# Comparison of the Josephson Voltage Standards of the INTI and the BIPM

(part of the ongoing BIPM key comparison BIPM.EM-K10.a)

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Abstract. A comparison between the Josephson array voltage standard of the Bureau International des Poids et Mesures (BIPM) and that of the Instituto Nacional de Tecnología Industrial (INTI), Argentina, was made at the level of 1.018 V in November 2011. For this comparison, both options of the BIPM.EM-K10.a comparison protocol were applied (A and B). Option B required the BIPM to provide a reference voltage for measurement by the INTI using its Josephson standard and measuring device. Option A required the INTI to provide a reference voltage for measurement by the BIPM using its analogue detector and associated measurement loop. In each case the BIPM array was kept floating from ground.

The final result observed is in good agreement with the combined relative standard uncertainty of 1.98 parts in 10<sup>9</sup> for the nominal voltage of 1.018 V.

#### 1. Introduction

Within the framework of CIPM MRA key comparisons, the BIPM performed a direct Josephson voltage standard (JVS) comparison with that of the INTI, Argentina, in November 2011.

The comparison followed the technical protocol of BIPM.EM-K10.a comparisons and followed options A and B of the protocol. This involved the BIPM measuring the voltage of the INTI JVS using its measurement loop with an analogue voltmeter as a detector for option A and the INTI measuring the voltage of the BIPM transportable JVS (BIPM JVS) using its own measurement chain for option B. The BIPM JVS was shipped to INTI, San Martin, Argentina, where an on-site direct comparison was carried out from 26 November to 2 December 2011.

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For both protocol options, the BIPM array was kept floating from ground and was biased on the same Shapiro constant voltage step for each polarity, which was necessary to maintain stability during the timeframe required for the measurement acquisition.

This article describes the technical details of the experiments carried out during the comparison.

## 2. Comparison equipment

#### 2.1 The BIPM JVS

In this comparison the BIPM JVS comprised a cryoprobe with a Hypres 10 V SIS array (S/N: 2538E-7), microwave equipment and the bias source for the array. The Gunn diode frequency was stabilized using an EIP 578B counter and an ETL/Advantest stabilizer. During the measurements, the array was disconnected from this instrument. The measurements were carried out without monitoring the voltage across the BIPM JVS. The RF biasing frequency was adjusted to minimize the theoretical voltage difference to zero between the two JVS.

The series resistance of the measurement leads was less than 4  $\Omega$  in total and the linear evolution of the thermal electromotive forces (EMFs) was eliminated by polarity reversal of the arrays. The leakage resistance between the measurement leads was greater than 5  $\times$  10<sup>11</sup>  $\Omega$  for the BIPM JVS.

#### 2.2 The INTI JVS

The main characteristics of the INTI JVS measurement set-up were:

- Resistance of the precision measurement leads:  $< 4 \Omega$  in total
- Leakage resistance between the precision measurement leads:  $> 1 \times 10^{11} \Omega$
- Josephson junction array: PTB SIS 1.2 V (S/N: Me 143/11)

• Bias source: PTB 3-91

Software: INTI-WINJVS version 2.0

Detector: HP34420A (S/N: US36002419)

## 3. Comparison procedures - Option B at 1.018 V

#### 3.1 First measurements

The BIPM JVS was set-up and checked for trapped flux. The array was biased to RF frequency  $f = 75.518\ 010\ \text{GHz}$  (n = 6519) to minimize the theoretical voltage difference between the two JVS.

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The INTI JVS was biased at f = 69.968~999~500~GHz (n = 7036). It is important to note that the INTI phase-lock loop was unable to achieve a frequency dispersion better than 100 Hz within a variation interval of  $\pm$  200 Hz.

The grounding configuration between the two systems was investigated, and only one configuration was found where both quantum voltages were stable at the same time. This configuration is explained as follows:

The voltage reference point of the INTI JVS reaches the BIPM JVS through the shielding to the leads that connect both systems. Furthermore, the BIPM probe and dewar were grounded.

