## NIST Technical Note 1764

# High-Temperature Guarded-Hot-Plate and Pipe Measurements: 2nd Operators Workshop (March 19-20, 2012) Co-sponsored by ASTM Committee C16 on Thermal Insulation

Robert Zarr Thomas Whitaker Frank Tyler

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U.S. Department of Commerce Rebecca Blank, Acting Secretary

National Institute of Standards and Technology Patrick D. Gallagher, Under Secretary of Commerce for Standards and Technology and Director Certain commercial entities, equipment, or materials may be identified in this document in order to describe an experimental procedure or concept adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the entities, materials, or equipment are necessarily the best available for the purpose.

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## 1. Introduction

The workshop on high-temperature guarded-hot-plate and pipe measurements was held on March 19-20, 2012 at the National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland, USA. The workshop was co-sponsored by the ASTM International Subcommittee C16:30 on Thermal Measurement and by the National Institute of Standards and Technology. This was the second workshop in a series that specifically focused on the needs of operators conducting these types of measurements.

## 2. Workshop Objective and Goals

The objective of the workshop was to examine and to improve the general understanding of the operation of the guarded-hot-plate and pipe apparatus at elevated temperatures (up to 650 °C).

The ultimate goal of the workshop was to examine and, hopefully, to reduce the present levels of variation in inter-laboratory comparisons of guarded-hot-plate and pipe apparatus measurements at elevated temperatures. To support this effort, five general areas of discussion were identified by the organizers and are presented below:

- 1) the role of NIST with respect to the development of high-temperature thermal insulation reference materials and/or measurement services for the public;
- the role of calibration in the metrological traceability for primary input (and some secondary) quantities that are required for the determination of steady-state thermal transmission properties;
- 3) the need for effective and accurate control strategies for the guarded-hot-plate and pipe apparatus;
- 4) the assessment of measurement uncertainties by using international guidelines such as the Guide to Expression of Uncertainty in Measurement (GUM) and,
- 5) the development and use of design of experiment approach in the planning of future interlaboratory comparisons.

## 3. Participants

The participants of the workshop included representatives from the following general categories: thermal insulation producers that utilize the equipment, equipment manufacturers, testing laboratories, consultants, academics, and government. Exhibit 1 presents a breakdown of the participants. Appendix A is a directory of the participants. There were seven international participants from Canada and Europe.

Category	Participants
Thermal insulation manufacturer	9
Equipment manufacturer or testing laboratory	7
Consultant	2
Academic	1
Government	8
TOTAL	27

Exhibit 1. Breakdown of workshop participants by user category

## 4. Agenda

The workshop was organized into four sequential sessions. The technical approach was based on the 1<sup>st</sup> Workshop Proceedings (Appendix B), which noted that the plate and pipe methods, although different in physical geometry, confront many of the same technical challenges. As a result, this workshop was organized along common generic issues. Each session included presentations followed by an open discussion period. The discussion minutes were recorded by the Session Secretary, edited by the Workshop Organizers, and described in Section 5. The presentations for Session 1 through Session 4 are represented with permission of their authors in Appendix C through Appendix F, respectively.

### 4.1. Session 1

Session 1 focused on the current status of high-temperature thermal insulation reference materials in North America and Europe. An overview of NIST research activities in this area were given as well as an overview of the NIST Standard Reference Data, Standard Reference Material, and Measurement Service Program.

Session 1 (Day 1)*					
Moderato	r: Bill Healy	Secretary:	Monyelle Mingo		
9:00	Welcome and Introductions		Bill Healy		
9:15	Workshop Overview		Robert Zarr		
A. <u>High-te</u>	emperature Reference Materials and Measurem	ent Services			
09:30	Industry needs, ASTM perspective		Tom Whitaker		
09:50	Europe outlook		Erik Rasmussen Roland Schreiner		
10:05	BREAK – 10 min.				
10:15	Summary of NIST research		Robert Zarr		
10:30	Overview of NIST SRM & Measurement Service	es	Robert Watters		
11:00	DISCUSSION				
12 Noon	LUNCH (NIST Cafeteria)				

\*Building 224, Room B245 unless otherwise stated

### 4.2. Session 2

Session 2 provided tours of the NIST metrology laboratories for electrical and dimensional measurements located in the Advanced Metrology Laboratory Complex (AML). (Unfortunately, the temperature metrology laboratories were unavailable due to a schedule conflict with the 9<sup>th</sup> International Temperature Symposium.) An overview of research activities at the Laboratorie national de métrologie et d'essais (LNE) were presented. The presentation was followed by a tour of the NIST guarded-hot-plate facilities which includ-

ed a short presentation on the construction of the 500 mm guarded-hot-plate apparatus and a preview of the PID control strategies for the apparatus. Summary discussions were organized afterwards on topics identified in the 1<sup>st</sup> workshop including thickness measurements, sensor reliability and accuracy, guard imbalance, and surface emissivity.

<u>Session 2</u> Moderator	<u>(Day 1)*</u> rs: Tom Whitaker, Frank Tyler	Secretary:	Leah Strohsnitter
13:00	Tour of NIST Metrology Laboratories (AML) Electrical (Building 218, Room F0013) Rand Dimensional (Building 219, Room G024)	Elmquist, Richar	d Steiner, Yi-hua Tang John Stoup
14:30	Laboratoire national de métrologie et d'essais (Building 224)	s (LNE) GHP	Alain Koenen
15:00	Tour of NIST GHP Facilities (Building 226) (advance demonstration of PID computer mo	del)	Robert Zarr, Bill Healy Bill Thomas
B. <u>Thickn</u> 16:00	<u>ess Determination</u> DISCUSSION Suggested topics: Plate: use of spacers Pipe: pin versus circumference metho Temperature effect (measure or use l	ods, bands literature values)	Attendees
C. <u>Sensor</u> 16:20	Reliability and Accuracy DISCUSSION Suggested topics: Uniformity check: number of sensors, Type of sensor: thermocouple versus Thermocouples: sheathed versus non Temperature effect – change with cyc	, locations platinum resista sheathed (grour cling, degradation	Attendees nce thermometer nding issues) n (how to check)
D. <u>Guard</u> 16:40	Imbalance Check DISCUSSION Suggested topics: Uniformity check: number of sensors, Thermocouples versus thermopile Imbalance experiment Gap insulation, emittance, expansion	, locations	Attendees
E. <u>Surface</u> 17:00	e(s) Emissivity DISCUSSION Suggested topics: How to measure Coatings – emittance, durability		Attendees
17:30 Adjo	urn Day 1	equested)	
18:00 Dinn	er at local restaurant (optional – reservations r	equested)	Return to notel

\*Building 224, Room B245 unless otherwise stated

### 4.3. Session 3

In Session 3, there were three presentations on PID (proportional, integral, derivative) control: 1) how to model the apparatus for simulation control strategies, 2) application to the plate method; and 3) application to the pipe method. After the Discussion period, two presentations were given on uncertainty analyses with applications on specific examples for the guarded-hot-plate method.

Session 3 (Day 2)*					
Moderato	rs: Tom Whitaker, Frank Tyler S	ecretary: Frank Tyler			
F. <u>Contro</u>	ol System Considerations and Steady-State Issues				
09:00	Simulation of PID control model (plate)	Bill Thomas			
09:30	Application of PID control model (plate)	Robert Zarr			
09:55	Industry issues PID control (pipe)	Tom Whitaker			
10:15	BREAK – 10 min.				
10:25	DISCUSSION Suggested topics: PID temperature control versus locking power (te Level of control at different temperatures (how pu How many data points per run How to define steady-state	Attendees mperature drifts) recise?)			
G. <u>Uncer</u>	tainty & Reporting				
11:00	Introduction to GUM	Robert Zarr			
11:20	Uncertainty Analysis using the GUM and GUM supplemen	t Blaza Toman			
11:45	DISCUSSION Suggested topics: How to use GUM uncertainty budget to rank sour How does uncertainty analysis complement DEX f Introduction in ASTM C16 test methods	ces of uncertainty? or inter-laboratory study?			
12 Noon	LUNCH (NIST Cafeteria)				

\*Building 224, Room B245 unless otherwise stated

### 4.4. Session 4

Session 4 presented the results of two inter-laboratory comparisons. The first presentation summarized 19 years of proficiency testing in North America by NVLAP for Test Method C 177 (guarded-hot-plate apparatus) and Test Method C 518 (heat-flow-meter apparatus). The second presentation summarized two European inter-laboratory comparisons. After the presentations, the session shifted focus to future inter-laboratory needs, specifically for the pipe test method. The first of these two presentations defined needs and goals of the proposed inter-laboratory comparison. The second presentation gave a

statistician's approach to the design of experiment (DEX) for a general inter-laboratory comparison with specific examples to the pipe inter-laboratory comparison under consideration.

Session 4 (Day 2)*Moderators:Tom Whitaker, Frank TylerSecretary:Leah Strohsnitter							
H. <u>Inter-l</u>	H. Inter-laboratory Round Robin Recommendations						
13:00	NVLAP C177/C518 Proficiency Tests	Jeff Horlick					
13:20	Europe Inter-laboratory comparison	Helge Hoyer					
13:40	BREAK – 10 min.						
13:50	High-temperature thermal insulation industry needs	Tom Whitaker					
14:30	Experimental design (DEX) for Pipe Round Robin	Jim Filliben					
17:00	Open Discussion about Workshop	Tom Whitaker, Frank Tyler					
17:15	Concluding Remarks	Tom Whitaker, Frank Tyler					
17:30	Adjourn Workshop						

\*Building 224, Room B245 unless otherwise stated

## 5. Discussion Summaries

Section 5 summarizes items of interest from the question-and-answer periods, and from the discussions that were encouraged by the moderators in each session. The minutes for each session were recorded, in outline form, by the Session Secretaries (Section 4) and also by William Healy, NIST. The notes were subsequently edited by the Workshop Organizers for review by the workshop attendees. In some cases, the names of the meeting attendees have been retained for clarity. Where appropriate, action items have been noted.

### 5.1. Session 1

### 5.1.1. Industry needs, ASTM perspective

Tom Whitaker voiced concern about initiating student interest in the field of industrial thermal insulation. [This issue was re-iterated in Session 4 with a follow-up question by Robert Zarr to Professor Thomas.]

### 5.1.2. European perspective

Erik Rasmussen and Roland Schreiner discussed expanded glass granulate as a round robin material for thermal conductivity to higher temperatures.

• *Question 1:* Frank Tyler asked if there were any issues with dimensional stability of the glass beads. *Answer*: They are dimensionally stable. The beads are commercially available from a company in Germany. The material is inexpensive.

- *Question 2:* What about the temperature differences (delta-T) during the tests? *Answer:* On the guarded-hot-plate (GHP) tests, the delta-T is constant. For the pipe tests, the delta-T is variable.
- *Question 3: W*hat about the glass, is there an opacifier? *Answer:* No opacifier. There is a special method to produce an expanded glass. Optical data about the glass are available from FIW.
- *Question 4:* Is the model given in the presentation part of any standard? *Answer:* No. The true thermal conductivity takes into account conduction, radiation, convection, [etcetera].
- *Question 5:* Is there a standard for the Nusselt sphere given in the presentation? *Answer:* No. FIW is probably the only one with the sphere test method.
- *Question 6:* Can the model be standardized? *Answer:* Could circulate the Ph.D. thesis, but tough to understand.
- *Comment 1:* Five different labs have been part of the testing with glass spheres. They hope to have all labs in Europe and some in North America. The five labs have different testing sizes. They measured at 50 mm and 100 mm thicknesses. If not capable of measuring at 100 mm, measure then at the highest thickness possible. Under 20 mm, an error arises because of glass spheres. They have not measured optical effects. The delta-T for the GHP testing was 50 K. Erik Rasmussen can get more detailed results of the testing to the [workshop] group, if desired.

#### 5.1.3. Summary of NIST research; Overview of NIST SRM & Measurement Services

Robert Zarr presented specific overview of NIST Guarded Hot Plate (GHP) activities and Robert Watters presented a general overview of the NIST SRD (Standard Reference Data), Calibration Services, and SRM (Standard Reference Material) programs. Questions and discussion were held to the end of both presentations.

- *Question 1:* Tom Whitaker asked if Europe is working to produce an SRM [using the glass spheres]. *Response:* The current effort is a private initiative.
- *Question 2:* Erik Rasmussen asked how do you determine the validity period of a reference material? *Answer* [from Robert Watters]: Some materials we know are inherently stable. Every statement on period of validity or expiration date includes the caveat that the SRM is stored and used as described in the paragraph on storage and use.. We do, on most occasions, use previous samples in the development of new reference materials. In other words, we may use a current SRM to help develop a new SRM. We do not do work to accelerate degradation. *Comment* [from Robert Zarr]: Instrument stability is important as well. To that end, the NIST GHP labs check instrument stability by establishing a measurement traceability chain for their primary sensors. The NIST GHP labs have begun a rigorous effort to send sensors back to the NIST metrology labs for control and stability checks. The stability check for the test material is more difficult.
- Continued discussion [from attendee]: European reference material typically has a validity period of only 5 years. Fibrous-glass boards should be (much) longer. For example, NIST certificate for 1450d has instructions for handing, storage, and use. If one follows these directions for the SRM, the certification is valid indefinitely (as stated on the 1450d certificate). Could some NIST SRM certificates have a date regarding expiration dates? If so, could these certificates extend expiration date?
- Continued discussion: Frank Tyler stated that ASTM Test Method C 518 requires that the SRM (or other transfer standard used for calibration purposes) be replaced every 5 years. There has been an effort to remove this text from Test Method C 518 but negative votes on the item have been found persuasive and the text has remained. The essential point in the negative votes is a request for any laboratory to demonstrate, with actual data, that the 5-year limit is either valid or invalid. Currently, no one has such data. Action Item: This discussion topic was identified as an action item (for the ASTM C 518 task group to collect such data).

- Continued discussion: Andrzej Brzezinski asked how to resolve the apparent contradiction given in the SRM [1450d] certificate and the 5-year text given in Test Method C 518. *Response:* It was stated that the Test Method C 518 document was probably written assuming that the reference materials were not always handled in accordance within their Instructions for use. *Comment:* Tim Rasinski noted that, as part of the NVLAP (National Voluntary Laboratory Accreditation Program) accreditation process, the laboratory does not need to discard [the reference material] at end of an expiration date. The NVLAP Assessor should not write a nonconformity against expired reference material as long as the lab has some process to show the reference materials have not drifted out of tolerance. The requirements of specific test methods will supersede this, however. One method to check [stability] could be to have two different units checked in two [different] devices.
- *Question 3:* Andrzej Brzezinski asked if there was a possible timeline for services by NIST. *Answer:*\_Robert Zarr responded that, depending on the outcome of the proposed inter-laboratory comparisons, the next stage of service from 20 °C to 200 °C is scheduled for the 2012 calendar year. The next temperature level would be scheduled afterwards.

