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Realisation of the triple-point of Argon: comparison between two devices

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Abstract

A new cryostat for the realization of the triple-point of the argon (83.8058 K), a defining fixed point of the International Temperature Scale of 1990 (ITS-90), was acquired at Italian National Metrological Institute (INRiM). The new system, manufactured by Fluke, is intended to substitute the current National reference, a model developed at BNM-INM in the 1975. The main difference between the two system is in the way to control the temperature. In the BNM-INM device the temperature is controlled adjusting the pressure of liquid nitrogen bath, in the Fluke system instead, an electrical heater wrapped around the argon cell is used, following cryogenic practice. This paper describes the result of the direct comparison and shows typical phase transitions obtained with the two argon systems. Then, a complete uncertainty budget is evaluated for the new Fluke system and compared with the National standard.

1. Introduction

The triple point of argon (Ar) is one of the defining fixed points ($T_{90} = 83.8058$ K) of the International Temperature Scale of 1990 (ITS-90) [1] and one of the most commonly used for the calibration of standard platinum resistance thermometers (SPRTs). In most European metrology institutes its realization is still based on the device designed by BNM-INM in 1975 [2, 3]. This system is reliable and easy to use which has made it famous and operational all over the world for many years although some improvements have been proposed over time to overcome some of its inconveniences such as the influence of atmospheric-pressure variations and the change in hydrostatic pressure [4, 5].

In recent years, various approaches to the realisation of the triple point of argon have been proposed by other researchers [6, 7] in order to overcome the above inconveniences. Furthermore, two different systems are now commercially available, one from Isotech and the other from Fluke.

The system developed by Fluke Corp. uses the design of the apparatus at NIST [8]. This system has some advantages, such as the possibility to calibrate four long-stem SPRTs simultaneously and exhibiting a longer plateau, thereby reducing the time for customer's calibration.

In this paper, we present the results of a direct comparison between the argon systems from BNM-INM and Fluke. In table 1 is listed some technical information regarding the two devices, better described in section 2. The main difference between them is in the way to control the temperature. In both systems, in fact, the argon cell is immersed in a liquid nitrogen (LN2) bath and the phase transition is realized using the 'permanent heating' method [3] but in the BNM-INM device the temperature is controlled acting on the LN2 pressure, while in the Fluke system uses an electrical heater, wrapped around the argon cell.

In section 2, we report the operating principles of the two systems and show some graphs of typical phase transitions. In section 3, the results of the comparison are illustrated and a complete uncertainty budget is evaluated.

Table 1. Comparison of technical characteristics of the two Ar systems (further information and references in section 2).

Technical characteristics	Ar-IMGC	Ar-Fluke
Ar quantity	2.5 moles	13.3 moles
Ar purity	99.9999%	99.9999%
Ar pressure at room temperature	6 × 10^6 Pa	1.17 × 10^6 Pa
Liquid nitrogen required	50 l	40 l
Height of Ar solid–liquid interface	75 mm	160 mm
Temperature control	By pressure	Electrical heater

2. Methods

2.1. Ar-IMGC system

The INRiM apparatus for the realization of Ar triple-point, hereafter Ar-IMGC, is one of the early devices produced by BNM-INM, serial number 27. It represents the National reference for Ar and it has participated repeatedly in international comparisons: EUROMET.T-K3 (Project 552) [9], CCT Key Comparison CCT-K3 [10], EUROMET Project 502 [11], Regional key comparison EURAMET.T-K9 (Draft B status) and CCT Key Comparison CCT-K9 [12].

The Ar-IMGC system (figure 1) uses an Ar cell totally immersed in a nitrogen bath, having 350 mm height and a volume of 1 dm^3. It contains 2.5 moles of Ar with a nominal purity of 99.9999% produced by Air Liquid Company [13, 14]. The internal pressure is almost 60 bar at room temperature. The performance of a complete experiment requires roughly 50 l of LN2. The distance between the free argon level and the lower end of the thermometer well is 75 ± 5 mm. Further information about the design can be found in [2–4].

To start the phase transition, the temperature of the thermometric cell is set higher than the transition temperature. The temperature of the LN2 bath is controlled by adjusting the pressure inside the cryogenic vessel to about 200 kPa through an external mechanical control valve. Pressurization occurs by applying a set of weights on top of the valve.

The INRiM procedure to realize the Ar triple-point is the following:

1. The SPRT is inserted in the cell when the system is at ambient temperature. The thermometric well is connected to a helium gas (He) reservoir to have a positive He flow ($p \geqslant 100$ kPa) in the well to avoid moisture condensation. The same flux is maintained also during change of thermometer.
2. The dewar is then filled with liquid nitrogen. When the thermometer displays a temperature below the argon triple point, the dewar is considered fully filled with liquid nitrogen.
3. After filling of the dewar with liquid nitrogen and the closure of the system, one waits for about 2 h in order to allow the pressure inside cryostat to stabilize to the optimal value.

