Calibration procedure for solar cells exhibiting slow response and application to a dye-sensitized photovoltaic device

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1. Introduction

The rapid improvement of dye-sensitized (DSSC) and perovskite (PSC) solar cell performances and the related growing interest for these emerging photovoltaic (PV) technologies [1,2,3] clearly indicate the need for methods and procedures to reliably measure and characterize PV devices known to possess a slow response to changes in photocurrent generation by incident light. Electrical power measurements of photovoltaic (PV) devices can only be accurately made if sufficient time is allowed for complete photocurrent generation. The generation time increases from typically a few milliseconds in the case of c-Si devices, over tens of milliseconds for technologies like back-contact and hetero-junction solar cells, to even longer times for dye-sensitized and some types of perovskite solar cells. In this work we propose a procedure for calibration of slow responding PV devices based on an accurate evaluation of their response time. Starting from a quantitative analysis of the photocurrent signal versus chopping frequency on the spectral responsivity set-up, the measurement of a dye-sensitized solar cell was performed at 1 Hz chopping frequency. Then current-voltage (I-V) measurements were performed at different sweep-times and directions, in order to determine the correct parameters for I-V characterization. Combining appropriate spectral responsivity (for determination and correction of spectral mismatch) and I-V measurements yielded a reliable calibration of the device including measurement uncertainty estimation. Based on this work criteria for a reliable calibration of slow responding PV devices are formulated, fulfilling all requirements specified in the standards (IEC 60904 series and IEC 60891).

IEC 60904-1 [9] contains a generic note warning that I-V measurements may be influenced by the voltage sweep rate and sweep direction and requests an analysis without giving much detail apart from the requirement for optimal overlap of current-voltage sweeps in opposite directions. Performance characterization of DSSC’s was described previously [10] focusing on the sweep speed and direction and presenting complete SR measurements at different chopping frequencies. However, no clear criterion was formulated how to determine the appropriate chopping frequency. In a review [6] the shape and distortion of the signal under monochromatic chopped light was presented, but again without a clear quantitative criterion on selection of appropriate chopping frequency apart from simple visual inspection. As in [10] full SRs measured at different chopping frequencies were presented as well as IV curves in opposite sweep directions. More recently [11] electrical characterization of PSC’s was discussed. The main feature of these devices was found to be their inherent instability, so that the measurement depended significantly on stabilization and degradation, both depended on the illumination of the devices prior to and during measurement. In this work we propose a procedure for precise calibration of these devices: it consists of firstly an evaluation of

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the response time of the device under test (DUT) and, consequently, a reliable calculation of the parameters to be used when characterizing them. The determination of the chopping frequency to be used in SR measurements is the first step of the procedure and is described in the time response analysis (Section 3.3). As opposed to previous works [6,10] we analyze the signals of the chopped light quantitatively to give a more objective criterion for the determination of the chopping frequency. The quantitative analysis is performed under chopped broadband light, providing a stronger output signal from the device under test. This condition allowed to freely and continuously vary the chopping frequency and to determine the device response in short time (5 min).

Once determined, the SR measurements were performed with the apparatus available at the European Solar Test Installation (ESTI) after necessary modifications and validation of the setup at the low chopping frequency. Prior to electrical characterization, I-V measurements were performed at different sweep times and sweep directions to ensure that the device was measured at equilibrium conditions. The combination of SR measurements with I-V characterization yielded a reliable calibration of the device under test (DUT) and, consequently, a necessary modification to complete the calibration procedure applicable to every PV device. For standard c-Si PV devices the procedures to perform reliable measurements are well established among reference laboratories like ESTI [15]. However, in case of emerging PV technologies like DSSC and PSC, new procedures need to be specifically developed taking into account the device characteristics, e.g. the slow time response. In this work the procedure developed at ESTI for the calibration of emerging PV technologies having slow response is presented. An example of its application to the particular case of a single DSSC involving a liquid electrolyte is presented in Section 3. It consists of an initial quantitative time response analysis needed to determine the chopping frequency suitable for SR measurements and consequently of a set of multiple I-V measurements performed at increasing sweep times and changing of the sweep directions.

