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Ensuring the validity of Zener calibration results based on historical calibration data

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ABSTRACT
This study introduces some techniques for ensuring the validity of Zener calibration results, which can be employed in other measurement analyses, based on the ISO/IEC 17025:2017 standard. We present and discuss several practical scenarios. Finally, we propose the use of a prediction technique to improve the calibration procedure, along with the error (normalised) $|E_\mathrm{N}|$-score as an acceptance criterion, which is the main contribution of this paper. This leads to a robust assurance of the validity of Zener calibration results. As a practical application of this proposal, we could verify two Zeners that have experienced power shortages; one of them had maintained its historical calibration data ($|E_\mathrm{N}|$ = 0.03), while the other had not ($|E_\mathrm{N}|$ = 2.19).

Section: RESEARCH PAPER

Keywords: calibration data; E_N-score; prediction techniques; Zener calibration

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1. INTRODUCTION

Zener-based DC voltage reference standards (hereafter called simply as Zeners) are commonly used as reliable standards for volt dissemination. Josephson Systems realise the volt, based on the Planck constant (h = 6.626 070 15 × 10^{-34} J s) and on the elementary charge constant (e = 1.602 176 634 × 10^{-19} C), and they are used to calibrate Zener standards, allowing expanded uncertainties of a few parts in 10^{9} (V/V). Zener standards are then used to calibrate voltage meters and voltage sources [1]–[5].

As many electronic instruments, Zener standards output voltages change in time (the so-called "time drift"). Each Zener unit has its own time drift, but it can be modelled using a linear drift model [6]. Although there is instant drift (which changes at each measurement, even changing the drift signal, either positive or negative, leading to either decreasing or increasing the output voltage value, respectively) and short-term drift (within a few days, which can also have changing signals), the drift of interest is the long-term one (within months and years). It brings higher impact (compared to the other ones) in the Zener voltages, leading to a consistent increase (or decrease, depending on the signal of the long-term drift) in its voltages; also, it is more stable and predictable, and it can be easily modelled.

Zener standards are also affected by room temperature, pressure, and humidity. Temperature and pressure can affect each measurement (such as the instant drift), but their effects are more predictable and can be modelled. For instance, R. Chayramy et al. [7] corrected the output voltages of the standards for their sensitivity to parameters such as their internal temperature (relative to the thermistor resistance of the Zener's oven) and atmospheric pressure. In 2016 and 2017, these sensitivity coefficients were determined again. The corresponding corrections vary between −7 parts in 10^{7} (V/V)/kΩ to +6 parts in 10^{7} (V/V)/kΩ, and from +8 parts in 10^{10} (V/V)/hPa to +2 parts in 10^{9} (V/V)/hPa, respectively [7]. Humidity also has predictable effects (which can also be modelled), but these are very slow compared to temperature and pressure effects. For instance, they can present a 20-to-40-day time constant [6], which can be more difficult to determine.

If temperature, pressure, and humidity coefficients are not available, a straightforward way is to keep such quantities under tight control. This allows the use of a classical linear time drift model for Zener standards over the calibration interval, as follows [6]:

$$V_\mathrm{Z} = V_0 + m \cdot t \,, \tag{1}$$

where V_Z is the voltage Zener value, in V, of the fitting line at time t; V_0 is the initial voltage Zener value, in V, of the fitting line at the reference time $t = 0$; m is the Zener drift rate, in V/d; t is the time in days from the calibration at $t = 0$ d.

Using a Programmable Josephson Voltage Standard (PJVS, primary system) and two Zeners (manufactured by Fluke, model 732B), Andrade and Landim [8] found that the room temperature variation impacted only 3 % in the Zeners' internal temperature. They also determined that the Zeners' output voltages changed only between -2 parts in 10^8 (V/V)/K to +2 parts in 10^8 (V/V)/K. This led to a normative room temperature variation of (23.0 ± 2.0) °C, at the Brazilian National Metrology Institute (Inmetro), assuring the laboratory is still capable of reaching its CIPM MRA Calibration and Measurement Capability (CMC), ± 0.1 μV (for 1.018 V) and ± 0.4 μV (for 10 V), for the secondary system (the client's Zener calibrated using the laboratory's working Zeners) [4], [8]. Another study at Inmetro (internal report) led to similar results, regarding the normative room humidity limits: (57.5 ± 12.5) %rh. However, at Inmetro laboratories, these quantities are under very tight control. For instance, we have recorded the following limits in the last 12 months: temperature between 21.6 °C and 24.0 °C, (22.8 ± 1.2) °C; humidity between 42 %rh and 56 %rh, (49 ± 7) %rh; pressure between 100 044 Pa and 102 644 Pa, (101 344 ± 1 300) Pa at Inmetro Quantum Electrical Metrology laboratory. These conditions allow the use of eqation (1) as the Zener prediction model.

