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## LETTER TO THE EDITOR

# Argon pressure is maintained in an aluminium thermometric fixed-point cell

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**Abstract**
The argon and helium pressure in two types of temperature fixed-point cell enclosure has been measured for over two weeks. It was found that there was no significant change in the argon pressure over this time, in contrast to the report of Ancsin (2003 *Metrologia* **40** 232–4). Helium was found to diffuse out of the cell as expected. We conclude that argon is a suitable ‘back-fill’ gas for fixed-point cells.

## 1. Introduction

The freezing point of aluminium at one standard atmosphere (at 660.323 °C) is one of the fixed-points specified for use in the definition of the International Temperature Scale of 1990 (ITS-90) [2].

The optimal realization of the fixed-point to obtain a precise calibration relies on many factors including the materials of the cell components. The high-purity metal is contained in a pure graphite crucible and this is held in a gas-tight container made from quartz (silica). An inert gas filling is an important part of a fixed-point cell to assist thermal exchange inside the cell, to protect the graphite from oxidation and to prevent metal vaporization. To obtain the specified condition, the pressure must be adjusted or corrected to one standard atmosphere, 101.325 kPa. In the ‘Supplementary Information for the ITS-90’ [3], it is suggested that argon should be used as the internal pure gas in high temperature fixed-point cells.

Recently, however, Ancsin [1] has observed the diffusion of Ar gas out of a quartz housing at high temperature. In his experiments, five different gases (air, He, Ar, $CO_2$, $N_2$) were used to back-fill the Al cell. Each of them showed a drop in pressure over time from the initial value of 1 atm, except nitrogen. Ancsin wondered if the gases, including argon, were being absorbed by graphite or aluminium. After further experiment, he concluded that the greater part of the pressure decreases were due to the permeability of the walls of the quartz tubes at high temperatures, and the best choice among the five gases tested, for use in sealed quartz cells, is nitrogen.

However, although nitrogen is basically non-reactive, it may form nitrides in fixed-point cells at high temperatures. Also data, such as used in thermodynamic calculation programs, show that freezing curves can be depressed due to the solution of nitrogen as an impurity [4]. Consequently, nitrogen should be avoided and it is therefore important to check whether the reported problem with argon is repeatable.

This study investigates the effects of argon and helium being used for back-filling an Al fixed-point cell. Our results show that the argon filling gas did not significantly change its pressure, when used in both a translucent silica tube containing a graphite crucible and in an empty quartz enclosing tube. On the other hand, with helium the pressure did drop, as expected.

## 2. Experimental apparatus

An NPL design of an open Al cell was used for this experiment. The Al sample was contained in a graphite crucible of length 250 mm, which was put into a translucent silica tube of length 530 mm. A single zone Carbolite furnace was used in conjunction with a potassium heat

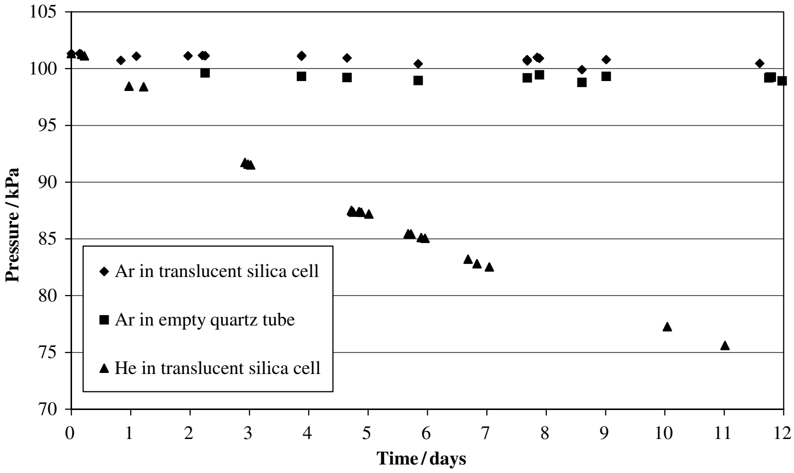

**Figure 1.** Graph of pressure over an Al fixed-point cell and in a separate empty quartz tube as a function of time in days.

pipe for maintaining a uniform temperature above or below 660 °C. A thyristor controller was used to control the furnace temperature, which applied only 120 V (through a transformer) to reduce the electrical noise. The resistance ratios of the platinum resistance thermometer (PRT) measurement were recorded from an ASL F18 resistance bridge, confirming the temperature stability of the fixed-point cell.

