PLATINUM / GOLD THERMOCOUPLE

User Maintenance Manual/Handbook

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ISOTECH

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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 surface contaminants is carried out.

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OPERATING INSTRUCTIONS AND PRECAUTIONS

Always keep the Gold/Platinum thermocouple in its case when not in use.

To prevent devitrification of the quartz outer sheath.

Prior to use ensure that all traces of grease or other surface contaminants are removed from the quartz outer sheath using a pure alcohol such as Ethanol or Acetone.

To prevent inducing strain into the pure thermocouple material.

The Gold/Platinum thermocouple has a fine coil of platinum at its measuring end. This coil allows for the different rates of expansion of the gold and platinum wires.

Due to these different expansion rates and the fact that we are dealing with pure materials the thermocouple must be heated and cooled in a controlled manner very much like that of a Standard Platinum Resistance Thermometer.

Using the thermocouple above 450°C; the thermocouple can be slowly introduced into a calibration volume or pre heating furnace set at 450°C and allowed to stabilize before heating slowly to the temperature of interest.

On completion of work at temperatures above 450°C the thermocouple must be cooled slowly in a preheating furnace or a calibration volume to 450°C and held there for 30 minutes before carefully removing the thermocouple to ambient temperature.

To prevent bending of the quartz outer sheath.

At 800°C and above the outer quartz sheath of the Gold/Platinum thermocouple softens and can bend if it is not supported along its length. At these high temperatures we recommend that the thermocouple is housed inside a close fitting, closed ended recrystallised Alumina tube, which has been pre-fired to 1000°C, or better still a silicon carbide tube.

Support the handle of the thermocouple with a lab stand will also help prevent any possible bending of the outer quartz sheath.

To prevent contamination of the pure thermocouple material.

At temperatures above 700°C metallic vapours can pass through the quartz outer sheath and attack the pure thermocouple material. For this reason the thermocouple is fitted with a fine tube that is open to atmosphere. The tube can be left open to atmosphere to provide a constant supply of oxygen to help protect the pure thermocouple material from contamination or the user can apply a flow of inert gas into the thermocouple again to protect from contamination.

To prevent moisture build up inside the thermocouple.

If the thermocouple is taken below the dew point (tested at 0.01°C) it is important that the fine tube is sealed prior, to avoid moisture from condensing inside the thermocouple.

GOLD/PLATINUM THERMOCOUPLE AND THE DC MEASUREMENT REQUIREMENTS

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Introduction

Thermocouples measure temperature differences.

ITS-90 removed them in favour of the Standard Platinum Resistance Thermometer.

The limiting factor with all normal thermocouple is that one or both of the thermoelements is an alloy and an alloy cannot be produced that is homogeneous. This means that, because the Emf of a thermocouple is generated along the wire where there exists a thermal gradient, if the thermal gradient is moved along the thermocouple the Emf will change.

This limits the best thermocouple accuracy to about ±0.3°C, 2 sigma.

If both thermo-elements were of pure metal, then this limitation would not exist, giving the possibility of more accurate thermocouple measurement.

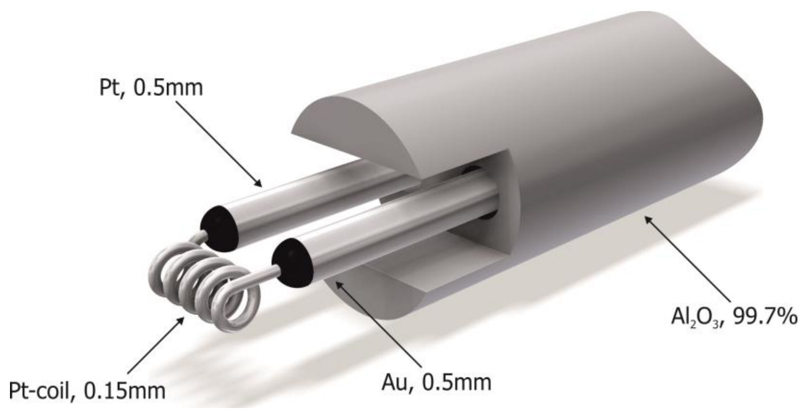
For good stability noble metals are preferable to base metals and in particular Gold/Platinum (Au/Pt) and Platinum/Palladium (Pt/Pd) have been investigated.

The first published tables for Au/Pt and Pt/Pd thermocouples dates to 1941 (Roesner & Wensel), however it was not until the 1980's with better purity metals available that the Au/Pt thermocouple was reconsidered.