It is worth mentioning that Zener linear time drift can only be characterised if it is always kept with power ON (either from its internal battery or from an outlet). This is so critical that Zeners have an "IN CAL" LED, which must always be ON, indicating that the calibration condition is preserved. If the Zener power is lost for only a few minutes, this LED will turn OFF, and there is no guarantee that the Zener will follow its normal behaviour: it may "jump" to another output value, and it might also change its long-term drift. We call it a "loss of Zener historical calibration data" (years of calibration work may be lost). This happens because the past measurements cannot be used for predictions anymore, since the parameters of equation (1) change. In this case, this Zener must be taken "out of order" until its power is restored and it is calibrated again (and the "IN CAL" LED is reset to the ON position again). This Zener can then be used for calibration, but its historical calibration data (related to its drift, one of its metrological properties) probably must be rebuilt. In this case, new calibrations must be executed (preferably every month or every other month); after at least four calibration points within a few months, a new linear time drift (historical calibration data) may be rebuilt.

According to one of the main Zener manufacturers, "Accumulated test data have shown that, once established, the measured drift is generally linear, provided the instrument continues to receive uninterrupted operating power. When drift rate is established, extrapolations of output voltage are possible, and allow certification of the standard with lower uncertainty than is obtainable from the stability specifications alone" [9]. That means Zener historical calibration data can be used to predict future values based on the past ones. This kind of prediction is usually done in several fields when time series are available. Indeed, Zener historical calibration data (considering that room temperature, humidity, and pressure are under tight control) are univariate time series (voltage dependent on time).

Zener historical calibration data can also be used to build control charts, which help the analysis of the validity of measurement results. One of the most important cornerstones of the ISO/IEC 17025:2017 is the assurance of the validity of measurement results. According to subsection 7.7.1 of this standard, "The laboratory shall have a procedure for monitoring the validity of results. The resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to review the results" [10]. Such monitoring of the validity of results may be carried out in a number of ways, for example:

- Use of checking or working standards with control charts, where applicable;
- Replicate tests or calibrations using the same or different methods;
- Review of reported results;
- Intermediate checks on measuring equipment;
- Intralaboratory comparisons.

In this article, we will discuss the applications of some of the above-mentioned techniques for the assurance of the validity of Zener calibration results, including the use of prediction techniques based on historical calibration data (which is also useful for other kinds of instrument calibration), presented in the next section.

2. ZENER PREDICTION USING LINEAR REGRESSION

We propose the use of a prediction technique for Zener voltage estimation, based on the historical calibration data, following the procedures of the simple linear regression method [11], [12]. The output values of the calibrations executed directly with the PJVS or secondary systems over the last few years were collected for each Zener. The corresponding date, output voltage value, uncertainty, effective degree of freedom, and coverage factor were recorded. Starting from the date and the voltage value information, the linear regression can be calculated as follows:

$$V_{\mathrm{pred}} = a + b \cdot \Delta t \, , \tag{2}$$

where V_{pred} is the predicted voltage Zener value of the fitting line at time t, in V; a is the linear coefficient (meaning the predicted voltage Zener value of the fitting line at the reference time $t = t_0 = 0$ s), in V; b is the angular coefficient (meaning the Zener drift rate), in V/d; Δt is the time interval between the predicted time (t_{pred}) and reference time (t_{ref}), in days. It is worth mentioning that Excel uses "one day" as its time unit, and its reference time t_0 is 00/01/1900 00:00:00 = 0 s.

The coefficients a and b are calculated by simple linear regression by the least square method so that equation (2) represents the straight line that better fits the recorded voltage values. Equation (2) is called the "predictor", since it allows to obtain the output value at any time ahead. On the other hand, its straight line is called the "prediction".