Ten measurements were made and the following preliminary result obtained:

 $(U_{\rm INTI}-U_{\rm BIPM})$  /  $U_{\rm BIPM}$  = -1.06 × 10<sup>-9</sup> with an experimental standard deviation of the mean of 1.6 × 10<sup>-9</sup> V.

This comparison result proves that the two standards were in good agreement. The stability achieved on the two JVS, even when connected together, was acceptable. Therefore, during the remaining period of the comparison, many experiments and measurement configurations were tested to achieve a lower voltage difference between the two JVS and a lower Type A uncertainty. Details of the experiments are described in Appendix A.

### 3.2 Description of the INTI measurement procedure

The BIPM array was disconnected from its bias source during the entire data acquisition process. The reference voltage of the chassis of the instruments that constituted the BIPM JVS was isolated from the reference voltage chosen for both JVS. To achieve this, the shielding of the biasing leads of the BIPM JVS was disconnected. The two arrays were connected in series-opposition via a dedicated switch (BIPM) and a polarity switch (INTI). The INTI reversal switch comprised a massive copper block in which the contact was realized by using copper bolts inserted into dedicated holes in the block. Due to the stabilization time of the thermal electromotive forces (EMFs), the same configuration was kept for the INTI switch and to open/close the measurement loop with the BIPM switch. In this comparison scheme (option B), the INTI JVS measurement setup measured the BIPM voltage as if it were a Zener voltage standard. During the comparison, only the biases of the two arrays were reversed, no mechanical switch reversal occurred. This operation was carried out manually on both JVS. Polarity reversal was typically completed in less than 5 s.

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## 3.2.1 Measurement procedure for the preliminary measurements (Option B)

The measurement loop was arranged so that both positive polarities of the arrays were connected together and the voltmeter (HP34420A, 1 mV range) was placed between the two negative polarities of the arrays. The "High" of the voltmeter was connected to the INTI array. An estimate of the theoretical voltage of the BIPM JVS was calculated using INTI software prior to the JVS being set to 1.018 V. A measurement point was acquired using the following procedure:

- 1 Positive polarity of the arrays;
- 2 Data acquisition of 5 points at NPLC=20;
- 3 Negative polarity of the arrays;
- 4 Data acquisition of 10 points at NPLC=20;
- 5 Positive polarity of the arrays;
- 6 Data acquisition of 5 points at NPLC=20.

<u>Note</u>: every measurement point is the mean value of 3 readings and every new reading must not differ from the previous one by more than a value defined by the operator, or the program will attempt to read a suitable value.

#### 3.2.2 Measurement procedure for the measurements following Option A.

The measurement loop was modified: both positive polarities of the arrays were still connected but a nanovoltmeter (EM N11,  $10~\mu V$  range) was placed between the two negative polarities of the arrays. The "High" of the nanovoltmeter was connected to the INTI array. The equipment included a voltage divider to prevent the detector from overload if both systems diverged from the same selected steps. The measurement software set-up was changed as follows:

- 1 Positive array polarity and reverse position of the detector;
- 2 500 data readings acquisition;
- 3 Positive array polarity and normal position of the detector;
- 4 500 data readings acquisition;
- 5 Negative array polarity and reverse position of the detector;
- 6 500 data readings acquisition;
- 7 Negative array polarity and normal position of the detector;
- 8 500 data readings acquisition;

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- 9 Negative array polarity and reverse position of the detector;
- 10 500 data readings acquisition;
- 11 Negative array polarity and normal position of the detector;
- 12 500 data readings acquisition;
- 13 Positive array polarity and reverse position of the detector;
- 14 500 data readings acquisition;
- 15 Positive array polarity and normal position of the detector;
- 16 500 data readings acquisition.

<u>Note:</u> Significantly more data could be read from the analogue detector compared to a digital detector during the same time window.

During the measurement process, the BIPM bias source was adjusted to manually select the same step after each polarity reversal. After each polarity reversal 10 seconds elapsed before beginning the data acquisition to avoid filter capacitor discharge effects.

Note: It was not possible to use a lower scale on the analogue detector because of a voltage offset that occurred when the reading command was sent on the IEEE 488 line. More details are provided in Appendix A.