In Canada the research was carried out by McLaren and Murdoch [2] who solved one of the problems with the thermocouple that of expansion mismatch. By adding a fine spring of platinum to the junction between the gold and platinum, the gold could expand freely without straining the platinum thermo-element.

McLaren and Murdoch considered all aspects of construction and performance publishing a lengthy two part article in 1987 which is still the standard work and essential reading to all who are interested in Gold/Platinum thermocouples.

Figure 1 – Measuring junction of the thermocouples 9/90, 11/90 and 12/90 (PTB design).



To summarise and simplify their work they found that this thermocouple if made according to their prescription was capable of accuracies of ±0.02°C from 0°C to 962°C.

Its output increases from some 6μV/°C at 0°C to about 25μV/°C at 962°C.

During the 1990's Burns et al of NIST have confirmed and extended their work. A reference list is attached at the end of the article [1].

During the 1990's, reluctantly at first, this author has been looking at the Gold/Platinum thermocouple to see from a laboratory and industrial point of view its advantages and limitations.

To construct such a thermocouple is a sophisticated project in itself.

Following the prescription of McLaren and Murdoch, wires of 99.999% purity are required and a long complex process of annealing taking many days.

However, making the thermocouple is only a small part of the project.

As the introduction mentions thermocouples measure temperature difference and so both the temperature at the reference and measuring ends must be considered.

Before selecting equipment to turn the thermocouple into a measurement system it is necessary to consider some electrical quantities.

At the silver point the Au/Pt thermocouple generates 25μV/°C and may be capable of accuracies approaching ±0.01°C. To match this, the total measuring system should have, in terms of voltage an uncertainty of ±0.25μV or in terms of temperature ±0.01°C.

Considering the temperature constraints, and firstly focussing on the reference junction, a good quality stirred ice bath make using distilled water would have an uncertainty of ±0.005°C, or half the total uncertainties. If a water triple point cell is utilized this reduces to ±0.0005°C and so a water triple point must be used.

Next, considering the upper calibration temperature, a pure (99.9999%) silver cell in a heat pipe apparatus can realise a temperature of 961.78°C within 1 to 4mK. This is adequate.

The third component of the measuring system is the voltage-measuring device. After research, the Wavetek 1281 7½ digit voltmeter was chosen, however the best UKAS uncertainty that could be issued with the meter was ±0.5μV 2 sigma, which is equivalent to ±0.02°C.

Combining these uncertainties it is possible to calculate the overall uncertainties at each calibration fixed point – four are used; *Water Triple Point, Zinc, Aluminium and Silver.*

Physical Constraints

The length of the thermocouple produced was two meters from measuring to reference junction and 3 meters from reference junction to digital voltmeter. This means that the reference junction and digital voltmeter must be mobile as the thermocouple measuring junction is calibrated. A mobile computer trolley was found to be ideal.

Platinum/Gold Thermocouple Results

A number of Gold/Platinum Thermocouples have been produced and calibrated. The Emf's at Silver, Aluminium and Zinc of some of them are tabulated in table 1 below.

Table 1: Gold/Platinum Thermocouples voltage at the Fixed Points.

Fixed Point	Expected µV	G001 µV	G002 µV	G009 µV	G010 µV	G012 µV	G013 µV	G015 µV
Ag	16,120.5	16,116	16,115.0	16,117.3	16,115.6	16,116.9	16,117.6	16,116.2
Al	9,320.44	9,316.3	9,316.3	9,318	9,316.8	9,317.65	9,317.8	9,317.3
Zn	4,945.63	4,943.7	4,953.7	4,944	4,934.1	4,943.65	4,943.7	4,943.6

Even at 5N purity the composition of the 10ppm impurities will influence the output Emf of the thermocouple. McLaren suggests a value of 20µV at the silver point as a typical spread of 5N material and all the results are within this expectation.

To establish international credence for the results, 3 thermocouples were sent to IMGC for intercomparison with the Italian laboratories fixed point and standard volt.

The results are presented in Table 2.

Table 2:

Fixed Point	G009 µV	G010 µV	G012 µV	k=2 U/C ±µV
Ag	16,116.9	16,115.3	16,116.9	0.9
Al	9,317.9	9,317.1	9,318.1	0.7
Zn	4,944.3	4,943.7	4,944.4	0.6
Sn	2,235.4	2,235.2	2,235.6	0.6

And lastly; one year after the first calibration Platinum/Gold Thermometer No. G012 was recalibrated at NTPL – see Table 3.

