STAR Protocols

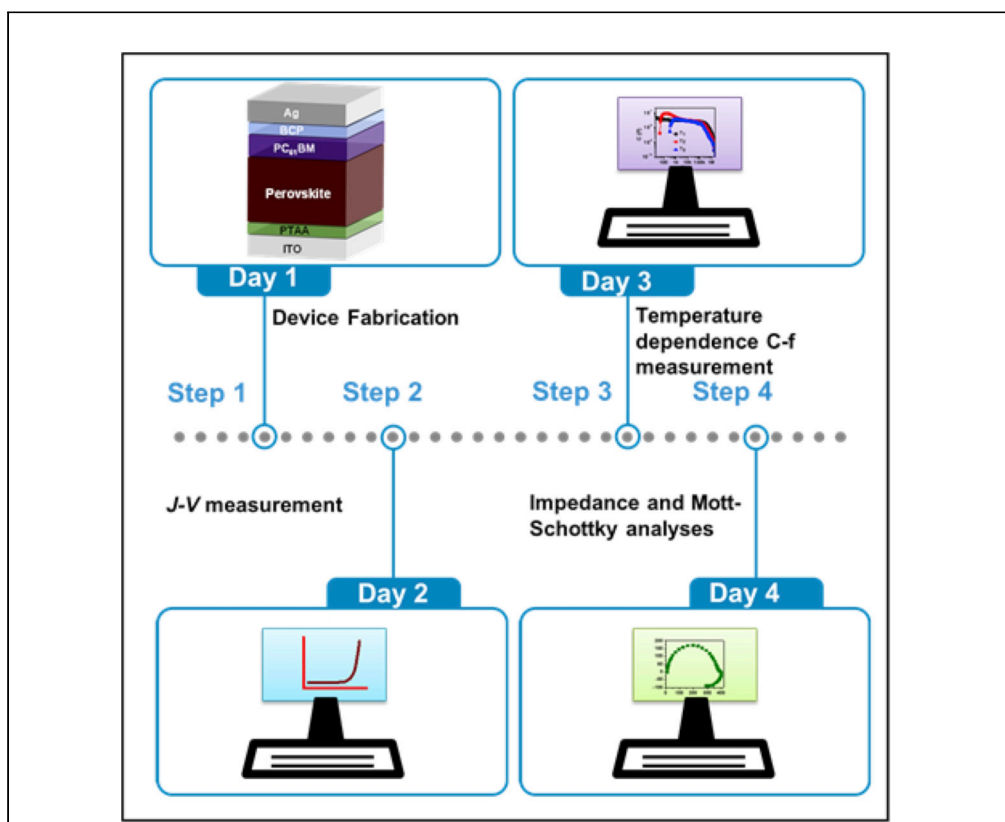**Protocol**

Protocol for deciphering the electrical parameters of perovskite solar cells using immittance spectroscopy

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Highlights

The electrical parameters of perovskite solar cells revealed by immittance spectroscopy

Intrinsic properties of materials and their correlation with device performance

Charge carrier dynamics and response of a working solar cell under illumination

Here, we present a protocol for the fabrication of inverted (*p-i-n*)-type perovskite solar cells, unraveling its electrical merits via immittance spectroscopy. The immittance spectroscopy is a prevailing technique for both qualitative and quantitative analyses of charge carrier dynamics in working devices. This technique integrates the temperature-dependent capacitance–frequency (*C-f*) spectra, impedance spectra, and Mott-Schottky analyses. This protocol is also applicable for typical (*n-i-p*) perovskite solar cells and other multilayer semiconductor devices.

Protocol

Protocol for deciphering the electrical parameters of perovskite solar cells using immittance spectroscopy

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SUMMARY

Here, we present a protocol for the fabrication of inverted (*p-i-n*)-type perovskite solar cells, unraveling its electrical merits via immittance spectroscopy. The immittance spectroscopy is a prevailing technique for both qualitative and quantitative analyses of charge carrier dynamics in working devices. This technique integrates the temperature-dependent capacitance–frequency (*C-f*) spectra, impedance spectra, and Mott-Schottky analyses. This protocol is also applicable for typical (*n-i-p*) perovskite solar cells and other multilayer semiconductor devices.
For complete details on the use and execution of this protocol, please refer to Khan et al. (2019, 2021).

BEFORE YOU BEGIN

Here we begin with the fabrication of perovskite solar cells and its characterization by temperature-dependent *C-f* measurements, Mott-Schottky analyses and impedance spectra.

1. It is advisable to use < 1 year old PEDOT: PSS received from manufacturer, and make sure by the visual examination that particles are not collected on the wall of the container.
2. Prepare the patterned Indium tin oxide (ITO) coated glass substrates (for *p-i-n* configuration) with less than 10 Ω/sq or patterned Fluorine doped Tin Oxide (FTO) substrates (for *n-i-p* configuration) with 7–15 Ω/sq. The substrates can be patterned by laser or chemical etching. We recommend customizing pre-patterned substrate from supplier. (Here is the simplified process for chemical etching: Preparation of an aqueous solution of 20% HCl in the fume hood. Mask the substrate using labeling tape. Add Zn powder and prepared HCl in the unmask area and wait ~ 10 min. Clean the substrates with Deionized water (DI) and check the resistance on both sides of the etching line via multi-meter)
3. Check the oxygen and moisture level in the glovebox, it should be approximately or less than 1 ppm of H_2O and 10 ppm O_2 level.
4. The thermal evaporator should be properly cleaned and in working condition, to avoid contamination. It is advisable to use designated thermal evaporator for electrode deposition.
5. Prepare substrate holder with metal grid mask as shown in Figure 2E for depositing silver electrodes on top of HTL layer.