#### 4. Uncertainties and results

### 4.1 Option B protocol

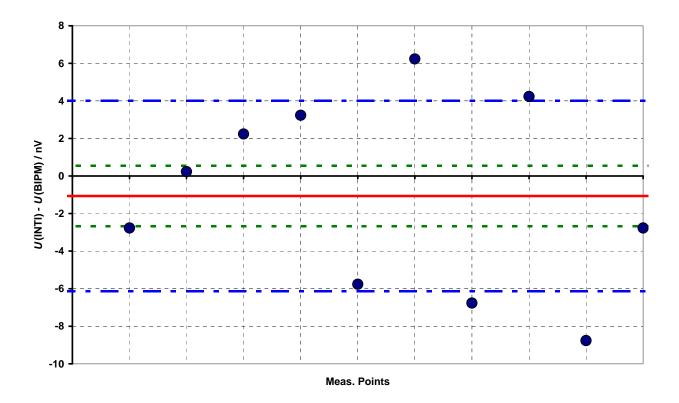
#### 4.1.1 Final result

The result using option B, expressed as the relative difference between the values attributed to the 1.018 V BIPM JVS ( $U_{BIPM}$ ) by the INTI JVS measurement set-up ( $U_{INTI}$ ) is:

$$(U_{\rm INTI} - U_{\rm BIPM}) / U_{\rm BIPM} = -1.06 \times 10^{-9}$$
 and  $u_{\rm c} / U_{\rm BIPM} = 1.98 \times 10^{-9}$ ,

where  $u_c$  is the total combined standard uncertainty and the relative Type A is  $u_A / U_{BIPM} = 1.6 \times 10^{-9}$ . All 15 individual measurements computed to calculate the final result are presented in Fig. 1.

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**Fig. 1**: Individual results obtained to calculate the option B comparison result at the level of 1.018 V. The solid line represents the mean value, the dotted dashed lines (- - - -) represent the experimental standard deviation, and dotted lines (- - -) are the experimental standard deviation of the mean.

## 4.1.2 Type B uncertainty components (option B protocol)

The sources of Type B uncertainty (Table 1) are: the frequency accuracy of the Gunn diodes, leakage currents, and detector gain and linearity. Most of the effects of detector noise and frequency stability are already contained in the Type A uncertainty. The frequency stability of the INTI RF source is the limiting factor in this comparison. A stability of ±100 Hz corresponds to an uncertainty of 1.5 nV. Both array polarities were reversed during the measurements, so the effect of the residual thermal EMFs (i.e. non-linear drift) and electromagnetic interferences are already contained in the Type A uncertainty of the measurements. Uncertainty components related to RF power rectification and sloped Shapiro voltage steps are considered negligible as no such physical effect was observed.

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		Relative uncertainty	
	Туре	BIPM	INTI
Frequency offset (A)	В	$8.0 \times 10^{-13}$	$8.25 \times 10^{-12}$
Leakage resistance (B)	В	$5.0 \times 10^{-12}$	2.31 ×10 <sup>-11</sup>
Detector (C)	В		1.16 ×10 <sup>-9</sup>
Total (RSS)	В	5.1 × 10 <sup>-12</sup>	1.16 × 10 <sup>-9</sup>

**Table 1:** Estimated Type B relative standard uncertainty components.

(A) Both systems refer to the same 10 MHz frequency reference, therefore only a Type B uncertainty from the frequency measured by the EIP is included. The frequency reference used for the comparison was provided by the INTI frequency standard (a caesium oscillator).

**BIPM JVS**: It has been demonstrated on many occasions that the EIP-578B is a very good frequency locker and the accuracy of the frequency can reach 0.1 Hz. Assuming a rectangular distribution, the relative uncertainty for the offset of the frequency can be calculated from the formula:  $u_f = (1/\sqrt{3}) \times (0.1/75) \times 10^{-9} = 8 \times 10^{-13}$ .