A typical plateau is shown in figure 2. For about 5 h, the temperature is stable within 0.30 mK. At times, a better stability of order 0.05 mK is obtained - as observed between the 4th and the 7th hour after the Ar system closure. After these 5 h, temperature dropped brusquely by 30 mK and changing erratically, meaning that the system exited the plateau. Therefore, we define as 'plateau' the region between 2.5th and 7.5th hour. In this time, we have a good stability of the phase transition and therefore the best period for sensor calibration.

The maximum at the beginning of the plateau is due to the procedure for realizing the phase transition. Indeed, by adjusting the pressure inside to the cryogenic vessel at about 200 kPa the temperature results higher than Ar transition temperature.

2.2. Ar-Fluke system

The Ar system acquired by INRiM is the model 5960A-EU s/n AR-60024 manufactured by Fluke Corp., hereafter Ar-Fluke. It contains an amount of 13.3 moles of Ar of high-purity grade (99.9999%), as declared by the manufacturer, at a pressure of 1.17×10^6 Pa at room temperature. The Ar gas is stored in two reservoir cylinders with a volume of 13.4 l each [15]. When the system is on the triple-point plateau, about 90% of the Ar gas is condensed inside the central cell. The system utilizes LN2, almost 40 l, in a Dewar vessel surrounding the Ar cell, while a thermal shield is placed between the LN2 bath and the Ar cell. The cryostat exhibits 4 re-entrant wells that allow the calibration of four SPRTs in sequence on the same plateau. The Ar cell depth is 160 mm, the

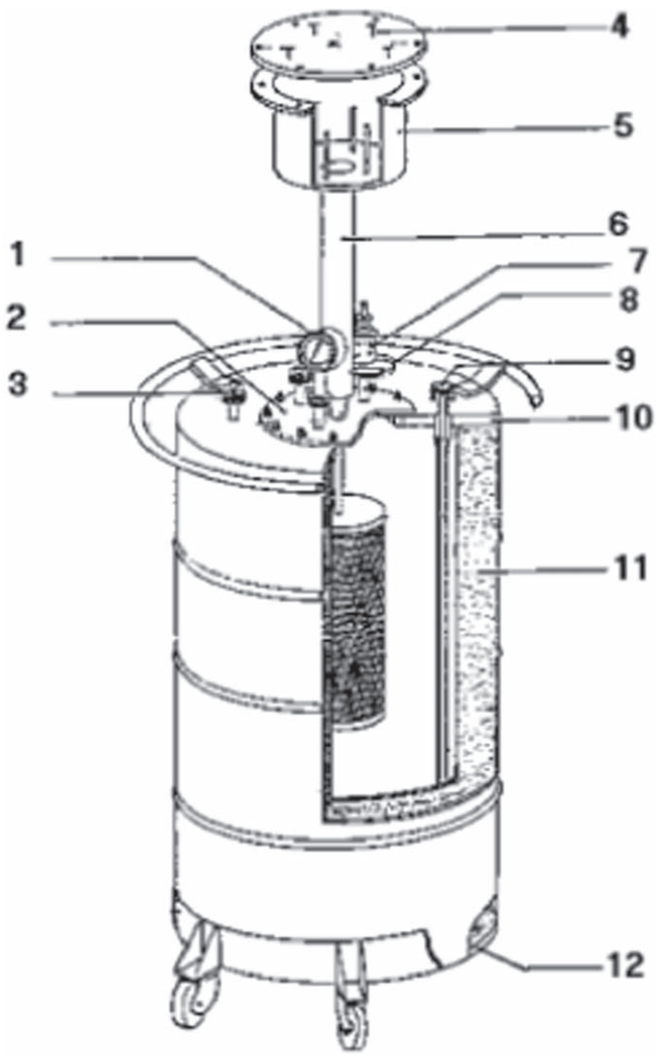

Figure 1. The Ar triple point apparatus [4]. (1) manometer, (2) flange, (3) LN2 outlet, (4) feedthroughs, (5) box for thermometer leads, (6) thermometer well, (7) pressure valve, (9) LN2 inlet, (11) dewar insulation. Reproduced with permission from [4].

inside diameter of the re-entrant wells is 8 mm. The characteristics of the cryostat and the working principles are illustrated in [15].

The main structure of the triple-point-of-argon system is shown in figure 3 [15]. A temperature controller is used with a sensor attached on the inside surface of the heater shield to control its temperature. This way the plateau is achieved without using pressure controls (the pressure during the operation is always kept constant) unlike the Ar-IMGC system.

He gas is used as exchange gas to improve heat transfer between the re-entrant wells and the SPRTs and also between the Ar cell and the heating shield. Additionally, during the insertion of SPRTs, the He gas flows through the re-entrant wells to avoid air to enter the re-entrant wells. Silicon rubber O-rings are used to seal the re-entrant wells with their SPRT from ambient.

