ITS-90 Fixed-Point Cells

FLUKE ®

Hart Scientific ®

- Best cell uncertainties commercially available
- Every ITS-90 fixed point available from mercury to copper
- Plateaus last days (gallium for weeks and TPW for months)
- Manufactured and tested by Hart's primary standards scientists

Hart scientists have designed and tested ITS-90 fixed-point cells for many years. Not only do we manufacture all the major fixed points, our metrologists have written extensively on the theory and use of cells and have created new designs covering a range of applications no other company can match.

Our testing of fixed-point cells is also unmatched. The scope of our accreditation includes the testing of ITS-90 fixed-point cells. Each cell may be purchased with this intercomparison option, which includes comparing the equilibrium value of your cell against that of a reference Hart cell.

Traditional freeze-point cells

If you want true primary temperature standards capability, you want metal freeze-point cells that are very close to the theoretical freezing temperature and provide plateaus that are both stable and long lasting.

Hart's metal freeze-point cells are the culmination of more than 20 years of primary standards experience. No other company has as much experience in the development of metal fixed-point cells as Hart. That's why you'll find Hart cells in many national metrology institutes around the world.

Each Hart cell is carefully constructed in our ultra-clean, state-of-the-art lab, using high-density, high-purity graphite crucibles containing metal samples with purity of at least 99.9999 % (six 9s) and, in many cases, 99.99999 % (seven 9s). The crucible is enclosed within a sealed quartz glass envelope that is evacuated and back-filled with high-purity argon gas. A special sealing technique is used to seal the cell at the freezing point. We measure and record for you the precise pressure of the argon gas to ensure the most accurate corrections for pressure.

Once manufactured, all Hart cells are tested and supplied with an assay of metal-sample purity. Every traditional size ITS-90 cell further undergoes more rigorous testing in our primary standards lab where we realize melt-freeze curves and perform a detailed "slope analysis" to confirm cell purity. If you want more data, we'll give you an optional intercomparison with our own reference cells.

Gallium cells

Gallium cells are a great reference for validation of instruments subject to drift (like SPRTs), and they're important for calibrating sensors used near room or body temperatures, in environmental monitoring, and in life sciences applications.

Hart's 5943 Gallium Cell is sealed in a stainless steel envelope. High purity gallium (99.99999 %) is enclosed in a plastic and metal shell. The stainless steel container is then filled with pure argon gas at one standard atmosphere at the melting-point temperature.

Gallium expands by 3.1 % when it freezes requiring the cell to have flexible walls. Unlike some manufacturers' cells, which are made from PTFE enclosure materials, our cells don't need pumping and refilling because they're not gas permeable. In fact, we guarantee our cells will maintain their uncertainty of < 0.1 mK for at least five years. Realization and maintenance of the cell is automated with our 9230 Maintenance Apparatus (see page 31). This apparatus will provide melting plateaus up to eight days and a convenient control to automatically achieve a new melt plateau each week with an investment of just five minutes. Never has the maintenance of a world-class gallium cell been easier.

Water cells

While simple ice baths are often used as a calibration point at 0 °C, their limitations include gradients, purity problems, repeatability issues, and variances in construction and measurement techniques. Triple point of water cells not only solve these problems; they represent the most used temperature on the ITS-90, and they're inexpensive to own and use.

Hart makes three traditional-size TPW cells (see page 14) that have been repeatedly proven in national labs to surpass their published uncertainty specification of ±0.0001 °C. Ice mantles may be formed using dry ice, LN_2, or immersion freezers and can last for up to two months when maintained in our 7012 or 7312 baths.

