NREL
Transforming ENERGY

"Round-Robin Verification of Specimen Temperature During Accelerated Testing for the PVQAT TG5 Studies"

David C. MILLER,[1*] Dennis DIETZ,[2] Brian HABERSBERGER,[3] Caitlin HALE,[2] Fabiana LISCO,[4] Wei LUO, Joshua MORSE,[1] Nancy PHILLIPS,[6] Lorenzo TYLER,[2] Sona ULICNA,[4] Doug VERMILLION,[7] John M. WALLS,[4] Peter WILLIAMSON,[2] Allen ZIELNIK,[2]

[1]National Renewable Energy Laboratory, Golden, CO, USA
[2]Atlas Material Testing Technology L.L.C., Mount Prospect, IL, USA
[3]The Dow Chemical Company, Lake Jackson, TX, USA
[4]Loughborough University, Loughborough, UK
[5]Solar Energy Research Institute of Singapore (SERIS), Singapore, SG
[6]DuPont Photovoltaic Solutions, Wilmington, DE, USA
[7]Eye Applied Optix, Mentor, OH, USA
[*]Presenting author

NIST-UL PV Materials Reliability workshop
Friday, 2019/12/13, 15:10-15:35
NIST, Gaithersburg, MD, Building 101, Portrait room

Specimen Temperature Verification: Topics for Today

- Motivation & introduction (2 slides)
- Quantification and analysis of **measurement precision** (4 slides)
- Specimen **temperature** verification in **artificial weathering**
  - -Discovery experiments (5 slides)
  - -Precision study (3 slides)
- Specimen **temperature** validation in **natural weathering** (5 slides)
- Summary & conclusions (3 slides)

Why Knowing the Specimen Temperature Is Critical to Accelerated Testing (Motivation)

- Photo- & hygro-degradation are typically thermally activated.
- A degradation mode's rate limiting (effective) chemical reaction may be modeled using Arrhenius representation.
- Example: EVA discoloration, E_a ~40 kJ·mol^{-1} (0.41 eV).

Miller et. al., Prog. Photovolt Res. Appl., 27, 2019, 391–409.

- Example: PET hydrolysis, E_a ~130 kJ·mol^{-1} (1.4 eV).

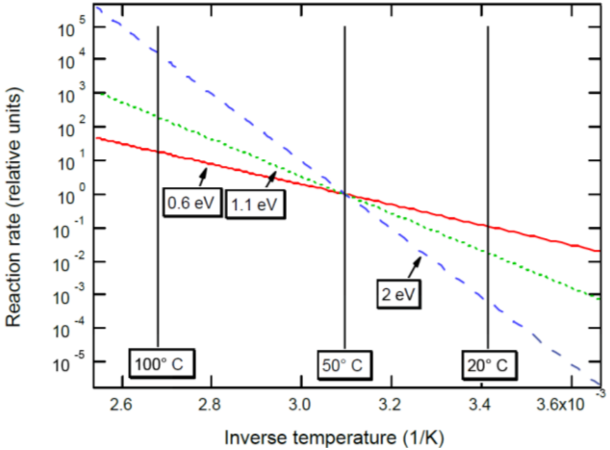
Pickett et. al., Polym. Deg. Stab. 98, 2013, 1311–1320.

$$k = A\left[\frac{T}{T_0}\right]^n e^{\left[\frac{-E_a}{RT}\right]}$$

The modified Arrhenius equation

- *k* varies *by orders of magnitude* with E_a & $T_{specimen}$.
- Specimen temperature is not directly controlled in most artificial weathering chambers!!!
- Ambient *T* variation complicates natural weathering. (Temperature varies readily outdoors).

⇒Today: verification of specimen temperature for IEC 62788-7-2 (artificial) and natural weathering.



Comparison of reaction rate as a function of E_a or T

Kurtz et. al., Proc. IEEE PVSC, 2009, 2399-2404.

Example From a Weathering Chamber Manufacturer (Motivation)

Specimens in Ci5000

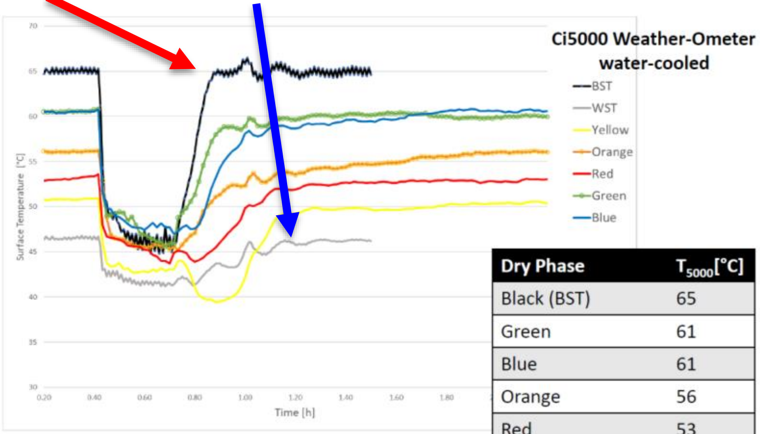
Feil, Proc. ASTM G03 Work., 2019.

- The most common PV material colors: transparent, white, black.
- Specimen color affects optical absorptance (radiant heating).

- Example: comparison of 5 different specimens in Xe chamber:
  - -5 distinct specimen temperatures realized!!!
  - -Bounded by black panel temperature and WPT.

- Specimen *T* verification is essential to quantitative accelerated testing and weathering science.

- Other industries may use many more colors than PV.
- Other industries may benchmark 1 specific product color (e.g., black runs hottest).



Dry Phase	T_{5000}[°C]
Black (BST)	65
Green	61
Blue	61
Orange	56
Red	53
Yellow	51
White (WST)	47

PV 3930 cycle in Ci5000 (Xe chamber).

Feil, Proc. ASTM G03 Work., 2019.

Interlaboratory (e.g., "Round-Robin") Verification of Measurement Precision

- **ASTM D7778** on interlaboratory studies **specifies to quantify** the ***precision*** and ***bias*** of each new test method developed. (preferred, not required in IEC/ISO system).
- ASTM E691 **defines** that "**precision**" means the ***repeatability*** and ***reproducibility*** with 95% confidence.
- <u>repeatability, r</u> – "What is the average of the variance for the laboratories", i.e., how tightly clustered are the data within each laboratory?
- <u>reproducibility, R</u> – "How well do the data sets overlap, and how tightly clustered are the data within each lab?" *(between labs & within labs)*
- Now conduct an interlaboratory ("round-robin") experiment to evaluate r and R. ☺

TABLE 11 Glucose in Serum—Precision Statistics

NOTE 1—This table (with the column for $s_{\bar{x}}$ omitted) is a useful format for the presentation of the precision of a test method as required by Section A21 of the *Form and Style of ASTM Standards*.