#### **INTI JVS**:

- (B) Assuming a rectangular statistical distribution, the relative uncertainty contribution of the leakage resistance  $R_L$  can be calculated from the formula:  $u_L = (1/\sqrt{3}) \times (r/R_L)$ . The values attributed to resistance have been measured during the comparison exercise on both JVS. The resistance of the INTI-JVS leads was measured prior to the comparison. Note:  $r = 4 \Omega$  for the INTI JVS.
- (C) A large proportion of the detector uncertainty is already contained in the Type A uncertainty of the measurements. This component expresses only uncertainty on the gain of the detector. The gain was not measured during the comparison procedure and no data are available on its spread. Therefore, an expansion factor of two to a typical uncertainty value for a similar device, 0.58 nV was applied [1].

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## 5. Discussion and conclusion

The results of the comparison are as follows:

The preliminary comparison result  $(U_{\text{INTI}} - U_{\text{BIPM}}) / U_{\text{BIPM}} = -1.06 \times 10^{-9}$  and  $u_{\text{c}} / U_{\text{BIPM}} = 1.98 \times 10^{-9}$  (Type A uncertainty = 1.6 nV). It was obtained using the original INTI measurement set-up with a HP34420A detector. This result supports the CMCs (Calibration and Measurement Capabilities) of the National Metrology Institute.

Different measurement configurations and instruments were tested during the week allotted to the comparison. Applying our expertise to the INTI measurement set-up did not result in any significant technical improvement. However, some experimental results indicated that the nanovoltmeter used in the measurement set-up was probably not the best in terms of induced noise in the measurement loop and related array voltage instability. Some technical aspects of the measurement set-up were identified as weaknesses in the accuracy of the measurement set-up to achieve a comparison result in the sub-nanovolt range. Of particular note were:

- a lack of flexibility concerning the grounding configuration of the dewar and the probe;
- an inability to physically disconnect the bias source from the array;
- the quality of the phase-lock system in terms of its efficiency and noise induced to the array;
- a failure to obtain best-case ground voltage reference continuity between the RF equipment and the probe.

These various aspects prevented any improvement to the preliminary result, which therefore constitutes the final result of the comparison:

 $(U_{\rm INTI} - U_{\rm BIPM}) / U_{\rm BIPM} = -1.06 \times 10^{-9}$  and  $u_{\rm c} / U_{\rm BIPM} = 1.98 \times 10^{-9}$  (Type A uncertainty = 1.6 nV).

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## **Acknowledgment**

The authors would like to thank Murray Early from MSL (New Zealand) for a valuable discussion on his experience with the origin of offsets observed on accurate Digital Multimeters.

## References

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#### **DISCLAIMER**

Certain commercial equipment, instruments or materials are identified in this paper in order to adequately specify the environmental and experimental procedures. Such identification does not imply recommendation or endorsement by the BIPM or INTI, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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## Appendix A

This part of the report describes the measurements performed in chronological order.

#### **26 November 2011**

The BIPM equipment was unpacked and the JVS assembled. The Hypres SIS array N° 2538E-7 was mounted on the probe and a critical current of  $I_{\rm C}$  = 95  $\mu$ A was measured at the first attempt to cool the array. During the cooling process the dewar was referred to the ground electrical potential. Laboratory air conditioning operated at the limit of its capabilities, causing significant temperature variations.

#### **27 November 2011**

The grounding configuration was investigated in the following way:

- 1 Each JVS was grounded to the earth potential of the mains powering its equipment. Shielding of the cables connecting both JVS was not connected. This configuration led to step instabilities on both arrays.
- 2 The BIPM probe was not grounded, giving an improvement in the stability of the INTI's steps. However, the stability of the BIPM voltage was unsatisfactory.
- 3 The voltage reference of the measurement loop was taken from the INTI JVS to the BIPM probe through the shielding of the cables connecting each JVS. This configuration brought acceptable stability to both JVS. Configurations whereby the INTI probe and/or dewar were not grounded could not be attempted because the set-up design prevented this option.

Furthermore, it was not possible to disconnect the INTI biasing source from the array. Therefore, even if the biasing source was powered from batteries, the existence of a leakage originating from the biasing source could not be cancelled.

The 10 MHz frequency reference was provided by a caesium oscillator provided by the INTI Time & Frequency laboratory. The signal was fed into an isolator before being plugged into the EIP578B 10 MHz external reference connector.

The first five measurement points were carried out using this configuration and the standard deviation of the mean of the corresponding frequency measurement was 100 Hz.