The associated uncertainty (of each predicted value) is calculated considering the angular coefficient and the linear coefficient uncertainties, as well as the dispersion of the recorded output values, as can be seen in equations (3) and (4):

$$u_{\mathrm{pred}} = \sigma \sqrt{1 + \frac{1}{n} + \frac{\left(t_{\mathrm{pred}} - \bar{t}\right)^2}{\sum\left(t_i - \bar{t}\right)^2}} \, , \tag{3}$$

$$\sigma = \sqrt{\frac{\sum (V_i - a - b\,\Delta t_i)^2}{n-2}}\,, \tag{4}$$

where σ is the standard deviation between the recorded output values and the prediction line; n is the number of recorded output values; V_i is each recorded output value; Δt_i is the time interval between the time of the recorded output value and the initial reference time (t_{ref}); and t is the average time of recorded output values.

Prediction expanded uncertainty (U_{pred}) is calculated by multiplying the uncertainty above, equation (3), by the coverage factor associated with the degree of freedom $n-2$ (k_{n-2}), with a 95.45 % confidence interval.

$$U_{\text{pred}} = k_{n-2} \cdot u_{\text{pred}}\,. \tag{5}$$

3. ENSURING THE VALIDITY OF ZENER CALIBRATION RESULTS

3.1. Use of checking or working standards with control charts

Although we have a primary standard (a NIST-designed PJVS) available, which realises the volt, we always check it at the beginning of each year, using check standards. Since we know the behaviour of our standards, based on several years of measurements using our PJVS, one simple way to quickly check if the measurements are consistent is to compare the latest measurement with the previous ones in a control chart. Figure 1 shows the previous measurements: in blue are the measurements related to more than one measurement on the same day, and in orange are the measurements related to the average value (of several days), which will be used for the traceability chain (for the secondary system). The solid black line represents the linear drift (in this case -7 parts in 10^{10} (V/V)/day, as it can be seen in the equation inset in this figure).

The same technique is used for the secondary system. In this case, it is important to have in mind that the control chart for the same Zener will present higher dispersion regarding its linear drift (which will be a little different from the equivalent one from the PJVS data, since the uncertainties are also higher in the secondary system, compared to the primary one). The important issue is to compare considering the same behaviour of the previous measurements in the same system. That will establish a baseline.

3.2. Replicate tests or calibrations using the same or different methods

In our case, the primary system is tested first. If the latest measurement does not fit the linear drift as the previous ones (Figure 1), it may be an indication of (Figure 2):

(a) **A problem with the electrical connections**: the measurement is repeated two more times, after checking the connections of the measurement circuit. If the two other measurements are consistent with each other and with the linear drift, the problem (solved) was a malfunctioning measurement circuit. If the three measurements keep produce similar results, we try the next step.

(b) **A problem with the checking standard**: we change the checking Zener and make three measurements. If the results are consistent with the linear drift of this previous checking Zener, the problem (to be solved) is the first Zener, whose status must be changed from “Calibrated” to “Out of order”, and it must be properly tested. If this second Zener also presents results different from its linear drift, we try the next step.

(c) **A problem with the calibration system**: we use a third checking Zener and make three measurements. If the results are consistent with the linear drift of this third Zener, the problem (to be solved) is also the second Zener, whose status must be changed from “Calibrated” to “Out of order”, and it must be properly tested. If this third Zener also presents results different from its linear drift, it is an indication of a problem with the calibration system, and it must be investigated (repeating the initial tests, quantum margin measurements, and so on, in the case of the primary system). After getting consistent results as a primary or secondary standard (whichever is the case), we go back to step (a) again.

Once the primary system and the Zeners have been tested and found OK, we check the secondary system, through steps (a) and (c).

