SIMPLE LIQUID NITROGEN COMPARATOR MODEL 461

User Maintenance Manual/Handbook

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GUARANTEE

This instrument has been manufactured to exacting standards and is guaranteed for twelve months against electrical break-down or mechanical failure caused through defective material or workmanship, provided the failure is not the result of misuse. In the event of failure covered by this guarantee, the instrument must be returned, carriage paid, to the supplier for examination and will be replaced or repaired at our option.

FRAGILE CERAMIC AND/OR GLASS PARTS ARE NOT COVERED BY THIS GUARANTEE

INTERFERENCE WITH OR FAILURE TO PROPERLY MAINTAIN THIS INSTRUMENT MAY INVALIDATE THIS GUARANTEE

RECOMMENDATION

The life of your **ISOTECH** Instrument will be prolonged if regular maintenance and cleaning to remove general dust and debris is carried out.

We recommend that this instrument to be re-calibrated annually.

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The company is always willing to give technical advice and assistance where appropriate. Equally, because of the programme of continual development and improvement we reserve the right to amend or alter characteristics and design without prior notice. This publication is for information only.

HEALTH AND SAFETY INSTRUCTIONS

1. Read this entire handbook before use.

2. Wear appropriate protective clothing.

3. Operators of this equipment should be adequately trained in the handling of hot and cold items and liquids.

4. Do not use the apparatus for jobs other than those for which it was designed, i.e. the calibration of thermometers.

5. Do not handle the apparatus when it is hot (or cold), unless wearing the appropriate protective clothing and having the necessary training.

6. Do not drill, modify or otherwise change the shape of the apparatus.

7. Do not dismantle the apparatus without disconnecting it from the supply and leaving time for it to reach ambient temperature.

8. Do not use the apparatus outside its recommended temperature range.

9. There are no user serviceable parts inside. Contact your nearest Isotech agent for repair.

11. Ensure materials, especially flammable materials are kept away from hot parts of the apparatus, to prevent fire risk.

12. Ensure adequate ventilation.

13. Each apparatus is protected by an over temperature circuit. Please consult handbook for details.

SIMPLE NITROGEN BOILING POINT APPARATUS

This device is intended for use with liquid nitrogen refrigerant supplied from a storage Dewar with a transfer device.

THE USER IS CAUTIONED NOT TO USE THIS APPARATUS WITH LIQUID OXYGEN.

The apparatus is intended for use by technical professionals skilled and trained in the techniques (and understanding the hazards) of storing, transferring, and otherwise handling liquid cryogens, including but not limited to liquid nitrogen.

This equipment is intended for use in the calibration of Standard Platinum Resistance Thermometers (SPRTs) such as the Isotech Model 909, Leeds and Northrup and YSI Models 8163 and 8167, etc.

It may be used to calibrate other thermometers having similar diameter, length and immersion characteristics. It provides an environment for the comparison of a thermometer under test with another thermometer having a known calibration, at the boiling temperature of liquid nitrogen.

UNPACKING THE COMPARATOR

DO NOT DISCARD ANY PACKING MATERIAL UNTIL ALL PARTS HAVE BEEN LOCATED AND IDENTIFIED. SOME PARTS ARE SMALL.

Because of the fragility of some parts, the Comparator is shipped in a partially-assembled form.

You should find in the package:

- One Stainless Steel Dewar flask.
- One Lid for Dewar Flask
- One Cylindrical block drilled with deep holes and a central threaded hole and partially covered with a porous blanket.
- Copy of this manual.

Please inspect the packing materials carefully, should any parts be found missing contact Isotech.

ASSEMBLING THE COMPARATOR

The Simple Liquid Nitrogen Comparator is easily assembled by lowering the equalising block into the insulation inside the Dewar.

The Dewer can then be carefully filled with liquid nitrogen to within 1 cm of the top of the vessel. Top up every 20 to 30 minutes, or between readings.

USING THE COMPARATOR TO CALIBRATE THERMOMETERS

Isotech practice in using the Comparator is as follows:

1. Use a resistance thermometer of known calibration (not necessarily an SPRT) to monitor the establishment of the boiling plateau. This thermometer will be called a "monitor" thermometer. Connect the monitor thermometer to a resistance measuring device - an ohm-meter, for example - with an adequate resolution (0.1Ù is sufficient for a 100Ù monitor thermometer)

2. Crack open the LIQUID VENT of the storage Dewar withdrawal system. As the liquid enters the Comparator, a substantial amount of liquid will be converted to gas until the Comparator is cold.