In the time response analysis we propose to illuminate the DUT with broadband light chopped at a specific frequency and measure the generated photocurrent. Varying the chopping frequency from low to higher values a threshold point can be found after which the peak-to-peak amplitude variation of the current signal decreases. This is the maximum chopping frequency that can be used when measuring the SR to ensure that the DUT is measured under suitable conditions. Standard chopping frequency values for DSSC devices are, for instance, around 1 Hz. Additionally, SR measurements were performed at three chopping frequencies around the threshold frequency point with different wavelengths of monochromatic lights. This was done to check possible differences in DUT response depending on whether the chopped light is broadband or monochromatic. If the relative signals are independent of wavelengths, such an effect can be excluded. To ensure reliable I-V curves, not influenced by the sweep time, we propose to perform multiple I-V measurements at increasing sweep times and changing the sweep directions. Reproducible I-V curves are required in this case and quantitative agreement between measurements performed in opposite sweep directions must be found.

3. Results

3.1. Devices

Three devices were involved in the measurements presented. Two reference devices used to measure irradiance both in SR and I-V measurements (REF) and one DUT. The reference devices consisted of two calibrated c-Si cells with an active area of 400 mm² and with an internal temperature sensor (PX302C for SR measurements and PX305C for I-V measurements). The DUT was a dye-sensitized single cell in a glass-glass construction having an active area of 77.4 mm² (total device area 550 mm²) (ESTI code PL84). The latter was provided by an external laboratory under a sample transfer agreement. Electrical wires were glued with silver paste to the electrodes. A sample holder was built to attach a PT100 temperature sensor to the rear of the DUT.

A picture of one REF and DUT taken during SR measurements is shown in Fig. 1.

3.2. Spectral responsivity measurements setup

The SR of a PV device is one of the key characteristics and represents the device response (in term of generated photocurrent) to quasi-monochromatic light signals at various wavelengths. Practically, in the calibration of a PV device the SR is one of the inputs used to calculate the spectral mismatch factor and to correct the measured I-V curves to STC accommodating for any spectral mismatch between the reference spectrum and the spectrum of the light source used for measurement. Techniques for SR determination are well known [16,17] and the procedure to perform measurements on a PV device is described in the IEC 60904-8 [12]. At ESTI the AC method is implemented and a dedicated setup is available for SR measurements of PV devices (cells and mini-modules). The schematic of the setup has been described in more details [16,18] and is shown here in Fig. 2. The setup consists of a continuous monochromatic illumination generated by a Xenon light source coupled with band-pass filters (mounted on five filter wheels with 16 filters each), covering the wavelength range from 300 to 1750 nm with a bandwidth from 8 to 20 nm. The monochromatic light is chopped and additional continuous broadband bias irradiation is normally used. The DUT and REF are placed next to each other (Fig. 1), illuminated with the same light (bias and monochromatic) and measured simultaneously. Two lock-in amplifiers are
used to distinguish current response due to the chopped monochromatic illumination from the total output current. The voltages of DUT and REF, generated by the photocurrent across two calibrated precision shunt resistors (5 Ω for DUT and 1 Ω for REF), are acquired after the stabilization of the signals is reached. An in-house LabVIEW software automatically controls the lock-in amplifiers parameters to acquire the signals and to automatically move the filter wheel once the signals are acquired. The temperature of the devices is controlled to be 25 °C ± 2 °C by a water cooled sample stage and is monitored by a PT100 placed behind the DUT and by the internal temperature sensor of the REF.

The existing SR apparatus runs typically at a chopping frequency around 70 Hz. This chopping frequency has been found to be optimal for standard c-Si PV devices, while it is certainly too high for slow responding PV devices. In order to investigate and find the optimal chopping frequency for these devices, we propose the time response analysis presented in the next section.