### 5.2. Session 2

### 5.2.1. Tour of NIST Metrology Laboratories (Advanced Measurement Laboratory Complex)

Rand Elmquest, Richard Steiner, and Yi-hua Tang provided tours of the electrical resistance and voltage measurement laboratories located in the Advanced Measurement Laboratory Complex. Afterwards, John Stoup provided a tour of a dimensional measurement laboratory for the evaluation of coordinate measuring machines.

#### 5.2.2. Laboratoire national de métrologie et d'essais (LNE) GHP – Situation in France

Alain Koenen presented an overview of current GHP activities at LNE.

- *Question 1:* For the inter-laboratory tests conducted at room temperature, was the variation of thickness known? *Answer:* No, the level of variation in the thermal conductivity data was not expected beforehand.
- *Comment 1:* In conjunction with the low-temperature thermal conductivity data presented, it was mentioned that NIST SRM 1450b was characterized to lower temperatures, down to liquid nitrogen temperatures (100 K).

### 5.2.3. Tour of NIST GHP Laboratory Facilities

Robert Zarr and John "Rusty" Hettenhouser presented a short photographic narration of the construction of the NIST 500 mm guarded-hot-plate apparatus. Afterwards, the attendees separated into three groups for tours of the NIST 1016 mm guarded-hot-plate facility, the NIST 500 mm guarded-hot-plate facility, and also a preview of the PID control model to be presented later in the workshop by Professor Thomas.

#### 5.2.4. Thickness Determination Discussion (Tom Whitaker and Frank Tyler, Moderators)

As part of this topic and subsequent discussions on the primary measurements, the moderators began the dialogue with an open request for information on how attendees used spacers for their guardedhot-plate apparatus. Subsequent questions addressed thermal expansion issues.

• *Question 1*: How do you keep your plates spaced? *Response:* Marinite<sup>1</sup> structural insulation machined to 0.001 in.<sup>2</sup> (0.03 mm) using drop gauge.

<sup>&</sup>lt;sup>1</sup> Certain commercial entities, equipment, or materials may be identified in this document in order to describe an experimental procedure or concept adequately. Such identification is not intended to imply recommendation or

- *Question 1a*: Do you change thickness as temperature increases or do you assume constant?
   *Response*: Assumed to be constant.
- Question 1b: Does test material have different thermal expansion, if so what happens? Response: Usually, the test material is compressible and preconditioned. If you try to correct for [test] thickness, you would have to know what the thermal expansion [of the test material] is. If you don't know, base it [test thickness] on room temp thickness.
- *Question 1c*: Are you going to change thickness calculation based on movement? *Response:* Average data over portion that is stable.
- *Question 2*: Do other attendees do anything different? In other words, are there any other thoughts or methods to be discussed?
  - Response: [In Section 7, ASTM C 177 states that, for rigid and high conductance specimens, the specimen surfaces should be made flat and parallel.] In actuality, the specimen surfaces will never be 100 % parallel. European standards warn against using the average [of thickness measurements] because you need full contact. It is important to use the separation between plates. The thermal resistance measurement is between flat surfaces so if they are off your measurement is off as well. You will have gaps, need to know that you will have good contact. Biggest source of mistakes.
  - Comment from Tom Whitaker: For calcium silicate (rigid material), users can't measure repeatable measurements, [inter-lab results show that we] got different thickness measurements.
- Question 3: Are there other ways that the attendees use to control the thickness of their plates, other than spacers, and that they consider to be more reliable? *Response*: For thermal conductivity measurements at 25 °C, the plates are stopped at whatever height you want. *Comment* #1: But if [the test specimen is] compressible, you may modify density. *Comment* #2: Characterize [the test] material at target density and then decide whether or not to use spacers.
- Question 4: Does everyone neglect to make corrections for [thermal] expansion [for the spacers]? Is it the norm to use room temperature thickness? *Response*: Most [attendees] agree that they use room temperature [thickness]. [The] expansion [effect is limited] up to 1% for temperatures up to 650 °C.
- *Question 5*: What about for high temperature? Do we make corrections for thermal expansion effects? *Response*: This is a source of error. *Comment #1*: If you make correction for thickness you have to change density. *Comment #2*: The NIST 500 mm GHP thickness system has capability to measure [in-situ thickness] during the test, but most people do not have this capability.
- *Question 6*: What about spacers that will experience thermal expansion and therefore change [the plate] spacing? *Response:* The specimen is expanding, not the spacer. *Another response:* What if both expand?
- *Question 7:* For writing [documentary] standards, what should we do? *Response:* Have a system that you can preload and that you can measure what's actually going on like NIST's system. Then you can calibrate your system (without specimen or one that you know the expansion of). Or do

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<sup>&</sup>lt;sup>2</sup> It is the policy of the National Institute of Standards and Technology to use the International System of Units (SI). However, in the North American construction and building materials industries, certain non-SI units are used and are reported in this publication (with SI equivalent units) to avoid confusion.

calibration with spacing material. Then you must assume the specimen will not expand more than spacer.

#### 5.2.5. Sensor Reliability and Accuracy Discussion (Tom Whitaker and Frank Tyler, Moderators)

[*Foreword to this Discussion:* Dr. Dean Ripple, formerly with the NIST Thermometry Group, provided answers to the temperature questions from the 1<sup>st</sup> Workshop in Granville, Ohio.]

- Discussion on RTDs (resistive temperature devices) versus type K thermocouples (fine wire in metal sheath): One attendee noted that type K thermocouples "drifted." Another attendee suggests [using] type N thermocouples because they don't drift as much. Robert Zarr states that NIST uses type N thermocouples in thermally insulated metal sheaths. As a result of the fabrication process during vacuum brazing, the type N thermocouples were exposed to temperatures (near 925 °C) well above operation temperatures.
- Question 1 for NIST: Over the long term can you check the ones [type N thermocouples] that are
  installed? No, they are brazed in [the plates and edge-guard rings]. [However,] if you know how
  stable the PRT is, you should be able to measure drift [of the adjoining thermocouples in the
  plate].
- Question 2 for attendee: What kind of thermocouples do you use? Type K on previous plate systems. In newer plate systems, 100 ohm PRTs moved away from thermocouples all together. Question 2a: How many [thermocouples] in the plates in the main heater (say 12 inch plate)? Response: For one plate system there are 3 in the main and 2 in the guard. For another plate system, there are 9 in the main heater and 8 in the guard. Again, these plate systems use PRTs, not thermocouples.
- Same Question (2) for other attendees: What kind of thermocouples do you use?
  - *Response 2b*: Type N, shunted thermocouples. Type K is not stable for long, so they switched to type N. Use 5 thermocouples [averaged] for one plate's temperature.
  - Response 2c: Type E for temperatures up to 500 °C; 4 thermocouples on each side of plate (2-sided plate). Question from a participant: Why do you use that type? Response: Highest output at lowest temperatures. But above 500 °C, you're 'pushing it'.
  - *Response 2d*: Type K, periodically push to 700 °C. At this temperature, a type K thermocouple drifts quickly.
- The moderators asked the attendees if there were other comments or questions to be discussed.
  - Question 3 for attendees: How long/often do you recalibrate? Response 3a: Some thermocouples cannot be removed, so whole probes are replaced. [In general, one attendee noted that the temperature measurement systems are re-built after a certain time period determined by the user.] Response 3b: Some participants have applied external calibrated thermocouples which are used as reference for internal thermocouples calibration. The external thermocouples are applied at the same position as the internal mounted thermocouple. Question from attendee: How can you be sure your plate is uniform? How can you get external thermocouple on the outside? Response: The external thermocouple is placed in contact with the plate surface (on the outside) at the same location as the plate thermocouple.
  - *Question 4 from an attendee*: Are there any issues with RTD's breaking by excessive thermal expansion? *Response*: No, they are strong. The sensors are in a metal sheath.

• It was suggested that [ASTM] could collect data on what [temperature] sensors were used, how thickness measurements were taken, how often and by what means do you calibrate, etc. and determine how this affects measurement readings.

#### 5.2.6. Guard Imbalance Check Discussion (Tom Whitaker and Frank Tyler, Moderators)

- Question for attendees: Do you control primarily on thermopile or surface thermocouples on plates?
  - Response 1: There are several stages; first, control temperature. After [the system is] in steady state, switch to gap thermocouples. Main [heater] is controlled by thermocouples and the guard is controlled by the thermopiles.
  - Response 2: Surface temperatures can give error, gap temperature and edge temperature will be different and gap temperature will be somewhere in between. Question from attendee: Is it significant? Response: We control on thermocouples, not thermopiles. It was significant enough to warrant concern for change. At 1 degree [unit unspecified], they were less than half a percent. Question from attendee: Did you run a guard imbalance test? Response: Yes, but does not have data on hand. Significance may have to do with size and thickness.
  - Response 3: Other imbalance test: check slope to see difference in imbalance between apparatuses. They have added correction in slope to adjust balance of guard and measuring area. Question from attendee: Do you offset it more? Response: Normally it is set to zero, but change is usually seen between plus and minus. You see imbalance between guard and area, test gives option to make adjustment. Question from attendee: Is test run at different temperature? Response: Yes, at different thickness and temperature. Question from attendee: Have you induced a degree change between guard and analyzed the result? Response: 1 degree [unit unspecified] causes about 10%. Half to one degree [unit unspecified] imbalance causes significant result. Should look into effects of ratio of metered area to plate area.
  - Comment: NIST conducts an imbalance check on each "new thickness specimen" received from a customer. We purposely do this as part of the uncertainty evaluation. [The gap thermopile voltage] never truly runs at zero volts, there is always some offset, usually small (on the order of a few tenths of a microvolt). Thus, there is always a small heat flow through guard gap. NIST typically combines the uncertainty in this small offset as part of the uncertainty analysis. We have discovered that, even though the gap voltage is small, the uncertainty can be large, especially as specimens get thicker (i.e., heat flow through the specimen is small relative to the heat flow across the guard gap).

#### 5.2.7. Surface(s) Emissivity (Tom Whitaker and Frank Tyler, Moderators)

- *Question for attendees*: How important is it to get 0.8 (or above) for the plate emissivity?
  - Response 1: No, because normally the bulk density of high temperature products is high so that radiation through specimen is minimal. For specimens having a low density, it is important to have high emissivity of plate.
  - Response 2: High emittance coatings for high temperature testing have been discussed in Europe. One laboratory has proposed raising the 0.8 limit to either 0.9 or 0.95. There was, however, no clear evidence of benefit, and the value was impossible to maintain in the long run. The conclusion was that there is no need to go higher than 0.8. Question from an attendee: Should this issue be researched more? Response: Yes, Helge Hoyer noted that he has a research report on the subject. It was suggested that the attendees can contact Helge Hoyer directly for the report.

Response 3: The NIST GHP laboratory uses a Gier Dunkle<sup>1</sup> DB-100 (ASTM E 408) to measure the near normal emittance of test samples and to check the plate emittances at room temperature. [Note: The instrument was on display during the GHP tour.] NIST uses a ceramic coating for their plate surfaces that measured 0.8 (ASTM E 408). The durability of the plate coating was the main issue. Question for NIST: How flat is surface after application? Answer: Very flat, as noted in the presentation, the vendor polishes the coating as part the fabrication process.

#### 5.3. Session 3

#### 5.3.1. Control System Considerations and Steady-State Issues

Bill Thomas, Robert Zarr, and Tom Whitaker presented back-to-back talks on PID control for plate and pipe apparatus. Questions and discussion were held to the end of the three presentations.

Professor Thomas described the PID objectives for the simulation model to allow faster tuning than the experiment as well as tighter control. For this discussion, the control elements and PID are *all* for center plate, not for cold sides. The model neglected gradients *within* plate as far as control surfaces. There were 9 knobs to play with; PID for each of 3 sections (main heater plus double guards). The outer guard is set at an offset of 5  $\mu$ V. The conduction across the gap was modeled as follows:

$$C = \frac{mC_p}{\Delta T}$$

where m = mass and  $\Delta T = \text{calculated}$  time step.

The heating rate, q was modeled as follows

$$q = \frac{V^2}{R_{el}}$$

where  $R_{el}$  = electrical resistance ( $\Omega$ ) of a heater element.

Knowing the power supply output, an incremental control algorithm for the simulation as well as for the apparatus. A key objective was to avoid overshoot because of the lengthy cool-down time required.

*Presentation 2*: Robert Zarr described the control strategies for the NIST 1016 mm GHP and for the 500 mm GHP apparatus. For the 500 mm GHP, the PRT in the center of the meter plate is used as the control sensor for the meter-plate heater. Two formulae for the discrete controller have been tried: the positional form and the incremental form. Initial attempts focused on the positional form using proportional and integral (PI) control. The control, however, was unsuccessful due to "integral wind-up," a condition in which the integral term over-accumulates error summation, and then under-accumulates, resulting in long-term oscillation. The result is theoretically removable, but NIST, after several months, was unsuccessful in removal of the oscillation. At the suggestion of co-worker, the researchers switched to the incremental form which has successfully been used in controlling the 1016 mm GHP for 20 years. For the 500 mm GHP, the scan rate has been set 60 s. The resistance ratio for each SPRT is measured using a DC bridge and converted by software to a temperature. The system takes 24 h to reach steady-state conditions. Data are collected for the last 4 h.

*Presentation 3*: Tom Whitaker presented control strategies developed by Industrial Insulation Group for the pipe method. Industrial Insulation Group has tried controlling two ways: thermopile versus surface-mounted thermocouples. Once the power is locked, there is some temperature drift but this is of no concern (see Discussion). Whitaker noted that the steady-state definitions per ASTM C177 and C335 need improvement. ISO 8302, he noted, is similar in that regard. Industrial Insulation Group (IIG) is ex-

ploring using alternating current (AC) for main heater. New kilowatt-hour technology (shown during NIST Electrical lab tour) may enable this and may be better at high temperatures.