A gas handling system was used to set the pressure in the cell to approximately 101.3 kPa. The cell was connected via a copper tube to the system, which was connected by reinforced plastic tube to the vacuum pump (Edwards XDS10 scroll pump) and a Wallace and Tiernan precision pressure gauge. Before the experiments, the whole system was tested using a Leybold Vacuum PhoeniXL 300 helium leak detector.

Argon and helium were the gases used to back-fill the cell in two separate experiments as both gases were conveniently available in the laboratory and both can provide an inert atmosphere for the Al fixed-point cell.

The fixed-point cell was maintained at the operating temperature, either −0.5 °C or +0.5 °C of the equilibrium temperature, which were the settings for the cell to be frozen and molten, respectively, for almost two weeks and the internal pressure was monitored during this time. The results are shown in figure 1.

Starting on day 2, in a separate experiment the permeability of argon through the wall of a clear close-ended quartz tube was investigated. Thus, a quartz tube, without an Al cell, was inserted into a three-zone Hart Scientific furnace and was connected to a duplicate gas handling system. This system was also leak tested. Then, the empty cell was back-filled with gas, sealed up and left to observe if any decrease in pressure occurred at the Al fixed-point temperature. The initial pressure was set at about 99.6 kPa, which was slightly below 101.325 kPa, to ensure that if there was any air leak then it would increase the tube pressure. These results are also shown in figure 1. When air was let into the empty cell at the end of the experiment, then the pressure increased to 1 atm. (In fact, the silica broke on cooling between 660 °C and room temperature.)

## 3. Results

As observed in figure 1, the pressure of argon gas was virtually unchanged during the time at high temperature. After 12 days, the pressure was about 100.3 kPa, only 1 kPa lower, or reduced approximately 1% from the initial pressure. For practical thermometric purposes, this is negligible. It may be due to some argon diffusing out or being absorbed by the graphite or Al, but it might also be a limitation of the pressure measurement system. In any case, the amount of pressure reduction is much less than that reported by Ancsin (approximately 25 kPa over 10 days) [1].

These results indicate that argon gas did not significantly diffuse out of the translucent tube or become absorbed into the aluminium or graphite.

For helium, figure 1 shows that the pressure kept decreasing with time. At the end of the measurement period, the pressure was about 75.6 kPa, i.e. it had dropped by about 25% below its initial value over 12 days, while Ancsin's report showed an 80% reduction within 4 days. That the pressure of the helium should drop is not a surprise, as it is well known [5] that a quartz tube is permeable to helium. The result confirms the ability of our system to detect a pressure drop and that there is no long-term leak from the outside.

The results for the empty quartz tube filled with argon gas, also shown in figure 1, indicate that the pressure had dropped by about 0.87 kPa, which is 1% reduced, over the period. This is similar to the result for the translucent silica cell and indicates that argon does not diffuse significantly through clear quartz at this temperature.

From these results, we surmise that Ancsin may have been using an unusually permeable quartz tube. We wonder if the nitrogen appeared not to diffuse due to back diffusion of nitrogen from the surrounding air. However, we are not able to confirm this hypothesis.

## 4. Conclusions

The changes in pressure in our experiments suggest that argon is not absorbed or lost within the Al cell, in contradiction to the results of Ancsin. Our results showed at most a very small permeation of argon, though the small pressure decrease may have been due to other effects such as limitations of the measurement system. This applies for both the translucent silica and the quartz tubes at the Al fixed-point temperature. Therefore, we conclude that argon is a suitable gas to use to back-fill the Al fixed-point cell.

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