Open metal cells

Made from the same materials and with the same manufacturing techniques as their sealed counterparts, Hart's new series of "open" metal fixed-point cells include a high quality valve for connecting to a precision pressure-handling system within your lab. Using such a system, the cell can be evacuated, charged, and purged several times with a pure inert gas, then charged again to a regulated

ITS-90 Fixed-Point Cells

Specifications

Model	Fixed Point	Style	Assigned Value (°C)	Outside Diameter	Inside Diameter	Total Outside Cell Height	Depth‡	Cell Uncertainty (mK, k=2)	Certification (mK, k=2)†
5900	Mercury	Stainless Steel	–38.8344	31 mm	8.2 mm	470 mm	200 mm	0.2	0.25
5904	Indium	Traditional Quartz Glass	156.5985	48 mm	8 mm	285 mm	195 mm	0.7	0.7
5905	Tin	Traditional Quartz Glass	231.928	48 mm	8 mm	285 mm	195 mm	0.5	0.8
5906	Zinc	Traditional Quartz Glass	419.527	48 mm	8 mm	285 mm	195 mm	0.9	1.0
5907	Aluminum	Traditional Quartz Glass	660.323	48 mm	8 mm	285 mm	195 mm	1.3	1.8
5908	Silver	Traditional Quartz Glass	961.78	48 mm	8 mm	285 mm	195 mm	2.4	4.5
5909	Copper	Traditional Quartz Glass	1084.62	48 mm	8 mm	285 mm	195 mm	10.1	12.0
5924	Indium	Open Quartz Glass	156.5985	50 mm	8 mm	596 mm	195 mm	0.7	0.7
5925	Tin	Open Quartz Glass	231.928	50 mm	8 mm	596 mm	195 mm	0.5	0.8
5926	Zinc	Open Quartz Glass	419.527	50 mm	8 mm	596 mm	195 mm	0.9	1.0
5927A-L	Aluminum	Open Quartz Glass (long)	660.323	50 mm	8 mm	696 mm	195 mm	1.3	1.8
5927A-S	Aluminum	Open Quartz Glass (short)	660.323	50 mm	8 mm	596 mm	195 mm	1.3	1.8
5928	Silver	Open Quartz Glass	961.78	50 mm	8 mm	696 mm	195 mm	2.4	4.5
5929	Copper	Open Quartz Glass	1084.62	50 mm	8 mm	696 mm	195 mm	10	12.0
5943	Gallium	Stainless Steel	29.7646	38.1 mm	8.2 mm	250 mm	168 mm	0.1	0.1

†Certifications at lower uncertainties are available for national laboratories.
‡Depth is measured from the bottom of the thermometer well to the top of the pure reference material.

pressure level while measurements are made with the cell.

Once assembled and tested, each Hart ITS-90 open cell further undergoes more rigorous testing in our lab, unlike cells from some manufacturers who provide their open cells as a kit of parts, without any test data.

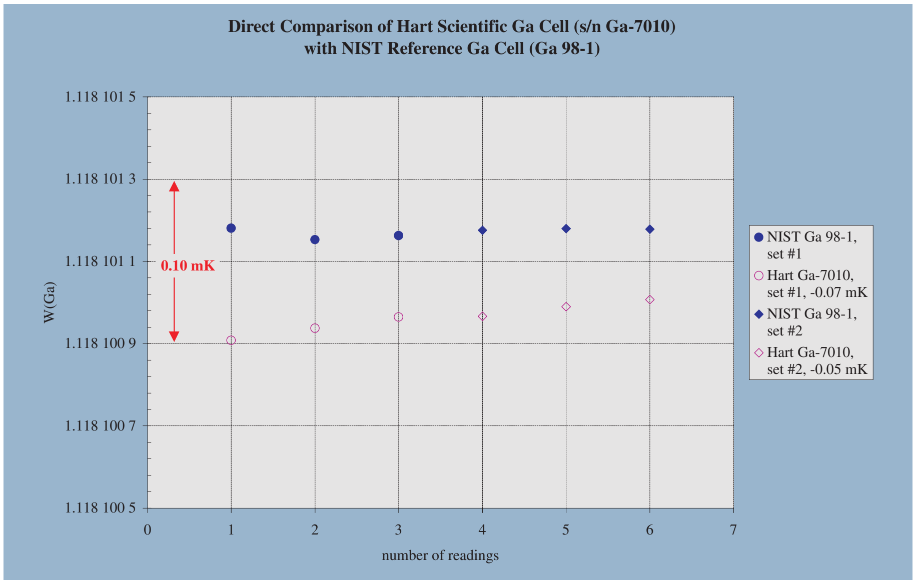
Because open cells allow users to measure the pressure within the cell, uncertainties due to pressure corrections may be minimized. Use of open cells is now being suggested by the CCT, and open cells can be used for demanding temperature-versus-pressure applications, as well as precision SPRT calibrations.