6. Confirm that the measuring devices i.e., impedance analyzer and low-temperature cryogenic system are working by measuring the impedance spectra of reference cell, make sure the devices are working properly and measuring the correct values.

7. Check that liquid nitrogen is available for low-temperature measurements.

KEY RESOURCES TABLE

REAGENT or RESOURCE	SOURCE	IDENTIFIER
Chemicals, peptides, and recombinant proteins		
Hellmanex solution	Sigma	Z805939
Acetone 99.6% GLR	Labbox	ACET-M0P-25K
Ethanol 96% AGR	Labbox	ETHA-6TA-5K0
Isopropanol	Labbox	PROL-0IA-1K0
PEDOT:PSS	Heraeus	CLEVIOS™ P VP AI 4083
PTAA (Mn=5,000–15,000 by GPC)	Xi'an Polymer	PLT502021T
Lead(II) iodide, ultra dry, 99.999%	Alfa Aesar	44314
MAI	GreatCell Solar	SKU MS101000
MACl (99.5%)	Xi'an Polymer	PLT501071I
[60]PCBM	Solenne BV	060995
BCP	Xi'an Polymer	PLT302031T
Silver wire (99.999%)	Alfa Aesar	11347117
Toluene, anhydrous, 99.8%	Alfa Aesar	41464
DMF, 99.8%	Acros Organics	348431000
Chloroform, ≥99%	Sigma	288306-1L
2-Propanol (High grade)	Alfa Aesar	41463
Other		
Sun Simulator	Newport/Oriel Instruments, AAA class	n/a
Glove box	MBRAUN	MB-Unilab Pro SP (2000/780)
Thermal Evaporator	Moorfield	MiniLab 060
Spin Coater	Laurell Technologies	Model WS-650Mz-23NPPB
Hot plate	Harry Gestigkeit GmbH	PZ28-3TD
Micro weighing balance	Sartorius	ENTRIS124I-1S
LCR meter	Keysight	E4980A
Heating /cooling stage with electrical probes	Linkam	Model LTS420E-PL8
Temperature Controller	Linkam	T96 S
Potentiostat	Biologic	SP300
Faradaic cage	Biologic	n/a
LED with variable power	Lamp fitted with power controller	n/a
Lux meter	n/a	n/a
UV ozone cleaner	Novascan	PSD-UV8
Indium Tin Oxide (ITO) coated glass slides	Xop Glass	n/a
Software and algorithm		
EC-Lab software	Biologic	n/a

Alternatives: If Potentiostat have the option to measure the *C-f* spectra along with impedance spectra (IS), then there is no need to have separate LCR meter, all the measurements can be performed with potentiostat. In the present protocol, we use ITO substrate, however, an alternative conductive substrates such as FTO can also be used. We have used poly triarylamine (PTAA) and poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS)] as hole transport layers (HTL) but other HTL materials e.g., poly [[4,8-bis[(2-ethylhexyl)oxy]benzo [1,2-b:4,5-b']dithiophene-2,6-diyl][3-fluoro-2-[(2-ethylhexyl)carbonyl]thieno[3,4-b]thiophe-nediyl] (PTB7) }, poly(3-hexylthiophene) (P3HT), etc. can also be used by taking into

consideration the energy level. Similarly, this protocol is also applicable to fabricate the perovskite layer with different cations and anions composition.

STEP-BY-STEP METHOD DETAILS
Device fabrication

⏱ Timing: (12–15 h)

The fabrication of planar perovskite solar cells device with the device architecture ITO/HTL/perovskites/ETL/Ag is depicted in Figure 1.

1. Substrate cleaning

⏱ Timing: 2–3 h

a. Mark the back of ITO substrates (1.5 cm*2 cm, as shown in Figure 2A) at the corner (10 Ω per sq., Xop Glass, Spain) for identification, and put in the holder for further cleaning (Figure 2B)

b. Ultrasonicate the substrates in 2% Hellmanex solution (by diluting in deionized water) for 20 min at 30°C.
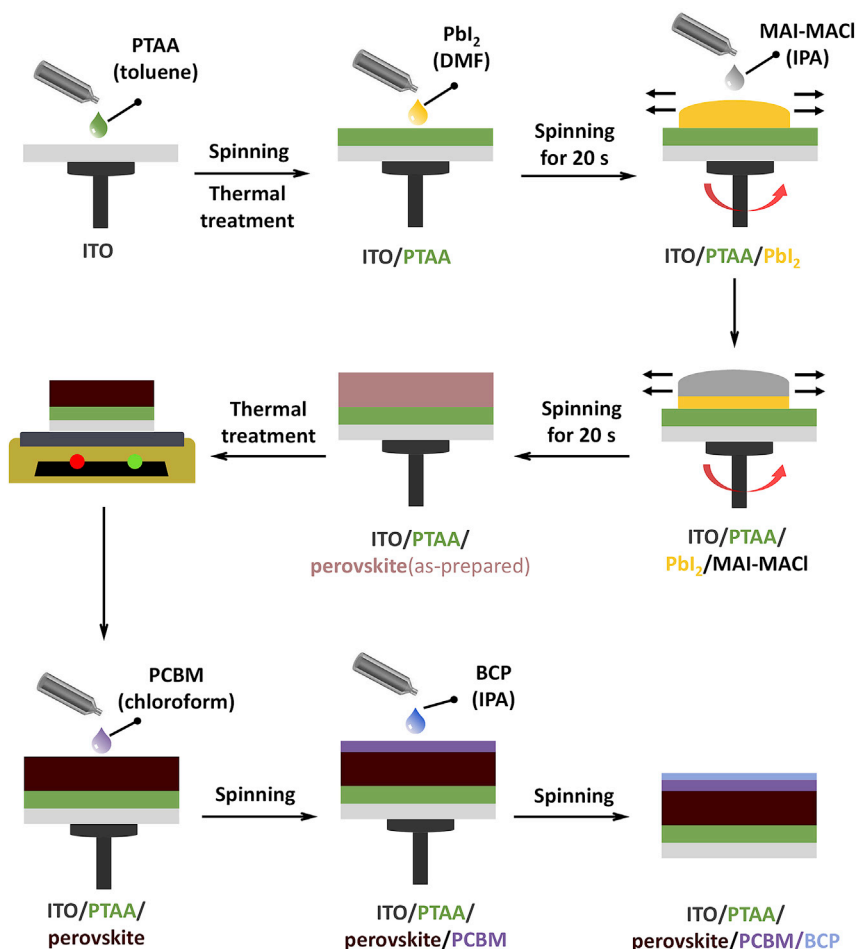
c. Wash the substrates with deionized water.