### 3.3. Time response analysis

The time response analysis presented here allowed us to determine the maximum suitable chopping frequency for the DUT. The setup implemented at ESTI for these measurements is part of the SR measurements setup described before. The filter wheel was removed in this case and REF and DUT were illuminated with broadband chopped light, giving significantly higher signals than with monochromatic light. The alternating voltage signals generated by REF and DUT across the shunt resistors were acquired simultaneously with a Yokogawa storage oscilloscope, without the use of lock-in amplifiers. No bias light was applied in this case. The chopping frequency varied from 0.5 to 80 Hz in this study (in about 5 min). The signals were acquired for at least three periods at each frequency and the peak-to-peak amplitude variation was calculated afterwards over the whole measured frequency range. In Fig. 3 the calculated values for REF and DUT normalized to the lowest frequency value for comparison are shown. The values of DUT were also divided point-by-point by the REF values (black squares) to compensate for any lamp instability. As shown in the figure the c-Si device (REF) reported a constant response in the measured frequency range, independently from the chopping frequency (standard deviation of the values ± 1.5% due to lamp instability), while the DSSC device (DUT) showed a strong distortion of the signals above 1 Hz with corresponding reduction of peak-to-peak values due to its slow photogeneration characteristic. If we consider acceptable a standard deviation within ± 1.5% also for DUT values, we can determine the suitable chopping frequency range for SR measurements where REF and DUT are both measured at equilibrium. To confirm this issue the two signals from REF and DUT were synchronized at low chopping frequency (below 1 Hz) and reported an increasing phase shift at higher chopping frequencies as shown previously [8]. The time response analysis presented (Fig. 3) suggests that a chopping frequency of 1 Hz is sufficiently low to obtain the full signal amplitude. Below this frequency the amplitude reached almost the maximum value and can be considered constant for our purposes. Above 1 Hz the DUT is not reacting fast enough to the chopped illumination and will exhibit under-estimated SR values if measured.

The measurements above were performed with broadband chopped light, due to its higher signal. To exclude a possible wavelength dependency of the time response, measurements with chopped monochromatic light were made. For this, a series of complete SR measurements were performed at 1.5 Hz, 2 Hz, and 10 Hz. The ratio of SRs measured at 10 Hz and 1.5 Hz showed a slight wavelength dependency. The same ratio of SRs measured at 2 Hz and 1.5 Hz did not show any more this wavelength dependency. This confirms that the chopping frequency determined with broadband chopped light by the time response analysis above, is adequate for the measurement of the complete SR.
3.4. SR measurements of DUT at low chopping frequency

Considering the results coming from the time response analysis, the system for SR measurements described in Section 3.2 was modified to work at low chopping frequency (≤2 Hz) by replacing the DC motor, which was limited to frequencies above 2 Hz, with a new stepper motor. The parameters of the lock-in amplifiers (time constant and waiting time) controlled by the LabVIEW software were chosen after observing carefully the signals from the lock-in amplifiers during their stabilization. The system was validated after modification using a chopping frequency of 1 Hz and comparing two SR measurements of a calibrated c-Si device performed at 78 Hz and 1 Hz [8]. After validation, the system described here was applied to measure the SR of the DSSC DUT. Measured data and interpolated validation, the system described here was applied to measure the SR of the DSSC DUT [14].

3.5. I-V measurements with different sweep times

A WACOM dual lamp steady-state solar simulator WXS 140 Super with an illuminated area of 14 cm by 14 cm and of class AAA (IEC 60904-9 [19]) was used for measuring the I-V characteristics. The procedure for measuring current-voltage characteristics of PV devices is described in IEC 60904-1 [9]. In the standard it is mentioned that depending on the technology, I-V measurements may be influenced by the voltage sweep rate and sweep direction. In these cases the effect should be carefully analyzed and problems can be excluded when measurements performed in opposite sweep directions (voltage ramp from short circuit conditions to open circuit and from open circuit to short circuit conditions) overlap optimally. Sweep rate dependency happens, typically, with highly capacitive devices like back-contact and hetero-junction c-Si solar cells and with DSSC’s [6]. Following the note included in the standard, the effect of the sweep time on the power measurements of the DUT has been studied here. The sweep time was calculated as the total time of the I-V sweep between the crossing-points of the current-axis (short circuit condition: V =0) and the voltage-axis (open circuit condition: I =0). The I-V measurements were performed supplying a linear voltage ramp going from voltage values slightly below zero to values slightly above V_{oc} (forward sweep) to ensure the crossing of both axes. All the measurements here reported were performed at an irradiance of about 990 W/m² and corrected to 1000 W/m². In Fig. 5 the electrical parameters extracted from the measured I-V curves of DUT at varying sweep time ranging from 70 ms to 10500 ms are shown. At each sweep time multiple measurements were made and their mean (as dots) and their standard deviation (as error bars) are represented in Fig. 5. All parameters (I_{sc}, V_{oc}, P_{max} and FF) increased up to 1000 ms reaching a plateau for longer sweep times.

