

International Round-Robin on Transport Properties of Bismuth Telluride

Hsin Wang

**Oak Ridge National Laboratory
Oak Ridge, TN USA**

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and DOE EERE VT Program - Propulsion Materials: Jerry Gibbs***

Annex VIII Participants

- **IEA-AMT Thermoelectric Annex**

- Annex lead: Oak Ridge National Laboratory (H. Wang)
- USA: Clemson (T. Tritt, S. Zhu); Marlow (J. Sharp); Corning (A. Mayolet, C. Smith, J. Senawiratne) and ZT-Plus (F. Harris)
- China: SICCAS (S.Q. Bai, L. Chen)
- Canada: Natural Resource Canada (J. Lo); University of Waterloo (Holger Kleinke); University of Quebec at Chicoutimi (Laszlo Kiss)
- Germany: Fraunhofer IPM (H. Böttner, J. König)



- **IEA-AMT members countries:**

- UK: NPL
- Finland: VTT (discussion on October 20)
- Israel:
- Australia:
- International Observer: Korea: KERI (H. W. Lee)



Annex VIII on Thermoelectric: Oct. 2009 - present:

- **Support DOE VT thermoelectric programs for vehicle applications**
- **Transport properties measurements**
- **Measurement standards and reliability**
- **Support the commercialization of thermoelectrics**
- **Annex VIII on thermoelectrics led by ORNL**
 - Round robin 1: 2009-2010 on n-type and p-type bismuth telluride
 - Round robin 2: 2010-2011 on p-type bismuth telluride
 - Round robin 3: 2012 at high temperatures n-type PbTe

IEA-AMT Focus: Bulk Thermoelectrics Used for Automotive Waste Heat Recovery

- **Significant gaps exist between literature ZT values and scalable materials**
- **Possible Issues:**
 - Measurement errors
 - No standards for calibration
 - Incomplete measurements
 - Data extrapolation
 - Materials non-uniformity
 - Orientation effect
 - Measurements on different samples



Marlow Materials Selected for Transport Properties Round-Robin Tests

- **Materials: $\text{Bi}_2\text{Te}_{3.005}$ (n-type) $\text{Bi}_{0.5}\text{Sb}_{1.5}\text{Te}_3$ (p-type)**
- **Four-sample Sets**
 - Thermal diffusivity: 12.7 mm diameter disk
 - Specific heat: 4 mm diameter disk
 - Seebeck coefficient and electrical resistivity: 2 x 2 x 15 mm³ bar, 3 x 3 x 12 mm³ bar
- **Temperature range: 20-200°C**
- **Round-robin plans:**
 1. Use best practice in each lab
 2. Focusing on one specific material
 3. Develop test procedures