#### Discussion

- Discussion by the attendees focused initially about permanent changes to the material with temperature, e.g., hydration/dehydration or decomposition resulting in mechanical movement such as shrinkage. Options included: 1) going up "the thermal conductivity/temperature curve," then coming down "the curve;" 2) pre-heating specimen (either in the apparatus or in another conditioning chamber), etc. Arguments, pro & con, were advanced by the attendees.
  - *One viewpoint*: Only going up the curve is valid as the material will not "see" temperatures beyond its application temperature.
  - *Counter viewpoint*: Processes are designed for temperature excursions at start-up or other periods. Therefore, the material will see higher than application temperature.
  - Comment 1: Ideally, measurements are conducted both up and down the thermal conductivity/temperature curve. It was noted that some materials exhibit two curves, depending on the direction; in essence, signifying that there is no unique thermal conductivity for the particular material.
  - *Comment 2*: Only going down the curve may be more representative of field application but the process is time intensive due to no active cooling to remove heat from the apparatus.
  - *Comment 3*: In some cases, this discussion is a moot point because the test point order is conducted as specified by the customer.
  - Comment 4/Open Question: If proceeding up the thermal conductivity/temperature curve, how long does one wait at each temperature for the binder, if present, to burn out?
  - Comment 5: Robert Zarr requested information on the quality of ASHRAE handbook data for industrial insulation materials and whether the data were obtained going up or going down with temperature.
  - Discussion followed on control technique: In order to minimize shocking the heaters with a power surge, IIG will ramp the set point, by software, to next value. IIG uses proportional and derivative control (PD) rather than proportional and integral control (PI). Allows [temperature] drift with [power] lock because precision is not a requirement based on overall curve. IIG uses Fuji controllers which will do auto-tuning at high temperatures [control] not so good at higher temperatures. The controller has ability to respond to software commands. Software can query controller as to the voltage output of the power supply and can switch it into constant power.

#### **Control Issues**

- New techniques: IIG is now looking at AC for main heater control.
- IIG stated that, for control, the controller requires different PID control constants for different temperature regimes.
- Several attendees discussed the requirements for the digital-to-analog (D/A) conversion. Attendees used different D/A converters having the following values: 24 bit, 12 bit, or 10 bit (among others). Some attendees noted that 10 bit and 12 bit D/A converters are unsatisfactory for precise control due to insufficient resolution capability. It was noted that 16 bit D/A are adequate, commercially available, and are currently inexpensive.
- One attendee noted that, because power is square over [electrical] resistance, if you change the control voltage by a factor of 2, the power will change by a factor of 4, which can compound control with a "hunting" problem.

- One of the issues is when you get to lower outputs because of high thickness insulation, it's hard to control very low power levels.
- Some labs use switchable in-line precision resistors for better control in low/high ranges.

### 5.3.2. Uncertainty & Reporting

Robert Zarr presented an introductory talk on the GUM and Blaza Toman presented an uncertainty analysis based on statistical approach recently approved in GUM supplement.

- *Question 1*: Bill Healy asked the attendees, with regard to the temperature range, what level of uncertainty are the attendees comfortable with at high temperature levels? *Response*: approximately 5% with some debate.
- Comment 1 and Question 2: Tom Whitaker stated he was somewhat uncomfortable about European EC Certification. He asked how do you go to a lab, get data, and know that you will meet that level? *Response*: Erik Rasmussen answered that the rules are made for consumer protection; not supposed to declare mean, but to declare curve with some kind of uncertainty. In Europe, [we are] aiming for 'safe values', not just mean, which reduces liability for the user. Uncertainty in testing is very important to Europeans.
- Comment 2: Tim Rasinski stated that as a NVLAP representative he is asked by participating labs, "why do we have to do an uncertainty calculation?" He stated that Germany is putting the uncertainty in the law; Europeans must use GUM and report coverage factor.
- Comment 3: Robert Zarr stated, should we not ask that, during an ASTM inter-laboratory study (ILS), each lab report its measurement uncertainty? He noted that this practice is currently required for inter-laboratory key and pilot comparisons among the national metrology laboratories.
- Comment 4: An attendee stated that one cannot attempt the uncertainty analysis to the level of commitment from NIST. Rasinski (NVLAP) stated that a laboratory need only have to consider primary contributors for the GUM, not necessarily all the sources cited by NIST (Zarr's presentation).
- *Comment 5*: The attendees stated most labs in the U.S. report a precision and bias (PB), based upon ASTM method's P&B section. Rasinski (NVLAP) stated that according to ISO 17025, if the equipment is built per prescribed ASTM method, then they can use the PB statement, but the reality is, some equipment built per spec is known to be outside the PB so there's a disconnect.
- *Comment 6*: Erik Rasmussen stated that this is an excuse (i.e., to use PB). That's why we're having this workshop. In Europe, they're demanding this type of exercise. They may skirt obligation in Europe as long as they can prove that they're 'working on the subject'.
- *Comment 7*: Rasinski (NVLAP) stated that if a manufacturer is considering shipping products outside of the U.S., you're going to have people (i.e., auditors) carefully examining your uncertainties. Rasinski cited a recent case with an auditor in Australia that examined, questioned, and ultimately rejected an uncertainty calculation from a domestic manufacturer.
- *Comment 8*: Helge Hoyer stated that we need statistics. You will always find a single item that will be below a standard, so statistics are needed not only for producers but for consumers.

### 5.4. Session 4

### 5.4.1. NVLAP C177/C518 Proficiency Tests

Jeffrey Horlick summarized 19 years of NVLAP proficiency test results for ASTM Test Methods C 177 and C 518.

• *Comment 1:* Tim Rasinski announced that NVLAP is currently seeking outside services to help run the NVLAP proficiency testing program. This individual person would need to coordinate the attainment of material, preparation of the material, and shipping of material to the laboratory

participants. Mr. Rasinski also stated that NVLAP is also seeking one more individual to conduct accreditation assessments of thermal insulation products.

- *Comment 2:* It is important to note that all of the results presented in the talk by Jeffrey Horlick were at room temperature.
- Action Item 1: Robert Zarr recommended that the results of this presentation (which have been previously published in the open literature) be forwarded to ASTM Task Group C 177 and Task Group C 518 for consideration as new item of business for inclusion as part of the precision and bias statement of the respective test methods.
- *Comment 3:* Tom Whitaker noted that over the course of 19 years, we have not made much improvements in the results.

#### 5.4.2. Europe Inter-laboratory comparison

Helge Hoyer presented results from two high-temperature inter-laboratory comparisons conducted in Europe by the European Mineral Wool Association.

- *Question 1*: Was there an attempt to plot thermal conductivity versus bulk density? *Answer*: Yes, but the results were not neat.
- *Question 2*: How much variation for the second material, 38mm? *Answer*: Four labs used 38 mm spacers; two labs 37.8 mm; and one lab 37.99 mm.
- *Question 3*: What was the range of variation? *Answer*: Some labs have fixed differences and some just use the spacers.
- *Question 4*: Given small number of labs, were there any statistical tests done? *Answer*: No.
- Comment 1: Looking at Lab G (on the slides), it's running low from 100 °C to 500 °C. That is a 1-in-6 chance of a lab running low. Raising that to the power of 4 will result in variation under 5 %.
- *Question 5*: The maximum deviations are all positive, does that mean anything? *Answer*: No.
- *Comment 2*: Tests are randomized; the labels A-G do not represent order.
- *Question 6*: On mineral wool round, was the material preconditioned? *Answer*: No.

#### 5.4.3. High-temperature thermal insulation industry needs

- Question 1: Where can we improve round robin testing?
  - Response 1: Now requiring use of calipers for measurement, give actual measurement. Requiring densities to be presented along with dimension measurements and mass. Asking for raw temperature (i.e., thermocouple) data as well as average temperature of the hot and cold surfaces.
  - *Response 2*: There is a broad variation in design of equipment and lengths (diameters are the same; all are 3 in. pipes). This may have an impact.
  - Response 3: The test material has not been selected: difficulties with calcium silicate and with mineral wool. Is there a material that works well in a high-temp round robin to be used on a pipe apparatus that can be passed from lab to lab, etcetera? With glass piece, you must have stronger mechanical support because of weight. Q: How do you get the glass beads around the ends? Outside 'jacket' is put on (usually plastic). At the last ASTM meeting, a decision was made for mineral wool to get the round robin going, objections?

#### 5.4.4. Experimental design (DEX) for Pipe Round Robin

• *Comment 1*: Comment from Jim Filliben based on the previous discussion:

- Observation 1: There is a large k, that is, a large number of factors that can affect the response. (In the previous Discussion [Section 5.3.2], Filliben counted the factors that were discussed and stopped counting when the tally reached 18 [← high enough].)
- Observation 2: n is small, that is the number of tests; and,
- *Observation 3*: sigma is unknown. Get question nailed down, specification of the problem as well.
- *Comment 2*: We probably need to do a sensitivity analysis before the inter-laboratory comparison. A what if? analysis to see what is important. Select a few primary and secondary factors. We do not fully understand how sensitive the results are to some of those factors. Prior to jumping into inter-laboratory comparison, it might be best to vet some of the issues in a sensitivity analysis.
  - *Response from attendees:* Agreed with that approach.
  - Response from Jim Filliben: Most common experiments here [at NIST] are sensitivity studies.
     Filliben typically uses 2-level fractional factorial designs; with 18 factors for this study, (2<sup>18</sup>) is a lot of runs. Wants 180 numbers (not sure where he got this number), would run a 2<sup>(18-10)</sup> = 256 numbers. Run this fractional factorial experiment, 2 levels. Constructing is relatively easy. Good information about main effects, but not necessarily interactions.
- *Comment 3*: As (mean) temperature increases, there is a curve in temperature [due to radiation effects]. Can run four equally spaced points either along the *X-axis* or *Y-axis*. For large multifactor experiments, look into interactions.
- *Comment 4*: Previous ASTM round robins (in the United States and North America) have not approached this design. Replication is important for these tests; if we can afford it, replicate everywhere.
- Action Item 2: Importance of sensitivity analysis, always lends valuable data/information about system/experiment. Look into doing 'in-house' sensitivity analysis. (Action item for next ASTM meeting).

### 5.4.5. Open Discussion about Workshop (Tom Whitaker and Frank Tyler, Moderators)

The following comments and questions were raised in the open discussion.

- Action Item 3: Request for electronic copy of presentations from this workshop to be made available.
- Action Item 4: The last workshop was held in 2007 which was 5 years ago. ASTM C16:30 should consider holding more frequent workshops. Question: Are similar workshops [on high temperature plate and pipe methods] held in Europe? Response from Erik Rasmussen: One already that has focused on 10 °C, another coming up in September in Belgium. This workshop has been inspiration for further work with high-temperature systems.
- *Comment 1:* Round robin presentation is similar to the results published by Mark Albers in ASTM STP 1426. Tom Whitaker is curious how results in Alber's paper compare to results presented in this workshop. He will e-mail an electronic copy of the paper from ASTM STP 1426.
- Action Item 5: 5-year time interval prescribed in C518, can it be taken out?
- Action Item 6: Temperature sensor variations: This workshop highlighted that, for thermocouples, several different kinds are used among the workshop attendees. Is there a better way? Look into platinum resistance.
- **Action Item 7:** Steady state determination: How to determine what algorithm or criteria is best for determining when steady state is reached. How do you define that you're in control?

- **Action Item 8:** Is it possible to have a guarded hot plate website for posting information and discussion that may occur between other workshop sessions?
- Question for Professor Thomas: As a follow-up to the Session 1 concern, Robert Zarr asked to Professor Thomas to comment on how to encourage more students to become involved in this area for study and employment. Professor Thomas noted that, quite simply, involve the faculty and the students will follow. Research grants are important. Thomas further noted that people will only pay attention when something is not working properly. No one pays attention when things are running smoothly.
- *Comment 2:* Good pace and good content during workshop, wide range of topics.

### 6. Summary and Recommendations

In general, this second workshop on high-temperature guarded-hot-plate and pipe measurements was well attended with participants from North America and Europe representing industry, consulting, academia, and government. The main objective of the workshop was to examine and to improve the general understanding of the operation of the guarded-hot-plate and pipe apparatus at elevated temperatures (up to 650°C). To accomplish this objective, the intention of the workshop was to present, and to discuss, the reasons and causes of differences between laboratory measurements with the ultimate goal to reduce the differences in laboratory comparisons.

To attain these objectives and goals, the workshop was organized into four sessions. Each session covered a particular subject considered important in achieving the overall goal of reducing the differences in laboratory comparisons. Session 1 covered the government's role in providing reference materials to the public and Session 2 covered issues of metrological traceability in relation to the calibration of primary measurements for the guarded-hot-plate and pipe apparatus. Session 3 covered instrument control issues and measurement uncertainty. Session 4 presented data from two recent inter-laboratory comparisons and how to design future inter-laboratory comparisons.

The general conclusion from the attendees was that the workshop was valuable and was also motivating with regards to further work with high-temperature systems. It was further noted that the first work-shop was held in 2007 (five years ago) and that ASTM C 16:30 should consider holding workshops of this type more frequently. It was also requested that the workshop proceedings be made available for electronic publication.

One of the tasks of the Session Moderators was the identification of "action items" for further attention. The remaining text in Section 6 summarizes the eight "action items" that were identified by the moderators in the group discussions (Section 5). Recommendations from the workshop organizers are included with each action item. The majority of these action items are to be forwarded to the appropriate ASTM task group or subcommittee for further discussion.

- The results of the NVLAP presentation (which have been previously published in the open literature) should be forwarded to ASTM Task Group C 177 and Task Group C 518 for consideration as new item of business for inclusion as part of the development of a modified precision and bias statement for the respective test methods.
- 2) The importance of sensitivity analysis always lends valuable data/information about system/experiment. The attendees should look into doing 'in-house' sensitivity analysis. (Action item for next ASTM meeting).
- 3) The last workshop was held in 2007 which was 5 years ago. ASTM C16:30 should consider holding more frequent workshops.

- 4) Request for electronic copy of presentations from this workshop to be made available.
- 5) The 5-year time interval prescribed in C518: can it be taken out?
- 6) Temperature sensor variations: This workshop highlighted that, for thermocouples, several different kinds are used among the workshop attendees. Is there a better way? Look into platinum resistance.
- 7) Steady state determination: How to determine what algorithm or criteria is best for determining when steady state is reached. How do you define that you're in control?
- 8) Is it possible to have a guarded hot plate website for posting information and discussion that may occur between other workshop sessions?

## 7. Acknowledgments

The success of the workshop was a direct result of the quality of the speakers and the participants. The organizers recognize the contributions from the speakers and the insightful discussions from the participants.