Figure 1. Schematics of the deposition of HTL, synthesis of perovskites layer over HTL, and spin-casting the ETL and buffer layer on top of perovskites layer during the fabrication process of inverted perovskite solar cells
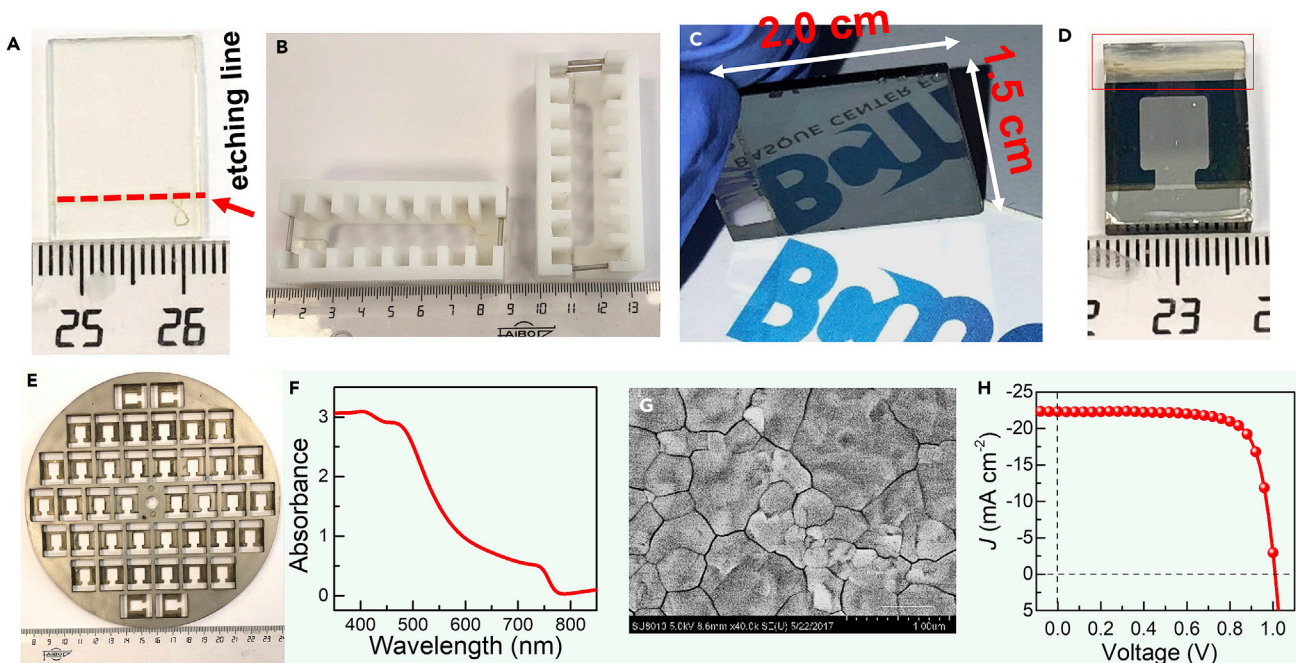
Figure 2. Perovskites solar cells fabrication and properties
(A) Photographs of the ITO substrate, (B) polytetrafluoroethene(PTFE) holder for cleaning substrates, (C) perovskite films after heating treatment, (D) perovskite solar cell (marked part should be removed by cotton bar with DMF), (E) substrate holder with metal grid mask, (F) the absorbance of perovskite film, (G) top-view scanning electron microscope image of perovskite film, and (H) typical *J-V* curves of perovskite solar cells .

   d. Ultrasonicate the substrates in acetone for 20 min at 30°C and process can be repeated If necessary.

   ⚠ CRITICAL: Acetone with a low boiling point and ultrasonication machine working for a long time will cause the temperature of water to rise rapidly. It is recommended to change the water or add ice regularly.
   e. Ultrasonicate the substrates in ethanol for 20 min at 30°C and repeat the process, if necessary.
   f. Ultrasonicate the substrates in isopropanol for 20 min at 30°C and repeat the process, if necessary.
   g. Dry the substrate rapidly with the help of dry air gun.

2. Preparation of hole transporting layers (HTL)-Day 1

   ⏱ Timing: 2–3 h
   Preparation of PEDOT:PSS film
   a. Treat the cleaned ITO substrates with UV-Ozone for 30 min.
   b. Stir PEDOT:PSS for around 30 min and filter with a 0.22 μm nylon filter before use.