Material	$\bar{x}$	s_x'	s_r	s_R	r	R
A	41.5183	0.6061	1.0632	1.0632	2.98	2.98
B	79.6796	1.0027	1.4949	1.5796	4.19	4.42
C	134.7264	1.7397	1.5434	2.1482	4.33	6.02
D	194.7170	2.5950	2.6251	3.3657	7.35	9.42
E	294.4920	2.6931	3.9350	4.1923	11.02	11.74

Example results of precision table (-your goal-) from ASTM E691.

<u>Where to document:</u>

- ASTM readily accepts precision & bias tables within the standard (as a section, possibly with an Appendix).
- IEC might include precision table in an Informative Annex.
- Publish the precision table (conference or journal) and reference it.

Analysis of Interlaboratory Verification of Measurement Precision

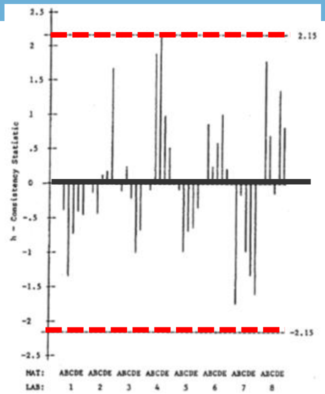
- Mandel's *h* and *k* statistics are an intermediate step allowing for the identification of outliers:

- *h* = between laboratory consistency.

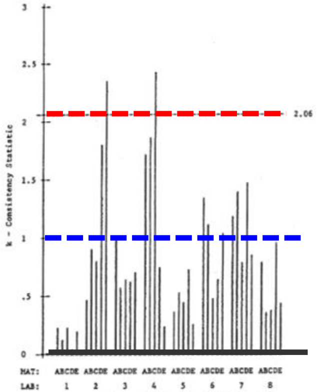
$$h = \frac{d}{s_{\bar{x}}}$$

- *k* = within laboratory consistency.
  k =1 is average (typical) variability.

$$k = \frac{s}{s_r}$$



Example h plot from ASTM E691 (between laboratory consistency).



Example k plot from ASTM E691 (within laboratory consistency).

- Arrange data tables in order of increasing result values (to identify value-related bias).
- Limits (0.5% significance) for *h* & *k* given in Table 5 of ASTM E691.

Treatment of Outliers (Mandel's Tests, Continued)

Look for in ***h* (between laboratory consistency)**:

- One lab is all positive (or negative) when other labs are not ⇒ verify results for that lab.
- All low values for *h* are one **sign**, whereas high values are opposite sign ⇒ value dependent trend?

Look for in ***k* (within laboratory consistency)**:

- One lab has very **high** (**or** very **low**) *k* **values** relative to others
  ⇒ poor repeatability or insensitive measurement scale?
- Values **all > 1**
  (within laboratory variability greater than average) ⇒ result of the test material?

Consider also:

- **Sort** and plot *h* and *k* **by lab** and look for a trend ⇒
  multiple outliers may indicate operator/equipment issue at a particular lab.
- Look for trends in histograms of the –**means**- and –**ranges**- of results
  (not a Mandel-based analysis).

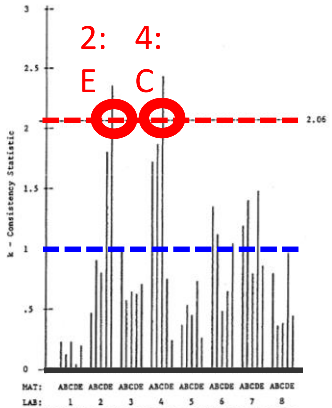


FIG. 2 Glucose in Serum: *k*—Materials within Laboratories

Example showing k sorted by material (within laboratory consistency). From ASTM E691.

Further Identification and the Treatment of Outliers

<u>Cochran's test</u> (for subtle differences within labs [*k, sort of*] :

$$C = \frac{s_{\max}^{2}}{\sum_{1}^{p} s^{2}}$$

- See Table 4 in ISO 5725-2.
- Requires same # of standard deviations obtained within r & R
(enforce performing complete experiments, else exclude a lab's results)!

<u>Grubb's test</u> (for subtle differences between labs [*h, sort of*] :

$$G_p = \frac{\left(x_p - \bar{x}\right)}{s}$$

- Arrange data values in ascending order.
- See Table 5 in ISO 5725-2.

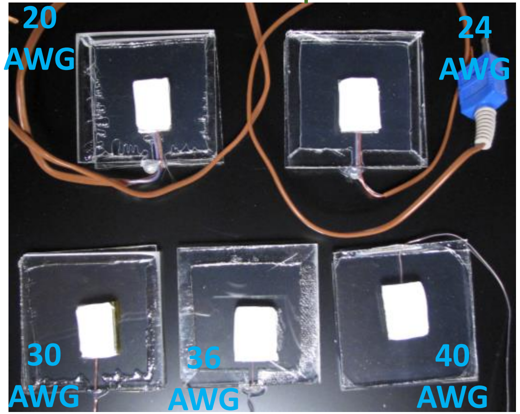
<u>What to do when outliers are observed</u>:

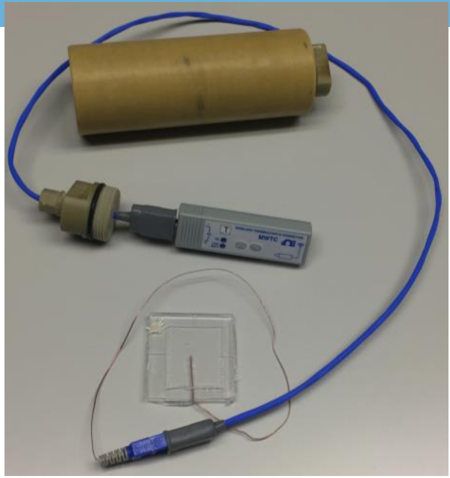
- Try to identify a possible reason (*e.g.*, particular material, technician, or instrument).
- Require retest (of material and/or laboratory). Then rerun analysis.
- Censor (remove) data. Option particularly when large number of other labs with similar results.
- Improve your test method, if applicable/appropriate.
- Mandel's, Cochran's, Grubb's tests:
If outliers are identified, rerun the analysis (with a lesser participants or materials) to identify additional outliers.