Table 3:

Fixed Point	G012 µK
Ag	16,117.6
Al	9,318.2
Zn	4,943.8
TPW	-0.1

Sensitivities

The Gold/Platinum Thermocouple has the following sensitivities to temperature.

Table 4:

°C	µV/°C
0	6.1
100	9.35
200	11.89
300	14
400	15.8
500	17.5
600	19.16
700	20.78
800	22.38
900	23.97
962	24.94

In graphs 1, 2 and 3 the results of the calibrations at NTPL are compared with those in IMGC at the silver point, the aluminium point and the zinc point.

All show agreement well within the combined uncertainties of the two laboratories. In fact agreement is in the majority of cases within the uncertainties of IMGC above.

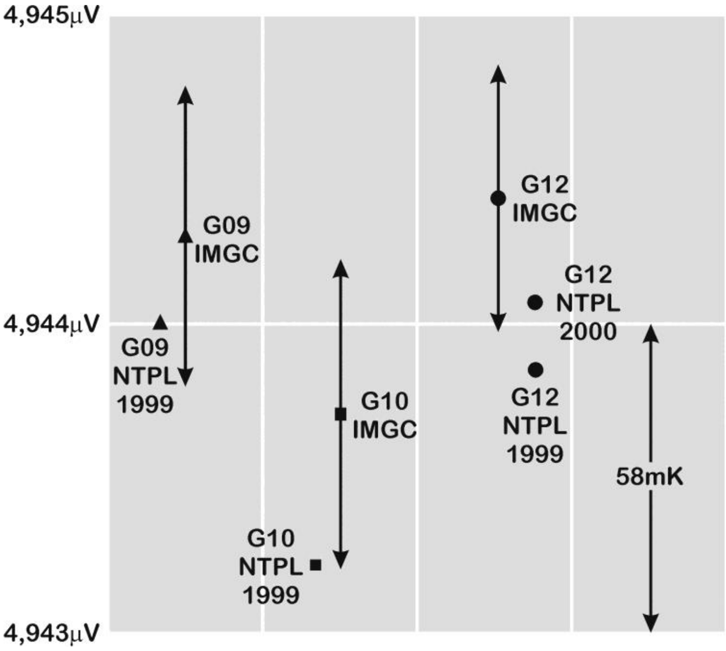
Uncertainty Budget

a) Realisation of Fixed Point ±0.006/2
b) Reproducibility of Thermocouple ±0.02/√3
c) Reference Junction ±0.001/√3
d) DC Voltage including Drifts ±0.041/√3 worse case (at zinc)
e) Polynomial Interpolation ±0.05/√3
f) Stray Thermal Emfs ±0.05/√3

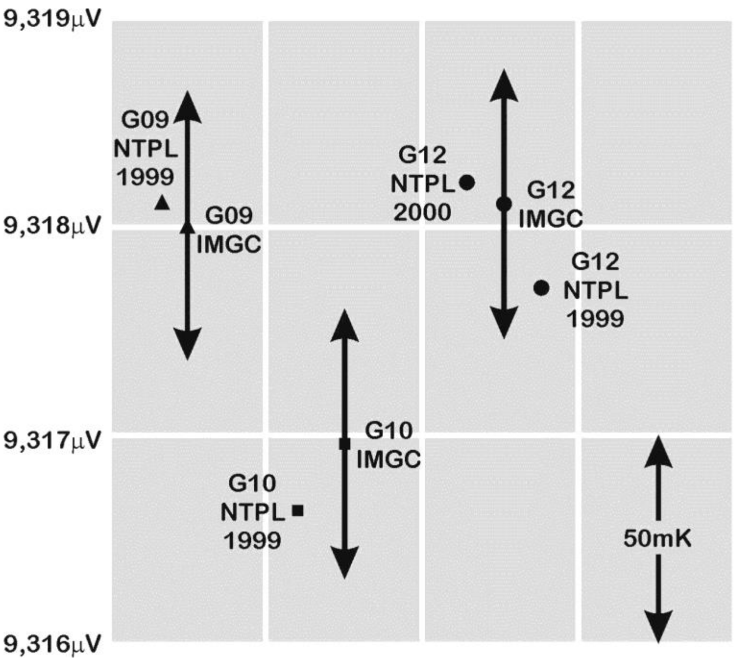
Gives an RSS value (*k*=2) of ±0.094°C or ±0.1°C in round figures.

On December 14th 2000 UKAS wrote agreeing to extend our accreditation to cover Gold/Platinum thermocouples to the above uncertainties.

