Standard Operating Procedure

(SOP)

On-Sun PEC Measurement

Test ID #PEC-P-9

Rev 1

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Revision History

This page documents the revisions over time to the SOP. The most recent iteration should be listed in the row space, with consecutive versions following.

Date of Revision	Page(s)/Section(s) Revised	Revision Explanation
04/05/2019	All	First Release

Outline/Table of Contents

1. Title Page
2. Table of Contents
3. Procedures
    a. Scope and Applicability
    b. Summary of Method
    c. Definitions
    d. Health & Safety Warning
    e. Cautions
    f. Interferences
    g. Personnel Qualifications / Responsibilities
    h. Equipment and Supplies
    i. Step by Step Procedure
        - Orientation of On-sun testing apparatus
        - Instrument or Method Calibration and Standardization
        - Sample Collection
        - Sample Handling and Preservation
        - Sample Preparation and Analysis
        - Troubleshooting
        - Data Acquisition, Calculations & Data Reduction Requirements
        - Computer Hardware & Software
    j. Data and Records Management
4. Quality Control and Quality Assurance Section
5. Reference Section

3. Procedures

a. Scope and Applicability – The purpose of this SOP is provide a protocol for "On-Sun Photoelectrochemical (PEC) Device Testing", i.e. outdoor environmental, testing of photoelectrochemical devices and cells. There are two different protocols: 1) benchmarking On-Sun performance at AM1.5G (or D) equivalent spectrum; 2) Testing On-Sun performance under real world conditions.

b. Summary of Method – The procedure consists of assembling the PEC cell on the testing rig, aligning the rig to a reference point, and initiating test.

c. Definitions –

- PEC: photoelectrochemical cell
- AM1.5G: Global Standard Spectrum = Air mass (AM) 1.5, global spectrum (G)
- AM1.5D: Direct Standard Spectrum = Air mass (AM) 1.5, direct spectrum (D)

d. Health & Safety Warning –

- Wear adequate personal protective equipment (PPE) for both the experimental apparatus as well as working outdoors, with environmental exposure.
- The motors that move the solar tracker have considerable torque, keep a foot away while the machine is swinging into position.
- If using electrolyte solutions be careful of splash hazards and ensure that solution will not spill from the cell as the solar tracker moves around during the course of a day.

e. Cautions – Not applicable for this SOP

f. Interferences – Not applicable for this SOP

g. Personnel Qualifications / Responsibilities – user should be familiar with indoor PEC testing, STH and IPCE measurements

h. Equipment and Supplies –

- Photoelectrochemical cell.
- On sun test stand: test stand capable of tracking the sun and holding the PEC cell, as well as a data acquisition system.
- Electrolyte or water supply as necessary depending on type of cell.

i. Step by Step Procedure

- Orientation of On-sun testing apparatus
    a. Orient the solar tracker relative to true magnetic north
        i. This is identified by an arrow on top of the solar tracker and the use of a compass.

b. Mount PEC cell to solar tracker
    i. Attach cell to the tracker with the photoactive component facing towards the light source
c. Fill with electrolyte, or water
    i. Depending on the test being performed, number of compartments, type of electrolyte, water or vapor feed. Fill or connect appropriate pump and water O_2 and H_2 separators.
d. Connect data acquisition cables to the PEC cell
    i. There are 4 "signal leads." 2 that sense voltage, 2 that sense current. Connect the working electrode (WE) voltage and current lead to the WE side of the PEC cell. Connect the counter electrode (CE) voltage sense to the CE side of the PEC cell
    ii. These leads are routed to a National instruments A/D module and a Hewlett Packard (Agilent) multimeter, respectively.
    iii. Labview does the data capture and analysis over long periods of time on a laptop.
    iv. The electronics are mounted inside the box underneath the solar tracker. There is a fan mounted inside the box to keep it cool while it's sitting in the sun.
e. Connect solar tracker to power cable.
    i. Initialize data acquisition software
f. Turn on the solar tracker
    i. When one starts the machine, it takes its GPS coordinates and uses this with the orientation information to set the correct angle for the solar altitude.
    ii. The solar tracker will then point towards where the sun should be, takes a measurement with its "peaking sensor," and proceeds to maximize its pointing accuracy. This way it's always pointed towards the brightest spot in the sky. WARNING: The motors that move the solar tracker have considerable torque, keep a foot away while the machine is swinging into position.