The Specimens Used in Discovery Experiments (Artificial Weathering)

•Quantify significance of (a)TC wire diameter (thermal conduction) and (b)view factor (radiative heating of TC bead) using easily shared measurement system.

TC wire diameter specimens





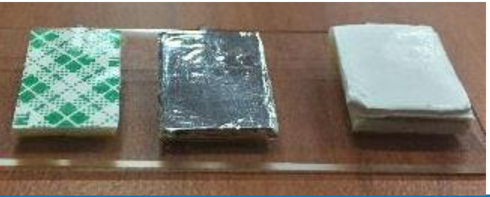
Components

Components:

-Laminated glass/EVA (1x)/glass coupon
(TC's include: 20, 24, 30, 36, 40 AWG wire)

-Extension cable
-G11 / FR5 housing (hermetic, with thermal capacitance)
-Wireless data logging
(Omega MWTC-D-T-915 **transmitter** & Omega MWTC-REC1-915 **receiver**)

-Mask
(double-sided foam adhesive tape, heavy duty Al, white PTFE)
Thanks John Sparks for the mask design!



Layers of the mask

Components in Ci5000

Details of the Specimen Temperature Verification Experiments

General procedure:

- Run chamber for ~90 min (no light), then ~90 min (Xe light). (Verify ChT then specimen & BPT).
- Data collected at 1 Hz.
- Extract data from last 1000 s of each experiment.

(chamber temperature typically stabilizes within 30 min).

Selecting the specimens:

- Compare 4 replicates to select "median" specimen

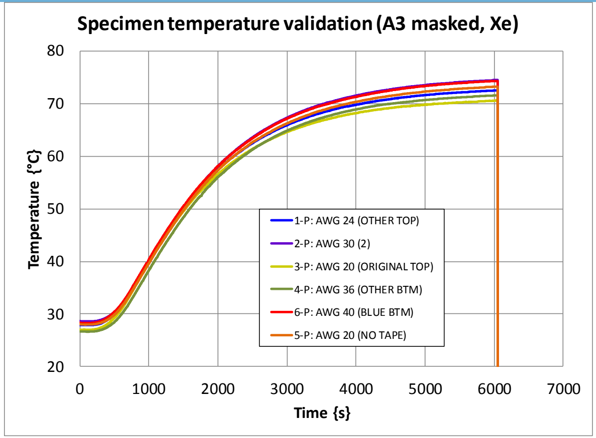
(20, 24, 30, 36, 40 AWG) for formal experiment.

Discovery experiment:

- Repeat at the IEC 62788-7-2 test conditions:

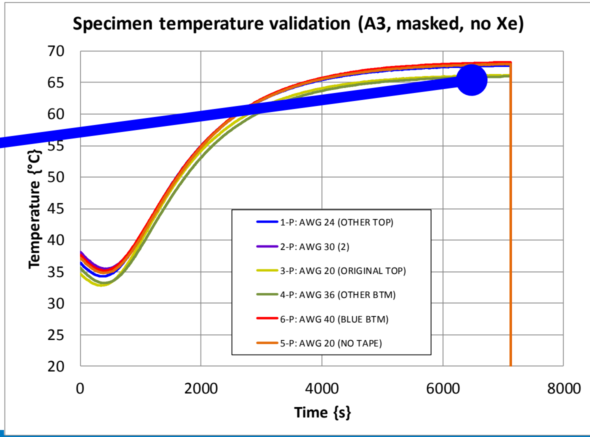
A1, A2, A3, A4, A5.

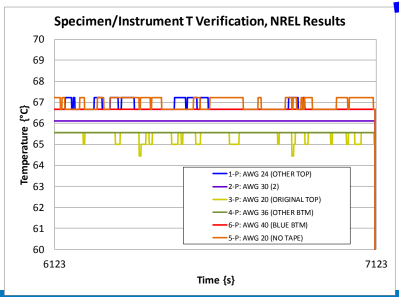
(ChT 45 – 85 °C).



A3 Xe light data

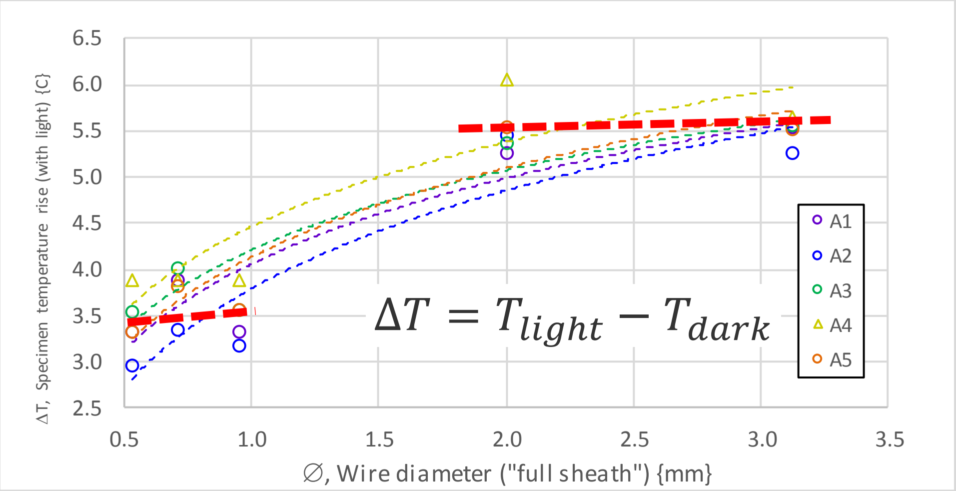
A3 dark data



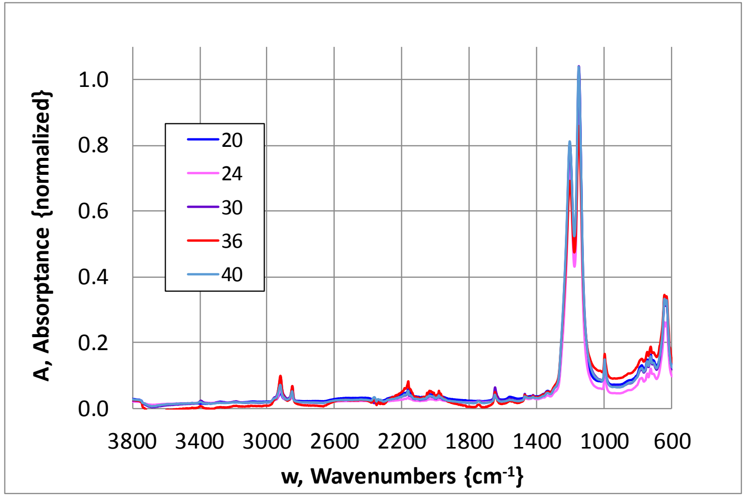


Temperature Was Discontinuous With Wire Diameter

- A gradual variation with wire diameter was expected (arbitrary exponential fit).
- A stepped variation was instead observed (dashed lines).
- Temperature discontinuity was observed at all chamber temperatures (A1-A5).