   Note: We did not find the correlation between stirring time and quality of PEDOT:PSS films, but the stirring time should be at least 15 min to make sure that the aggregates disperse homogeneously without precipitation.
   c. Place 50 μL PEDOT:PSS solution on the center of the ITO substrate and wait for ~ 3 s to spread the solution all-over the substrate and spin-coat it at 5,000 rpm for 30 s with acceleration (2,500 rpm/s).

   ⚠ CRITICAL: UV-Ozone treatment will heat the substrate and raise the temperature. It is recommended to allow the substrates to cool down before the spin-casting of PEDOT:PSS precursor solution on it. If the substrate with PEDOT:PSS shows dots or radial stripes, it's advisable to check if the substrate is clean or not, and filter the PEDO:PSS precursor solution again. In case of unsatisfactory results, please use new cleaned substrates.

d. Transfer the deposited PEDOT:PSS films to a hot-plate for annealing at 150°C for 15 min. For further use transfer the PEDOT:PSS films into the glovebox immediately.
Preparation of PTAA film

e. Prepare the PTAA solution with a concentration of 5×10^{-3} g/mL in toluene and stir at 22°C–25°C for 2 h in glove box.

f. Cast 50 μL PTAA solution in the center of the ITO substrate and spin-coat at 5,000 rpm for 30 s with acceleration (2,500 rpm/s) and the PTAA film was left to anneal at 100°C for 5 min.

⚠ CRITICAL: The ambient temperature and humidity can change, which may affect the solvent volatilization and thickness of PTAA film. We recommend preparing PTAA films in dry air glovebox at a constant low temperature (around 20°C).

3. Fabrication of Perovskite active layer and electron selective contacts-Day 1

⏱ Timing: 2 – 3 h

a. Dissolve 1 M PbI_2 (600 mg/mL) in 1 mL dimethyl formamide (DMF) at 70°C and stir it at 400 rpm/s for 12 h. The mixed organic cation solution of MAI: MACl (50: 5 mg/mL) was dissolve in 1 mL IPA at 22°C–25°C and stir at 400 rpm/s for 12 h. Prepare $PC_{61}BM$ solution with a concentration of 10 mg/mL in chloroform and stir at room temperature for 2 h. Prepare BCP solution with a concentration of 0.5 mg/mL in IPA and stir it at room temperature for 24 h.

b. Filter the PbI_2 solution by a 0.45 μm PTFE filter and keep the solution at 70°C for further use. Before use filter also the mixed organic cation solution by a 0.45 μm PTFE filter.

c. Cast 30 μL hot PbI_2 solution on the center of the ITO/HTL and spin-coat (See Figure 1) at 4500 rpm for 20 s with acceleration (9,000 rpm/s), then 50 μL of the mixed organic cation solution was dynamically cast on the wet PbI_2 film without stopping and continue the spin-coating at 4500 rpm for 30 s. The as-prepare film is annealed at 100°C for 5 min and the post anneal film is shown in Figure 2C, record the absorbance and morphology of perovskite film as shown in Figures 2F and 2G, respectively.

⚠ CRITICAL: To obtain uniform perovskite films, wettability of PTAA is essential, for this pre-wetting by DMF before casting PbI_2 solution, dynamics casting of PbI_2, or low molecular weight PTAA can overcome such issue. The prepare perovskite changes color from brown to dark within 2–4 s when the substrate is place on the hot plate, if this process is delay, it can due to the perovskite solvent. The DMF vapors should be clean at regular interval, during the continuous deposition of perovskite films. We recommend removing DMF vapors by purging the glovebox, and prepare less than 20 devices every time.

d. Cast 60 μL $PC_{61}BM$ solution on perovskite film to spin-coat at 1500 rpm for 30 s with acceleration (750 rpm/s).

⚠ CRITICAL: Chloroform is a low boiling point solvent, it is critical to control the temperature of the glovebox (below 30°C) during the preparation of $PC_{61}BM$ electron transporting layer. The alternative is replacing chloroform with chlorobenzene or more green solvent such as toluene to prepare $PC_{61}BM$ solution. The concentration of $PC_{61}BM$ in chlorobenzene/toluene should be increase to 20 mg/mL, and can be spin-coat on perovskite film at 4000 rpm for 30 s. Further, leave the as-prepare $PC_{61}BM$ film for around 2 h in the glovebox or keep it in vacuum condition for more than 30 min for full solvent volatilization.

e. Cast 60 μL BCP solution on top of $PC_{61}BM$ film and spin-coat at 4000 rpm for 30 s with acceleration (2,000 rpm/s).

4. Deposition of top electrodes Day 1

⏱ Timing: 6–8 h

The samples are left to rest for close to 2 h in a glovebox, to allow solvent evaporation, and

then transfer in the thermal evaporation chamber to prepare for electrode outside of the glovebox.

a. To prepare the anode, the perovskite film mark by a red rectangle in Figure 2D was remove by scraping with a cotton bar with DMF (5 – 10 μL).

b. Clean the samples with an air gun to dry the solvent residues and remove unwanted dust particles before placing the samples into the substrate holder for evaporation (Figure 2E).

c. Control the rotation speed of the substrate holder to 20 rpm/s during the evaporation process.

d. When the vacuum values reaches 5×10^{-6} mbar, the evaporation of the silver will be made in the following way: the first 5 nm with the speed of 0.1 Å/s and then at 1.0 Å/s up to 100 nm. The silver wire will melt first under the low heating current and start to evaporate with an increase in the current.

e. If the thickness monitor is not working or not installed, thickness of the silver layer can be deduce by the cross-sectional scanning electron microscope or profilometer, or spectroscopic ellipsometer.