The procedure for triple-point realization in the user manual prescribes that, each time the system is used, it is pumped down using a vacuum pump to purge all latent gases or moisture, and then filled, at room temperature, with fresh He gas up to approximately 34.5 kPa. The He pressure is adjusted before the phase transition realization in order to compensate for the variation in temperature. The second step is to fill the system with LN2 one day in advance in order to freeze the whole system. It takes about eight hours for the Ar cell to be completely frozen into the central cell and the temperature to equilibrate to LN2 temperature (−196.7 °C). As the third step, the shield temperature is set to − 188 °C for about 1 h to pre-melt the outer layer of the Ar cell allowing to obtain a stable and flat triple-point plateau. In the fourth step, the Ar system temperature is lowered to a maintenance temperature at −189.312 °C, i.e. 32 mK higher than the triple-point of Ar (including compensation for controller off-set).

Figure 4 shows an Ar plateau obtained with the Fluke system. The long duration was achieved with daily nitrogen refilling (downwards peaks in the figure). Apart from the unusual overall noise during this particular

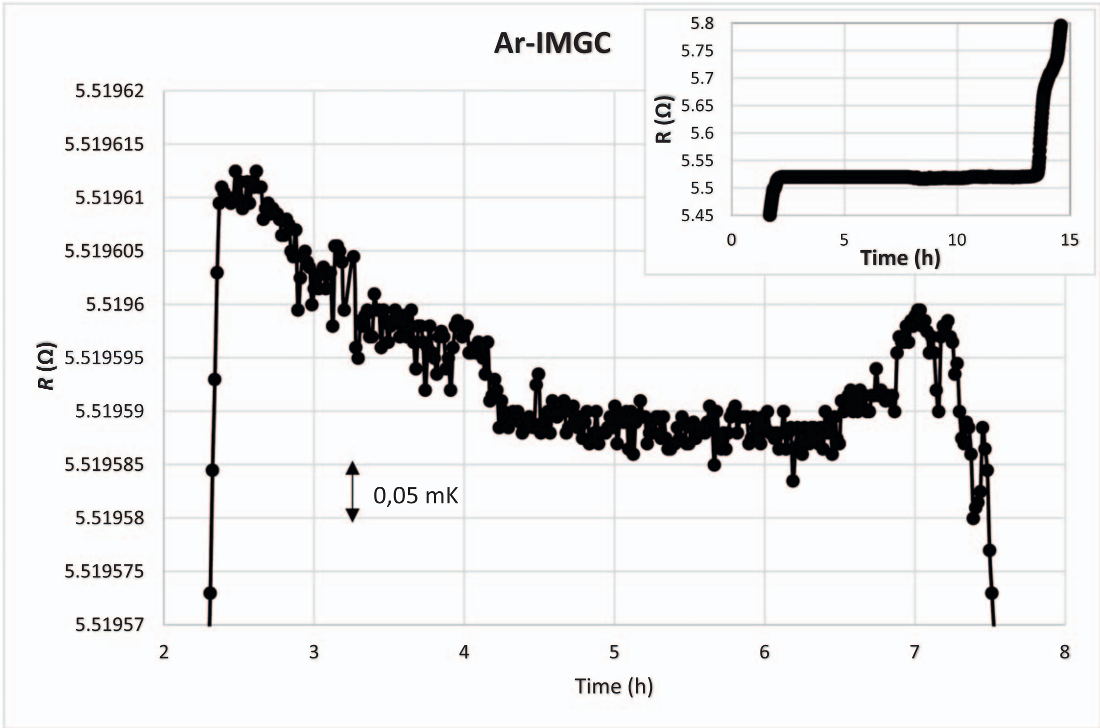

Figure 2. Ar-IMGC plateau recorded on 13 January 2021. In the insert, the whole phase transition. Recording starts immediately after the closure of the system.

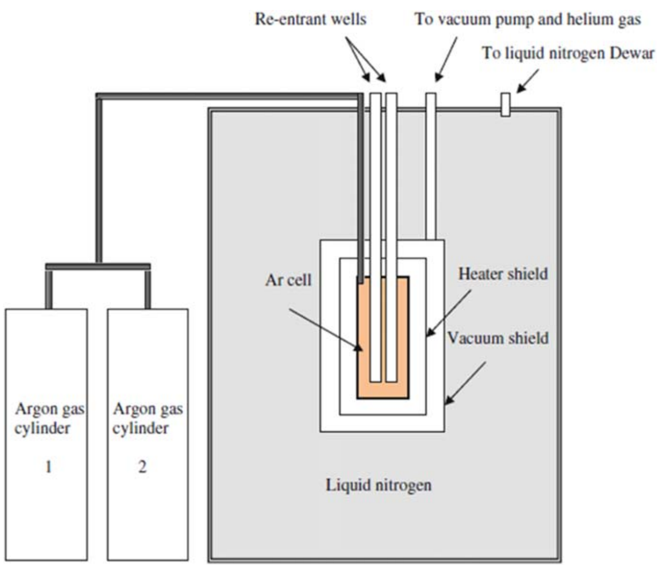

Figure 3. Schematic drawing of the triple-point-of-argon system. Reproduced with permission from [15].

transition, the plateau is stable within 0.10 mK for more than 10 h, and an even better stability is obtained for smaller time intervals.