## **Appendix A: Participants**

Name	Affiliation
Franz Bauer	Exova
David Bell	Southern Research Institute
Andrzej Brzezinski	LaserComp
Rob Campbell	Netzsch Instruments, North America, LLC
Rick Dolin	Industrial Insulation Group, LLC
Jim Filliben	NIST Statistical Engineering Division
William Healy	NIST Energy and Environment Division
Jeff Horlick	NIST Standards Coordination Office
Helge Hoyer	Rockwool International
Timothy Jonas	Knauf Insulation
Alain Koenen	Laboratoire national de métrologie et d'essais (LNE)
Niina Lehtinen	Paroc Oy Ab R&D
Mark Mantonya	Owens Corning
Bhavesh Patel	Southern Research Institute
Olivier Pons Y Moll	Isover Saint-Gobain CRIR
Tim Rasinski	National Voluntary Laboratory Accreditation Program (NVLAP)
Erik Rasmussen	Rockwool International
Mitch Rose	Netzsch Instruments, North America, LLC
Roland Schreiner	FIW München
Stephen Smith	SES Consulting Services
Marc Thermitus	Consultant
Bill Thomas	Virginia Polytechnic Institute and State University (Virginia Tech)
Blaza Toman	NIST Statistical Engineering Division
Frank Tyler	Owens Corning
Robert Watters	NIST Measurement Services Division
Tom Whitaker	Industrial Insulation Group, LLC
Robert Zarr	NIST Energy and Environment Division



Fig. A1 – Attendees at 2<sup>nd</sup> Operators Workshop on High-Temperature Guarded-Hot-Plate and Pipe Measurements

# Appendix B: 1<sup>st</sup> Workshop Proceedings

ASTM C177 / C335 Discussion Points from Workshop #1 Page 1 of 3

	C177 (Plate)	C335 (Pipe)	
Α	Thickness Determination	Α	Thickness Determination
1	C177 Annex A1 addresses thickness issues. It is recommended to measure thickness before & after testing. For rigid materials, contact resistance can be an issue, in addition to thickness change with temperature.	1	C335 method states that the insulation be installed, the outer circumference measured, inner circumference is assumed to be the outside of the pipe, and the thickness is calculated
2	For an ILS, it is recommended that a semi-rigid or compressible material is used. Each lab should measure thickness but test at assigned thickness. Spacers should be provided for stack designs.	2	Use wider Pi tape (2") to improve measurement of semi-rigid material
3	For rigid material, thickness should be measured insitu at start and at max temp, or pre-determined at max temp	3	Measure pipe with Pi tape to get actual pipe thickness
4	Labs can check their systems with traceable standards, at temperature, if possible	4	Use paper tape to wrap around, poke hole through overlap and then measure distance between the holes
5	Measure thickness (of semi-rigid material) at 5 locations using a calibrated vernier caliper & assign average value to specimen	5	Possibly calculate pipe dimension at temperature, @ 650C, pipe expands ~10 mm in length
6	How do you verify that plates are parallel and that this is not changing either at max temp or with time?	6	Band straps could compress material resulting in varying insulation thickness
7	Have you evaluated sample thickness measurement reproducibility & repeatability for your lab?	7	Use minimum number of bands, preferably, no more than one on test area
8	How is the test area determined?	8	Pin gauge material at temp at the top of the pipe to determine slump after the test is completed
в	Sensor Reliability & Accuracy	В	Sensor Reliability & Accuracy
1	How is 'uniformity of plate temperature' defined and measured? What type of thermometry & location?	1	What type of thermometry is used to measure temperature. Typical response were grounded end TC in a SS sheath. Other suggestions are ungrounded TC in metal sheath.
2	How do you address potential contact resistance issues with rigid test specimens?	2	How do you assure good thermal contact with the metal pipe? At end of groove, TC end cemented in groove touching pipe for grounding
3	Temperature measurement locations & method of measurement should be reported, sensor raw data should be reported	3	Anchor TC along pipe for at least 150mm from bead. Applies to thermocouples mounted in the pipe and on the outside surface of the test sample
4	How do you check for thermal or other degradation of sensors?	4	Bead mounted with masking tape or other tape that has emittance similar to the test material.
5	Do you use control charting [at elevated temperatures] with dedicated laboratory reference material? What material do you use? How is it verified / tracable? How do you know the control reference material is not changing?	5	Many wrap TCs around pipe test sample to maintain good contact
		6	Sometimes double layer to handle metal pipe expansions and evaluate samples at a higher mean temperature
		7	Rotate pipe to determine if TC location makes a difference
		8	Grounding helps eliminate 60 Hz hum if the data acquisition system does not have filtering

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#### ASTM C177 / C335 Discussion Points from Workshop #1

C177 (Plate)			C335 (Pipe)		
			9	Extra insulation over guards was added to reduce the	
				power going to the guards resulting in lower TC noise.	
C	Guard Imbalance Check		C	Guard Imbalance Check	
1	What is the difference between Thermonile and TC		1	How significant is the choice of using an interior	
l '	averages for guard balancing? Le when thermopile		Ľ	thermopile versus surface mounted thermocouples to	
I	is control what are surface temperature			control the guard?	
	differences?			oontor the guard.	
2	Impact of number & location of thermopile junctions		2	If TC difference is 0, thermopiles are not zero	
3	Impact of number & location of TCs		3	Should gaps be insulated? (NPL)	
4	Should gaps be insulated? (NPL)		4	Joints opening between the sample over the metered	
				section and the guard section could affect results	
<b>├</b>			5	How much care is taken to assure joints are closed as	
I			Ť	pipe expands	
D	Control System Considerations		D	Control System Considerations	
1	How is the temperature controlled? If the control		1	How is the temperature controlled? If the control uses	
÷	uses PID control, how do you assure accurate		÷	PID control, how do you assure accurate power	
	power readings (see 3)			readings.	
2	Do you have different PID settings as the pipe		2	Do you have different PID settings as the pipe	
	temperature is changed.			temperature is changed.	
3	How are you measuring power and what are		3	C335 allows use of TC or thermopile for control	
	sources of error?				
4	If you 'lock' the power, does the system drift?		4	Heater concentricity is very important	
			5	What is the resolution of the data acquisition system,	
				especially when measuring guard balance	
I			6	How are voltage and amps measured and how do you	
<u> </u>			7	assure a constant standard	
<b>└</b>			8	Voltage Taps at test area to eliminate lead resistance	
			0	voltage raps at lest area to enminate lead resistance	
E	Steady State Issues		E	Steady State Issues	
1	In some instances, the test meets the steady state		1	Steady state could be different (sec 8.4 in C335)	
	conditions per the standard but the temperature is				
	still changing				
2	Check equilibrium criteria, especially in automated		2	The system software may detect "steady state", but is	
	systems			there still a longer term drift?	
3	8.8.1.3 and Note 19 gives criteria using system time		3	vvnat is the sampling period? What is the time	
4	constant		4	Constant of the apparatus?	
4	control? How is 'in-control' defined & reported?		4	Does FID contor cause any fluctuations?	
	wondor: now is in-conduct defined a reported?				
5	Delta T should not change by 0.2%		5	how is the ambient controlled and does that have an	
				effect on the measurements?	
F	Surface(s) Emissivity		F	Surface(s) Emissivity	
1	Where is it important and how is it validated?		1	Check emittance of pipe	
2	We need to make sure we are using a consistent		2	Could be really important if a gap between insulation	
I	procedure and reporting what the measured			and pipe exists	
	emissivity is.				
4	NPL uses HE23 (Rolls-Royce paint)				
5	NIST has a ceramic coating that they are using	$\vdash$	<u> </u>		
	177 225 Workshop1 2007			2/24/2012	

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#### ASTM C177 / C335 Discussion Points from Workshop #1

C177 (Plate)				C335 (Pipe)		
6	C177 6.12.5 gives a method for checking emissivity but it is difficult					
G	Uncertainty & Reporting		G	Uncertainty & Reporting		
1	GUM - "Guide to the Expression of Uncertainty in Measurement" (NIST Technical Note 1297)		1	Thickness has largest single impact		
2	http://physics.nist.gov/pubs/guidelines/TN1297/tn12 97s.pdf		2	RR density variation is probably due to thickness measurement		
3	Research / provide data for random / systematic components of Q, dT, Thickness, Test Area		3	Research effect of pipe diameter; most use 3-inch pipe		
			4	Possibly use wider Pi tape to help reduce variability on semi-rigid materials		
			5	Should we be taking into account pipe diameter thermal expansion?		
			6	Measurements influenced by fit & joint openings		
			7	Measurements effect by environment and must be closely controlled		
н	Interlaboratory RR Recommendations		н	Interlaboratory RR Recommendations		
1	Choice of material? A semi-rigid material is recommended (see thickness issues)		1	Choice of material? Mineral Fiber pipe has been chosen		
2	Definition of the Goal of the RR is important. Goal for next RR is to help identify apparatus variability		2	Definition of the Goal of the RR is important. Goal for next RR is to help identify apparatus variability		
3	The inter-laboratory measurement & reporting protocol should be cross-checked with the findings of this workshop in order to ensure that all key target data is captured		3	Contact previous participants and try to identify more participants		
4	ASTM ILS will be contacted once critical parameters are defined which are inclusive of participant equipment characteristics, and materials & temperatures established.		4	TG for review questionair and agree to specific method		
5	Temperature measurement locations & method of measurement should be reported, sensor raw data should be reported		5	limit the RR max temperature to allow more participation and improve quality of data.		
6	Consider sharing heater designs for possible guide to best practices		6	ASTM ILS group has agreed to do the analysis and prepare a P&B statement		
7	Type of control (thermopile vs temp averaging) should be reported, or both should be used if user's system permits.		7	Proficiency testing to help monitor performance. Can we get enough participation to establish a proficiency program		
8	Ruggedness testing should be done by each lab		8	Ruggedness testing should be done by each lab		

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## **Appendix C: 2nd Workshop Proceedings - SESSION 1 Presentations**





## 2<sup>nd</sup> Operators Workshop on High-Temperature Guarded-Hot-Plate and Pipe Measurements

ASTM C16:30 Thermal Measurements Subcommittee Test Methods C 177 and C 335

March 19-20, 2012

### WORKSHOP OVERVIEW

Organizers Frank Tyler, Tom Whitaker, Robert Zarr





## Technical Objective, Goals, and Outcomes

#### Objective:

To improve our understanding of the operation of the guarded-hotplate and pipe apparatus at elevated temperatures (up to 650°C)

#### Goals:

- Examine, discuss, and evaluate:
  - role of NIST (reference materials and/or measurement services)
  - metrology for input (and secondary) quantities
  - · control strategies for apparatus
  - sources of measurement uncertainty → GUM
- Reduce variation in inter-laboratory comparisons
- Outcomes:
  - Notebook (copies of slide presentations, 1<sup>st</sup> Workshop summary)
  - Addendum (written summary of Discussion edited by Workshop Organizers and reviewed by WERB for distribution 05/31/2012)
  - Action items will be referred to the appropriate ASTM task group

2

## Background and Approach

#### History

- 2007 1<sup>st</sup> Operators Workshop, Owens Corning, Granville
- 2012 2<sup>nd</sup> Operators Workshop, NIST Gaithersburg

### Background:

- 1<sup>st</sup> Workshop:
  - Presented plate and pipe methods separately
  - Summary report, however, noted commonality of methods
- Current Approach:
  - 2<sup>nd</sup> Workshop builds on the results of the 1<sup>st</sup> Workshop
  - Technical sessions presented generically taking advantage of common issues

3

## **Comparison of Plate and Pipe Methods** Plate Pipe $\lambda_{a} = \frac{QL}{A(T_{h} - T_{c})} \qquad \lambda_{a} = \frac{Q\ln\left(\frac{r_{out}}{r_{in}}\right)}{2\pi L_{p}(T_{in} - T_{out})}$ Equations (C 1045) axial heat flow radial heat flow Differences (shape factor) (shape factor) power (DC voltage and resistance), temperature, Similarities thermopile gap measurements, (PID) control, GUM, inter-laboratory design 4



## **Guidelines and Announcements**

· Please turn off cell phones, beepers, pagers, etc.

#### Session format

- Speakers: 15 minute presentation; 5 minute Q/A
- Audience: hold questions until Q/A time slot (additional Q/A time during Break or Discussion)
- Amenities
- Monday night dinner (optional)
  - Local restaurant
  - Reservations to Monyelle by 12 noon

## Questions?

8

7





## US Government Commitment

DOE Awards More Than \$30 Million to Help Universities Train the Next Generation of Industrial Energy Efficiency Experts

September 13, 2011

WASHINGTON, D.C. – U.S. Energy Secretary Steven Chu today announced more than \$30 million for 24 universities in 23 states across the country to train undergraduate- and graduate-level engineering students in manufacturing efficiency to help them become the nation's next generation of industrial energy efficiency experts. Each school will receive \$200,000 to \$300,000 per year for up to 5 years to help university teams to gain practical training on core energy management concepts through DOE's successful Industrial management concepts through DOE's successful Industrial Assessment Center program.

5

6

## Energy Efficiency Experts, Design Engineers, Reliability Engineers

How reliable are the Thermal Properties of High-Temperature Insulation Materials?

What is the impact of the quality of the Thermal **Properties?**
### ASTM C335 – Pipe Apparatus

Tests performed at seven different laboratories using the horizontal guarded-end apparatus and at one laboratory using an unguarded cylindrical screen test apparatus on two samples of calcium silicate insulation in the range of mean temperatures from 35 to 390°C did not vary by more than 6.3 % of the average

### ASTM C177 Guarded Hot Plate

In 1988, results of a interlaboratory comparison were reported for seven high-temperature guarded-hot-plate apparatus. The plates ranged in size from 203 to 406 mm in diameter and 300 to 610 mm<sup>2</sup>. Different matched pairs of fibrous alumina-silica and calcium silicate were measured by each laboratory over a mean temperature range from 330 to 701 °K. Reference equations based on NIST-Boulder corrections were fit to the data. Imprecision in the deviations from the model were 15 and 16 % (2s level) for fibrous alumina-silica and calcium silicate, respectively. It was established that a significant percentage of the standard deviation in this comparison was due to material variability and not apparatus error.



# Impact of the Reliability of High Temperature Thermal Properties

- Safety, Personnel Protection, Fire Protection
- Some Processes require critical control
- Control Energy costs
- Control Greenhouse Gas Emissions by reducing waste heat loss

# What Does the Industry Need?

- ASTM needs to hear from you regarding changes to the method and/or apparatus;
  - reduce variability
  - improve the accuracy & repeatability of measurements
- Participation from NIST to provide an SRM and provide Transfer Standards that can be used to demonstrate traceability.
- Encourage NIST to participate in comparisons with other National Laboratories
- Encourage research projects into measuring and verifying the quality of high-temperature thermal conductivity measurements.