⚠ CRITICAL: When removing the perovskites film, the minimum amount of DMF should be used and care needs to be taken not to accidentally remove the whole layer.

⚠ CRITICAL: The vacuum value needs to be less than 10^{-5} mbar, as the vacuum values can impact the increase in temperature during the evaporation process. The lower the vacuum degree, the higher the super-heating rate.

Characterization of devices

⏱ Timing: (10–15 h)

5. Testing of device performance-Day 2

⏱ Timing: 30 min – 1 h

The current density characteristics of the devices was measure under AM 1.5G (100 mW cm^{-2}) illumination at the ambient condition with (~ 25°C and ~ 40% relative humidity) with the help of AAA grade solar simulator (Newport) as shown in Figure 3.

Before starting the J-V measurement, calibrate the light illumination by using standard Silicon (Si) cell. For this, the standard Si solar cell was kept at the center of the simulated xenon light source, at a similar height as of devices.

a. The shadow mask was adhered to the un-encapsulated solar cells to define the active area (0.09 cm^2) to reduce the influence of scattered light.

b. Measure J-V curves under reverse scan: from 1.2 V to -0.1 V, scan-rate 100 mV s^{-1}, delay time 100 ms, and pre-sweep delay: 10 s.

6. Capacitance vs. frequency measurement-Day 3

⏱ Timing: 8–10 h

Perform C-f measurements at variable temperatures using Keysight precision LCR meter linked to a Linkam heating / cooling stage filled with liquid nitrogen (shown in Figure 4). The temperature was control with the help of software. Firstly, cool down the devices to 150 K, and thereafter start measurements in the heating mode in the temperature range 150 K − 350 K.

a. Place the sample inside the Linkam heating/cooling stage.

b. Connect the probes to the top and bottom contact and check the connectivity from a multimeter.
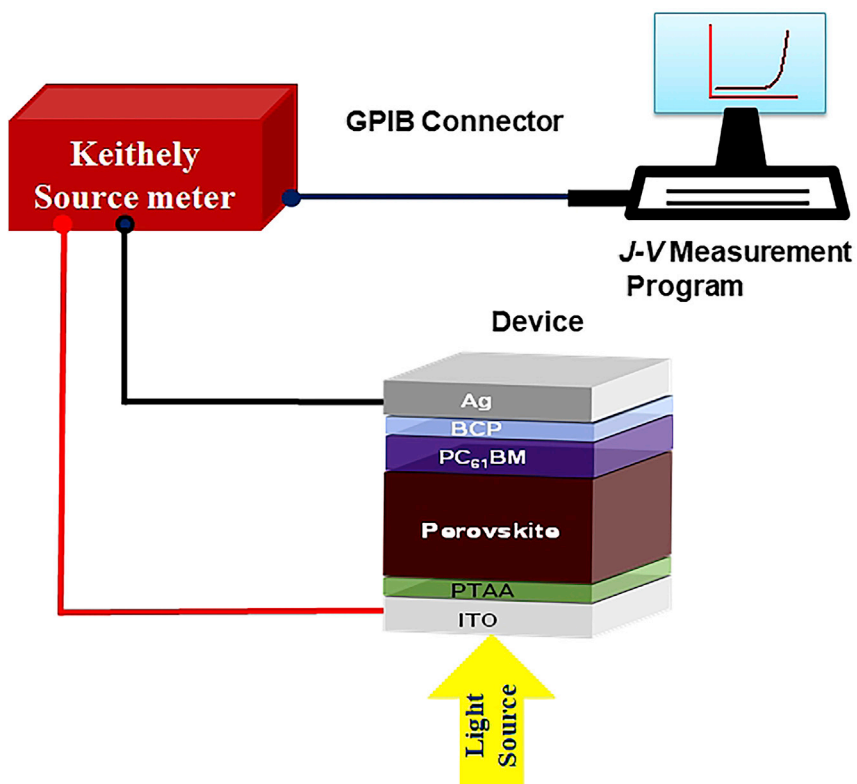
c. Fill the liquid nitrogen in DF2 Flask.

Figure 3. Illustrating the measurement of *J-V* characteristics of solar cells under illumination

d. Set the desire temperature and run the temperature controller with a ramp rate of 2°C/min.
e. After acquiring the desire temperature, wait for a while (3–5 min) to stabilize the temperature of device and then measure the capacitance in the frequency range 100 mHz−2 MHz and repeat the same at different temperatures.
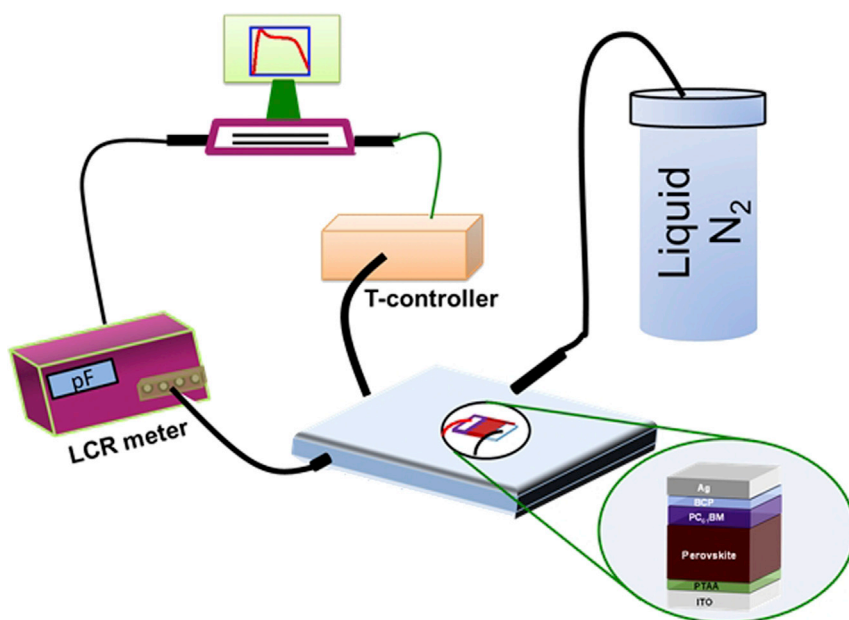