3.3. Review of reported results

In this situation, everything looks right: the primary system, the secondary system, and the Zener reference standards are working fine, and the calibrations are being executed normally. In this case, each calibration is composed of several measurements, carried out within a day (for the primary system) or within several days (either for the primary or for the secondary systems), which will allow the statistical analysis (mean, standard deviation, and so on). Not only the raw calibration data, but also the statistical one and the calibration report (calibration results and administrative information), are checked by the calibration performer. Next, a properly trained second professional will check all the calibration data (raw and final ones), as well as the

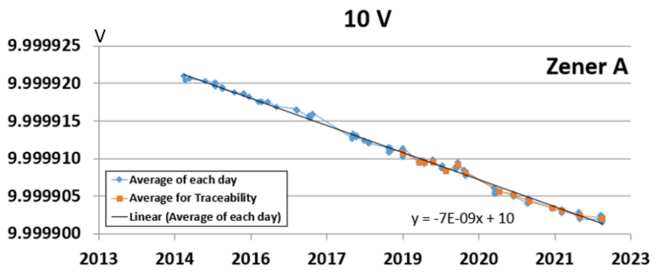

Figure 1. The control chart of Zener identified as “Zener A”. The blue dots represent the historical calibration data of all measurements (even more than one on the same day). The orange dots represent the average value, which will be used for the traceability chain (for the secondary system). The solid black line represents the linear drift. Abscissa = year.

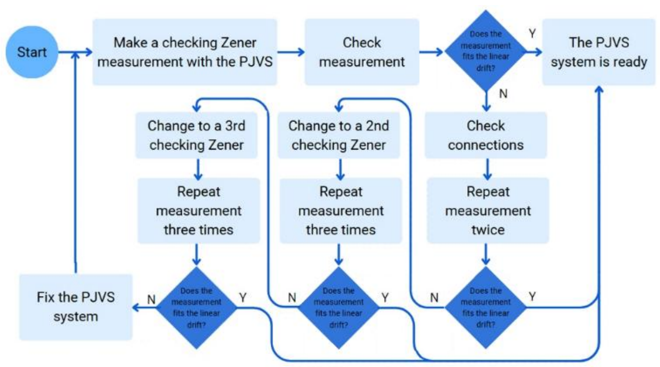

Figure 2. Flowchart of the procedure described in section 3.2 for the PJVS system.

calibration certificate. If any error is detected, the calibration performer must correct it.

3.4. Intermediate checks on measuring equipment

Zener standards are usually carried out in between calibration intervals, such as one quick measurement, with the aid of a control chart. If there are any suspicions about the Zener status (for instance, if something unusual happens, such as the loss of the "IN CAL" status, meaning the loss of historical calibration data), an intermediate check must also be done immediately.

3.5. Intralaboratory comparisons

We use this technique when everything seems to work properly, in a "triangulation process": if two systems or calibration techniques are working, both must present equivalent calibration results. Since we have two calibration systems (a primary and a secondary one), we regularly conduct calibrations of the same DUT (Device Under Test) Zener (checking standard) using both systems, as well as the classical error (normalised) $|E_\mathrm{N}|$-score as a flag.

A new approach we are using is prediction techniques and two systems, as presented in subsection 3.5.2. This is the main contribution of this paper.

3.5.1. Using two calibration systems without prediction techniques

This is used to check our secondary system, considering our primary system (PJVS) as the reference. One of our Zener reference standards is calibrated using our secondary system, generating the calibration result $V_{\mathrm{Z(Sec)}}$, with the expanded uncertainty $U_{\mathrm{Z(Sec)}}$. Next, this same Zener is calibrated using our primary system (PJVS), generating the calibration result $V_{\mathrm{Z(PJVS)}}$, with the expanded uncertainty $U_{\mathrm{Z(PJVS)}}$. The $|E_\mathrm{N}|$-score modulus must be lower than (or, at most, equal to) 1, so that both systems are considered consistent with each other [13]:

$$|E_\mathrm{N}| = \left| \frac{V_{\mathrm{Z(Sec)}} - V_{\mathrm{Z(PJVS)}}}{\sqrt{U^2_{\mathrm{Z(Sec)}} + U^2_{\mathrm{Z(PJVS)}}}} \right| \leq 1 \,. \qquad (6)$$

For instance, it is possible to see in Table 1 that the $|E_\mathrm{N}|$ was lower than 1 in both output voltages (1.018 V and 10 V).

Table 1. $|E_\mathrm{N}|$-score modulus of an interlaboratory comparison between Inmetro's primary system (PJVS) and secondary system.