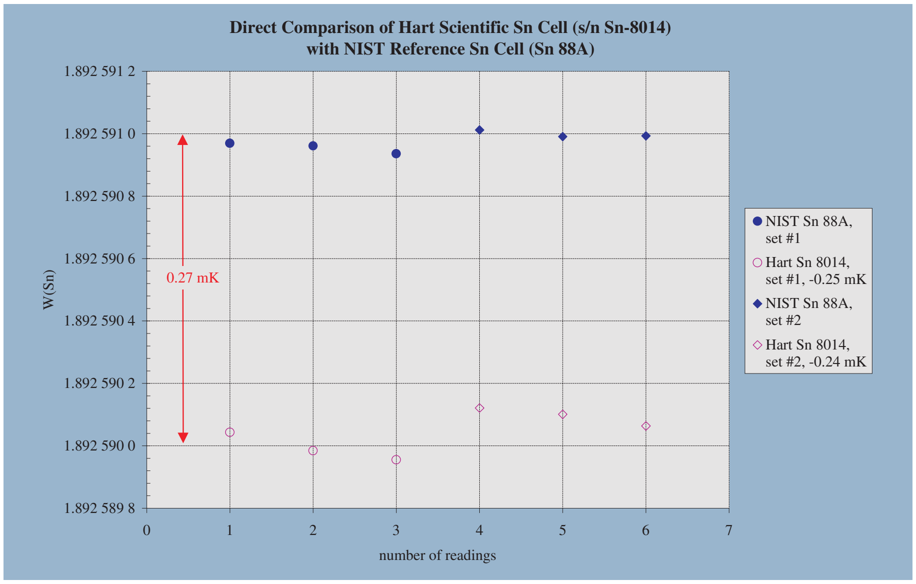
The height of these cells has been extended to allow easy access to the gas valve while the cells are in use. Pure quartz-wool insulation and four high-purity graphite discs prevent heat loss from the metal sample to the pressure regulation system while optimizing vertical temperature gradients within the cell. Each cell has an outside diameter of 50 mm (2 inches) and a height of 600 mm (23.5 inches)—(silver and copper cells are 700 mm [27.6 inches] tall).

When it comes to primary temperature standards, Hart supplies more equipment than all of our competitors combined. If your goal is to reduce uncertainty, start by buying from the company that supports its products better than any other metrology company in the world. Why trust your primary standards to any other company?

Ordering Information

5900	Mercury Cell, Stainless Steel
5904	Indium Cell, Traditional Quartz Glass
5905	Tin Cell, Traditional Quartz Glass
5906	Zinc Cell, Traditional Quartz Glass
5907	Aluminum Cell, Traditional Quartz Glass
5908	Silver Cell, Traditional Quartz Glass
5909	Copper Cell, Traditional Quartz Glass
5924	Indium Cell, Open Quartz Glass
5925	Tin Cell, Open Quartz Glass
5926	Zinc Cell, Open Quartz Glass
5927A-L	Aluminum Cell, Open Quartz Glass, Long
5927A-S	Aluminum Cell, Open Quartz Glass, Short
5928	Silver Cell, Open Quartz Glass
5929	Copper Cell, Open Quartz Glass
5943	Gallium Cell, Metal Cased
2068-D	Stand, Fixed-Point Cell, Black Delron





Open cells allow users to minimize the uncertainty from pressure corrections by regulating cell pressures themselves.

What is the uncertainty of my cell?

Fixed-point cells are standards which embody reproducible physical phenomena. The uncertainty associated with these standards can be viewed in two ways.

The first way is based on the purity of the constituent components only. Unfortunately, most of the assays provided by manufacturers of pure metals are not of sufficient quality to make a determination of the purity of the supplied metal to the level of uncertainty required. To be used in realizing the ITS-90, it would typically be necessary to have a high quality traceable assay capable of verifying 99.9999% purity or better. Even with an assay, additional evidence of the purity is necessary. This evidence includes an analysis of the slope of the freezing and melting curves and a comparison against another cell which makes similar or better claims of purity. Finally, because the temperature of a fixed point cell is defined at one atmosphere, pressure traceability is required as well.