The following five measurement points were performed using the INTI EIP 578B internal 10 MHz crystal as the frequency reference of both JVS RF sources. The standard deviation of the mean of

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the corresponding frequency measurement was reduced to 20 Hz. However, the stability of the INTI array deteriorated and the corresponding results were unsatisfactory.

Using the external 10 MHz signal without its isolator gave corresponding measurement points that were noisy and discrepant.

It was decided that later in the experiment an improved RF locked signal would be attempted, because the 100 Hz standard deviation on the INTI frequency signal was comparable to a 1.5 nV Type A uncertainty on the Josephson voltage. Priority was given to improving the stability of the measurement set-up particularly the behaviour of the nanovoltmeter.

#### **28 November 2011**

We attempted to increase the stability of the INTI array as it was the only course of action within the option A comparison protocol where the detector is an EM N11. Measurements obtained with the HP34420A on 27 November were repeated with this set-up.

The voltage difference measured for the first two points was unacceptable and the analogue filter of the nanovoltmeter was engaged. This modification had no impact on the quality of the measurements.

The INTI array jumped from its quantum voltage step as soon as the computer started to run the data acquisition process. The computer was not equipped with an IEEE 488 bus isolator because no significant improvement had previously been observed when using this device.

The INTI measurement software was written using C++, so an executable file was prepared from the source code and run on the BIPM laptop, which operated using batteries.

This modification had no positive impact on the quality of the measurements.

We returned to the configuration whereby the INTI computer controlled the data acquisition but an optical fibre isolation bus was installed to decouple the ground of the computer from the voltage reference point of the measurement set-up.

Note: it was not possible to compute any result from the acquired data using BIPM software because the calculation is based on fixed frequency values from the RF sources (with a resolution of 1 Hz). The INTI software was designed to record the frequency from the counter at different times in the measurement process and the quantum voltage relies on the mean value of these measurements.

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#### 29 November 2011

The measurement configuration described previously using the INTI desk computer and associated IEEE 488 bus isolator was used to obtain measurement points.

Three acceptable measurement points were recorded but the measurement time significantly increased to more than 10 minutes per point due to INTI array instability.

Switching to the BIPM laptop to control the measurement (Labview<sup>©</sup> software) resulted in the voltage across the INTI array becoming stable again. The assumption was made that the commands sent on IEEE 488 bus were responsible for its instability rather than the grounding distribution.

The HP34420A detector was changed to the BIPM Keithley 2182A. The INTI software was modified to comply with the commands from this device.

The stability of the INTI array was considerably improved and 5 equally spaced time points were carried out with excellent results. The mean value of the five points was: m = +0.93 nV and the corresponding standard deviation of the mean was  $\sigma_A = 1.2$  nV. This result is not considered a comparison result because the DVM belonged to the BIPM. This illustrates the importance of the nanovoltmeter choice in a measurement set-up with regard to the associated stability of the quantum voltages and electrical noise in the measurement loop.

No such nanovoltmeter was available at the INTI; however a Keithley 182 was tested. The first measurement point was found to be an outlier as the external part of the DVM connector was not grounded. A second measurement point was no better. Engaging the analogue filter resulted in a worse measurement, probably due to the relaxing time period of the filter not being sufficiently taken into account. A fourth point was measured with longer stabilization time and a fifth with the filter disengaged but for each, the relaxing time was of the same order. Both results were acceptable. The main improvement was the voltage stability of the INTI Josephson array.

<u>Note</u>: A similar large offset measured by an HP34420A had already been observed during a BIPM comparison at MSL[2]. Early and Roberts[3] noted that high quality DMMs show some abnormal behavior in the presence of AC signals at their input related to the use of a discrete front end amplifier. Filipski and Boecker also reported some problems was attributed to a common mode effect related to the noise compensation system[4].

## **30 November 2011**

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The INTI dewar ran out of liquid helium. While the dewar was being refilled leakages at the level of an extender on the transfer tube were encountered.

#### 1 December 2011

The INTI array was cooled and the measurement set-up assembled.