The possibility to re-fill the cryostat with LN2 during phase transition without inducing a major perturbation in temperature (the system is able to compensate the variation in a few minutes) increases the plateau duration, especially in comparison with the Ar-IMGC system.

2.3. Comparison method

In order to compare the performance of the new argon system, Ar-Fluke, with the national standard, Ar-IMGC, an internal comparison, was organized. The data were taken with a 25.5 Ω long-stem SPRT, Hart Scientific model 1283, normally used as the lab check thermometer for the Ar triple-point. A Standard Resistance bridge

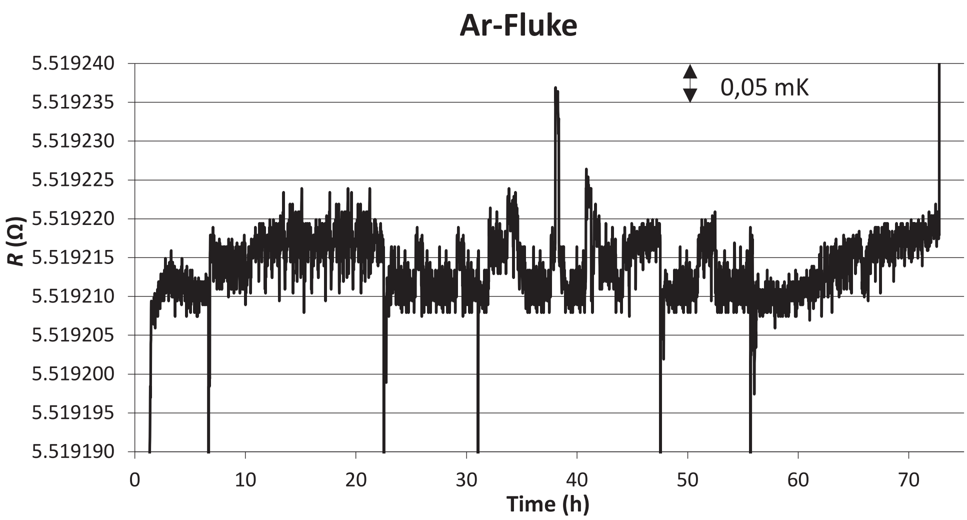

Figure 4. An Ar-Fluke plateau, recorded on 22 November 2022. The record starts when the melting temperature is settled.

ASL F18 was used to read the data. The reference resistance employed with the bridge was a 100 Ω Tinsley model 5685A enclosed in its thermal case. Unfortunately, the non-optimal choice of this reference resistor caused a loss in bridge resolution of one order of magnitude.

The comparison ran in two assessment rounds, the first in January 2021 and the second in January 2022. The same thermometer was used during both rounds. The only difference was in the realization sequence of the plateaus. In the first round, one Ar system was used per day, so, the first day of the comparison the plateau was induced in Ar-IMGC, the second day in Ar-Fluke, and so on. In total, 4 plateaus were recorded, two for each system, and during each plateau, the SPRT resistance was determined several times on plateau.

In the second round instead, the plateaus were induced in the two cells at the same time switching quickly from one argon system to the other. Four plateaus were performed in both systems in 4 different days. During each plateau, the resistance of the SPRT was determined several times for each cell, alternating the two cells in measurement.

The measurement current was 2 mA and the self-heating effect was determined switching the current to 2.83 mA. The results given here are fully corrected for thermometer self-heating and for immersion depth. The hydrostatic correction is equal to −0.000019 Ω (0.19 mK) for Ar-IMGC and equal to −0.00005 Ω (0.5 mK) for Ar-Fluke. This correction was calculated considering a distance between the free level of the Ar and the lower end of the thermometer well of 7.5 cm for Ar-IMGC and 16 cm for Ar-Fluke system, while the distance between the center of the sensing element and the lower end of thermometric well is 2.3 cm.

3. Results and discussion

3.1. Recorded plateaus during comparison

Figure 5 shows the plateau recorded for the Ar-IMGC system on the second day, 20 January 2021. The whole phase transition was recorded and the breaks in the graph correspond to the determination of the zero-current resistance (R_0) values used for the comparison of the two systems. The first value of R_0 was not considered for the comparison because the transition was not stable yet. The plateau was stopped at the fifth hour, and the maximum variation in the day was about 0.4 mK.

