc/°C.)

PRACTICAL EXAMPLE OF SPECIAL CALIBRATION

The following calibration was performed by Dymec for a customer who wished to use the quartz thermometer in calorimetry. It illustrates how, given special measurement conditions, accuracies higher than the all-purpose figures quoted on the data sheet are obtainable with the quartz thermometer. It will be noted that the hysteresis uncertainty is smaller by an order of magnitude.

2. Preset Numbers

The preset numbers for the instrument's slope thumbwheel switches are derived from these formulas:



Where S = Slope in $kc/^{\circ}C$.

The calibration report read as follows:

1. Requirement

Two 2850A probes to be specially calibrated at 0, 20, 30, 40, 60° C. Special emphasis given to accurate determination of hysteresis effects over this range.

2. Description of Calibration

Reference temperatures for calibration were determined using a certified platinum resistance thermometer and a Mueller bridge.

Calibration was performed in constant temperature baths having typical stabilities of $\pm .005^{\circ}C$. Stability of the temperature environment actually imposed on the thermometers was further enhanced by mounting the quartz thermometer probes and the platinum thermometer in a copper block suspended in a can immersed in the bath. Helium was circulated around the copper block to improve the heat transfer characteristics of the system. Estimated uncertainty in measurements due to instability of the controlled temperature was no greater than $\pm .002^{\circ}C$.

The calibration was carried out in the following order of nominal temperatures:

In Table 2, "Exact Temperature Average" is simply the mean of exact temperatures for a given nominal temperature, as recorded in Table 1. "f - f_T " is the corresponding mean of error frequencies, computed as follows:

 $(f - f_T) = (f - f_0) - f_T$

where f_0 is the average zero offset for the probe.

To illustrate: for a nominal temperature of 20° C, the temperature average is the mean of 20.410, 20.104 and 19.921, or 20.145°C. The actual probe

a.	$0^{\circ}C.$	h.	40°C.
b.	20	i.	30
c.	0	j.	40
d.	20	k.	60
e.	30	1.	40
f.	20	m.	0
g.	30		

Description of Data and Data Reduction 3.

Table 1 is a listing of the exact reference temperatures, actual probe output frequencies, and hysteresis effects. The reference temperature in °C is numerically equal to frequency in kc, because the slope thumbwheel switches are set at 8150. Hysteresis effect (H) is the change in probe output frequencies at those temperatures are 19.960, 19.653 and 19.472, the mean of which is 19.695 kc. The average zero offset for the probe is the mean of -. 197, -. 195 and -. 198, or -. 197 kc. Therefore $(f - f_T)$ is equal to:

(19.695 + .197) - 20.145 = -.253

TABLE

EXACT	PROBE "A"		PROBE "B"	
±.002°C	f	н	f	н
0.000 °C	197	-	175	-
20.410	19.960	-	19.993	Н
0.000	195	+.002	173	+.002
20.104	19.653	005	19.689	+ .002
30.241	29.665		29.707	-
19.921	19.472	+.001	19.506	+.001
30.152	29.577	+.000	29.615	004
40.066	39.366	-	39.409	-
30.352	29.775	000	29.813	000
39.389	38.702	+.003	38.747	+.006
59.843	58.888	-	58.941	-
40.167	39.466	004	39.508	007
0.000	198	003	167	+.006

frequency due to hysteresis, from the previous observation at a given temperature to the current one. For example, for the first two measurements at 20° with probe "A", H was computed as follows:

$\mathbf{T}_1 = \mathbf{f}_{T_1} =$	20.410	$f_1 =$	19.960
$T_2 = f_{T_2} =$	20.104	$f_2 =$	19.653
$f_{T_2} - f_{T_1} =$	306	$f_2 - f_1 =$	307

At this point there would appear to be a hysteresis effect of $(f_2 - f_1) - (f_{T_2} - f_{T_1}) = .001$ kc.

BUT the slope of the frequency-temperature characteristic must be accounted for, as follows:

 $Slope \frac{\Delta f}{\Delta T} = .987 \text{ kc/}^{\circ}C$ (taken from probe standard calibration chart.)