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# European situation on high temperature testing

Erik Rasmussen Public Affairs Rockwool International A/S

#### ROCKWOOL FIRESAFE INSULATION

### Legal background

- EN standards for Thermal insulation products for building equipment and industrial installations have been published in December 2009 and CE marking will be mandatory as of August 1, 2012
- Thermal conductivity shall be declared for the temperature range for which the products can be used.
- Testing shall be performed by an accredited and notified laboratory for the initial type testing

## Voluntary initiatives

- The current VDI certification is expanded to a VDI & Keymark cooperation for a joint European product certification with focus on thermal conductivity accuracy in testing (both for manufacturers and 3rd parties)
- Roland Schreiner will present the work of FIW on defining a material to be used for testing as no reference material is available
- Helge Høyer will later present the results of a recent round-robin on GHP on the same one set of test specimens.











Forschungsinstitut für Wärmeschutz e.V. München
Future perspectives
<ul> <li>Improvement of measured values by means of the Gaussian correction to evaluate the uncertainty of the reference curve of ± 3% (now only 9% of all measured values exceed the ± 3% limit)</li> </ul>
<ul> <li>Add more values to the Round Robin (Labs and apparatus)</li> </ul>
<ul> <li>Improve the heat transfer model and the properties of the expanded glass beads with the focus on radiation and uncertainty budgets</li> </ul>
Bring the Round Robin to 3. level stage
<ul> <li>Extend the temperature range to the very low temperatures (-180 °C)</li> </ul>
Start of a new Round Robin this year ("AKT Thermophysik")
• Discussion of "true thermal conductivity" to higher/lower temperatures
ASTM Workshop 2012, NIST 9
Forschungsinstitut für Wärmeschutz e.V. München
Thank you for your attention!
R. Schien
ASTM Workshop 2012, NIST 10

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### **NIST High-Temperature GHP**

#### 1996 to 2000 – Evaluation of previous GHP equipment

- Bora Rugaiganisa, Guest Worker, evaluated 200 mm low-temperature GHP
  - Daniel Flynn, Contractor, evaluated 250 mm high-temperature GHP
    - Electrical cross-talk noted between heaters and PRTs
    - o Difficulty with constant-power and constant temperature control
    - o Analysis of plate temperatures and edge heat flows
    - o Mechanical operation unwieldy

#### 2001 to present – 500 mm GHP apparatus

- 2001 to 2004: specifications, design, components construction
- 2005 to 2006: assembly of apparatus
- 2007 to 2010: verification and PID control testing

2nd Operators Workshop on High-Temperature Guarded-Hot-Plate and Pipe Measurements 9

### **Future Considerations**

#### Inter-laboratory comparisons with other NMIs

- Temperature: 20 °C to 200 °C (ΔT: 25 K and 50 K)
- Pressure: ambient to  $10^{-4}$  torr (fixed mean temperature and  $\Delta T$ )
- Calibration Services (U.S. customers)
  - Individual measurements (initially at cost, several \$K)
  - Temperatures: ambient to 200 °C (ΔT: 25 K and 50 K)
  - Thicknesses: 25 mm to 50 mm
  - Different material(s) for each customer
- SRMs
  - Benefit: one material for everyone
  - Batch certification (100 % sampling possible)

2nd Operators Workshop on High-Temperature Guarded-Hot-Plate and Pipe Measurements

### High-Temperature SRM Candidates

#### ASTM input

- ASTM Questionnaire distributed to C16, E06, and E37
- Response preferences:
  - > 300 mm square, 25 mm thick, board form
  - Maximum temperature: 650 °C (mean or face)
- Working with C16:30.1.2 to identify potential candidate materials

### Potential candidates (in no specific order)

- Mineral wool board
- Unbonded (needled glass-fiber) blanket
- Calcium silicate board
- Fumed silica board (SRM 1449, 1459)

#### Assessment



- HFM tests (24 °C) before and after conditioning in air at 650 °C
- Thermogravimetry analysis (TGA)

2nd Operators Workshop on High-Temperature Guarded-Hot-Plate and Pipe Measurements





- Metrology training
- Measurement practice guides
- User facilities (CNST and NCNR)

NIST

MATERIAL MEASUREMENT LABORATORY



- Calibrations
  - Service in NIST technical lab
  - Customer sends instrument to NIST
- Standard Reference Data (SRD)
  - Evaluated numeric data on physical or chemical properties
  - Scientific algorithms on behavior of systems
- Standard Reference Materials (SRMs)
  - Physical artifacts with certified physical or chemical properties
- Laboratory Accreditation
  - Formal assessment of quality systems



















# SRM – Overall Process

- Identification of stakeholder measurement needs
- · Underlying research needed to address the issues
- · Decision on the best channel to fulfill the needs
- SRM development work to determine feasibility
- Submission of production plans
   Statements of Work (SOWs)
- Business case check
- Material Hazard Assessment
- Production measurements and certification



8. Value assignment based on selected data from interlaboratory studies	•	•	23
<ul> <li>What happens if the user community gets "hooked" an SRM?         <ul> <li>Lab committed to renewals forever?</li> <li>Other means to achieve the same driver goals</li> </ul> </li> </ul>	" o	on	

### **Appendix D: 2nd Workshop Proceedings - SESSION 2 Presentations**






















NIST 500 mm Guarded-Hot-Plate Apparatus

2<sup>nd</sup> Operators Workshop on High-Temperature Guarded-Hot-Plate and Pipe Measurements

March 19-20, 2012

#### **Construction Photograph Record**

#### **Robert Zarr and John Hettenhouser**

National Institute of Standards and Technology 100 Bureau Drive Gaithersburg, Maryland, United States

#### Superstructure and Vacuum Cart

**NIST Welding Shop** 



## Installation of Vacuum Baseplate and Bell Jar

Building 226, Room B107

# Installation of Vacuum Baseplate and Bell Jar

Building 226, Room B107





















Helium Leak Check











### Hot Plate Components Fabrication

NIST Machine Shop and NIST EDM Shop































### **Hot Plate Construction**

Post brazing NIST Machine Shop






# **Dimensional Inspection**

NIST Machine Shop Quality Inspection CMM (Coordinate Measurement Machine)



# Dimensional Reference (Baseline)

NIST Dimensional Metrology Moore M48 CMM



## Cold Plate Assemblies – Thermometer Plate, Heater Plate, and Coolant Plate

Original Sketches, Fabrication, Vacuum Brazing, Post-brazing Machining, Coating





## Thermometer Plate – Vacuum Brazing, Side 1



## Thermometer Plate – Vacuum Brazing, Side 1



## Thermometer Plate – Vacuum Brazing, Side 1



#### Thermometer Plate – Vacuum Brazing, Side 1



## Thermometer Plate – Vacuum Brazing, Side 1



#### Thermometer Plate – Vacuum Brazing, Side 2





#### Thermometer Plate – Excess Braze Removal



## Thermometer Plate – Coating Vendor







## Cold Plate Heater – Vacuum Brazing



Cold Plate Heater – Vacuum Brazing









# Edge Guard Assemblies – Heater Ring, Coolant Ring, and Water Jacket Ring

Fabrication, Vacuum Brazing

#### Edge Guard Rings – Initial Bending, Machining and Welding





## Edge Guard Rings – Initial Bending/Welding



## Edge Guard Rings – Finish Lathe Cutting



#### Edge Guard Rings – Finish Lathe Cutting





## Edge Guard Heater Rings – Heater Grooves



Edge Guard Heater Rings – Thermocouple Grooves



#### Edge Guard Heater Rings – Vacuum Brazing



#### Edge Guard Heater Rings – Vacuum Brazing





Edge Guard Heater Rings – Vacuum Brazing



## Edge Guard Heater Rings – Vacuum Brazing



Edge Guard Heater Rings – Vacuum Brazing



## Edge Guard Heater Rings – Electrical Terminations



Edge Guard Heater Rings – Electrical Terminations





## Edge Guard Coolant Rings – NIST Welding



# Water Jacket Rings

## Edge Guard Coolant Rings – NIST Machining



#### Edge Guard Coolant Rings – NIST Machining





#### Assemble Large to Small Diameters Flexible Thermal Insulation Between Rings



Assemble Large to Small Diameters Flexible Thermal Insulation Between Rings



#### Assemble Large to Small Diameters Flexible Thermal Insulation Between Rings



#### Hand-cut Flexible Thermal Insulation





# Assemble Cold-Plate Components

# <section-header><section-header><image>

## Thermometer Plate – Opposite Surface





## Next Layer of Flexible Thermal Insulation



#### Install Coolant Plate




### Install Flexible Wrap Around Water Jacket



Weight Check







### Initial Fit Check Inboard Cold Plate Assembly





### Install Inboard Cold Plate Assembly





### Install Inboard Cold Plate Assembly





### Install Inboard Cold Plate Assembly





## <section-header><image>



### Install Outboard Cold Plate Assembly





### Install Outboard Cold Plate Assembly





### Install Outboard Edge Guard Assembly



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### Install Outboard Edge Guard Assembly



### Install Outboard Edge Guard Assembly



### Install Outboard Edge Guard Assembly



### Install Outboard Edge Guard Assembly



### Plates and Rings Installed on Overhead Rails





## Original Hot Plate Thermopile Non-functional

## (KP vs. 65 % Pd - 35 % Au)

**Removal and Replacement** 

### Prep for Sandblasting of Coating



Prep for Sandblasting of Coating













## Machine out 1<sup>st</sup> Thermopile













## <section-header><image>

Outboard and Inboard Thickness Measurement and Force Application Backstop





### Terminals for Thermocouple and Voltage



## Isothermal Zone Box for External Thermocouple Wiring

### Pre-wire Terminals (copper telephone wire)



Instrument Panel (Retracted)



### Install Terminals (copper telephone wire)



### Install Thermocouple Wire (Bell Jar Feedthroughs)



### Install Lower Portion of Isothermal Box



1. C.

Insulate Top





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### **Appendix E: 2nd Workshop Proceedings - SESSION 3 Presentations**

Simulation of PID Control for Guarded-Hot-Plate Apparatus

William C. Thomas Professor Emeritus Mechanical Engineering Virginia Polytechnic Institute & State University Blacksburg, Virginia

### Main Objective

Minimize Traditional Trial & Error Tuning Operations by Simulating Response with a Mathematical Model

Reduce Time and ExpenseImproved Set of Controller Gains



- Describe Simulated Control Method
  - Mathematical Model Basis
    - Emphasis on the (Less Complex) 1-m GHP
  - Tuning Procedure for PID Gain Settings
- Typical Response Results
- Modeling Details

### NIST 1-m Guarded-Hot-Plate










$$e = Error = Setpoint - Process Variable  $\Delta t_{sr} = Controller Sample Rate  $K_p, K_i, \& K_d = Proportional, Integral, and Derivative Gains$$$$

# Programmed Control Algorithm

$$v' = max \left( 0, min \left\{ v_{hl}, \left[ v + \kappa_P \Delta t_{sr} e' + \kappa_D [e' - e] \right] \right\} \right)$$

 $v_{hl} = Maximum Allowable$  $\kappa_P, \kappa_D = Renamed Gains$ 

## **Tuning Procedure**

Recall:  $v' = max \left( 0, min \left\{ v_{hl}, \left[ v + \kappa_P \Delta t_{sr} e' + \kappa_D [e' - e] \right] \right\} \right)$ 

- Info on Incremental Method Tuning Lacking in Literature
- Desired Response
  - "Reasonable" Initial Approach to Set Point
  - Tight control during Final Hour
- Procedure Adopted
  - Set  $\kappa_p$  to Zero & Manually Increase  $\kappa_p$  to Approach Set Point
  - Increase κ<sub>D</sub> to Minimize R-Value Excursions in Quasi-steady Period
    - Maximum Deviation
    - RMS Deviation

# Summary

### Simulation Results

- Improved Set of Controller Gains
- Greatly Reduced Required Time vs Traditional Operation
- Gain Parameters Satisfactorily Used for Many Years
  - Nominal R-Values 0.4 4.8 C m<sup>2</sup>/W
  - Thicknesses 13 230 mm

Simulation Model Has Been Extended to NIST's 500 mm GHP

### Mathematical Model for NIST's Extended-Range 500-mm GHP

- Same General Modeling Approach
- 16 PID controllers (vs 3 for 1-m GHP)
  - Not Intended for Two-Sided Operating Mode
  - Symmetry Enables Simplifying
    - **10** Controllers & Heaters
    - 44 Thermal Bridges
- Highest Uncertainty Source: Evaluation of Thermal Bridges
  - Types as for 1-m GHP
  - + SPRT, TC, TP and Heater Sheaths ("Fins")



# References

- W. F. Stoecker and P. A. Stoecker, *Microcomputer Control of Thermal and Mechanical Systems*, Van Nostrand Reinhold, 1988.
- W. C. Thomas and R. R. Zarr, *Thermal response simulation for tuning PID controllers in a 1016 mm guarded hot plate apparatus*, ISA Transactions 50 (2011) 504-512.













- Description of apparatus
- Control strategy
  - Temperature (PID control)
  - Gas pressure
- Results and discussion
- Summary



- Discrete equivalent Pl analog controller (Raven, 1978)
  - Positional form of the control equation

$$m(k) = K_1 e(k) + K_2 \Delta t \left( \frac{1}{2} (m) + m(0) \right)$$
(1)

engineering laboratory

- Incremental or velocity form of the control equation

$$\Delta m(k) = m(k) - m(k-1) = K_1'[e(k) - e(k-1)] + K_2' \Delta t e(k)$$
<sup>(2)</sup>

where:

- *k* is current sampling interval;
- $K'_1, K'_2$  are control constants (incremental version);
- e(k), e(k-1) are errors from setpoint for current and previous intervals; and,
- $\Delta t$  is the sampling interval (s)
- 16 separate heaters → 16 PI control equations (32 control constants (K'<sub>1</sub>, K'<sub>2</sub>) to be determined)



# **Outline**

- Motivation
- Description of apparatus
- Control strategy
  - Temperature (PID control)
  - Gas pressure
- Results and discussion
- Summary









# <section-header> Summary and Future Work Control stability achieved within stated goal Additional work may be required for the control constants at low pressures Thermal conductivity results consistent with previous SRM 1450 results (temperature and pressure) Future work: Inter-lab comparisons with other NMIs (National Metrology Institutes) at : Elevated temperatures Low gas pressures

engineeri

# 2<sup>nd</sup> Operators Workshop on High-Temperature Guarded-Hot-Plate and Pipe Measurements

High-Temperature Thermal Insulation Industry Needs

Thomas Whitaker Chairman, ASTM Committee C16 on Thermal Insulation





# Control System Considerations Steady State Issues

• PID Control

- PID model for the NIST high temperature plate
- Application of that model to the NIST high temperature plate
- Pipe PID control
  - IIG R&D Center Pipe
  - 3 temperature controllers with thermocouple input
  - Output of the controller connected to the main power supply is a proportional DC voltage.