V_n (V)	Secondary			PJVS			$\lvert E_\mathrm{N} \rvert$
	$V_{\mathrm{Z(Sec)}}$ (V)	$U_{\mathrm{Z(Sec)}}$ (µV)	$U_{\mathrm{Z(Sec)}}$ (V)	$V_{\mathrm{Z(Sec)}}$ (V)	$U_{\mathrm{Z(Sec)}}$ (nV)	$U_{\mathrm{Z(Sec)}}$ (V)	
1.018	1.018 097 8	0.1	1×10^{-7}	1.018 097 830	40	4.0×10^{-8}	0.00
10	9.999 873 8	0.4	4×10^{-7}	9.999 873 680	250	2.5×10^{-7}	0.25

The corresponding Figure 3 shows that both results are compatible in both situations (1.018 V and 10 V), including the error bars.

3.5.2. Using a prediction technique

When Zeners have a recorded voltage x time database, which was built using previous measurements, it is possible to make good enough predictions of their current values, as well as estimations of the corresponding uncertainties, according to equations 2, 3, and 4.

Figure 4 (a) shows an intralaboratory comparison using only the PJVS system, at 10 V. In this case, a predicted value (green dot) of a Zener (identified here as "Zener A") in our laboratory, based on all previous measurements using the PJVS system, is compared to the last measurement (the last blue dot). It is possible to see that the error bars are consistent. The dashed line is the fitting line based on the previously measured (blue dots) values. The dotted lines (right above and below the fitting line) are the limits regarding one standard uncertainty. The orange lines (right above and below the dotted lines) are the limits of the expanded uncertainty. The $|E_\mathrm{N}|$ between the predicted value and the last measured one was 0.28, indicating a very good consistency of the method.

Figure 4 (b) shows an intralaboratory comparison using only the secondary system, at 10 V, for calibration of a Zener (identified as "Zener B") in our laboratory. In this case, the current measurement (red X) is compared to the predicted value (green cross, behind the red X), based on the previous measurements of the secondary system, showing that they are coincident. The $|E_\mathrm{N}|$ between the current measurement and the predicted value was 0.03, indicating an excellent consistency of the values. It is worth noting that this Zener experienced an "IN CAL" indicator OFF a few days before this calibration, but this intralaboratory comparison between the current calibration and the prediction point proved its historical calibration data was preserved. So, we decided to include the prediction checkpoint in our measurement procedure.

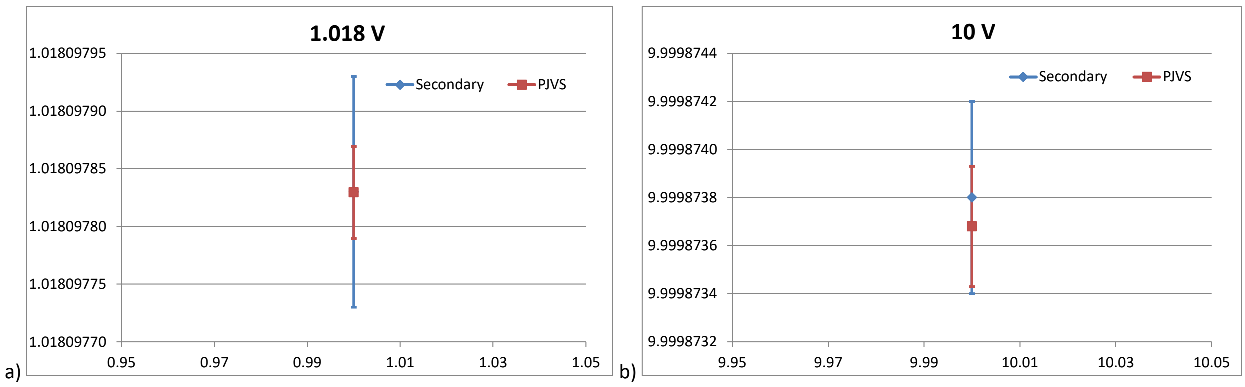

Figure 3. Graph with error bars of an interlaboratory comparison between Inmetro's primary system (PJVS) and secondary system for a) 1.018 V and for b) 10 V outputs. Abscissa and ordinate = voltage in V.