Interestingly the standard deviation of I_{sc} and FF values when measuring I-V curves too fast was significant (about 1% for I_{sc} and 3% for FF below 300 ms). At higher sweep time (>300 ms) the repeatability of the measurements increased as expected. In some cases at long sweep times the standard deviation bars are not visible in the graph because they are smaller than the dimension of the point.

The unusual high standard deviation measured on V_{oc} values (about 0.4%) at 10500 ms of sweep time could be linked to temperature effects. In such long measurements the device was heated up by the light during the sweep and consequently the V_{oc} varied. The set of measurements reported suggested that a sweep time between 1 s and 10 s should be suitable for performing reliable power measurements of the DUT under investigation.

3.6. I-V measurements varying the sweep direction

In order to fulfill the requirements of the IEC 60904-1 [9] a good overlap between measurements performed in opposite sweep directions must be verified. I-V measurements performed in opposite sweep directions using three different sweep times (432 ms, 1460 ms, 5760 ms) are presented in Fig. 6. Here the forward sweep is defined as the sweep from short circuit I_{sc} to open circuit V_{oc} and the reverse sweep the one scanned in the opposite direction from V_{oc} to I_{sc}. The temperature was monitored and controlled to be around 25 °C ± 0.3 °C during the sweep. No correction for temperature, nor for MMF was applied. The hysteresis observable at the two lower sweep times disappeared in the third case when the sweep time was above 5 s. Quantitatively the difference in P_{max} between measurements performed in opposite sweep directions with the same sweep time decreased from 8.7%, at a sweep time of 432 ms, to 0.5%, at a sweep time of 5760 ms. The electrical parameters extracted from the measured I-V curves shown in Fig. 6 are presented in Table 1.

The relative difference of the values corresponding to I-V curves measured in opposite sweep directions was calculated for comparison and is reported in Table 1. It is noticeable that at short sweep time (432 ms) there is a significant difference between the parameters extracted from I-V curves scanned forward and reverse. As the sweep time increases both I-V curves (scanned forward and reverse) converge to an intermediate I-V curve. The I-V curves measured in forward direction at any sweep time is closer to the intermediate I-V curve at

![Fig. 4. Absolute spectral responsivity measured data and interpolated of the DUT (device ESTI code PL84).](image)

![Fig. 5. Electrical parameters calculated from I-V curves measured at STC and normalized to the maximum value. Every point represents the average of three values with its relative standard deviation due to repeatability of the measurement.](image)
long sweep times than the respective curve measured in reverse (Fig. 6 and Table 1). Nevertheless the difference between forward I-V curve at fast sweep speeds and the one at slow sweep speed suggested that long sweep times were necessary for this type of device. A sweep time of about 5 s appeared to be sufficiently long to guarantee reliable I-V measurements.

### 3.7. Electrical power measurements with corrections included

Finally three I-V measurements of the DUT performed near STC conditions using a forward sweep time of 5760 ms were selected for the calibration of the device. In Fig. 7 the I-V characteristics are plotted (solid lines) together with the power at each point (dashed lines). The electrical parameters ($I_{sc}$, $V_{oc}$, $I_{mp}$, $V_{mp}$, $P_{max}$ and FF) calculated from the I-V curves shown in Fig. 7 are reported in Table 2, as average values and their standard deviations due to repeatability. The standard deviations are well below the overall measurement uncertainties of the ESTI apparatus. This is expected as the small standard deviations are due to the good repeatability of the measurements as they have small random variations, whereas the overall uncertainty of the measurements is dominated by systematic contributions [20].

In order to complete the calibration of the DUT and obtain final electrical values corrected to STC, MMF correction was applied. The MMF value as calculated in Section 3.3 for the DUT was 1.0191 and the corrected parameters are reported in Table 3 together with their uncertainties. The calculated combined expanded uncertainties ($k=2$) of the ESTI apparatus come from the analysis presented by Müllejans [20]. The UCs for a c-Si device of the same dimensions would be even smaller. But in the case of a DSSC device the following additional UC contributions were considered: difference between actual device junction temperature and temperature sensor (about 1 °C), larger uncertainty of the MMF due to significant difference between the technology and, consequently, the SR’s of DUT and REF and the hysteresis effect.