Figure 4. Experimental setup for the measurement of temperature dependence *C-f* spectra and impedance spectra

⚠ CRITICAL: If high variation in the measured spectra at different temperatures is observe, check V_{OC} of the device under illumination, and in case of over 5% change in V_{OC} replace the device.

7. Impedance measurement -Day 4

⏱ Timing: 2–3 h

Carry out the impedance measurements in the desire frequency range (e.g., High frequency up to 2 MHz, low frequency 1 Hz − 10 mHz) under the perturbation of 10−30 mV ac signal using Biologic SP300 Potentiostat. If the select frequency range is not ideal, only part of spectra will display, as shown in Figures 5A and 5B. Illuminate the device with a white LED and illumination intensity can be control by a controller and the range of light intensity for different photovoltage was c.a. 0.1 − 0.5 sun illumination. To minimize the noise and external interferences, we suggest using a faradaic chamber for sample measurements.

Measuring IS under short circuit condition

a. Turn on Biologic SP 300 Potentiostat and wait for 30 min to stabilize the system.
b. Place the sample inside the faradaic cage and connect the device electrodes with Biologic SP 300 Potentiostat.
c. Open EC Lab software and load setting for Potentiostatic Electrochemical Impedance Spectroscopy (PEIS).
d. Set E_{we} to E = 0 V vs. Ref, Scan frequency from f_i = 2 MHz to f_f = 0.1 Hz, with N_d = 10 points per decade in Logarithm spacing.
e. Set AC perturbation V_a = 20 mV, Wait for p_w = 0.1 s period before each frequency measurement, average measure per frequency N_a = 2 and corrects the drift of the system.
f. Record the impedance spectra in dark.
g. To record the IS under illumination, vary the current of LED and record the illumination intensity via Lux meter, thereafter measure the IS.
h. Measure *J-V* before and after IS measurement.
   Measuring IS under open circuit condition
i. Vary E_{we} to E = 0.2 V to 0.8 V and keep other parameters as above.
j. Record the IS in dark as well as under illumination.

⚠ CRITICAL: Measure *J-V* before and after IS measurement, if there is a variation of more than ±5% in V_{OC}, replace the device

⚠ CRITICAL: AC perturbation has to be high enough to produce a noise-free signal but small enough to ensure linearity. For more detail please see the troubleshooting section and read the work by Pitarch-Tena et al., (2018).

⚠ CRITICAL: There are different ways to reduce the noise in low-frequency impedance spectra either by increasing the AC perturbation, or by increase of the integration time, and/or the increase of the number of Integration cycles during the measurement.
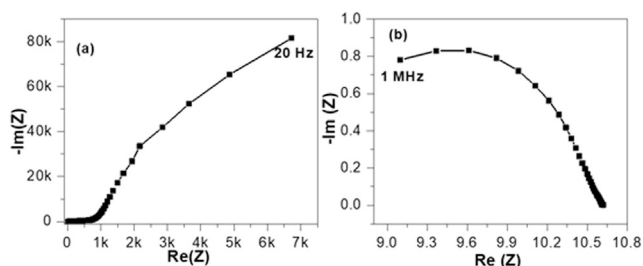

Figure 5. Incomplete impedance spectra
Impedance spectra showing incomplete measuring range (A) low frequency, (B) high frequency

△ CRITICAL: V_{ac} should be maintained as such that it does not affect the linearity of the signal. As the perovskites are not electrically stable, thus increase of measurement time is not advisable.

△ CRITICAL: Give sufficient time interval (2–5 min) between two successive measurements for the stabilization of mobile ions.

8. Mott-Schottky measurement -Day 4
   For this experiment, one should first identify the measuring frequency at which Mott-Schottky plot should be measure. The measuring frequency can be identified from the stable plateau region of the *C-f* spectra shown in Figure 6A. The measurement steps are:
   a. Load setting for "staircase potential electrochemical impedance spectroscopy (SPEIS)" technique in EC Lab software.
   b. Measure in single "single sine" mode.
   c. Set the scan voltage from −0.5 V to +1 V with adequate number of points.
   d. Choose the scan frequency (from *C-f* spectra).
   e. Set AC perturbation V_a = 20 mV, Wait for p_w = 0.1 s period before each frequency measurement, average measure per frequency N_a = 2 and corrects the drift of the system.
   f. Record Mott-Schottky plot.

   △ CRITICAL: Occasionally, linear region in Mott-Schottky plot (Figure 6B) is not visible thus we cannot determine the slope and offset voltage from it. This can occur due to higher ionic capacitance as compare to depletion capacitance. To avoid this, perform the measurement at low temperature where all ionic motion is diminished.