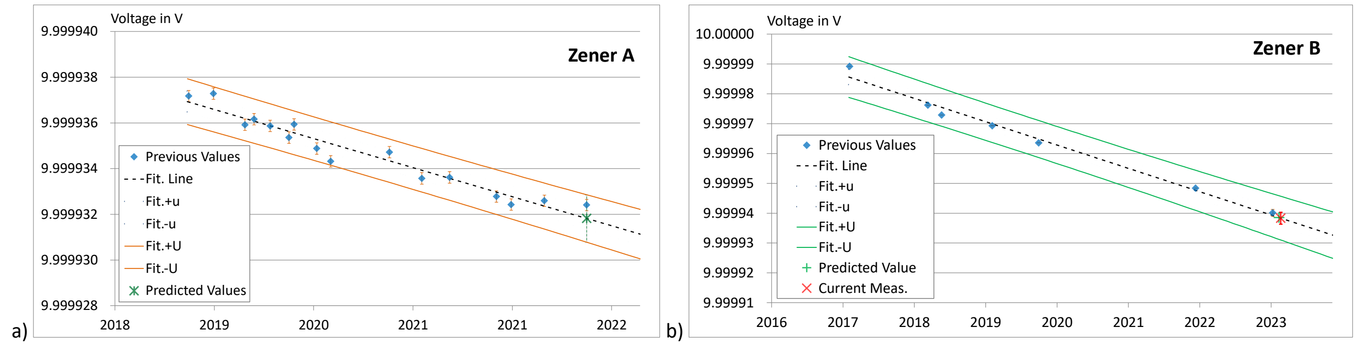

Figure 4. a) Intralaboratory comparison using Inmetro's primary system (PJVS), at 10 V, comparing a predicted value (green dot) to the last measurement (the last blue dot), using "Zener A"; $|E_N|$ = 0.28. b) Intralaboratory comparison using Inmetro's secondary system, at 10 V, comparing the current measurement (red X) to the predicted value (green cross), using "Zener B"; $|E_N|$ = 0.03. Abscissa = year.

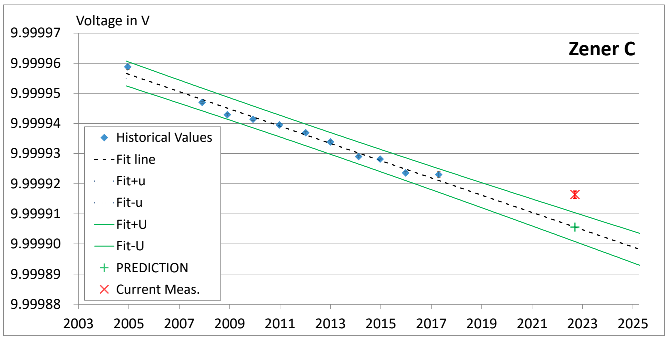

Figure 5. Intralaboratory comparison using Inmetro's secondary system, at 10 V, comparing the current measurement to the predicted value (green cross); $|E_N|$ = 2.19. Abscissa = year.

Figure 5 shows the same situation described for Figure 4 (b) but using a different Zener (identified as "Zener C"). This time, it is possible to see that the current measurement (red X) and the predicted value (green cross) are far from each other. The $|E_N|$ between them was 2.19, indicating an inconsistency between these values. It is also worth noting that this Zener experienced an "IN CAL" indicator OFF a few days before this calibration, and this intralaboratory comparison between the current calibration and the prediction point proved its historical calibration data was not preserved in this case. So, a new historical calibration data set must be rebuilt for this Zener.

4. CONCLUSIONS

Some techniques for ensuring the validity of Zener calibration results (that can be employed in other measurement analysis), based on the ISO/IEC 17025:2017 standard, were presented. Some practical situations were presented and discussed. We proposed the use of a prediction technique, which improved the calibration procedure, leading to a robust assurance of the validity of calibration results. The normalised error was also used as an acceptance criterion. It was possible to verify two Zeners that have experienced "IN CAL" LED OFF situation, where one of them has maintained its metrological characteristics ($|E_N|$ = 0.03), while the other one has not ($|E_N|$ = 2.19).

AUTHORS' CONTRIBUTION

Regis P. Landim's contribution to this work was related to conceptualisation, data curation, formal analysis, investigation, methodology, project administration, resources, supervision, software, validation, visualisation, and writing (original draft, review, and editing).

Helio Carvalho's contribution to this work was related to data curation, formal analysis, investigation, methodology, software, validation, visualisation, and writing (original draft).

Renato da Costa's contribution to this work was related to visualisation and writing (original draft).

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