EXACT TEMPERATURE AVERAGE	f - f _T * PROBE "A"	f - fT * PROBE "B"
0.000 °C	.000	.000
20.145	253	244
30.248	379	365
39.874	499	481
59.843	758	730

* These values normalized to account for average zero offset.

For $\Delta T = -.306^{\circ}C$, $\Delta f = -.302$ kc

Thus, if there were no hysteresis, the $(f_2 - f_1)$ above should be -. 302; instead it is -. 307. Therefore the hysteresis effect is -.005 kc, which is equivalent to $-(.005/.987)^{\circ}C$, essentially $-.005^{\circ}C$.

Table 2 is derived from Table 1 and is used as an intermediate step in calculating the probe frequency-temperature slopes over restricted temperature ranges; these slopes are shown in Table 3.

PROBE "A"			
RANGE	SLOPE	PRESET	MAX. HYSTERESIS
0 - 20	.98744	9271	±.002
20 - 30	.98753	9263	±.001
30 - 40	.98753	9263	±.002
40 - 60	.98703	9308	±.002

PROBE "B"			
RANGE	SLOPE	PRESET	MAX. HYSTERESIS
0 - 20	.98789	9230	±.001
20 - 30	.98802	9219	±.002
30 - 40	.98795	9225	±.003
40 - 60	.98753	9263	±.003

The use of $(f - f_T)$ to calculate slope is an arbitrary choice made for convenience; the plot of $(f - f_T)$ vs. T is the same as f vs. T with 1 kc/°C subtracted out. Thus it follows that the actual slope of the frequency-temperature plot between two temperatures T_1 and T_2 is:

$$S_{1-2} = 1.0 + \left[\frac{(f - f_T)_2 - (f - f_T)_1}{T_2 - T_1} \right]$$

Table 3 gives the slope over each of the limited ranges for which hysteresis data were obtained. "Preset" gives the corresponding settings of the



preset switches on the rear panel of the DY-2801A which compensate for slope. "Max. Hysteresis" is the largest uncertainty due to hysteresis that may be expected when working between the extremes of the given range. It is taken as half the maximum H observed in Table 1.

For example, over the range 20 to
$$30^{\circ}$$
C,
 $S_{1-2} = 1.0 + \left[\frac{-.379 + .253}{10.103}\right]$
 $= .98753 \text{ kc/}^{\circ}$ C

The preset is given by

$$PRESET = \frac{88,150}{SLOPE} - 80,000$$

FIGURE 1

Where the highest accuracy is required, both distilled water ice and distilled water should be used. Any materials in contact with the water-ice slush of the bath should, of course, be free from contaminants. When first prepared, the ice should extend to the bottom of the dewar.



Maximum hysteresis for the $20-30^{\circ}$ span is $.005^{\circ}$, so a mean value of $.002^{\circ}$ is listed in Table 3. In comparison, the hysteresis specification for the quartz thermometer over its full operating span is $.05^{\circ}C$.

APPENDIX 1 - PRACTICAL ICE POINT REFERENCE

A practical 0° C reference may be established by using a simple ice bath, consisting of an intimate mixture of shaved ice and water. A convenient form of ice bath is illustrated in Figure 1. A large mouth dewar (preferably one quart or larger) is first filled with shaved or finely cracked clear ice, and enough water is added to fill all the spaces between the pieces of ice, but not enough to float the ice. Any potable water and ice are sufficiently pure for this purpose, and will provide a bath that is within a few hundredths of a degree of 0° C. Each thermometer probe is inserted through a hole in the cork of the dewar. The tips of the probes should be at least 4-1/2 inches below the surface of the slush, and the probes should be at least 1/2 inch from the side of the dewar.

Excess water should be poured off, and more ice should be added, as indicated above, at intervals sufficiently short that the bottoms of the probes are never surrounded by water alone. A safe practice is to keep a minimum of one inch of ice below the probes, because the temperature of the water below floating ice may be several degrees above the ice point. How often such renewal is required depends upon many factors, including the quality of the dewar and the rate at which heat is conducted downward along the wires and probes.