Temperature rise vs. wire diameter for unmasked specimens.



FTIR spectra for the TC jackets. Each wire gage is indicated.

- FTIR suggests TC cable jackets are composed of the same base polymer material.
- Weathering of the cable jacket not examined. ;b

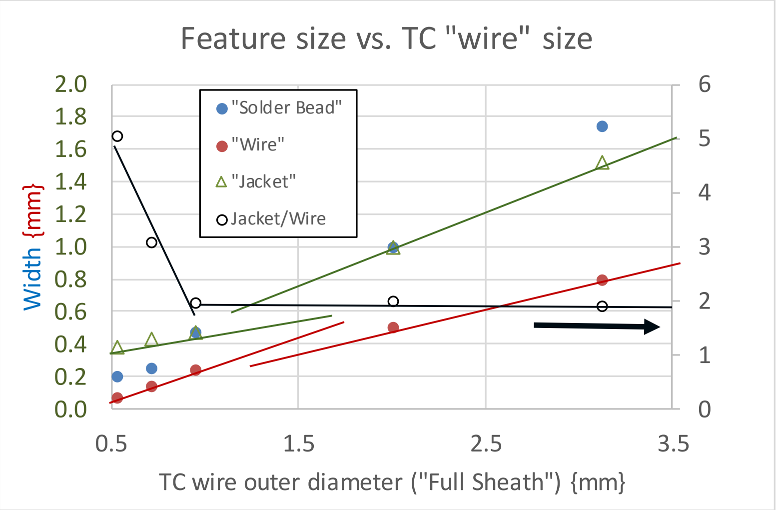
An Explanation? Cable Jacket Thickness Varied Between the TC's!

Examine physical dimensions of the TCs:

- Solder bead, wire, jacket ∅ vary with TC AWG.

- Jacket thickness (**Jacket:Wire**) becomes distinct with AWG.
  ⇒Presumably jacket thickness affects heat transfer.

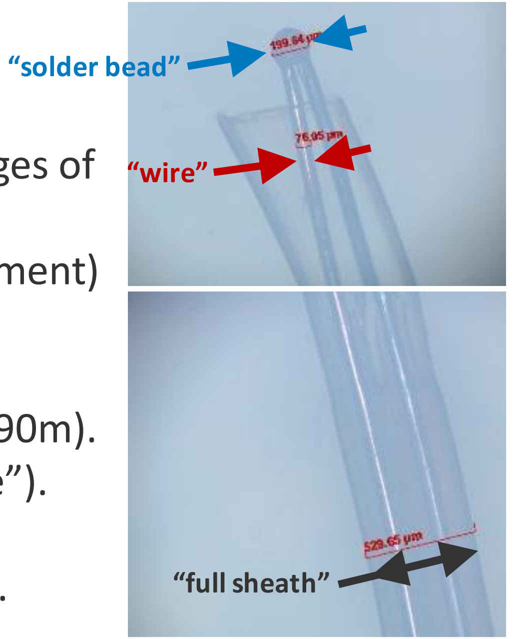
Analysis of TC feature size



- Shown here: example of challenges of common equipment.
- Pyranometer ($$$ add-on equipment) may be used instead of TC's.
- Other concerns:

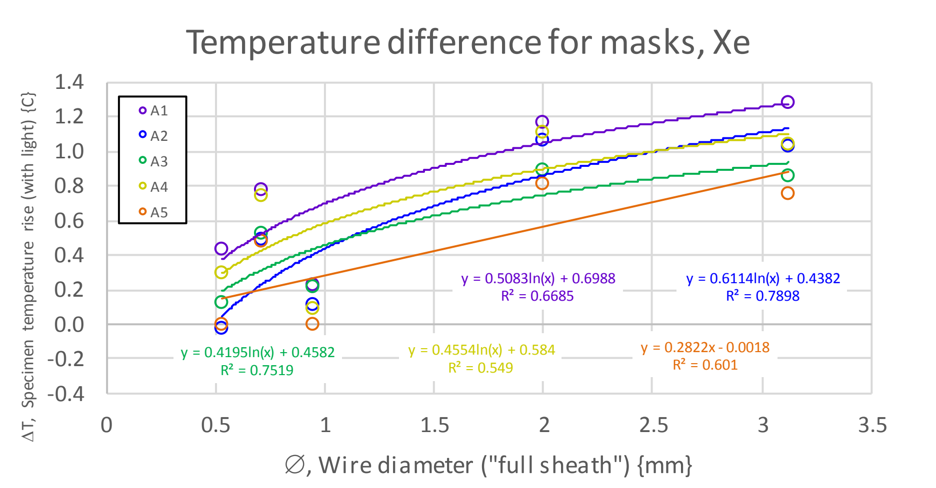
-Overheating transmitters (use *t*~90m).
-Transmitter range (in metal "cage").
-Fragility of coupon with TC wire.
-*T* rise radiant heating (next slide).

Microscope images of 40 AWG specimen



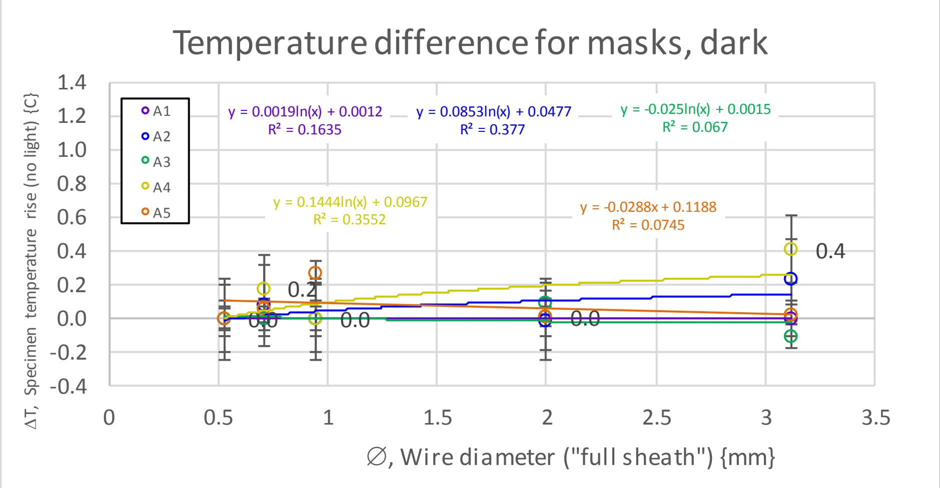
"jacket" = ("full sheath" – 2 · wire)

Effect of Radiative Heating Limited to ~ 1°C for Thickest Wire



Temperature difference (in Xe, with and without mask).