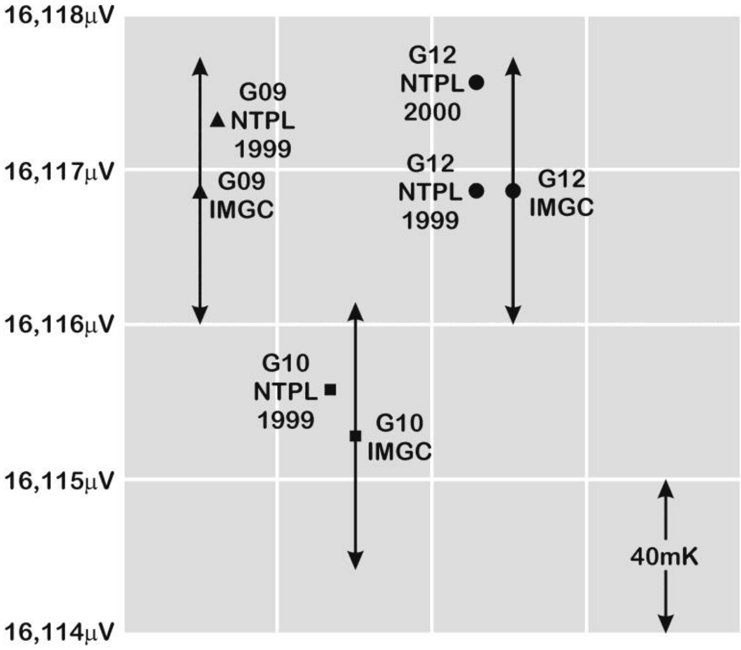
Gold/Platinum Thermocouple Intercomparison NTPL vs IMGC – Zinc Point



Gold/Platinum Thermocouple Intercomparison NTPL vs IMGC – Aluminium Point



Gold/Platinum Thermocouple Intercomparison NTPL vs IMGC – Silver Point



Discussion

With considerable care in construction and annealing, with patience and skill in calibration, the limiting factor in the use of Gold/Platinum thermocouple is the accuracy of the digital voltmeter. This and the allowance for uncertainties in the polynomial make practical uncertainties about 0.1°C 2 sigma. This is some 3 x better than any other thermocouple.

Conclusion

The Gold/Platinum thermocouple is worthwhile considering for accurate temperature measurements over the range 0 to 962°C where uncertainties of ±0.05°C are required. However, close attention needs to be given to the cold or reference junction and to the accuracy of the voltage measuring device.

The Gold/Platinum thermocouple has the potential to reproduce the silver point ±0.01°C from day to day and so if the voltmeter is sufficiently accurate and the uncertainties associated with the polynomial can be reduced this thermocouple has the potential to have smaller uncertainties than are currently claimed.

Note: All work so far has done with platinum and gold of 99.999%.

The 10ppm impurities vary from lot to lot and account for variations in the Emf vs Temperature, being about 20μV or 0.8°C at the silver point. One route towards a more reproducible thermocouple would be to use 99.9999% pure wires. These can be purchased. However it is not exaggeration to say that wire of this purity is so weak mechanically that it becomes impractical to produce a robust thermocouple.

So here is the ultimate limitation to the thermocouple.

Suggested Reading

[1] *G. W. Burns, G. F. Strouse, B. M. Lui and B. W. Mangum.*
Gold versus Platinum Thermocouple: Performance Data and an ITS-90 based Reference Function.
National Institute of Standards and Technology, Gaithersburg, Maryland, 20899 copyright American Institute of Physics.

[2] *E. H. McLaren and E. G. Murdoch*
The Pt/Au Thermocouple Part 1: Essential Performance
Part II: Preparatory Head Treatment, Wire Comparisons and Provisional Scale.
Ottowa, Canada, K1A 0S1

RETURNING YOUR THERMOMETER TO ISOTECH

Due to the fragility of the Gold/Platinum Thermocouple we strongly recommend that this is not transported unless personally hand carried.

In the event that the unit must be shipped by courier then the following instructions must be adhered to.

Packing Instructions for the Gold/Platinum Thermocouple

See illustration below for reference.
Place the two foam blocks over the closed case and lower into inner box. Pad out ends with polystyrene chips to stop the unit from moving - seal the box, lower it into the outer box and fill with chips, then lift the inner box slightly so the polystyrene chips completely surround it. Place the protective sheath if supplied on top of the chips and seal the box.

Always remember to label the box thoroughly with “fragile” and “this way up” labels and arrange adequate insurance cover.

Your unit should now be ready to send.