   Continue to fill the Dewar until the level is 1cm from the top.

3. Monitor the thermometer connected to the ohm-meter. Note when the resistance of the thermometer indicates approximately the liquid boiling point, and no longer changes. (The resistance will depend upon the thermometer's calibration, but will be approximately 25% of the room temperature resistance).

4. Add enough liquid nitrogen to reset the level to 1cm below the top.

5. When the Comparator has been adequately filled and chilled, little evidence of boiling-off gas will be seen and the liquid will be retained in usable quantity for 30 minutes before it is topped up again.

A BRIEF TECHNICAL TUTORIAL

COMPARISON CALIBRATIONS AT THE BOILING POINT OF NITROGEN (OR ARGON)

Temperature is defined by the International Temperature Scale of 1990 (ITS-90), which replaces the International Practical Temperature Scale of 1968 (IPTS68) and all previous International Scales. The Scale requires the realization of a series of defining fixed points, phase equilibria of pure materials, to which a value of temperature has been assigned. Thus the state in which pure water exists simultaneously in its liquid, solid and vapour phases (the triple point of water) defines the temperature 273.16K or 0.01°C; the state in which pure tin exists simultaneously in its liquid and solid phases under 1 standard atmosphere pressure defines the temperature 231.928°C.

To calibrate a thermometer over the region of temperature from -189.3442°C to 0.01°C, the Scale specifies a calibration at the triple point of argon (-189.3442°C), at the triple point of mercury (-38.8344°C) and the triple point of water (0.01°C). The measurements obtained are then compared with a reference function and the resulting differences used to calculate the coefficients of a deviation function which can then be used for interpolation. An Isotech computer program (Daedalus) is available that will enable all relevant ITS 90 data to be calculated.

An alternative to the realization of the triple point of argon, employed by many Laboratories including National Laboratories, is a comparison calibration in boiling nitrogen or argon.

COMPARISON VERSUS ABSOLUTE CALIBRATION AT THE COLD END OF THE LONG-STEM SPRT RANGE

In theory, the triple point of argon is a simple point to establish. It has been realised in sealed cells by Pavese, Bonnier, Furukawa and others, particularly for capsule thermometers. Sealed cells have the drawback that, at room temperature, they exist under relatively high pressure (e.g, 3000 psi), and must be regarded as pressure vessels in handling and for transportation. Also, the cryostat required is not simple, and the prevention of heat transfer via the cell mountings and the tubes of long-stem thermometers must receive special attention. In practice, the realisation of the argon triple point can be costly and complicated.

Most laboratories will elect to calibrate at this end of the SPRT range by the simpler and less costly comparison method, Isotech Model ITL-M-18205 is designed specifically for this purpose. In this method, a thermometer under test is compared to a thermometer with a known calibration that is traceable to a National Laboratory. Indeed, many National Laboratories will calibrate thermometers submitted to them by comparison with their own thermometers, realising the argon triple point itself only infrequently, and only for the calibration of their own reference SPRTs.

This policy has been announced by the National Physical Laboratory of England, in its publication "Adoption of the ITS-90", as follows:

"Most thermometers (submitted for calibration) will involve measurements at the tin and zinc freezing points, plus a comparison with NPL standards in a bath of liquid nitrogen."

Other National Laboratories will follow similar practices.

THE EFFECT OF PRESSURE (E.G. LABORATORY ALTITUDE) ON THE NITROGEN AND ARGON BOILING POINTS

The temperature at which a pure liquid boils is that temperature at which the vapour pressure and the ambient pressure are equal.

The normal boiling point of nitrogen is -195.798°C; a mere 6.45K below the argon triple point. The normal boiling point of argon is -185.88°C. However, since these are two-phase (liquid-vapour) equilibria rather than triple points, their temperature will vary with pressure, for example as a function of laboratory altitude above standard sea level. Table 1 shows the variation of the nitrogen and argon boiling temperatures with altitude.