"*Difference*" is $T_{no\ mask} - T_{mask}$



Temperature difference (no Xe, with and without mask).
Error bars (2 S.D.) shown for reference.

- Temperature *difference* 0.4±0.2 °C (~11% of *T rise)*, with masks present, 36 & 40 AWG.
- Temperature *difference* 1.0±0.2 °C (~18% of *T rise)*, with masks present, 20 & 24 AWG.
- Voids adjacent to TC *wire* did not greatly affect result. (Replicates gave consistent *T*'s.)

- Modest *T* rise. Masked configuration selected for more accurate representation.
- 30 AWG selected for formal coupon specimen temperature verification.

The Specimens Used in Verification of the Artificial Experiments

Components:

-Transmitter, housing, receiver same as before.

Coupons (front/encapsulant (1x)/back):

-**Silica/EVA/Silica**

"**SES**", as in majority of TG5 S2

-**Float glass/EVA/Float glass**

"**FEF**", architectural glass for maximum T_{rise}

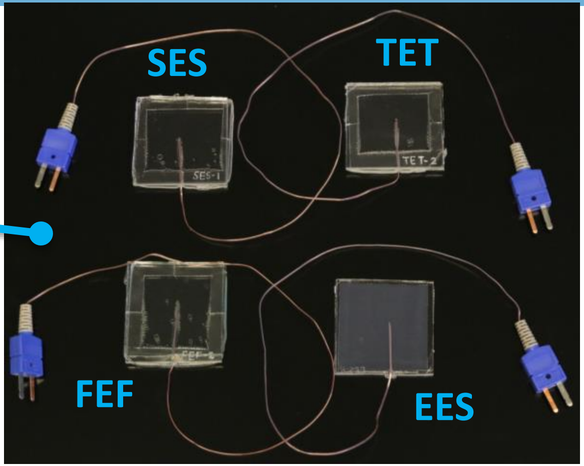
-**Textured glass/EVA/Textured glass**

"**TET**", rolled Solite glass as in PV module

-**EFTE/EVA/Silica**

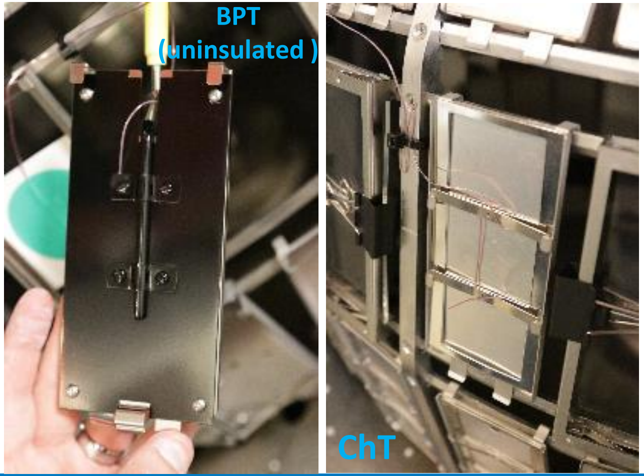
"**EES**", including (1x) frontsheet

The four coupon configurations.



- Glass was first solarized for 500 h in SunTest (Xe chamber).
- TG5 Study 2 must verify $T_{specimen}$ before artificial weathering R-R.

Layers of the mask



Black panel and chamber verification configurations.

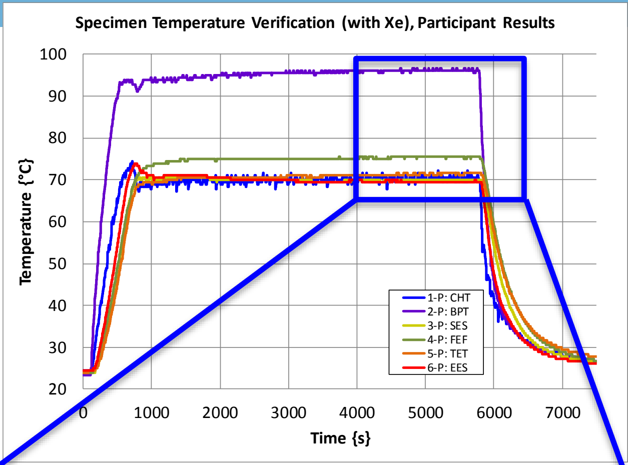
Lessons Learned From Representative Data in the Interlaboratory Study

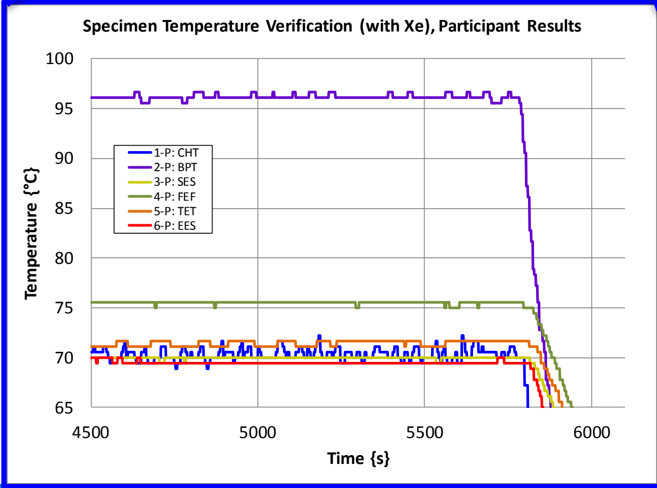
- ChT and BPT are the least stable channels.
  TC's directly exposed to the chamber environment (including blowing air for temperature control).
  Specimen TC's have added thermal mass of laminated coupon.

- The periodicity of the temperature control algorithm may show up in ChT and BPT data.
  It is not clear why this is not evident for all participants/labs (as shown in the example).