Figure 6 shows another plateau with Ar-IMGC recorded during the second round of assessment, 20 January 2022 (9th day). We followed the beginning of plateau and then stopped recording to make the first determination of the day, then the same SPRT was moved to Ar-Fluke and then back to make the second determination followed by the end of the transition. Also in this case, the maximum variability of the plateau, between the 2.5 and 7.5 h, is about 0.35 mK.

This plateau was recorded one year after those of figures 2 and 5, so the (absolute) differences in R_0 could be due to thermometer drift.

Figures 7 and 8 show similar graphs as figures 5 and 6, but for the Ar-Fluke system. Figure 7 gives the results from the first assessment round. The whole plateau was recorded with SPRT Hart 1283 where the breaks are due to the R_0 determinations (3 values on 21 January 2021).

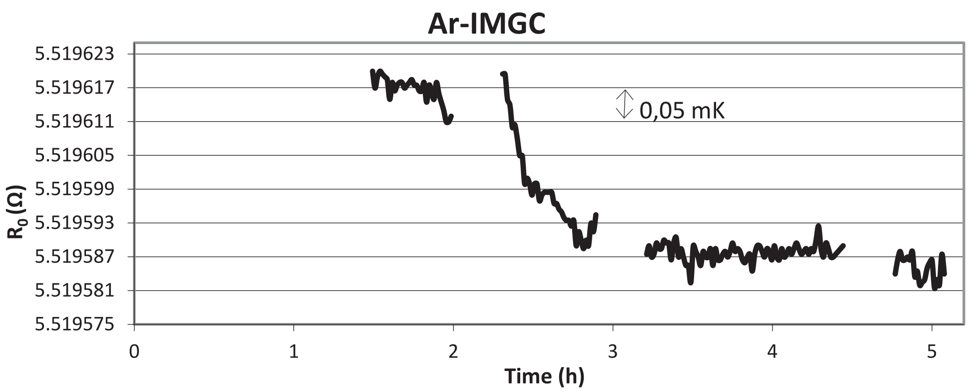

Figure 5. Triple-point plateau for Ar-IMGC, recorded on 20 January 2021. The record starts 1 h after the closure of the system.

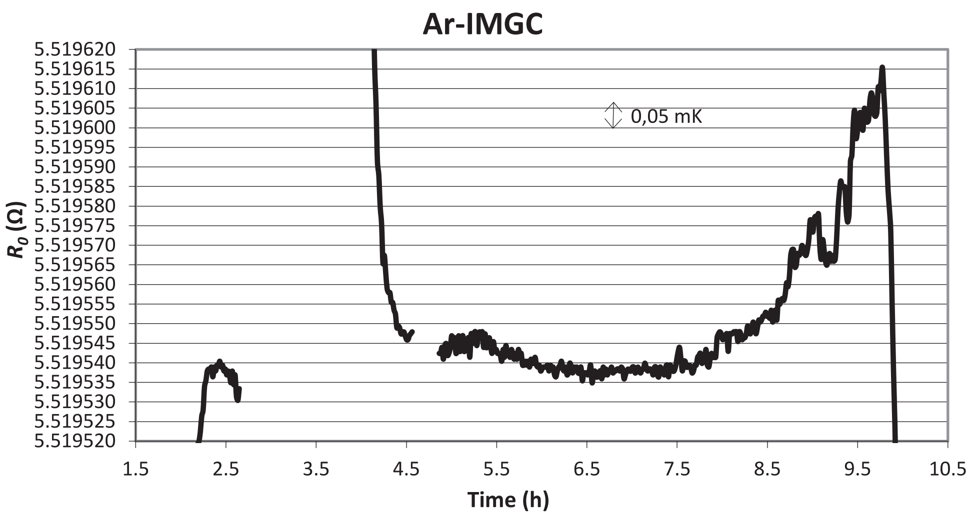

Figure 6. Triple-point plateau for Ar-IMGC. Data recorded 20 January 2022. The record starts immediately after the closure of the system.