The second approach to fixed-point uncertainties is similar but shifts the emphasis away from the traceable assay and derives its traceability through inter-comparison with another traceable fixed-point cell. In this case, the assay and the slope analysis become the supporting evidence for the observed difference against the traceable cell. This second approach represents actual observed performance in a laboratory rather than unproved claims and weakly justified hopes. This approach is particularly important with sealed cells because there is no way to verify the pressure within a sealed cell after it has been sealed.

Hart's published specs are guaranteed and can be verified through an optional accredited certification in our primary temperature lab. Are the values assigned to your fixed-point cells traceable?

Traceability and thermometric fixed-point cells

Reprinted from *Random News*

What is an intrinsic standard?

Intrinsic standards are defined by the NCSL as "standard(s) recognized as having or realizing, under prescribed conditions of use and intended application, an assigned value the basis of which is an inherent physical constant or an inherent and stable physical property." Thermometric fixed-point cells are included in the NCSL "Catalog of Intrinsic and Derived Standards." Some other well-known intrinsic standards include the Josephson-array voltage standard, the Quantum Hall resistance standard, and the Cesium atomic frequency standard.

The definitions themselves do not directly address the issues of uncertainty, traceability, or accreditation. However, in the case of thermometric fixed points, these issues are covered in the notes to the definition. The notes indicate that the value is assigned by consensus and need not be established through calibration. The uncertainty is said to have two fundamental components: (a) that associated with its consensus value, and (b) that associated with its construction and application. Traceability and stability are said to be established through verification at appropriate intervals. Verification can either be based upon application of a consensus approved test method or through intercomparison. Furthermore, the intercomparison may be accomplished with standards in a local quality control system or external standards including national and international standards.

Sealed metal freeze-point cells

Do fixed-point cells fit the definition?

The basic parameter of the cell, the phase transition, is believed to be an inherent and stable physical property of the cell when used under prescribed conditions. The generally accepted values for the temperatures of the phase transitions along with corrections due to pressure and hydrostatic head are assigned by the ITS-90, the current temperature scale adopted by the BIPM. From the values given and by taking a few measurements, the theoretical temperature of the phase transition can be calculated. Also defined by the ITS-90 is the intended application, namely, as defining thermometric fixed points to be used in conjunction with an appropriate interpolation instrument and associated equations. Finally, the conditions of use are described by supplementary information to the ITS-90 as well as a significant body of literature. Although not everyone agrees on the exact procedures, for the most part, they are quite well understood and accepted. It appears, then, that fixed-point cells can indeed be considered intrinsic standards.

However, several issues arise: First, the ITS-90 discusses fixed points based on phase transitions of pure substances. An ideal substance behaves differently than the real materials that we are able to obtain. The departure depends on the impurity content of the sample once it is assembled into a cell. For very highly pure materials, the slope of the plateau can be used to approximately determine the purity, but the absolute temperature remains difficult to predict. Second, the ITS-90 does not directly specify an optimum cell design, furnace or cryostat design, or minimum purity requirements. Quite to the contrary, many designs and options are presented in the literature. Although experiment results may suggest one design over another, the conclusions regarding uncertainty are not always clear. Third, the measurement results obtained from a cell are highly dependent upon experimental conditions. Having a good cell is only part of the exercise.

Issue	Verification via SPRT	Verification via Industry Intercomparison	NIST MAP	Cell Certification
Uncertainty for Cells	maybe	no	maybe	yes
Traceability for Cells	maybe	no	maybe	yes
Laboratory Apparatus	yes	yes	yes	maybe
Laboratory Equipment	yes	yes	yes	maybe
SPRT Calibration	no	maybe	yes	no
Procedure Evaluation	maybe	maybe	yes	no
Computation Evaluation	no	maybe	yes	no

Fourth, since a thermometer always measures its own temperature, the thermometer must be able to come to thermal equilibrium with the cell. This is affected by the cell, its apparatus, the thermometer, and the technique used to realize the phase transition. Finally, since we wish to perform traceable calibrations, knowing only the theoretical temperature is not adequate.