- Make sure to include ChT and BPT in your study!
  These "results" help to diagnose each experiment/lab.
  Analyze precision of ChT and BPT along with your specimens.

- Thermal time constant of stabilization typically 15-30 minutes.
  Verify that data is stabilized prior to analysis.





Data from the interlaboratory study, shown for masked specimen with Xe light.

Results: the Precision Study Gives Numbers to the Typical Temperatures

- Last 1000 seconds analyzed in 200 s readpoint "replicates".
- r (within lab repeatability) ≤ 0.5 °C! (2σ) ☺
- R (between lab reproducibility) ranges from 3-6 °C.

-Hunt et. al. (Proc. ASTM G03 Work., 2019):

10 °C specimen temperature variation between machines.

5 °C specimen temperature variation within same machine.

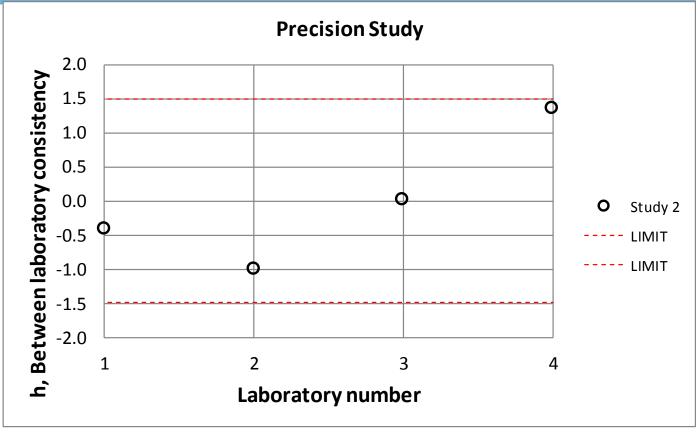
-T-type thermocouple is ±2 °C.

- ChT verified in dark and with irradiation.
- Can you also participate? Please help IEC 62788-7-2!

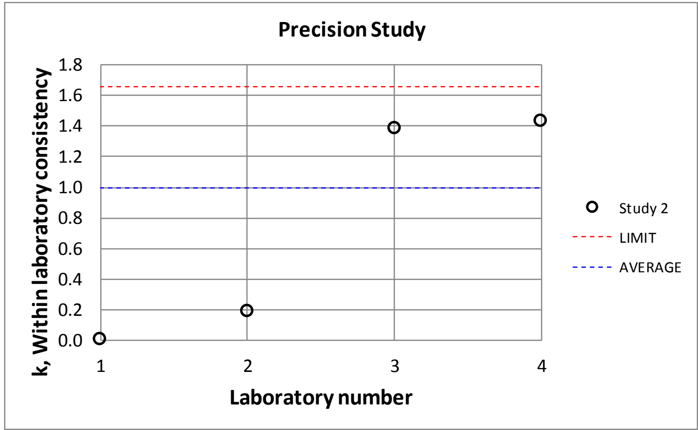
(To be applied to encapsulants, backsheets, …)

SPECIMEN	DESCRIPTION	C_1'' {°C}	s_x'	s_r	s_R	r	R
SES	Silica/EVA/Silica	70.9	0.1	1.0	1.0	0.4	2.8
TET	Textured glass/EVA/Textured glass	72.5	0.2	2.1	2.1	0.5	5.9
FEF	Float glass/EVA/Float glass	76.0	0.1	1.2	1.2	0.3	3.4
EES	EFTE/EVA/Silica	71.4	0.1	2.5	2.5	0.3	6.9
ChT_E	chamber temperature (illuminated)	70.5	0.2	0.9	0.9	0.5	2.5
BPT	black panel temperature	94.7	0.0	1.8	1.8	0.1	4.9
ChT_d	chamber temperature (dark)	67.9	0.1	1.1	1.1	0.2	3.0

Precision for the various coupon specimens as well as the chamber and black panel.

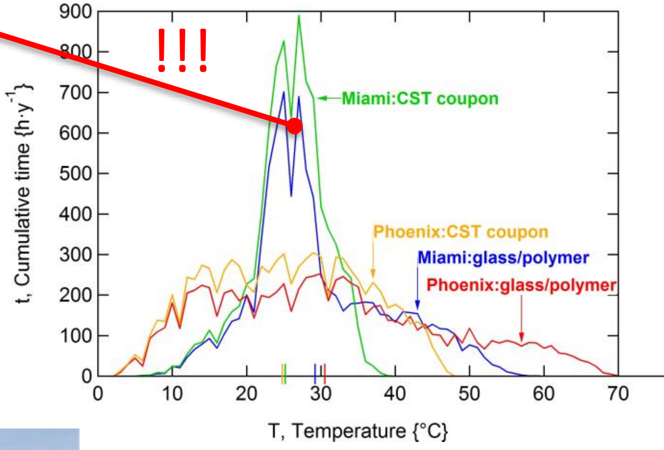


Precision for the various coupon specimens as well as the chamber and black panel.



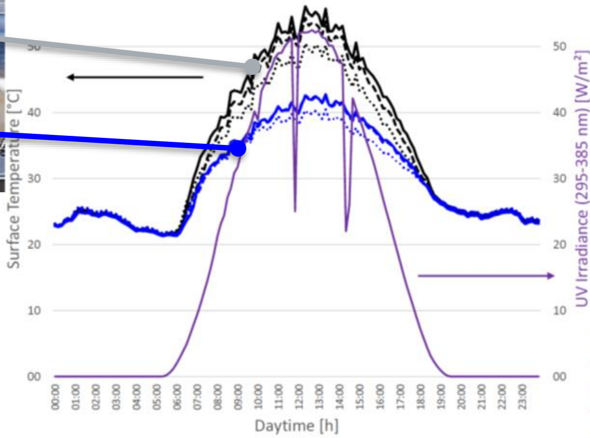
Why Knowing the Specimen Temperature Is Critical to Natural Weathering

- Once coupon specimens are used, be aware they may function differently outdoors than the complete product! (Example from TG5 Study 1, relative to King model/TMY3 for PV).



Unbacked transparent coupon specimen may reach very different temperatures than PV modules (TG5 Study 1).

- Temperature can vary significantly, depending on absorptance (color). (Consider example of white-, black-panel temperatures).



White panel, black panel, other… temperature is still a concern in natural weathering.

Feil, Proc. ASTM G03 Work., 2019.