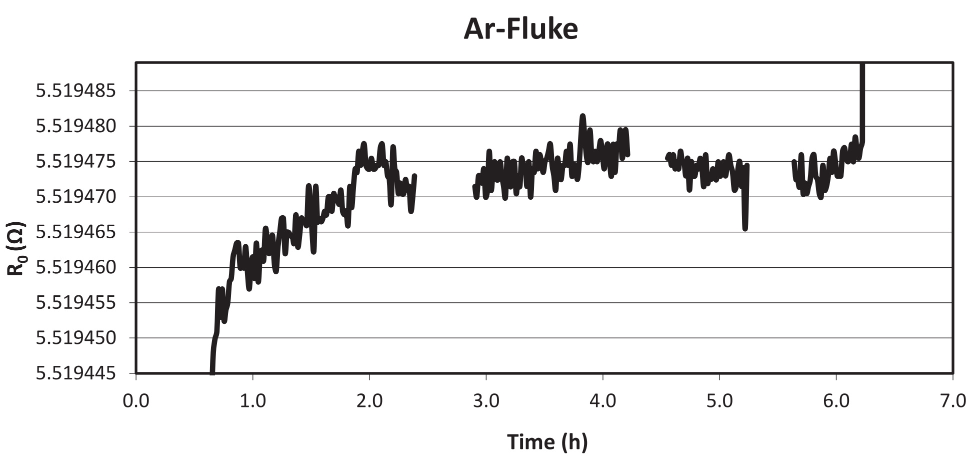

Figure 7. Triple-point plateau for Ar-Fluke. Data recorded 21 January 2021. The record starts 30 min after settling of the melting temperature.

The graph in figure 8 instead was recorded with another thermometer: SPRT 25.5 Ω model 1218 long stem from Hart Scientific, kept in one of the free wells. A system from Measurement International, type 6010 B, performed the recording. In fact, during the second round of the assessment the SPRT Hart 1283 was moved

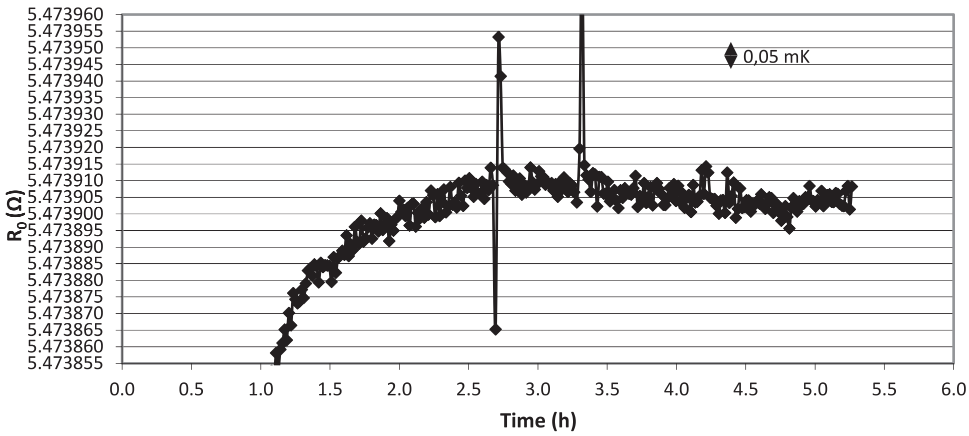

Figure 8. Triple-point plateau for Ar-Fluke. Data recorded 19 January 2022 by SPRT Hart 1218. The record starts when the melting temperature is settled.

Table 2. Results of the comparison between Ar-Fluke and Ar-IMGC.

Data	Day number	Ar system	R_0 (Ω)	ΔR_0 Fluke-IMGC (μΩ)	ΔT Fluke-IMGC (mK)
19-Jan-21	1	Ar-Fluke	5.51884	−37.9	−0.35
19-Jan-21	1	Ar-Fluke	5.518843		
20-Jan-21	2	Ar-IMGC	5.518893		
20-Jan-21	2	Ar-IMGC	5.518872	−29.2	−0.27
20-Jan-21	2	Ar-IMGC	5.518873		
21-Jan-21	3	Ar-Fluke	5.518839		
21-Jan-21	3	Ar-Fluke	5.518846		
21-Jan-21	3	Ar-Fluke	5.518841		
22-Jan-21	4	Ar-IMGC	5.518847		
13-Jan-22	5	Ar-IMGC	5.518873	−19.2	−0.17
14-Jan-22	6	Ar-IMGC	5.518909		
14-Jan-22	6	Ar-Fluke	5.518883		
14-Jan-22	6	Ar-IMGC	5.518923		
18-Jan-22	7	Ar-IMGC	5.518892	−36.4	−0.33
18-Jan-22	7	Ar-Fluke	5.518868		
18-Jan-22	7	Ar-IMGC	5.518917		
19-Jan-22	8	Ar-IMGC	5.518904	−23.8	−0.22
19-Jan-22	8	Ar-Fluke	5.51888		
20-Jan-22	9	Ar-IMGC	5.518901	−21.9	−0.20
20-Jan-22	9	Ar-Fluke	5.518879		
20-Jan-22	9	Ar-IMGC	5.518901		
	Mean			**−28.06**	**−0.26**

from one Ar system to another to make the R_0 determinations. The two spikes in the graph are due to perturbation of the Ar-Fluke system temperature due to the insertion and removal of the SPRT Hart 1283. The determination of R_0, for the comparison, was performed during this interval. The use of another thermometer does not influence the comparison results significantly because this graph serves only to visualize the transition trend, to see the noise level and/or possible perturbations in the plateau.