To certify or not to certify?

So, how do we demonstrate that our cells embody the ITS-90 definitions and how do we establish traceability? Before we tackle those points, there are three issues that we must consider. First, whatever method we choose, we must perform a robust uncertainty analysis on our measurements. The uncertainty associated with the temperature of the phase transition is only one component among many that should be considered. Second, statistical process control (SPC) is critical whenever the measurement relies upon physical processes (such as the realization of a phase transition). Through SPC, we can quantify the repeatability of our process and show that the test experiment represents the process. Third, although SPRTs and sealed cells can be used as transfer standards, intercomparison of fixed-point cells over long distances is problematic.

That having been said, the simplest method is to perform measurements in your cells using a calibrated SPRT. If the uncertainty of the measurement is sufficiently small, the temperature can then be shown to be within the estimated uncertainty based upon the theoretical considerations of the cell construction. In the case of low purity cells (five 9s or lower), it may be appropriate to "assign" a temperature and uncertainty to the realization obtained from the cell. These methods may be considered the least robust and will typically result in the largest uncertainties, but they can be shown to be traceable determinations.

A similar but more complicated method is to intercompare calibration results with peer laboratories or a reference laboratory using a suitable transfer standard. Although this type of analysis cannot directly "certify" the performance of a fixed-point cell, it will show your laboratory's capability to calibrate SPRTs using them. In many cases, this is what you are attempting to illustrate. A well-designed intercomparison will evaluate the results of the calibration, the raw and intermediate data, and the computations. Much insight can be obtained from such scrutiny.

Water triple point cells

NIST offers a measurement assurance program (MAP) to satisfy this need.

Finally, the cells can be tested by an experienced laboratory that has the capability to provide traceable results with uncertainties in the neighborhood of your requirements. If the laboratory performing the test is using its own equipment and apparatus, this type of test will show the performance of the cell only. Additional experiment and uncertainty evaluation may be required for use in your laboratory. Also, this method will not illustrate your laboratory's ability to use the cells properly. The major advantage of this method is that it can provide the lowest overall uncertainty. Often, this is referred to as "direct comparison."

So, what should we do?

Only a few years ago, it was considered acceptable to use a fixed-point cell with plateau analysis to show that the cell was behaving itself. This approach has proven to be inadequate as our understanding evolves and we try to improve our laboratories. Moreover, laboratory accreditation requires that we follow rigorous procedures in evaluating our uncertainties. If our uncertainties approach National Metrology Institute level, our data and analysis must justify this. And, the fixed-point cell is a critical component in the uncertainty evaluation. The intrinsic standard argument provided by the NCSL does state that some level of intercomparison is necessary. Presumably, the NCSL expects the intercomparison to be appropriate to the uncertainty claimed and based on the most current practices.

We must choose the method that makes economic sense and that satisfies our requirements. For example, if we are calibrating secondary PRTs using mini fixed-point cells, we may be justified in using a calibrated SPRT to verify the performance of our cells. Many laboratories (several accredited) use this method with success. Traceability can easily be demonstrated and the uncertainty analysis is straightforward. On the other hand, if we have spent tens of thousands of dollars on a system to calibrate SPRTs and these SPRTs are used for critical measurements, the NIST MAP program is a very good option, provided we qualify. Finally, if we wish to provide cell certifications, we will require a set of certified reference cells along with a robust uncertainty evaluation. At Hart, we use a combination of all three methods.

Conclusion and recommendations

So, are fixed point cells intrinsic standards or certified artifacts? It really doesn't matter. Both viewpoints require testing and traceability. Both approaches require rigorous uncertainty analysis that must satisfy the scrutiny of our accreditation assessors, our customers, and our peers. And each perspective can be logically justified. At Hart, we treat some as intrinsic standards and others as certified artifacts. Our uncertainty analyses are as rigorous as we can make them and we welcome comment from our peers. Additionally, the NIST thermometry staff is available to assist in the development of uncertainty budgets, meeting traceability and accreditation requirements, as well as unique testing requirements. Finally, we use the approach that will result in the lowest uncertainties for a given set of equipment and techniques. After all, isn't that what it's all about?