The Outdoor Fixture and Its Use for Natural Weathering

<u>Components</u>:

-Al backplane (mechanical support).

-Foam core (reduce heat transfer).

-Outdoor rated front absorber.

⇒Silicone rubber top layer next time.

-PMMA clips (specimen attachment, use PEEK with concentrator).

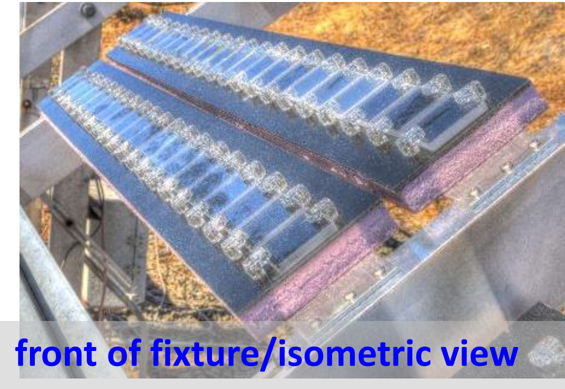
•Attach to rack at latitude tilt.

<u>Coupons more delicate than modules!</u>:

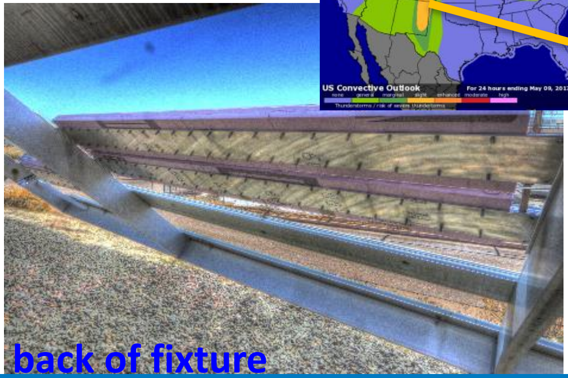
•Hail shield
Essential in CO, deploy for day based on convective weather forecast.

•Safe storage in case of hurricane (Miami, Singapore).

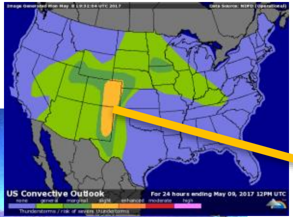
fixture on rack

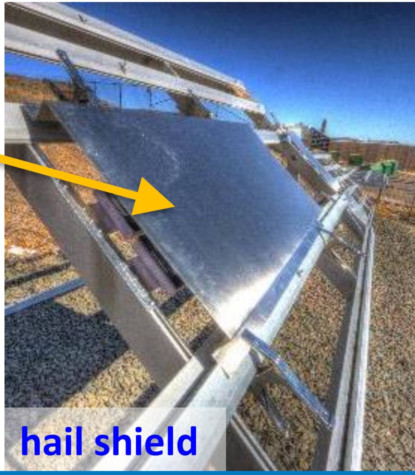


front of fixture/isometric view



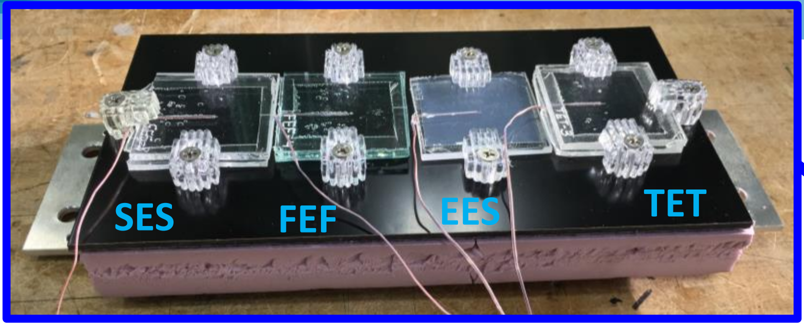
back of fixture



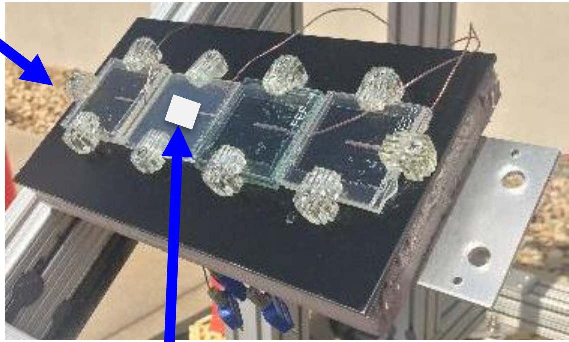


hail shield

The Specimens Used in Verification of the Natural Weathering Experiments



Assembled prototype, with the four coupon configurations



Components:

-Laminated coupons (with TC)
(SES, FEF, TET, EES)
-Mask
(double-sided foam adhesive tape, heavy duty Al, white PTFE)
-Wired data logger
(Omega RDXL4SD)
-Sign/foam/Al sample fixture geometry
(simulate T_{module})

Layers of the mask

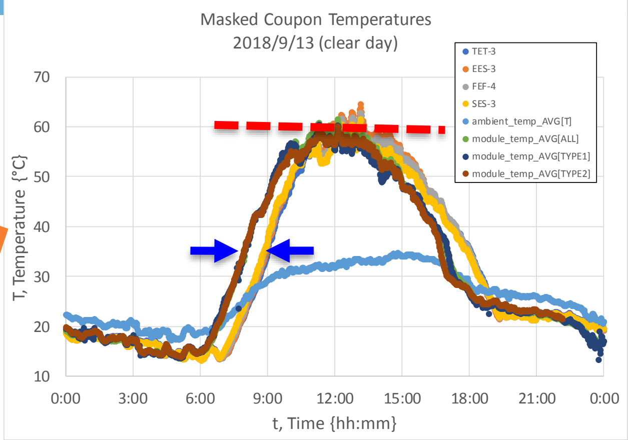
Data logger

- Obtain data for multiple hot (summer) days.
- Compare to NREL OTF PV array, including mono-Si, poly-Si, and thin film PV modules.
- Identify representative $T_{specimen}$ during natural weathering R-R.