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**Table 1**

<table>
<thead>
<tr>
<th>Sweep time (ms)</th>
<th>Sweep direction</th>
<th>$I_{sc}$ (mA)</th>
<th>$V_{oc}$ (V)</th>
<th>$P_{max}$ (mW)</th>
<th>FF (%)</th>
</tr>
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<tr>
<td>432</td>
<td>Forward</td>
<td>8.686</td>
<td>0.716</td>
<td>3.962</td>
<td>63.74</td>
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<td>Reverse</td>
<td>8.928</td>
<td>0.724</td>
<td>4.321</td>
<td>66.85</td>
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<td>1.20%</td>
<td>8.70%</td>
<td>4.80%</td>
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<td>1460</td>
<td>Forward</td>
<td>8.72</td>
<td>0.719</td>
<td>4.016</td>
<td>64.07</td>
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<td>Reverse</td>
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<td>0.723</td>
<td>4.184</td>
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<td>4.10%</td>
<td>2.80%</td>
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<tr>
<td>5760</td>
<td>Forward</td>
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<td>0.72</td>
<td>4.025</td>
<td>64.06</td>
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<td>0.10%</td>
<td>0.50%</td>
<td>0.30%</td>
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**Table 2**

<table>
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<th>Electrical performance data</th>
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<tr>
<td>$I_{sc}$ [mA]</td>
</tr>
<tr>
<td>----------------</td>
</tr>
<tr>
<td>Measurement 1</td>
</tr>
<tr>
<td>Measurement 2</td>
</tr>
<tr>
<td>Measurement 3</td>
</tr>
<tr>
<td>Average</td>
</tr>
<tr>
<td>Standard dev.</td>
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</table>
4. Discussion and conclusions

In this work a general procedure for accurate calibration of slow responding PV devices is described and discussed. It allows for a precise evaluation of their response time and, consequently, for a reliable calibration fulfilling all requirements specified in the standards (IEC 60894 series). The procedure consists of an initial quantitative time response analysis needed to determine the chopping frequency suitable for SR measurements and then performing this measurement. Then a set of multiple I-V measurements performed at increasing sweep times and changing of the sweep direction. The procedure has been applied as an example to a single dye-sensitized solar cell incorporating a liquid electrolyte and a demonstration of its practical applicability is given. However, the method is not limited to this type of device and can be generally applied, in particular to PV devices which are known to have a slow response or whose response is not known. For the time response analysis a set-up needs to be able to operate at sufficiently low frequencies (here below 1 Hz), which in our case required a modification of the hardware of the SR set-up normally operated above 50 Hz. The measurement of the response to varying chopping frequencies is rather fast as the signal is high due to the broadband chopped light. Therefore the chopping frequency can be continuously varied over a wide range of values and data acquired by a storage oscilloscope. The analysis here was done manually, but could easily be performed by a dedicated software so that the result is available immediately. Therefore, our analysis is improved from previous work [6,10,11], as it uses quantitative analysis of the signal and is much faster than previous methods which compared essentially complete SR measurements at various chopping frequencies.

Furthermore, for I-V measurements a light source which provides stable irradiance for sufficiently long times is required. For the sweep times required here (5 s) this is only provided by a steady-state solar simulator, as pulsed systems are limited to tens of milliseconds (and in exceptional cases may reach about 1 s). For high capacitive c-Si devices attempts have been made to compensate the short pulse in exceptional cases may reach about 1 s). For high capacitive c-Si simulators, as pulsed systems are limited to tens of milliseconds (and times required here (5 s) this is only provided by a steady-state solar simulator. The use of steady-state simulators poses a challenge in controlling the device temperature during the measurements, as the continuous illumination with the irradiance required by STC inevitably heats up the device. While the device can be shielded from the light between measurements, the relatively long measurement times will likely result in device heating. This in turn will alter the measurement results, as PV device parameters exhibit temperature coefficients typically of a few per mille per Kelvin. Therefore temperature control is mandatory. Here this was achieved by an actively cooled sample stage with a thermal mass much larger than that of the DUT, acting as a heat sink. For larger DUTs, and in particular modules, this challenge will be larger. At the present time it is not clear how to adequately deal with it.