For this Table, the pressure (p/mm Hg)-temperature (t/°C) relationship has been computed from the Antoine vapour pressure equation

$t = (B/(A - \log p)) -$ (Eq. 1)

in which, for nitrogen, A = 6.4946, B = 255.68, C = 266.558, and for argon A = 6.6165, B = 304.277, C = 267.328.

TABLE 1: THE BOILING POINT OF NITROGEN AND ARGON AT VARIOUS LABORATORY ALTITUDES

FEET OF ALTITUDE	PRESSURE/ MM HG	NITROGEN BOILS °C	ARGON BOILS °C
-1000	787	-195.50	-185.54
0	760	-195.80	-185.88
1000	733	-196.11	-186.22
2000	707	-196.41	-186.56
3000	681	-196.72	-186.90
4000	656	-197.02	-187.24
5000	632	-197.33	-187.58
6000	609	-197.63	-187.92
7000	586	-197.93	-188.26
8000	564	-198.24	-188.60
9000	543	-198.53	-188.94

(Pressure-Altitude data from "Pressure-Altitude Tables Based on the United States Standard Atmosphere", W. Brombacher, NACA Report No. 538, 1948)

THE METHODOLOGY OF COMPARISON CALIBRATION

Comparison calibration is done by placing two thermometers in an isothermal situation at an approximately known temperature. One of these thermometers (the standard) must have a known calibration (such as is obtained from a UKAS or National Laboratory), that includes data for the temperature range under consideration.

METHOD 1

After the two thermometers have reached a condition of thermal equilibrium with the bath, the resistance of the standard thermometer is measured, the resistance of the unknown is measured, and the resistance of the standard is measured again, to assure that no change in temperature has taken place (possibly caused for example by change in ambient pressure).The temperature of the bath, t_b, is then determined from the measured resistance and the calibration table for the standard, and the measured resistance of the unknown at the same temperature is taken to be its resistance at t_b.

It is possible to take account of slow steady drifts by taking a succession of measurements at regular intervals, alternatively with each thermometer. The contrasting feature of a "thermostatically controlled" bath is that temperature fluctuations occur as the control function fluctuates. In this case, each measurement should be an average value taken over a sufficient number of control cycles to avoid possibility of swings of equal magnitude but in opposite directions between successive instantaneous measurements leading to an erroneous interpretation.

METHOD 2

Outlined below is a potentially more satisfactory way than Method 1 to perform a calibration transfer, if there is available a resistance measuring bridge or instrument equipped with appropriate terminal connections and the facility to allow the ratio of two resistances to be determined. Examples of such bridges are the Guildline Model 9975 and the Automatic Systems Laboratories F-16, F-17 and F18 Bridges.

With a thermometer to be calibrated and a standard thermometer (with a known calibration) immersed in (and in thermal equilibrium with) the bath, the temperature (t) maybe found from the standard's calibration data, being that value corresponding to the resistance R_s determined by comparison with a known fixed resistance.

A measurement of ratio, r, of the resistance (R_u) of the thermometer intended for calibration to that (R_s) of the standard thermometer will then furnish the calibration point (t, R_u). In principle, a transfer of calibration carried out in this manner depends not only on equality of temperature of the thermometers at the time of the transfer but also on this temperature being identical to the vale t, determined by comparison of R_s and the fixed resistance.

In this context there is no advantage immediately evident over Method 1. However, if the two measurements relate respectively to slightly different bath temperatures (and, therefore marginally different values of R_s and R_u), the ratio r can, in certain circumstances, be construed still to apply to that value of R_s used for temperature determination and, consequently, can yield a value of R_u belonging to that temperature, without introducing significant error. The condition that renders r relatively insensitive to small temperature differences is that the two thermometers have similar rates of change of resistance with temperature. For example, a discrepancy of 1°C in a bath temperature of about -200°C is equivalent to a calibration measurement error no greater than a few mK for any thermometers that would qualify as standards, within the constraints defined by ITS-90. However, spatial uniformity of temperature is still vital.

By way of illustration the results are presented below of some calculations on the correspondence between 17 thermometers calibrated at various times. The thermometers represented a fair selection of Leeds and Northrup 8163 and 8167 thermometers, of various ages and provenance. The reference standard for each comparison was one of two Leeds and Northrup Model 8163 thermometers having long and honourable histories and current NIST calibrations.