- Setting the PID values was not trivial
  - The controller Auto Tune worked well at the lower temperatures but not well at the higher temperatures
- I use different PID settings for each target temperature.

# **Steady State**

- Collect Data every minute
- Average at 5 minutes, then at 30 minutes
- Monitor for changes in
  - Hot surface temp
  - Guard offset
  - Measured thermal conductivity
    - Change <0.5% over a 2 hour period
    - Monotonically decreasing or increasing

# **Questions & Discussion**

PID temperature control versus locking power (temperature drifts) Level of control at different temperatures (how precise?) How many data points per run How to define steady-state

### Estimating Measurement Uncertainty of Thermophysical Properties

### Robert Zarr

National Institute of Standards and Technology Engineering Laboratory Energy and Environment Division

2<sup>nd</sup> Operators Workshop on High-Temperature Guarded-Hot-Plate and Pipe Measurements

> Gaithersburg, Maryland, United States March 20, 2012





### "Classic" NBS Measurement Uncertainty Statement





 1972 – Dr. H. H. Ku, NBS Statistician, reported that C. H. Meyers, NBS physicist, originally stated the uncertainty for his measurements on the heat capacity of ammonia as follows:

"We think our reported value is good to 1 part in 10,000: we are willing to bet our own money at even odds that it is correct to 2 parts in 10,000. Furthermore, if by any chance our value is shown to be in error by more than 1 part in 1000, we are prepared to eat the apparatus and drink the ammonia."

 The statement was not approved by the NBS Editorial Board and is only preserved anecdotally (by Ku).

### **Complications – Multitude of...**



# **GUM Philosophy and Approach**

### **Philosophy**

- International consensus uniform method so that measurements in different countries can be compared (analogous to SI units)
- Universal applicable to all kinds of quantitative measurements and to all types of input data

### Approach

- GUM does not distinguish between random and systematic components
- Instead, evaluations are classified as either:
  - Type A those evaluations by statistical analysis of the data, or
  - Type B those evaluations by other means.















15 µV

Vm S 0.087 W (Reference [13]) (Reference [13]) (Reference [13]) B B

### Example – Thermal Conductivity (continued)



Relative combined standard uncertainty,  $u_{c,r}(\lambda)$ 

$u_{c,r}(\lambda) = \frac{u_{c}(\lambda)}{\lambda} = 1$	$\left(\frac{u_{\varrho}(Q)}{Q}\right)^{2} + \left(\frac{u_{L}(L)}{L}\right)^{2} + \left(\frac{u_{A}(A)}{A}\right)^{2} + \left(\frac{u_{\Delta T}(\Delta T)}{\Delta T}\right)^{2}$
---	--

### Example – Thermal Conductivity (continued)

### 4) Compute expanded uncertainty, U, for statement

Example

 $λ = (0.04500 \pm 0.00041)$  W·m<sup>-1</sup>·K<sup>-1</sup>, where the number following the symbol ± is the numerical value of an expanded uncertainty  $U = ku_c$ , with U determined from a combined standard uncertainty (i.e., estimated standard deviation)  $u_c = 0.00020$  W·m<sup>-1</sup>·K<sup>-1</sup> and a coverage factor k = 2. Because it can be assumed that the possible estimated values of the standard are approximately normally distributed with approximate standard deviation  $u_c$ , the unknown value of the standard is believed to lie in the interval defined by U with a level of confidence of approximately 95 %.

Relative expanded uncertainty, U<sub>r</sub>

 $U_r = (0.00041/0.04500) \times 100 = 0.9\%$  (NIST guarded-hot-plate measurements for customers are typically rounded up to nearest 0.5%)



# Uncertainty Analysis using the GUM and GUM Supplement

Blaza Toman Statistical Engineering Division ITL, NIST

# Outline

- Example Measuring Thermal Conductivity
- Uncertainty definitions
- Probability short review
- Uncertainty Analysis using GUM Supplement
- Uncertainty Analysis using GUM
- Modeling trends and uncertainty based on repeated measurements
- Conclusions

2



# **Other Considerations**

From previous work it is known that thermal conductivity  $\lambda$  may be a function of

- 1. bulk density  $\rho$  not part of the measurement equation
- 2. Temperature  $T_m = \frac{T_h + T_c}{2}$

As in equation

 $\lambda = a_0 + a_1 \rho + a_2 T_m$ 

Example – NIST SRM 1450d Fibrous-Glass Board Thermal conductivity from 280K to 340K [1]

### Sample data from this experiment

T <sub>m</sub> (K)	ρ (kg m <sup>-3</sup> )	T <sub>h</sub> (K)	T <sub>c1</sub> (K)	T <sub>c2</sub> (K)	Q/2 (W)	A (m <sup>2</sup> )	L (mm)	λ (Wm <sup>-1</sup> K <sup>-1</sup> )
280	113.5	292.50	267.50	267.50	3.895	0.12980	25.93	0.03112
295	114.3	307.50	282.50	282.50	4.050	0.12989	26.02	0.03245
295	119.0	307.50	282.50	282.50	4.086	0.12989	25.82	0.03249
340	123.8	352.50	327.49	327.50	4.736	0.13016	25.85	0.03782

The total data set included 15 measurements in a 3x5 factorial experiment with three density  $\rho$  (low, mid, high) levels and five temperature  $T_m$  levels (280, 295, 310, 325, 340).

-

# Thermal conductivity calculation

For each experimental observation (row in the table)  $\lambda$  (called the measurand) is computed according to

$$\lambda = \frac{\frac{Q}{2}L_{Average}}{A \ \Delta T_{Average}}$$

This gives the value, we need an accompanying uncertainty

# Uncertainty according to the GUM [2]

- measurement uncertainty "reflects the lack of exact knowledge of the value of the measurand".
- The corresponding state of knowledge is best described by means of a <u>probability distribution</u> over the set of possible values for the measurand.
- Standard uncertainty is the standard deviation of this distribution, expanded uncertainty is the 95% probability interval.

### 8

# So what exactly is a probability distribution?

Imagine flipping a coin 2 times and recording Y=number of heads in two flips

- Y = 0 with probability 0.25
- Y = 1 with probability 0.5
- Y = 2 with probability 0.25

This is an example of a probability distribution

# Another example - Human Height



Usually described using a Gaussian probability distribution (bell curve)

For American males - mean is 70 in, standard deviation is 3 in.

Probability that randomly selected male's height is in (63in,73in) is 0.68
## What exactly is uncertainty then?

Without considering  $\rho$  and  $T_m$  for the moment,

Recall that

$$\lambda = \frac{\frac{Q}{2} L_{Average}}{A \ \Delta T_{Average}}$$

We need to obtain a <u>probability distribution</u> for  $\lambda$ , or at least the <u>standard deviation</u> of this distribution.

But isn't  $\lambda$  a constant? Not if the input quantities have uncertainties.

# Uncertainty using GUM Supplement [3]

- For each input variable (Q, L, A,  $\Delta$ T) measured with uncertainty obtain a probability distribution using data and/or uncertainty budget.
- Generate values of (Q, L, A,  $\Delta T$  ) from the distributions and compute  $\lambda$
- This gives draws from the distribution of  $\lambda$

Compute mean and standard distribution to get estimate of  $\lambda$  and its standard uncertainty.



 $\Delta T \sim Gaussian(mean = 25.005, sd = 0.077)$ 



## Uncertainty Analysis via GUM

- GUM method <u>approximates</u> the standard deviation of the probability distribution of the measurand, using a Taylor series expansion which linearizes the measurement equation.
- This results in a formula for the standard deviation (uncertainty):

$$sd(\lambda) = \sqrt{c_{Q}^{2}sd^{2}(Q) + c_{\Delta T}^{2}sd^{2}(\Delta T) + c_{L}^{2}sd^{2}(L) + c_{A}^{2}sd^{2}(A)}$$

where the coefficients are partial derivatives such as

$$c_{Q} = \frac{\partial \lambda}{\partial Q} = \frac{L}{A \Delta T} \bigg|_{L=25.85, A=0.13016, \Delta T=25.001}$$

## Result via GUM for $\lambda$ for T<sub>m</sub> = 340K, $\rho$ = 123.8 kg·m<sup>-3</sup>

• The mean =  $0.03782 \,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$ 

• The standard deviation (standard uncertainty ) = 0.0001626

• The relative standard uncertainty = 0.0043

Recall results via GUM Supplement:

 $mean = 0.03781 \, W \cdot m^{-1} \cdot K^{-1}$ 

relative standard uncertainty = 0.0043

# Additional statistical modeling

- Density  $\rho$  and temperature  $T_m$  are not directly part of the measurement equation, but are known to have an effect on  $\lambda$ . This relationship is evaluated via the 3x5 experiment.
- The "data" at each experimental setting are the GUM results for  $\boldsymbol{\lambda}$

For example at  $T_m = 340$ K: low  $\rho$  : mean = 0.03753, sd = 0.1607 (0.43%) med  $\rho$  : mean = 0.03761, sd = 0.1606 high  $\rho$  : mean = 0.03782, sd = 0.1626

## Relationships - p

 Based on analysis done in NIST SP260-173 - there is <u>no</u> <u>systematic</u> relationship between ρ and λ. But there is random variability in λ due to ρ. This can be accounted for in the uncertainty.

 For example at T<sub>m</sub> = 340K, there are three Gaussian curves. These can be combined [4] into a single one which has mean = 0.03765, std = 0.1998 (0.53%).

# Relationships - T<sub>m</sub>

• Based on analysis done in NIST SP260-173 - there is a <u>linear relationship</u> between  $T_m$  and  $\lambda$ , through the origin.

$$\lambda = a_1 T_m$$

• Need to estimate the coefficient  $a_1$ 

# Estimation of $a_1$

At each temperature  $T_m$ , we have a value of  $\lambda$ , an uncertainty and a probability distribution (GUM Supplement)

#### Monte Carlo Procedure:

• At each of the 5  $T_m$  values, generate a value of  $\lambda$ , from the corresponding Gaussian.

For example for  $T_m = 340$ K: Gaussian(mean=0.03765, sd=0.1998).

- Fit the linear regression
- Repeat.



## Results

$$\lambda = (1.10497 \text{ x} 10^{-4})T_m$$

with uncertainty of the slope  $(2.467 \times 10^{-7})$ 

Predicted value at  $T_m = 340$  K from this model is 0.038 W·m<sup>-1</sup>·K<sup>-1</sup>, with uncertainty 0.00023 (0.60%)

This uncertainty includes components from Q, L, A,  $\Delta T$ ,  $\rho$ , and the regression on  $T_m$ 

## Conclusion

- Demonstrated both traditional GUM analysis and GUM Supplement analysis on example from SRM 1450d.
- GUM Supplement analysis of uncertainty is straightforward and enables more complex statistical modeling which can produce estimates of systematic effects such as linear trends, as well as account for additional uncertainty.

## References

- [1] NIST SP 260-173
- [2] JCGM 100:2008. Evaluation of measurement data guide to the expression of uncertainty in measurement. Joint Committee for Guides in Metrology (JCGM), Sèvres, France (2008) <u>http://www.bipm.org/en/publications/guides/gum.html</u>.
- [3] JCGM 101:2008. Evaluation of measurement data supplement 1 to the "Guide to the expression of uncertainty in measurement" – propagation of distributions using a Monte Carlo method. Joint Committee for Guides in Metrology (JCGM), Sèvres, France (2008)

http://www.bipm.org/en/publications/guides/gum.html.

 [4] Toman, B. Bayesian Approaches to Calculating a Reference Value in Key Comparison Experiments, Technometrics, vol. 49, 81 – 87, 2007.

#### **Appendix F: 2nd Workshop Proceedings - SESSION 4 Presentations**

### Thermal Conductivity Proficiency Testing Results – Nineteen NVLAP Proficiency Testing Rounds from 1986 – 2004

-- or --

### 19 Years in 19 Minutes

Jeffrey Horlick and Dr. Lawrence Knab ASTM C16.30 – Workshop 2 March 19-20, 2012

National Institute of Standards and Technology •



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National Institute of Standards and Technology •

## What is NVLAP?

- A process for accreditation of testing and calibration laboratories – described in NIST Handbook 150
- Established in the U.S. Code of Federal Regulations (Title 15, Part 285) in 1976
- Administered by NIST
- Linked to NIST research units
- Based on international (ISO/IEC) standards
- Available to any qualifying laboratory (public private)
- Fee supported
- Signatory to several international MRAs
- 850+ labs in the system today

National Institute of Standards and Technology •

### **NVLAP Assessment of Laboratories**

(Tim Rasinski is the NVLAP Program Manager for this program)

- Review of laboratory management system documentation and NVLAP assessment history
- On-site visit by NVLAP Assessors for management system and technical assessment
- Proficiency testing



#### **Standard Test Method**

In 1945, the American Society for Testing and Materials (ASTM) formally adopted the guarded-hot-plate method as a standard test method based, in part, on NBS's design. In 1947, NBS engineers Henry Robinson and Thomas Watson extended the temperature range of the guarded-hot-plate apparatus and in the next few years, completed the first interlaboratory comparison of thermal conductivity tests of insulation (jointly sponsored by the American Society of Heating and Ventilating Engineers and NBS). These tests demonstrated the need for suitable means to calibrate the apparatus of industrial and other

	rear	Material Used in Each Proficiency Test Round
11	1986	1-inch thick, fibrous glass board
12	1988	1-inch thick, expanded polystyrene (EPS) board, 2.5 pcf density
13	1989	The test data for Round 13 is not included in this paper.
14	1990	0.75-inch thick, closed cell neoprene foam sheet stock
15	1991	1-inch thick, closed cell polyisocyanurate foam board with bonded faces
16	1991	0.75-inch thick, thermal insulation board composed of expanded vermiculite binders and fibers
17	1992	1-inch thick, unfaced, polyimide foam board
18	1992	0.5-inch thick, unfaced, rigid, closed cell PVC foam board
19	1993	2-inch thick, unfaced, rigid, cellular, polystyrene bead board (Type C Bead) (RCPS), nominal 3 pcf density
20	1993	1-inch thick, unfaced, coated one side, fibrous glass duct liner, nominal 2 pcf density
21	1994	1-inch thick, foil-faced fibrous glass board, nominal 3 pcf density
22	1995	2-inch thick, unfaced mineral wool board, nominal 6 pcf density
23	1996	1.5-inch thick, foil-faced fibrous glass duct wrap, 0.75 pcf density
24	1997	2-inch thick, unfaced fibrous glass board, 3-pcf density
25	1997	1-inch thick, open cell polyurethane foam, 2.5 pcf density
26	1999	1-inch nominal thickness, expanded polystyrene (EPS) board, 2.6 pcf density
27	2000	2-inch nominal thickness, expanded polystyrene (EPS) board, 2.6 pcf density
28	2001	combined 1-inch and 2-inch nominal thickness to make a 3-inch specimen; expanded polystyrene (EPS) board, 2.6 pcf density. In most cases, labs used the one-inch EPS test sample from previous R26 and likewise, the two inch EPS test sample from previous R27.
29	2003	1-inch nominal thickness, fibrous glass duct liner insulation, 1.6 to 1.8 pcf density
30	2004	1.5-inch nominal thickness, polyisocyanurate rigid-board insulation, 2.5 pcf density
brief sui	mmary of	possible factors affecting proficiency testing results
a) Time: 1986 thro	this report this 2004	possible factors affecting proficiency testing results t covers 19 rounds of NVLAP Proficiency Testing: Rounds 11 through 30, - 19 years
a) Time: 1986 thro b) Test n 2004 the	this report ough 2004 nethod star re were thr	possible factors affecting proficiency testing results t covers 19 rounds of NVLAP Proficiency Testing: Rounds 11 through 30, - 19 years ndards: ASTM C177 and ASTM C518 have changed. Between 1986 and ree revisions to ASTM C177 and five revisions to ASTM C518
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"trimmed Grand Mean"

is the mathematic average of the k-factor values

with the outliers excluded

### Percent Deviation from trimmed Grand Mean

=

100 \* (laboratory value - trimmed Grand Mean)

trimmed Grand Mean

NIST National Institute of Standards and Technology .