- Instrument or Method Calibration and Standardization –
    a. For accurate on-sun benchmarking measurements require exclusion of diffuse radiation that is readily coupled into PEC cells and inflates photocurrent measurements. The following additional, optional procedure was established for using the direct solar radiation only, which being nearly collimated, also mitigates error from refraction at and optical concentration by the PEC cell interfaces.
    b. Measure direct solar irradiance every minute by a Kipp & Zonen pyroheliometer and spectral data ever five minutes by a NIST traceable Prede PGS-100 Direct Normal Spectroradiometer. Matching that of the pyroheliometer, a 5° field-of-view collimating tube is (constructed following ASTM E1125-9940) placed on the on sun side of the PEC cell through which the device being benchmarked is

illuminated. Shadow alignment marks ensure a collimating tube pointing accuracy better than 1°. With clear skies (broadband turbidity ~0.2 or lower), the spectral mismatch between the solar illumination provided by this configuration and AM1.5D is 1% or less between ~9 am and ~11 am. Regardless, the minor spectral correction was performed using IPCE and real-time spectral data following established procedures.

c. The spectral correction procedures are based on those used for a primary reference cell (RC) calibration (reference SOP), where the short-circuit photocurrent of a reference cell, measured outdoors, is translated to reference illumination (for example AM1.5D or AM1.5G). Here, the device to be benchmarked takes the place of the reference cell. The measured photocurrent I_{sc}^{meas} is translated to reference conditions at 1000 W/m^2 following

   i. $$I_{sc}^{0} = I_{sc}^{meas} \frac{1000\ \mathrm{W\,m^{-2}}}{E_{meas}} \times CN,$$

d. where I_{sc}^{0} is the desired photocurrent, E_{meas} is the total measured irradiance in W/m^2, and CN is the so-called calibration number, given by

   i. $$CN = \frac{\int_{300}^{4000} E_{REF}(\lambda)\, SR^{RC}(\lambda)d\lambda \cdot \int_{300}^{4000} E_{INC}(\lambda)d\lambda}{\int_{300}^{4000} E_{REF}(\lambda)d\lambda \cdot \int_{300}^{4000} E_{INC}(\lambda) SR^{RC}(\lambda)d\lambda},$$

e. The CN accounts for the spectral mismatch between the measured outdoor spectral flux $E_{INC}(\lambda)$, the reference spectral flux $E_{REF}(\lambda)$, and the IPCE or spectral response of the device, $SR^{RC}(\lambda)$, and as noted above is on the order of ~1% for our measurements. The integration is taken over the wavelengths 300 - 4000 nm of the full solar spectrum. In principal, $E_{meas} = \int_{300}^{4000} E_{INC}(\lambda)d\lambda$, but we use different subscripts to distinguish between the two quantities, since in practice they are measured on separate instruments, each with its own accuracy.

f. For tandem devices, I_{sc}^{0} is first calculated for each junction individually using its subcell $SR^{RC}(\lambda)$, assuming that it is the current limiting junction. The quoted I_{sc}^{0} for the tandem device is then taken as the lower value between the two subcell values.

j. Data and Records Management – Not applicable for this SOP.

4. Quality Control and Quality Assurance Section

Not applicable for this SOP.

5. Reference Section

R1. Döscher, H., Young, J. L., Geisz, J. F., Turner, J. A. & Deutsch, T. G. Solar to hydrogen efficiency: Shining light on phoelectrochemical device performance. *Energy Environ. Sci.* **9,** 74–80 (2015).