3.2. Comparison Ar-Fluke versus Ar-IMGC

Table 2 reports all results of the comparison: column 1 gives the day of measurement, column 2 the progressive day number (to make the graphs easier to read), and column 4 the R_0 for each system. Columns 5 and 6 report the difference between Ar-Fluke and Ar-IMGC, both in resistance (ΔR_0) and in temperature (ΔT), respectively. The differences are evaluated, in the first round, as the mean value between data recorded in consecutive days, while in the second round as the mean difference between data recorded in the same day. Finally, the mean values were calculated.

The transition temperature for Ar-Fluke is, on average, 0.26 mK lower than for Ar-IMGC with a (purely statistical) standard deviation of 0.07 mK.

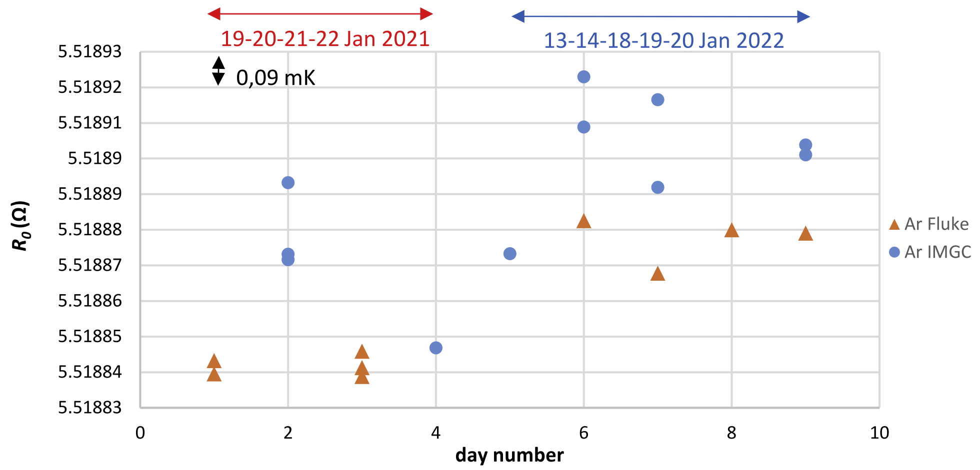

Figure 9. Zero current resistance measured during each day of the comparison.

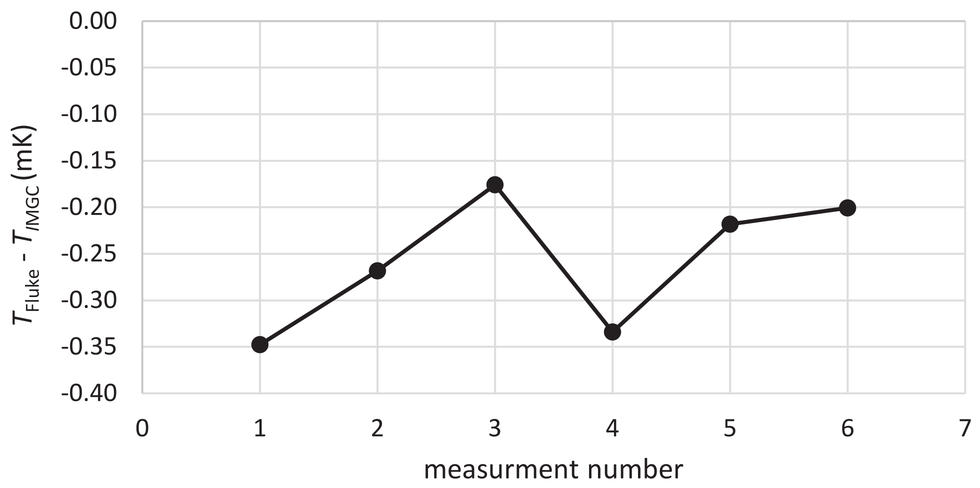

Figure 10. Difference in transition temperature between Ar-Fluke and Ar-IMGC systems. The *x*-axis represents the progressive measurement sample.

A graph of R_0 is given also in figure 9. It shows a shift in data recorded in the second round of assessment. This general increase in resistance is attributed to thermometer drift (no check at the triple point of water).

Figure 10 reports the difference in transition temperature between Ar-Fluke and Ar-IMGC, obtained from the measurements carried out during the comparison (column 6 table 2).

3.3. Uncertainty budget

In this section, three different uncertainty budgets are evaluated: one pertaining to the triple-point realization with each Ar system and the uncertainty of the comparison.

Table 3 reports the complete uncertainty budget for the realization of the Ar triple-point with the two systems.