ASTM C177	Round	11	12	13	14	15	16	17
Year of testing	1986	1988	1989	1990	1991	1991	1992	
Trimmed Grand Mean of	0.2304	0.2348		0.2619	0.1604	0.3844	0.3297	
Standard Deviation of k	0.0043	0.0033		0.0039	0.0048	0.0135	0.0099	
%CV of k-factor value	1.	1.87	1.38		1.50	2.98	3.52	2.99
No. of labs / No. of labs	9/8	11 / 10		10/9	10/10	10/9	9/9	
ASTM C518	Round	11	12	13	14	15	16	17
Year of testing		1986	1988	1989	1990	1991	1991	1992
Trimmed Grand Mean of	0.2288	0.2354		0.2592	0.1638	0.3721	0.3227	
Standard Deviation of k	0.0033	0.0034		0.0042	0.0045	0.0114	0.0070	
%CV of k-factor value	1.43	1.43		1.61	2.73	3.08	2.16	
No of labs / No of labs	outliers excluded	29/26	27/27		20/19	21/21	21/20	20/20

ASTM C177	Round	11	12	13	14	15	16	17	18	19	20
Year of testing		1986	1988	1989	1990	1991	1991	1992	1992	1993	1993
Trimmed Grand Me	an of k-factor value	0.2304	0.2348		0.2619	0.1604	0.3844	0.3297	0.2338	0.2369	0.2484
Standard Deviation	of k-factor value	0.0043	0.0033		0.0039	0.0048	0.0135	0.0099	0.0088	0.0069	0.0063
%CV of k-factor valu	ue	1.87	1.38		1.50	2.98	3.52	2.99	3.75	2.91	2.52
No. of labs / No. of I	labs outliers excluded	9/8	11 / 10		10/9	10/10	10/9	9/9	10/9	10/9	10/9
ASTM C518	Round	11	12	13	14	15	16	17	18	19	20
Year of testing		1986	1988	1989	1990	1991	1991	1992	1992	1993	1993
Trimmed Grand Mea	an of k-factor value	0.2288	0.2354		0.2592	0.1638	0.3721	0.3227	0.2334	0.2346	0.2456
Standard Deviation	of k-factor value	0.0033	0.0034		0.0042	0.0045	0.0114	0.0070	0.0116	0.0036	0.0084
%CV of k-factor valu	10	1.43	1.43		1.61	2.73	3.08	2.16	4.99	1.54	3.41
VI IS IMOSOF YUIL	10	1.40									
No. of labs / No. of I	abs outliers excluded	29/26	27/27		20/19	21/21	21/20	20/20	21/21	21/20	21/21
ASTM C177	abs outliers excluded	29 / 26	27 / 27	23	20 / 19 24	21 / 21 25	21 / 20 26	20 / 20	21/21	21 / 20	21 / 21
ASTM C177 Year of testing	Round	29 / 26 21 1994	27 / 27 22 1995	<b>23</b> 1996	20 / 19 24 1997	21 / 21 25 1997	21 / 20 26 1999	20 / 20 27 2000	21 / 21 28 2001	21 / 20 29 2003	21 / 21 30 2004
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No. of labs / No. of l ASTM C177 Year of testing Trimmed Grand Me Standard Deviation %CV of k-factor val	Round Round ran of k-factor value of k-factor value ue	29 / 26 21 1994 0.2236 0.0042 1.88	27 / 27 22 1995 0.2304 0.0061 2.63	23 1996 0.2673 0.0177 6.61	20 / 19 24 1997 0.2283 0.0032 1.40	21/21 25 1997 0.2562 0.0051 2.00	21 / 20 26 1999 0.2278 0.0006 0.28	20 / 20 27 2000 0.2301 0.0024 1.03	21 / 21 28 2001 0.2325 0.0036 1.56	21/20 29 2003 0.2432 0.0042 1.75	21/21 30 2004 0.177 0.004 2.36
ASTM C177 Year of testing Trimmed Grand Me Standard Deviation %CV of k-factor val No. of labs / No. of	Round Round ean of k-factor value of k-factor value ue labs outliers excluded	29 / 26 21 1994 0.2236 0.0042 1.88 8 / 8	27 / 27 22 1995 0.2304 0.0061 2.63 8 / 8	23 1996 0.2673 0.0177 6.61 8 / 8	20 / 19 24 1997 0.2283 0.0032 1.40 7 / 6	21 / 21 25 1997 0.2562 0.0051 2.00 7 / 7	21 / 20 26 1999 0.2278 0.0006 0.28 7 / 5	20 / 20 27 2000 0.2301 0.0024 1.03 7 / 6	21/21 28 2001 0.2325 0.0036 1.56 7/7	21/20 29 2003 0.2432 0.0042 1.75 7/6	21 / 21 30 2004 0.177 0.004 2.36 5 / 5
ASTM C177 Year of testing Trimmed Grand Me Standard Deviation %CV of k-factor val No. of labs / No. of ASTM C518	Round Round ean of k-factor value of k-factor value ue labs outliers excluded Round	29 / 26 21 1994 0.2236 0.0042 1.88 8 / 8 21	27 / 27 22 1995 0.2304 0.0061 2.63 8 / 8 22	23 1996 0.2673 0.0177 6.61 8/8 23	20 / 19 24 1997 0.2283 0.0032 1.40 7 / 6 24	21/21 25 1997 0.2562 0.0051 2.00 7/7 25	21 / 20 26 1999 0.2278 0.0006 0.28 7 / 5 26	20 / 20 27 2000 0.2301 0.0024 1.03 7 / 6 27	21 / 21 28 2001 0.2325 0.0036 1.56 7 / 7 28	21/20 29 2003 0.2432 0.0042 1.75 7/6 29	21/21 30 2004 0.177 0.004 2.36 5/5 30
ASTM C177 Year of testing Trimmed Grand Me Standard Deviation %CV of k-factor val No. of labs / No. of ASTM C518 Year of testing	Round Round ean of k-factor value of k-factor value ue labs outliers excluded Round	29 / 26 21 1994 0.2236 0.0042 1.88 8 / 8 21 1994	27 / 27 22 1995 0.2304 0.0061 2.63 8 / 8 22 1995	23 1996 0.2673 0.0177 6.61 8/8 23 1996	20 / 19 24 1997 0.2283 0.0032 1.40 7 / 6 24 1997	21/21 25 1997 0.2562 0.0051 2.00 7/7 25 1997	21/20 26 1999 0.2278 0.0006 0.28 7/5 26 1999	20 / 20 27 2000 0.2301 0.0024 1.03 7 / 6 27 2000	21/21 28 2001 0.2325 0.0036 1.56 7/7 28 2001	21 / 20 29 2003 0.2432 0.0042 1.75 7 / 6 29 2003	21/21 30 2004 0.177 0.004 2.36 5/5 30 2004
ASTM C177 Year of testing Trimmed Grand Me Standard Deviation %CV of k-factor val No. of labs / No. of I ASTM C518 Year of testing Trimmed Grand Me	Round Round ran of k-factor value of k-factor value ue labs outliers excluded Round ran of k-factor value	29 / 26 21 1994 0.2236 0.0042 1.88 8 / 8 21 1994 0.2230	27 / 27 22 1995 0.2304 0.0061 2.63 8 / 8 22 1995 0.2328	23 1996 0.2673 0.0177 6.61 8/8 23 1996 0.2745	20 / 19 24 1997 0.2283 0.0032 1.40 7 / 6 24 1997 0.2291	21 / 21 25 1997 0.2562 0.0051 2.00 7 / 7 25 1997 0.2548	21/20 26 1999 0.2278 0.0006 0.28 7/5 26 1999 0.2291	20 / 20 27 2000 0.2301 0.0024 1.03 7 / 6 27 2000 0.2301	21/21 28 2001 0.2325 0.0036 1.56 7/7 28 2001 0.2335	21 / 20 29 2003 0.2432 0.0042 1.75 7 / 6 29 2003 0.2408	21/21 30 2004 0.177 0.004 2.36 5/5 30 2004 0.1814
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#### Thermal Insulation LAP ASTM C17<u>7</u> and ASTM C51<u>8</u> using trimmed Grand Mean



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2 13	14	15	16 AS Ro 148 	TM C und 1 3 data within +/- 2° +/- 3° +/- 4° +/- 5° +/- 6° TM C und 1 7 data within +/- 2° +/- 6° +/- 6° +/- 3° +/- 4° +/- 3° +/- 4° +/- 3° +/- 4°	18 1777 11 th a poir % % % % % % % % % % % % %	19 rougi nts (12 rougi nts (12 rougi nts (14 rougi	20 h 30 (e 2 outlie 2 outlie 2 outlie 133 139 142 h 30 (e 6 outlie cour 227 266 288 298 303	21 excep ers no nt 3 3 2 excep ers no nt 7 5 9 3 3 3	t Rou t inclu	23 md 1 (ded) % wi 6 8 89 93 95 93 95 93 95 93 95 93 95 95 95 95 95 95 95 95 95 95 95 95 95	24 3) ithin 2 0 .9 .9 .9 .9 .9 .9 .9 .9 .9 .9	25	26	27	28	29	3





# Next

- Data from NVLAP Proficiency Testing have been used in the development of precision and bias statements for ASTM C177 and C518. The data analyzed in this presentation has been made available to ASTM Subcommittee C16.30.
- Reprints are available from Jeffrey Horlick.
- NVLAP will continue to conduct proficiency testing for thermal conductivity when a contract proficiency testing provider is found. The last round was #31 in February 2010.

# EURIMA European Mineral Wool Association

High Temperature Thermal Conductivity Measurment Comparisons

# **EURIMA Comparison 1996**

The tests were performed on individual specimens taken from a selected lot of high density Rockwool boards resulting in a density range for the specimens from 134 kg/m<sup>3</sup> to 152 kg/m<sup>3</sup>.

		1996			
Mean temp	100 C	200 C	300 C	400 C	500 C
Н	48.6	63	79.6	99.2	122.4
I	39.8	51.8	65.1	80.7	99.7
К	43	57	72	89	108
	43	56	72	91	112
М	43.1	56.4	71.3	87.8	
N	45	58.1	73.6	91.7	112
0	40.2	51.6	65.2	81.1	99.2
Р	44	57	72.2	90.8	114
Q	39.4	46.1	57.8	74.6	
Mean	42.9	55.2	69.9	87.3	109.6
Max deviation from mean %	- 8	- 16	-17	- 14	- 8



# **EURIMA Comparison 2011**

Contrary to the 1996 comparison the 2011 one used the same specimen with the limitation due to different plate sizes for the used equipment. The specimen was circulated to the laboratories in order of decreasing plate dimensions. The specimen was a ISOFRAX blanket measured at a thickness of 38 mm. The densities stated by the laboratories varied from 124 kg/m<sup>3</sup> to 135 kg/m<sup>3</sup>

		2011			
Mean Temperature	100 C	200 C	300 C	400 C	500 C
А	46.3	65.3	88.8	120.0	161.6
В	49.0	66.0	89.0	119.0	159.0
С	49.9	68.2	91.1	120.8	159.0
E		67.6	90.0	119.0	156.7
F	47.9	67.6	94.8	128.4	171.6
G		62.0	83.7	111.4	145.1
Mean	48.3	66.1	89.6	119.8	158.8
Max deviation					
from mean %	4.1	6.2	6.6	7.0	8.6



## 2<sup>nd</sup> Operators Workshop on High-Temperature Guarded-Hot-Plate and Pipe Measurements

High-Temperature Thermal Insulation Industry Needs

Thomas Whitaker Chairman, ASTM Committee C16 on Thermal Insulation





# C16 Round Robin C335 Pipe Test

- The goal of this RR is to evaluate equipment differences. The participating laboratories all have different designs.
- In previous RR there have been issues with the material chosen for the RR. There were "fit issues" when Calcium Silicate was circulated. When we used Mineral Wool, the pieces were characterized by dimensions and weight but there were no assurances the pieces were identical.

- There participants have agreed to providing construction details about the apparatus
- The temperature has been restricted to 300C as the upper limit due to limits of the participants.
- The participants have agreed to supply detailed measurements not just the "steady state" data.

# Issues

Drift is an issue and has caused some discussion. For both the test methods we are looking at in the workshop, the "definition" of steady state is change of <0.5% and not monotonically increasing or decreasing. If an operator does not have computer controlled equipment that collects data and proceeds to the next point, he can see a longer term, slow drift. I have allowed my apparatus to sit at one temperature and plot the measured conductivity. Can meet the definition of "steady state" in about 1 day, but after 5 days, the answer is different and sometimes by more than 1%. My apparatus sets in a room measuring about 4m x 5m. If I plot measured conductivity over time, I can pinpoint when the night crew comes in to empty the trash containers. That visit could trigger a change in the slope and provide an inflection point that breaks the "monotonically changing" rule.</li>

- Applies to both Pipe and Flat test methods. At higher temperatures, lots of things change. If the test sample has some organic or combustible component, that can shift the measured conductivity.
- Some materials will have a reaction somewhat quickly and fit within the definition of "steady state". Others a delayed reaction that can cause a shift, but it occurs slowly and may or may not be complete when the computer considers the apparatus to be at "steady state".