A first measurement using the K182 as a detector was started but not completed because of instability with the INTI array. The cause was found to be in the low value of the critical current indicating that magnetic flux was trapped during the cooling process. This phenomenon was unusual for this array suggesting a high level of electromagnetic interference in the laboratory.

After the INTI system was rechecked, 5 measurements were made that were all discrepant. The INTI array required a RF power adjustment after each polarity reversal. The critical current value of the array junctions was checked and found to be too low again, requiring the JVS to be warmed up and re-cooled. Moreover, the software was difficult to run properly because the step number calculation failed frequently. No reason could be found for this problem.

A set of measurement points was performed but all the results gave a voltage difference between the two arrays of larger than several tens of nanovolts. This was despite waiting for several seconds to allow the input impedance of the detector to reach equilibrium after each polarity reversal.

Changing the K182 to the HP34420A nanovoltmeter gave a result equivalent to that previously observed when the INTI array was unstable.

The measurement set-up for Option A of the protocol was changed whereby the BIPM JVS and its associated software measures the participant JVS with an EM N11. The idea was to see if the INTI array remained stable with an analogue detector inserted in the measurement loop.

It was possible to carry out 3 consecutive points on the 10  $\mu$ V range but once again, because the INTI frequency was not measured during the data acquisition process, the results were unreliable. It was not possible to use a lower scale than the 10  $\mu$ V range of the N11 because an offset larger than 1  $\mu$ V appears at the time the program starts to read. The origin of the offset was obviously linked to the IEEE 488 command but no explanation could be found because no obvious ground loop was discovered. The grounding configuration of the measurement set-up was checked but no ground loop was discovered and the present configuration was the only one where both arrays were sufficiently stable to perform a comparison.

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It was concluded that the noise induced by the navoltmeter in the measurement loop was the critical point of the measurement configuration. We returned to the Option B protocol with the K182 as the detector. No satisfactory results were obtained, so the K182 was exchanged for the HP34420A device. The result from this configuration was worse, giving an offset of 12  $\mu$ V on the measured voltage difference. The BIPM K2182A gave a positive response on the stability of both arrays. Five consecutive points were performed and gave a satisfactory result (m = +0.48 nV and the corresponding Type A uncertainty of  $\sigma_A = 1.34$  nV).

It was decided to return to the option A comparison protocol and to improve the phase lock of the INTI RF signal at the same time. To achieve this, INTI was provided with a spare BIPM Gunn diode that was locked at f = 72.880 GHz using BIPM ETL phase lock electronics [5].

Note: the INTI Gunn is biased at 5 V and the BIPM ETL phase lock electronics produced a 10 V DC regulation signal, therefore it was not possible to use it on the INTI Gunn diode.

The effect on the INTI quantum voltage steps was spectacular. The size of the steps was more than doubled and 4 measurement points were performed using the EM N11 on its 10  $\mu$ V range (m = -0.11 nV and the corresponding Type A uncertainty of  $\sigma_A = 2.1$  nV). We concluded from the accuracy capability ( $\pm$  200 Hz) and the noise induced to the Josephson array that the phase lock of the INTI RF source was not working properly.

#### 2 December 2011

As the behavior of the INTI array was much more satisfactory using the BIPM RF source associated to its phase lock system, we returned to option B of the comparison protocol using the HP34420A as the detector. Six measurement points were carried out under good stability conditions (m = -1.5 nV and the corresponding Type A uncertainty of  $\sigma_A = 2.6$  nV).

<u>Note</u>: these results cannot be used to compute the final result of the comparison because part of the INTI JVS was modified using BIPM equipment.

As good stability conditions had been achieved modification of the reference potential distribution configuration in the measurement loop was attempted. No configuration tested resulted in an improvement on the one selected at the beginning of the exercise. The reason for this lies in the rigidity of the INTI set-up configuration in two respects: the dewar is always grounded and the bias source cannot be disconnected from the array.

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Closer investigation of the INTI JVS found that the RF source voltage reference was not disconnected from the voltage potential reference of the probe. This implies a ground loop that could induce an offset to the results or can be responsible for additional noise in the measurement set-up.

Time constraints meant that it was not possible to test the effect caused by implementing the isolation of the RF source from the INTI probe.

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