EXPECTED OUTCOMES

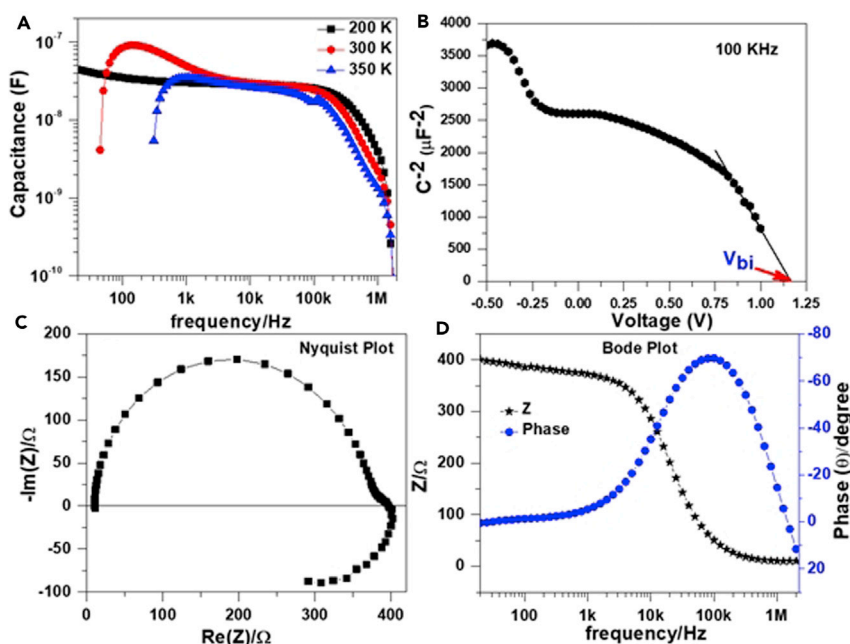The important outcomes of the present protocols are illustrated below:

Figure 6. Immittance spectra
(A) Temperature dependence *C-f* spectra, (B) Mott-Schottky plot, (C) Nyquist plot and (D) bode plot

J-V characteristics: Key photovoltaic parameters: short-circuit current (J_{SC}), open-circuit voltage (V_{OC}), fill factor (FF) and power conversion efficiency are derive from *J-V* characteristics under illumination. Moreover, *J-V* characteristics can also provide the information about parameters affecting device photovoltaic performance including: series resistance (R_S), shunt resistance (R_{Sh}), ideality factor (n), photo current density (J_{ph}), and reverse saturation current density (J_0).

Capacitance spectroscopy: The *C-f* measurement reveals the different capacitive phenomenons in perovskite solar cells such as electronic dipole polarization and charge accumulation at perovskites/charge selective contact interface [Chen et al., (2016)], possible ferroelectric effects [Pockett et al. (2015)]. By using temperature dependence *C-f* spectroscopy (Figure 6A), we extract important charge transport parameters such as distribution of density of states, the attempt-to-scape frequency, traps activation energy. Moreover, the capacitance spectroscopy also constitutes information about the mechanism of undesired *J-V* hysteresis in perovskite solar cells. Further, the temperature dependence *C-f* spectroscopy provides the information on the effect of phase transition of perovskite on its electrical properties. The Mott-Schottky graph is plotted $1/C^2$ vs. E_{we}, from which we can deduce the information about interfacial depletion at perovskites/charge selective contact interface by extrapolating the linear region to voltage axis as shown in Figure 6B. The Mott-Schottky plot provides the information of dopant density (N_{eff}), distribution of trap density, depletion layer width (W), built-in potential (V_{bi}), at perovskites/charge selective contact interface.

Outcomes of impedance spectroscopy: In perovskite solar cells different physical processes occurs at different time scales e.g., the ionic migration is slow as comparison to electronic migration. Similarly, interfacial phenomenon appear at high frequency, whereas bulk phenomenon is slower. Therefore by performing a simple experiment of impedance spectra over a frequency domain, different physical process occurs in perovskite solar cells could be reveal which are not easy by other complicated techniques. Impedance spectroscopy measurement yields the Nyquist and Bode plot as shown in Figures 6C and 6D. These graphs provide both quantitative and qualitative insight into the charge transport phenomenon occurring in a perovskites solar cell. Qualitatively, we can derive the insight of electronic and ionic charge transport, interfacial charge accumulation and recombination, dielectric relaxation process, lattice distortions, electrode polarization in perovskite solar cells. The study will also separate the ionic and electronic transport features from each other. Quantitatively, Nyquist plots are analyzed by fitting with an equivalent circuit. The fitting provides us several important electrical parameters including, charge transfer resistance, recombination resistance, interfacial charge exchange time constant, interfacial double-layer capacitance, bulk capacitance, dielectric constant, ionic, and electronic diffusion coefficient, and mobility, the time constant.

QUANTIFICATION AND STATISTICAL ANALYSIS

The electrical parameters can be derived from thermal admittance (*C-f-T* spectra) using following steps:

1. Plot (–fdC/df) vs. f plot at different temperatures.
2. Extract the frequencies (f_{peak}) of the peak value of - fdC/df at different temperatures.
3. Draw Arrhenius plot $\ln(f_{peak}/T^2)$ vs. 1000/T and evaluate the slope and intercept of the graph.
4. Calculate the activation energy: $E_A = k_B \times slope$ and attempt-to-escape frequency (ATEF) $v_0 = e^{intercept} \times T^2$.
5. Draw Mott-Schottky plot as shown in Figure 6B, and choose the best linear region corresponds to the depletion region in as describe by Almora et al., (2016).
6. Find the interfacial built-in-voltage V_{bi} by extrapolation of the linear region of Mott-Schottky plot to voltage axis and calculate doping density N through slope of straight line.
7. Evaluate depletion width corresponds to zero bias: $W = \sqrt{\frac{2\epsilon\epsilon_0 V_{bi}}{qN}}$

Carry the analysis of impedance spectra by fitting the measure spectra to an equivalent circuit. Different equivalent circuit are being use to model the impedance spectra of perovskite solar cells. Most common equivalent circuit use to analysis the impedance spectra of perovskite solar cells are: (i) Voight, (ii) matryoshka, (iii) Maxwell, and (iv) hybrid Voight-matryoshka (Khan et al. 2020). As an example, here we demonstrated the analyses of impedance spectra exhibits inductive loop using matryoshka circuit contain both capacitance and inductance as shown in Figure 7. Make the fitting using Z-view software. For the extraction of important parameters and elements from equivalent circuit, consult the protocols by Guillen et al., (2014).