The first block ('Cell') is related to those items pertaining to the cell only, it includes the following terms:

- The main item 'Impurities' accounts for temperature change due to the presence of impurities in the argon gas. It is equal for the two systems having the same nominal purity grade.
- 'Variability of the plateau realization': accounts for the differences between the fixed-point temperature evaluated from repeated plateaus. It is the standard deviation, converted in temperature, of all R_0 values recorded–for each system - during the comparison, with the same SPRT.
- 'Hydrostatic head' accounts for the hydrostatic pressure effect on the equilibrium temperatures, considering only the vertical dimensions, with an overall uncertainty of 0.8 cm.

Table 3. Uncertainty budget for the realization of the Ar triple-point with the Ar-IMGC and Ar-Fluke systems.

Source of uncertainty	Contribution to the combined standard uncertainty/mK	
	Ar-IMGC	Ar-Fluke
Cell:		
Chemical impurities (type B)	0.028	0.028
Variability in plateau realization (type A)	0.23	0.177
Hydrostatic head (type B)	0.016	0.016
Heat flux (type B)	0.115	0.015
Slope of plateau (type B)	0.112	0.045
Re-entrant wells consistency (B)	0	0.098
Resistance Measurement:		
Bridge (type A)	0.042	0.042
Reference resistor stability (type A)	0.054	0.054
Thermometer Self-heating (type B)	0.022	0.022
Electrical noise (type A)	0.029	0.058
Combined standard uncertainty, ($k = 1$)	**0.29**	**0.23**
Expanded uncertainty, ($k = 2$)	**0.58**	**0.46**

Table 4. Uncertainty budget for the comparison of the Ar systems.

Source of uncertainty	Contribution to the combined standard uncertainty / mK
Standard deviation of the comparison	0.07
Variability in plateau realization	0.23
Bridge repeatability	0.016
Reference resistor stability	0.054
Thermometer Self-heating	0.022
Electrical noise(type A)	0.058
Combined standard uncertainty, ($k = 1$)	**0.25**
Expanded uncertainty, ($k = 2$)	**0.51**

- 'Heat flux' term was obtained from the temperature change when temporarily increasing the cryostat temperature while on plateau.
- 'Slope of the plateau' is the uncertainty component linked to the variation of temperature measured during a day long comparison. In other words, it is the mean difference, for each system, between the first and the last measurement on the same day. A rectangular probability distribution is assumed.
- 'Re-entrant wells consistency' is linked to temperature inhomogeneity between thermometer walls. It was evaluated only for Ar-Fluke having 4 wells. The SPRT was moved between wells on the same plateau.

The second block, 'Resistance Measurement', reports the terms related to the determination of resistance:

- 'Bridge' includes all terms linked to the bridge (resolution, accuracy, non-linearity, AC quadrature). The terms are the same because in both cases the same ASL F18 Resistance Bridge was used.
- 'Reference resistor stability' considers the effects of slight temperature variations of the standard resistor. It is equal for both systems.

- 'Thermometer self-heating' term reflects the uncertainty in the extrapolation of the SPRT resistance to zero current.
- 'Electrical noise' term considers the noise present while on plateau.

Based on these calculations, the Ar-Fluke system presents only a slightly smaller expanded uncertainty for the Ar triple-point realization.

Regarding the direct comparison of the two Ar systems, its expanded uncertainty is evaluated equal as 0.51 mK, see table 4. This relatively high value is mainly due to the higher variability of the Ar-IMGC plateaus.

4. Conclusions

A new cryostat, Ar-Fluke, was acquired at INRiM for the ITS-90 realization of the triple-point of Ar and it was compared with the National standard, Ar-IMGC. The new cryostat presents a different working principle compared to Ar-IMGC: the temperature is controlled by an electrical heater, following cryogenic practice, instead of acting on the LN2 pressure. The new design allows some important advantages in comparison with the older system: longer phase transition plateaus and reduced customer's calibration time, due to 4 re-entrant walls for calibration of several SPRTs on the same plateau.

The Ar-Fluke temperature is, on average, 0.26 mK lower than that Ar-IMGC, with an expanded uncertainty of the comparison equal to 0.51 mK ($k = 2$). Therefore, the difference being well within the uncertainty, two systems measure essentially the same Ar triple point temperature. The uncertainty in Ar-triple point realization is slightly smaller for Ar-Fluke ($U = 0.46$ mK) than for Ar-IMGC ($U = 0.58$ mK).

The predominant contribution to the uncertainty in the difference is due to the variability of the Ar-IMGC system. This could be attributed to the influence of atmospheric-pressure variations or heat flux variations along thermometric well, as pointed out by other authors [3–5].

The next step is to participate in an international comparison in order to confirm the Ar-Fluke uncertainty budget and substitute the national standard for the Ar triple-point realization.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Conflicts of interest

The authors have declared no conflict of interest.

Compliance with ethics requirements

This article does not contain any studies with human or animal subjects.

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