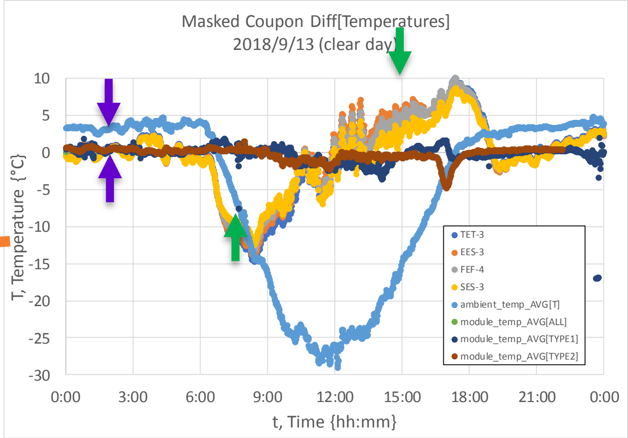
Lessons Learned From Representative Data in the Outdoor Study

Data shown for NREL (masked coupons), relative to glass/BS modules:

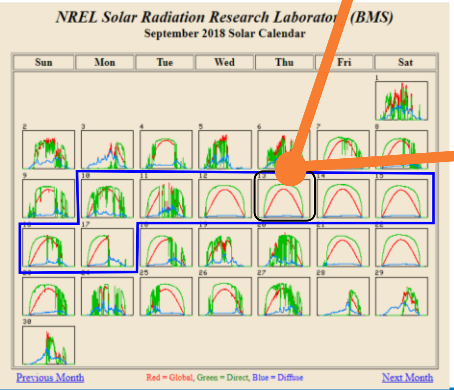
- Hot, clear sky day chosen as example.
- ~30 minute time lag for coupons relative to PV modules. (Different components… different thermal time constant).
- Coupons achieve *T* similar to PV modules. Coupons accelerated relative to modules (ΔT_{max} of ~5°C).
- Coupon temperature is less than module at beginning of day; greater than module temperature at end of day (sunset).
- Effect of radiative cooling observed at night (ΔT of ~3°C, coupons & PV).



Temperature data for clear day



Temperature for clear day (relative to PV)



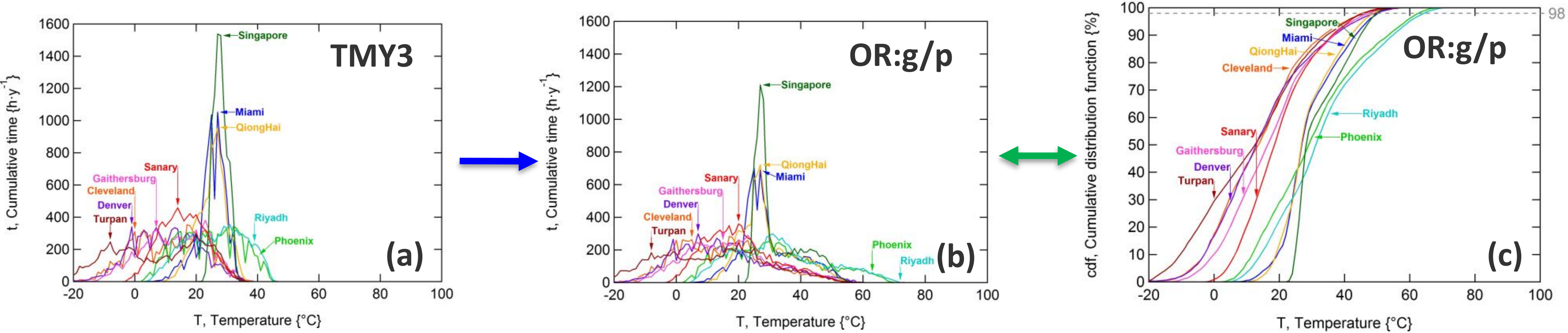
Validating Specimen Temperature Relative to the Natural Weathering Sites

•Module temperature can be estimated, e.g., TMY3 data and e.g., King model.

•Module *T* estimates obtained for PVQAT TG5 S1 & S2 natural weathering sites.

• US & world benchmark locations: hot-dry sites (BWh) & hot-humid sites (Af & Am) readily distinguished from other locations.

•Hope to verify $T_{fixture}$ is accelerated/similar relative to T_{module} (estimated or measured).



Time-temperature history: (a) of TMY3 data and (b) King model for open rack glass/polymer backsheet module. The cdf is shown in (c) for the data from (b).

Summary and Conclusions

- Temperature can vary with geometry & optical absorptance (color) $\Rightarrow$ verify $T_{specimen}$.
  Important in both artificial & natural weathering!!!
- Precision (repeatability and Reproducibility) should be evaluated in an interlaboratory study using the methods in ASTM D7778, ASTM E691, ISO 5725-2.
- Details of experiment (vendor's wire jacket thickness) may complicate *T* verification.
- Modest *T* rise (0.4 °C) observed from radiative heating of TC bead $\Rightarrow$ use mask.
- *r* of $\leq$ 0.5 °C and *R* of 3-6 °C observed so far for artificial weathering.

Can you help with the $T_{specimen}$ study (IEC 62788-7-2 for Xe weathering)?

- Distinct temperature difference can be observed between coupon and module specimens, when no cell is present.
- Initial validation of temperature for natural weathering is encouraging.
  (30 min time lag, <5 °C acceleration for specimen fixture relative to PV).

Getting Involved… The PVQAT TG5 Efforts

PVQAT
International PV Quality Assurance Task Force

There are two regional TG5's (UV weathering).
Each group focusing on different supporting activities (experiments).
You may participate/follow more than 1 of the groups. ☺

- TG5 "X" (encapsulants; now looking ahead to backsheets and test sequences).
  We welcome participants from other regions!
  Contact: David MILLER <David.Miller@nrel.gov>

- TG5 Japan (sequence of tests; MiMo study; encapsulant delamination).
  Combined work with TG2 & TG3.
  Contact: Tsuyoshi SHIODA <Tsuyoshi.Shioda@mitsui-chem.co.jp>

See: http://www.pvqat.org (PVQAT effort)

also: http://pvqataskforceqarating.pbworks.com (minutes, references, attachments, meeting recordings)

Acknowledgements

☞Thanks also to Amanda FLORENDO, Jorge RIVERA, Audrey THOMAS, Kristin WILKINS, and Erika WUNDERLICH (Atlas/Ametek).

- If interested in today's experiments or TG5 activities, please contact David.Miller@nrel.gov

This work was supported by the U.S. Department of Energy under Contract No. DE-AC36-08-GO28308 with the National Renewable Energy Laboratory. This material is based upon work supported by the U.S. Department of Energy's Office of Energy Efficiency and Renewable Energy (EERE) under the Solar Energy Technologies Office (SETO) office.

Your questions and feedback are much appreciated! Please help me to cover the important details & perspectives.

NREL STM campus, Dennis Schroeder