LIMITATIONS

Impedance spectroscopy is voltage-dependent base characterization techniques, and it discards the dynamic processes at the various internal interfaces, furthermore, the analysis of data requires complex theoretical models that also neglect the interface roughness.

TROUBLESHOOTING

Problem 1
Low vacuum in the chamber (steps 4).

Potential solution
Solution 1: Check for leakage in the chamber. Sources of leaks can be from, open drain valves, blown gaskets, loose flanges, poorly packed valves, broken pipes and etc.

Solution 2: Impurity/dust present in the chamber from previous deposition cause low vacuum, we recommend proper cleaning the chamber before placing the devices for electrode deposition.

Solution 3: Restriction in the pump inlet piping e.g., close or partially close valves, object in the line, plugged inlet screen, etc. This restriction will cause pressure drop that gives low vacuum in the chamber and a higher vacuum in the pump

Solution 4: Make sure, the films deposited inside the glove box are completely dried otherwise solvent present in films prevent reaching the desired vacuum.

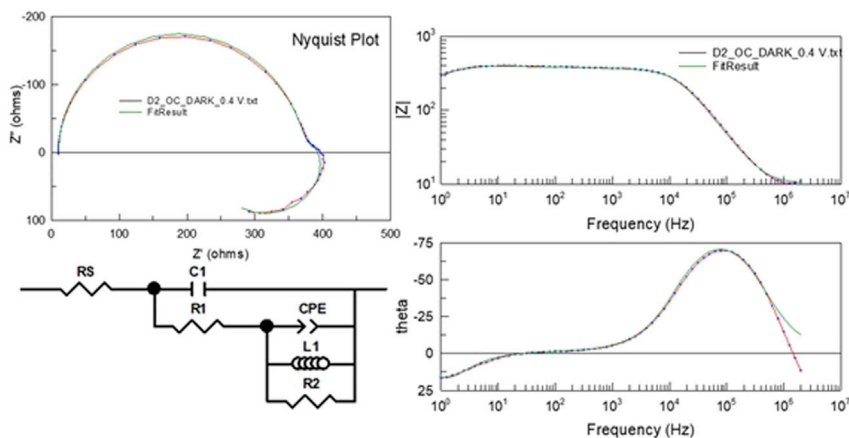Solution 5: Occasionally deposition also take place on vacuum gauge electrode that cause display of false vacuum.

Figure 7. Representative fitting of impedance spectra with equivalent circuit using Z-view software

Problem 2
No or flawed measurement in *J-V* curves (steps 5).

Potential solution
Solution 1: If the *J-V* curves (as shown in Figure 2h) are not typical, or absence of any curve profile, check the cathode and anode contact properly with the help of multi-meter.

Solution 2: Measure the *J-V* characteristics of reference cell and confirm the instrument is giving correct parameters i.e., parameters match with reference values.

Solution 3: Check V_{OC} and J_{SC} under illumination with the help of multi-meter. Very low values indicates, fabricated solar cell is not working.

Problem 3
No or flawed measurement in C-f measurement (steps 6).

Potential solution
Solution 1: In the absence of any spectra, check the connections to device by multi-meter.

Solution 2: Measure the *C-f* spectra for reference capacitors and make sure instrument is giving correct value.

Solution 3: In case of flawed spectra (*C-f* profile), check the device status by measuring *J-V* curve and compare the performance parameter with previous measurement. In case of variation of more than $\pm 5\%$, one should replace the device or repeat the measurements.

Problem 4
No or flawed spectrum in Impedance measurement (steps 7 and 8).

Potential solution
In case of flawed spectrum or absence of any curve, following steps should be performed:

Check connections using multi-meter.
Check V_{OC}
Measure *I-V* characteristics
Total harmonic distortion (THD) indicator can be used to assess the validity of impedance measurement and linearity problem. Before start of experiment check the box "EIS quality indicator". The spectra with low THD value indicates there is no issue related to the linearity of signal. Whereas, if THD changing with frequency, attributed to loss of linearity of the signal. This problem can be resolved by decreasing the V_{ac}.
If part of spectra is missing as shown in Figures 5A and 5B), extend the frequency range.

RESOURCE AVAILABILITY

Lead contact
Further requests for resources and materials should be directed to and will be fulfilled by the lead contact, Shahzada Ahmad (shahzada.ahmad@bcmaterials.net).

Materials availability
This study did not yield any new unique reagents.

Data and code availability
This study did not produce datasets/code.

AUTHOR CONTRIBUTIONS

M.T.K. prepared the initial draft, P.H. contributed to device fabrication, A.A. provided assistance to M.T.K., S.K. has contributed to characterization, and S.A. supervised and directed the research. All authors contributed to the draft and prepared the final version.

DECLARATION OF INTERESTS

The authors declare no competing interests.

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