Each of the 17 thermometers was calibrated relative to the standard thermometer at the boiling point of oxygen (it might equally have been done at the boiling point of nitrogen, or of argon) by using the standard thermometer as the standard resistor and the unknown thermometer as the unknown resistance of a Guildline Model 9975 Precision Current Comparator. In addition, each unknown thermometer was calibrated at two fixed points of the ITS-90, the mercury triple point and the triple point of water, and a table of values calculated (from all the data) at intervals of 1°C over the range from -200° to -179°C, using the Isothermal Technology Daedalus MS-DOS interpolation program.

From the table of resistance ratios for each thermometer, at 1°C intervals between -200°C and -179°C, are calculated increments of resistance ratio, and then the distribution of increments for the same temperature interval for each of the thermometers. Table 2 provides a summary of the results. (The detail is available, and will be sent to any reader on request).

From this information, it was possible to derive the mean and range of increments for the group at each temperature, and the standard deviation at each temperature in terms of increment and of temperature.

In addition estimates were made of bath temperature uncertainties that are tolerable, at nominal values of -186°C and -196°C in order that calibration uncertainty for a particular thermometer be maintained within about 1mK.

TABLE 2: RATIO AND INCREMENT OF 17 THERMOMETERS BETWEEN -200°C AND -179°C

1 TEMP DEG°C	2 MEAN INCR PER °C	3 RANGE OF INCR	4 2 STD DEV LEVEL OF INCR POPULATION °C	5 2 STD DEV LEVEL °C	6* CAL ERROR ERROR °C FOR UNACC DEV IN BATH TEMPERATURE
200	.004304	.000003	.00000150	.0003665	
199	.004310	.000004	.00000174	.0003772	.00115
-198	.004316	.000004	.00000172	.0003849	.00077
-197	.004322	.000003	.00000150	.0003896	.00039
-196	.004326	.000004	.00000177	.0003917	.00000
-195	.004333	.000003	.00000165	.0003915	.00038
-194	.004333	.000004	.00000177	.0003894	.00077
-193	.004336	.000003	.00000162	.0003856	.00115
-192	.004338	.000004	.00000193	.0003805	
-191	.004340	.000003	.00000137	.0003743	
-190	.004341	.000003	.00000150	.0003674	
-189	.004342	.000003	.00000140	.0003601	.00106
-188	.004342	.000003	.00000165	.0003526	.00071
-187	.004342	.000003	.00000158	.0003452	.00036
-186	.004342	.000003	.00000154	.0003384	.00000
-185	.004341	.000003	.00000165	.0003323	.00033
-184	.004340	.000002	.00000118	.0003273	.00065
-183	.004339	.000002	.00000139	.0003238	.00087
-182	.004338	.000003	.00000150	.0003219	.00130
-181	.004337	.000002	.00000112	.0003220	
-180	.004334	.000003	.00000160	.0003244	
-179	.004333	.000003	.00000140	.0003294	

*Over the temperature range of column 1, the calibration error per degree deviation of bath temperature from its assumed value is not particularly sensitive to the temperature itself.

Comment

It is clear that the thermometers reported on have very closely matching characteristics which fact appears to render superfluous knowledge of the precise temperature of the bath. However, the extent of agreement between characteristics is not discernible without, detailed measurement. In any event, care must be taken to assess the demands imposed on the measurement environment by the calibration accuracy required for a thermometer with given or approximately known, characteristics.

For industrial resistance thermometers, there will generally be a considerable difference in the slope of the resistance/temperature characteristic from that of an SPRT calibration standard.
However, the calibration accuracy required is unlikely to be at the level of a few mK, thus providing a wider latitude in acceptable bath temperature deviation than would otherwise be the case.

The methods, with appropriate modifications, are obviously applicable to other types of thermometer, e.g. thermocouples.

USEFUL REFERENCES

H. Preston-Thomas, The International Temperature Scale of 1990, Metrologia 27, 3-10 and 107 (correction) 1990. Isotech will furnish a copy of this paper upon request.

H. E. Sostmann, Fundamentals of Thermometry Part I, pp 1-18, also Practical Calibration of Thermometers on the International Temperature Scale of 1990, pp 19-30, Isotech Journal of Thermometry, Vol. 1 No. 1, (1990).