# **Questions & Discussion**

2<sup>nd</sup> Operators Workshop on High-Temperature Guarded-Hot-Plate and Pipe Measurements

> ASTM 16:30 Thermal Measurements Subcommittee Test Methods C 177 and C 335

Experiment Design (DEX) of Round Robin

Jim Filliben, NIST

03/20/12 (Tuesday): 1:50-2:30 224/B245

> Bob Zarr jjfzarrtalk032012.pptx Handout Notes

> > 2

1

The deliverable of this talk is

1. to describe a structured and generic 2part <u>framework</u> that has proved to be useful and insightful for scientific problem-solving at NIST; and

2. to <u>apply</u> that structure to the problem of assessing whether a set of laboratories are equivalent wrt their thermal conductivity measurements.

# Outline

- 1. DEX/Stat Framework
- 2. Questions
- 3. DEX Problem Classification
- 4. DEX Problem Translation
- 5. DEX Principles & Techniques
- 6. DEX Criteria & Interlab Designs



# NIST Inter-Governmental Collaborations:



World Trade Center



Bullet proof Vests



Iris-scan Identification



Consumer Testing



Radiation Detection



Food Gamma-ray Spectrometry



. GSA LADAR Building Inspection



Lung Tumor Metrology



**Gulf Oil Spill** 

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# 1. DEX/Stat Framework













### Within-Lab Questions

23

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#### For my lab ...

- Q1. What is the uncertainty of my thermal cond. (TC) measurements?
- Q2. Does my TC value change with material?
- Q3. Does the unc(TC value) change with material, or temp, or ...?
- Q3. What are the most important factors affecting the uncertainty, bias, and precision of my TC measurements?
- Q4. What are the optimal settings for all relevant factors?
- Q5. What is a good predictive model, that describes my TC measurements? Is such a model good enough for simulation?
- Q6. Are the above answers robust over all relevant other ("robustness") factors that may effect my system?
- Q7. What is the temperature scope ("range") of my TC readings?
- Q8. To test any of the above, how many observations do I (stat) need?
- Q9. To test any of the above, how many observations can I afford?
- Q10. Will my sample (of readings, of materials) be representative of the population?
- Q11. What is the population? (of readings, of materials, of ...)
- Q12. Are my TC readings in stat control?
- Q13. Does ground truth exist?

### Within-Lab Questions

#### For my lab ...

Q1. What is the uncertainty of my thermal cond. (TC) measurements	2# +- #
Q2. Does my TC value change with material?	Y/N
Q3. Does the unc(TC value) change with material, or temp, or?	Y/N
Q3. What are the most important factors affecting the	List
uncertainty, bias, and precision of my TC measurements?	
Q4. What are the optimal settings for all relevant factors?	Vector
Q5. What is a good predictive model, that describes my TC	Function
measurements? Is such a model good enough for simulation?	
Q6. Are the above answers robust over all relevant	Y/N
other ("robustness") factors that may effect my system?	
Q7. What is the temperature scope ("range") of my TC readings?	[#,#]
Q8. To test any of the above, how many observations do I (stat) need?	'#
Q9. To test any of the above, how many observations can I afford?	#
Q10. Will my sample (of readings, of materials) be representative	Y/N
of the population?	
Q11. What is the population? (of readings, of materials, of)	{}
Q12. Are my TC readings in stat control?	Y/N
Q13. Does ground truth exist?	Y/N
<b>Between-Lab Questions</b>	25
---	---------------------
For labs 1 and 2 (and beyond)	
Q1. Are the 2 labs equivalent?	Y/N
Q2. What does "equivalent" mean?	<discuss></discuss>
Q3. Does our equivalence conclusion change with material,	Y/N
or temp, or? (interactions?) (robustness)	
Q4. What are the most important factors affecting the	List
our equivalence conclusion?	
Q5. What is a consensus TC value valid over both labs?	# +- #
Q6. Are the devices used by the 2 labs equivalent?	Y/N
Q7. If the labs are not equivalent, what are the correction factors	# +- #
which map one lab into another?	
Q8. What is the temperature scope ("range") for my equivalence	[#,#]
conclusion?	
Q9. To test equivalence, how many observations do I (stat) need?	#
Q10. To test equivalence, how many observations can I afford (\$/time	e)?(\$, days)
Q11. To test equivalence, how many factors should I vary?	#
Q12. To test equivalence, How many levels of a factor (e.g., Temp)	#
should I use? What such levels?	
Q13. What is the uncertainty for TC measurements in each lab?	# +- #
Q14. Do I have data-based estimates of such uncertainties?	Y/N

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3. DEX Problem Classification

















# 1. Problem Classification

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**Usual DEX Sequence pre-Comparative:** 

- **1. Define Scope**
- 2.1 Lab, many reps
- 3.1 Lab, Sensitivity Analysis Design
- 4. Multi-Lab, Comparative Design

4. DEX Problem Translation



## **2. Problem Translation:** Minimal Info to Start the DEX Process

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#### Specificity is key ....

1. Title:

- 2. Problem/Question:
- 3. Response:
- 4. Number k of Factors to Vary:
- 5. Number n of Runs Affordable:

## 2. Problem Translation: Minimal Info to Start the DEX Process

=

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For	your	proj	ect/	prob	lem:
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1. Title =\_\_\_\_\_

2. Problem/Question = \_\_\_\_\_

- 3. Response Y
- 3. Number k of Factors to Vary =\_\_\_\_
- 4. Sample Size n

	Ехр	eriment Des	sign Work:	sheet	D ate:
1. Project/Problem Title:	0 4				
2. Researcher:					
3. Project Background &	Importance:				
4. General Project Quest	tion:				
5. Specific Project Quest	ion (This Expe	riment Only):			
6. (Generic) Stat Goal(s)					
7. Scope of Copelusions:					
r. coope or contractore.	5				
DEX Essentials 8. Response Variable Y:		Gener	ic Stat Mode	al: Y = f( X1	X2,,Xk) + e
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X4: X5:					
X7: X8:					
X9: X10:					
18. General DEX Catego 19. Specific DEX:	ny: Comp, Scr	'Sens, Regr, O	ptim, Ver/Fa	ilt, Unc:	
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# **DEX Principles & Techniques**

Principles Construct Efficient Designs Construct Effective Designs Infer about Population Avoid Biased Factors

Maximize Test Sensitivity Estimate All Model Parameters Allow for Expanded Model

Estimate Sigma Model-Free Estimate Main Effects & Int.

Save \$ Make Conclusions Robust

Assess Drift Avoid Bias from Drift Avoid Confounding

Minimize SD(Estimates) Realistically Sample the Process

Reduce Effect Uncertainty Assess Repeatability Assess Reproducibility

Techniques Elicit Dominant Project Goal(s) Elicit Project Scope & Constraints Randomize Randomize, Block, Balance Orthogonal Balance n(distinct) >= k+1 Record Additional (e.g., Ambient) Variables Replicate Full Factorial or High Res. **Fractional Factorial Designs** Pilot Study, Fract. Fact. Designs Design in Many **Robustness Factors** Controls, Replicate across Time **Drift-Reducing Designs** Full Factorial or High Res. **Fractional Factorial Designs** Sample Where Variation Is **Design in Vicissitudes** (Multiple Sets) Youden Pairs for Homogeneity Replication Multiple Sets (Across Days)

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# **DEX Principles & Techniques**

Principles	Techniques
Construct Efficient Designs	Elicit Dominant Project Goal(s)
Construct Effective Designs	Elicit Project Scope & Constraints
Infer about Population	Randomize
Avoid Biased Factors	Randomize, Block, Balance Orthogonal
Maximize Test Sensitivity	Balance
Estimate All Model Parameters	n(distinct) >= k+1
Allow for Expanded Model	Record Additional (e.g., Ambient) Variables
Estimate Sigma Model-Free	Replicate
Estimate Main Effects & Int.	Full Factorial or High Res. Fractional Factorial Designs
Save \$	Pilot Study, Fract. Fact. Designs
Make Conclusions Robust	Design in Many Robustness Factors
Assess Drift	Controls Replicate across Time
Avoid Bias from Drift	Drift-Reducing Designs
Avoid Confounding	Full Factorial or High Res
Avoid Comountaining	Fractional Eactorial Designs
Minimize SD(Estimates)	Sample Where Variation Is
Realistically Sample the Process	Design in Vicissitudes
realistically sumple the rissess	(Multiple Sets)
Reduce Effect Uncertainty	Youden Pairs for Homogeneity
Assess Repeatability	Replication
Assess Reproducibility	Multiple Sets (Across Days)

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## 1 & 2. Equivalance & Effect

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#### An effect is ...

- 1. By default, "effect" means shift in location.
- 2. Our ability to detect an effect depends on the intrinsic (within-lab) variability of the data  $\sigma_i$
- 3. "Effect" could also mean shift in variation (within lab  $\sigma_{ij}$
- We <u>conclude</u>: "a factor has an effect" by computing a minimum <u>statistical</u> significant difference (via statistical hypothesis testing).
   Of equal importance, is the minimum engineering significant difference.

#### 1 & 2. Equivalance & Effect

#### Equivalence ...

- 1. By default, 2 labs are "equivalent" if they are statistically identical in location.
- 2. Our ability to detect equivalence depends on the intrinsic (within-lab) variability of the data  $\sigma_i$
- 3. Equivalence could also mean statistically identical in variation (within lab  $\sigma_i$ )
- We <u>conclude</u>: "2 labs are equivalent" by computing and comparing location estimates (via statistical hypothesis testing, e.g., t, ANOVA). Of additional interest is whether the location differences exceed the "minimum engineering significant difference" (scientist-provided).













## 6. Balance

#### t-test

To determine if a statistically significant difference in location exists, the t statistic is as follows:

$$t_{stat} = \frac{\overline{y}_2 - \overline{y}_1}{s\sqrt{\frac{1}{n_1} + \frac{1}{n_2}}}$$

 $t_{stat}$  is compared to  $t_{crit}$  where  $t_{crit}$  is theoretical value taken from  $t_{n1+n2-2}$  distribution

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## 6. Balance

For example, if total n = 6 (2 labs and 3 TC measurements at a fixed temp per lab)  $\overline{y_1} = 6$   $\overline{y_2} = 8$ Is there a statistically significant shift in location?

n1	n2	t <sub>stat</sub>	$\wedge$	t <sub>crit</sub> =t <sub>4,0.975</sub>	Shift?
3	3	$t_{stat} = \frac{\overline{y}_2 - \overline{y}_1}{s\sqrt{\frac{1}{n_1} + \frac{1}{n_2}}} = \frac{8 - 6}{0.85\sqrt{\frac{1}{3} + \frac{1}{3}}} =$	2.88	2.78	Yes
4	2	$t_{stat} = \frac{\overline{y}_2 - \overline{y}_1}{s\sqrt{\frac{1}{n_1} + \frac{1}{n_2}}} = \frac{8 - 6}{0.85\sqrt{\frac{1}{4} + \frac{1}{2}}}$	2.72	2.78	No
5	1	$t_{stat} = \frac{\overline{y}_2 - \overline{y}_1}{s\sqrt{1/n_1} + 1/n_2} = \frac{8 - 6}{0.85\sqrt{1/5} + 1/1}$	2.15	2.78	No

## The t-test is sensitivity to imbalance





## 7. Orthogonality











#### 8. Scope & Robustness

**Robustness Factors are the Key...** 

1. The robustness factors define our <u>scope</u> of conclusions

about the primary factor.

Choice/decision for you: What scope do you want--narrow or broad?

If fix all robustness factors, then scope = narrow

Your data-based conclusions are limited to where you collect data

2. Ideally, the robustness factors do not interact with

the primary factor.

If not interact, then have robustness.

If do interact, then have non-robustness (& insight)

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### 8. Scope & Robustness

Robustness Factors: What we do (DEX) ...

1. Let R.F. float: do not vary them (and do not record them)

2. Let R.F. float: do not vary them (but do record them)

3. Fix all Robustness Factors

4. Vary/control one Robustness Factor

5. Vary/control two Robustness Factors

6. Vary/control "all" Robustness Factors



#### 8. Scope & Robustness

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**Thermal Conductivity Models** 

**Our Model: Y** = f(X1, X2)(k=2, n=20)**Y** = Thermal Conductivity X1 = Laboratory (5 levels: 1, 2, 3, 4, 5) X2 = Temperature (4 levels: 100, 200, 300, 400) Nature's Model: Y = f(X1, X, XB = 20)**Y** = Thermal Conductivity (5 levels: 1, 2, 3, 4, 5) X1 = Laboratory X2 = Temperature (4 levels: 100, 200, 300, 400) X3 = ?X4 = ?...

#### 8. Scope & Robustness

Us vs. Nature: Choosing the Correct Robustness Factors ...

Our model:  $Y = f_{SE}(X_1, X_2, X_3, ..., X_k)$ 

Nature's model:  $Y = f_N (X_1, X_2, X_3, ..., X_k, X_{k+1} ..., X_N)$ 

There is no substitute (or recovery) from the scientist/

engineer choosing an <u>incomplete</u> set of robustness factors.

This requires skillful S/E expertise.

## 9. Confounding

 Confounding occurs in a design when levels of one factor are directly correlated with levels of another factor

#### Temp = 500

Lab	Material Type	Result           7.24		
1	1			
1	1	8.15		
1	1	6.98		
2	2	10.23		
2	2	11.05		
3	2	10.78		

<u>Lab - Material Type</u> <u>Confounding:</u> The observed difference in results cannot be directly attributed to either Lab or Material Type 93

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## 9. Confounding

• Confounding occurs in a design when levels of Confounding is a curse to any experiment inasmuch as valid, crisp and unambiguous results cannot be drawn.

Confounding factors may not always be obvious:

Material, Material Type Environmental factors Time (devices may drift over time) Learning effect/run order

Local power sources drifting


## 12. Sample Size n:

Y = f(X1, X2, X3, ..., Xk)

n will grow as number of robustness factors grow

n (theoretical) >= k+1

n (theoretical) >=  $(2*\sigma/T)**2 = g(\sigma,T)$ 

n (max) is dictated by scientist \$/time

## 4 Ways to Reduce n:

1. Reduce the number of factors (but poorer scope)

- 2. Reduce the number of levels (but poorer scope)
- 3. Reduce the number of reps (but poorer  $\sigma$ )
- 4. Orthogonal fractional factorial design for the robustness factors (but lose higher-order interaction info)

**Null Designs** 

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## 6. DEX Criteria & Interlab Designs