Based on the work and results presented, we propose the following criteria for determining suitable measurement conditions for slow responding PV devices:

a) perform time response analysis (see Section 3.3) using broadband chopped light over a wide frequency range. Select a chopping frequency such that the peak-to-peak signal is within 3% of the equilibrium.

b) measure SR at least at three representative wavelengths (near the maximum of the SR, at a wavelength around the lower boundary plus 50 nm and at a wavelength around the higher boundary minus 50 nm) selecting the chopping frequency determined in a) and its half. Check that the ratio of the SR measured at the two chopping frequencies is constant for the three selected wavelengths; if not choose lower chopping frequency until condition met.

c) perform complete SR measurement at the chopping frequency determined in b).

d) acquire I-V curves in forward and reverse sweep directions at increasing sweep times. For each sweep time analyze the difference of the three main I-V curve parameters (I_{sc}, V_{oc} and P_{max}) extracted from I-V curves measured in forward and reverse sweep directions and select the sweep time with the following criteria: ΔP_{max} < 0.5%, ΔI_{sc} and ΔV_{oc} < 0.1%

e) measure multiple I-V curves at sweep time determined under d) (minimum 3) and check for repeatability with the following criteria: I_{sc} and V_{oc} better than 0.1% and P_{max} better than 0.5%.

Only when all criteria are fulfilled and the respective measurements have been made, a valid calibration of the PV device is achieved.

In this work appropriate chopping frequency for SR measurement and appropriate sweep-times for I-V curves acquisition were determined and then the respective measurements made. Together with the correction for spectral mismatch this constitutes a complete PV device calibration at STC. We propose to apply this procedure, specifically developed for devices which have long time response (like back-contact and hetero-junction solar cells, DSSC’s and some types of PSC’s), also for the calibration of devices with unknown time response. If the criteria outlined above are fulfilled, a reliable calibration of the PV device is achieved. There will be cases where this approach is not applicable. For example in the case of some types of PSC’s where the situation might be even more complex [11], requiring additional steps as the devices are inherently unstable and require light soaking which at the same time might already contribute to device degradation. Moreover the response in PSC’s is highly dependent on the type of perovskite material and also on the device configuration [21]: problems can occur due to the ionic-electronic character of the halide perovskite. In these cases different approaches have been tested and proposed in the literature: the simplest is to hold the device at the voltage at P_{max} (V_{mp}) and wait for stabilization [11]. However, this voltage is not known a priori and therefore the result might deviate significantly from the real maximum power point. Maximum power point tracking as proposed by Zimmermann et al. [21] has in our opinion the disadvantage that the algorithm might lead to oscillation, because the device is inherently unstable. We suggest to apply in these cases the method proposed by Christians et al. [22], which consists in the measurement of steady-state photocurrent at several voltages around the presumed V_{mp} and then check the self-consistency with the I-V curves measured at various scan rates in forward and reverse directions and the steady-state I_{sc} and V_{oc} values determined beforehand. We propose to do this first starting from voltages below V_{mp} to above and then to repeat in the

Table 3
Final electrical parameters values after MMF correction (MMF =1.0191) and corresponding expanded uncertainties of the measurements in absolute and relative units.

<table>
<thead>
<tr>
<th></th>
<th>UC</th>
<th>Units</th>
<th>k=2 [%]</th>
</tr>
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<tbody>
<tr>
<td>I_{sc}</td>
<td>8.890</td>
<td>± 0.089</td>
<td>± 1.0</td>
</tr>
<tr>
<td>V_{oc}</td>
<td>0.7208</td>
<td>± 0.0029</td>
<td>± 0.4</td>
</tr>
<tr>
<td>FF</td>
<td>64.05</td>
<td>± 0.64</td>
<td>± 1.0</td>
</tr>
<tr>
<td>P_{max}</td>
<td>4.104</td>
<td>± 0.062</td>
<td>± 1.5</td>
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</table>

opposite direction. The two $P_{\text{max}}$ values obtained in this way should be consistent within 0.5%.

Following the steps described here will ensure a reliable electrical calibration of slow responding PV devices. The criteria proposed should be considered for inclusion of a respective future standard for measurement of such devices.

References