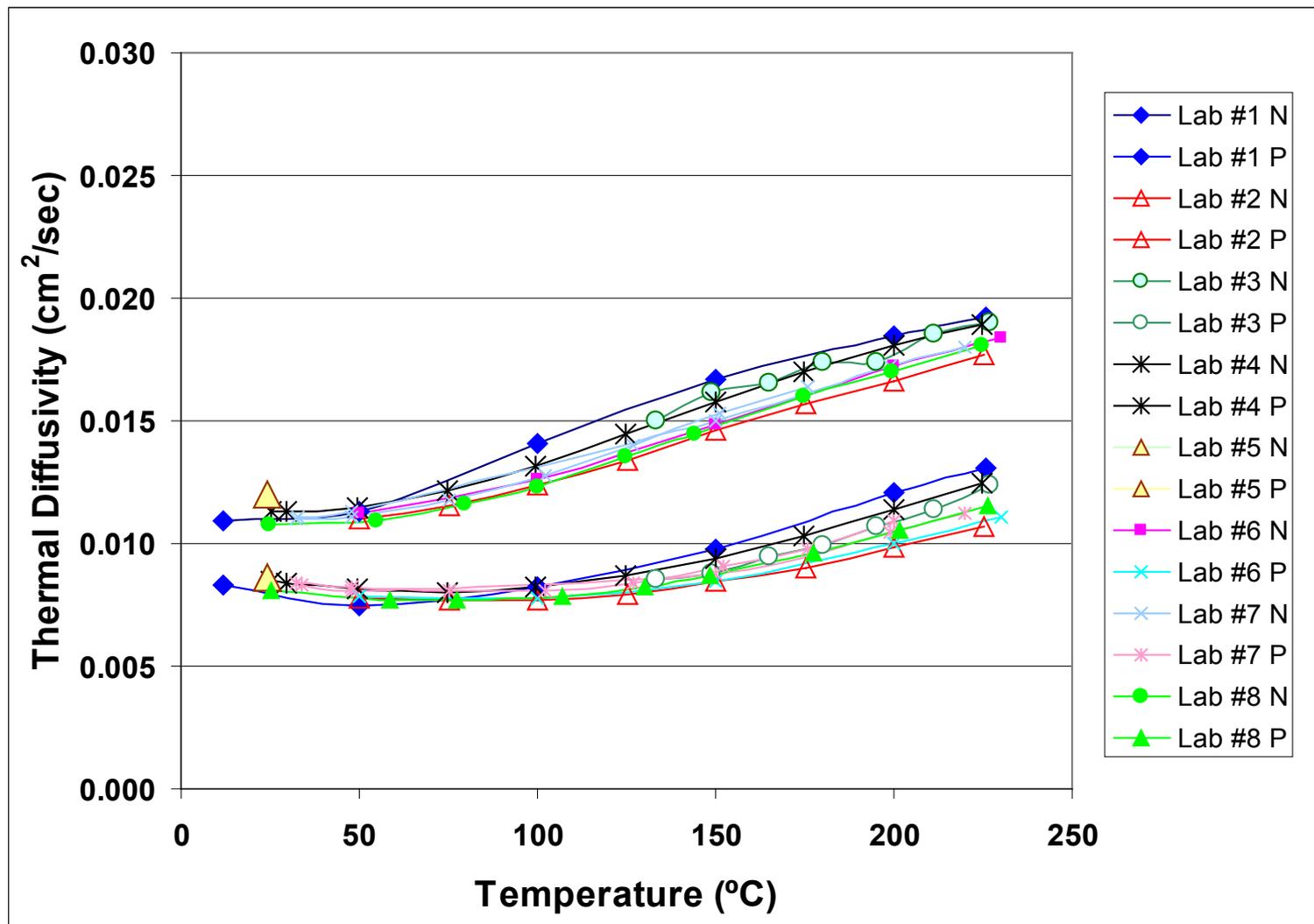
NIST Standards and Thermoelectrics

- Five internationally recognized standards:
 - Temperature (K); Distance (m); Current (A); Frequency (Hz) and Mass (Kg)
- Thermoelectric properties for ZT:
 - Seebeck coefficient: V/K
 - Electrical resistivity: Ohm-m
 - Thermal conductivity: (W/mK)
 - Thermal diffusivity: m²/sec
 - Specific heat: J/gK
 - Density: Kg/m³
- All TE properties are “derived”



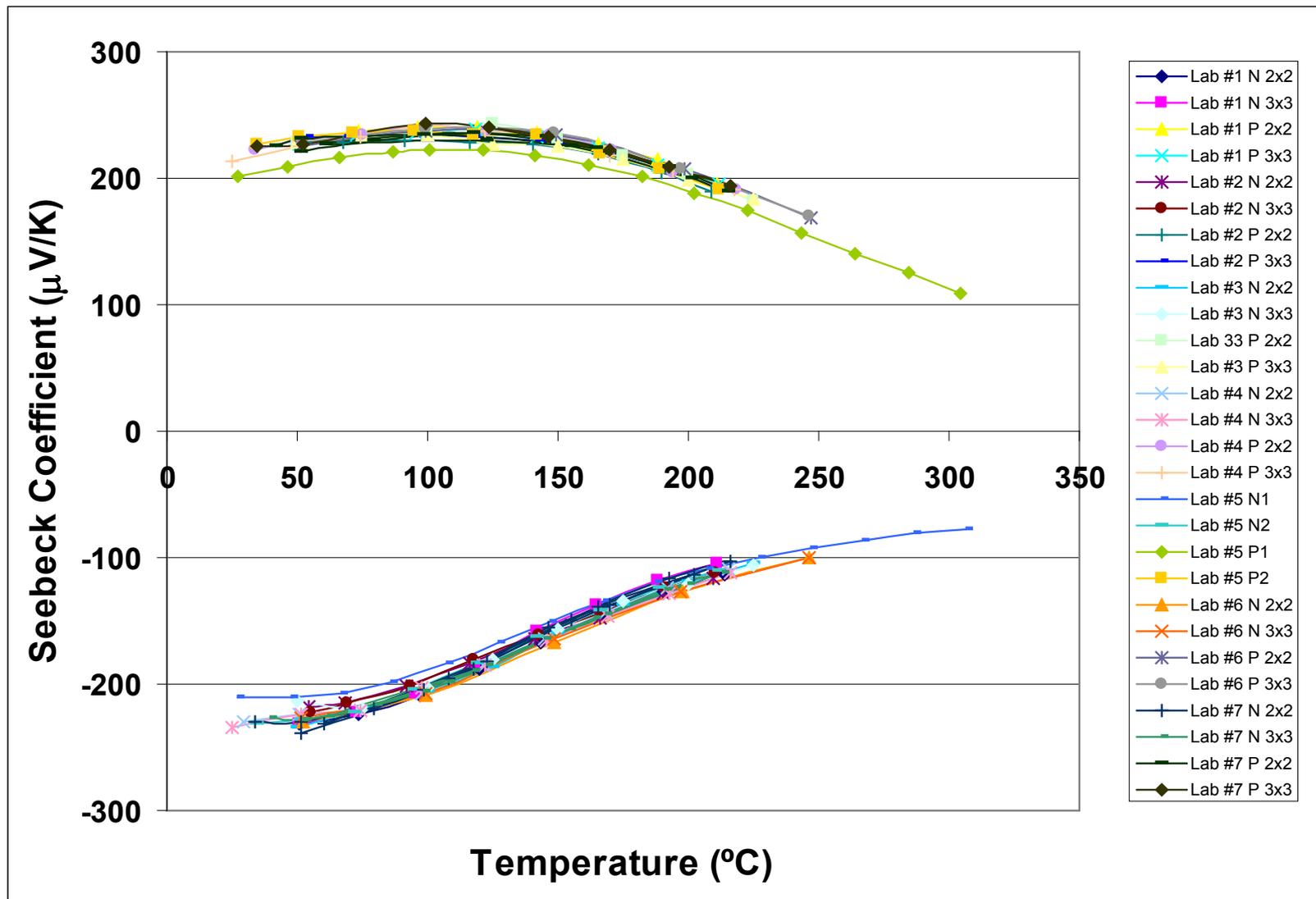
$$ZT = s^2 T / \rho k$$
$$k = \alpha C_p D$$

Round-robin 1: Thermal Diffusivity

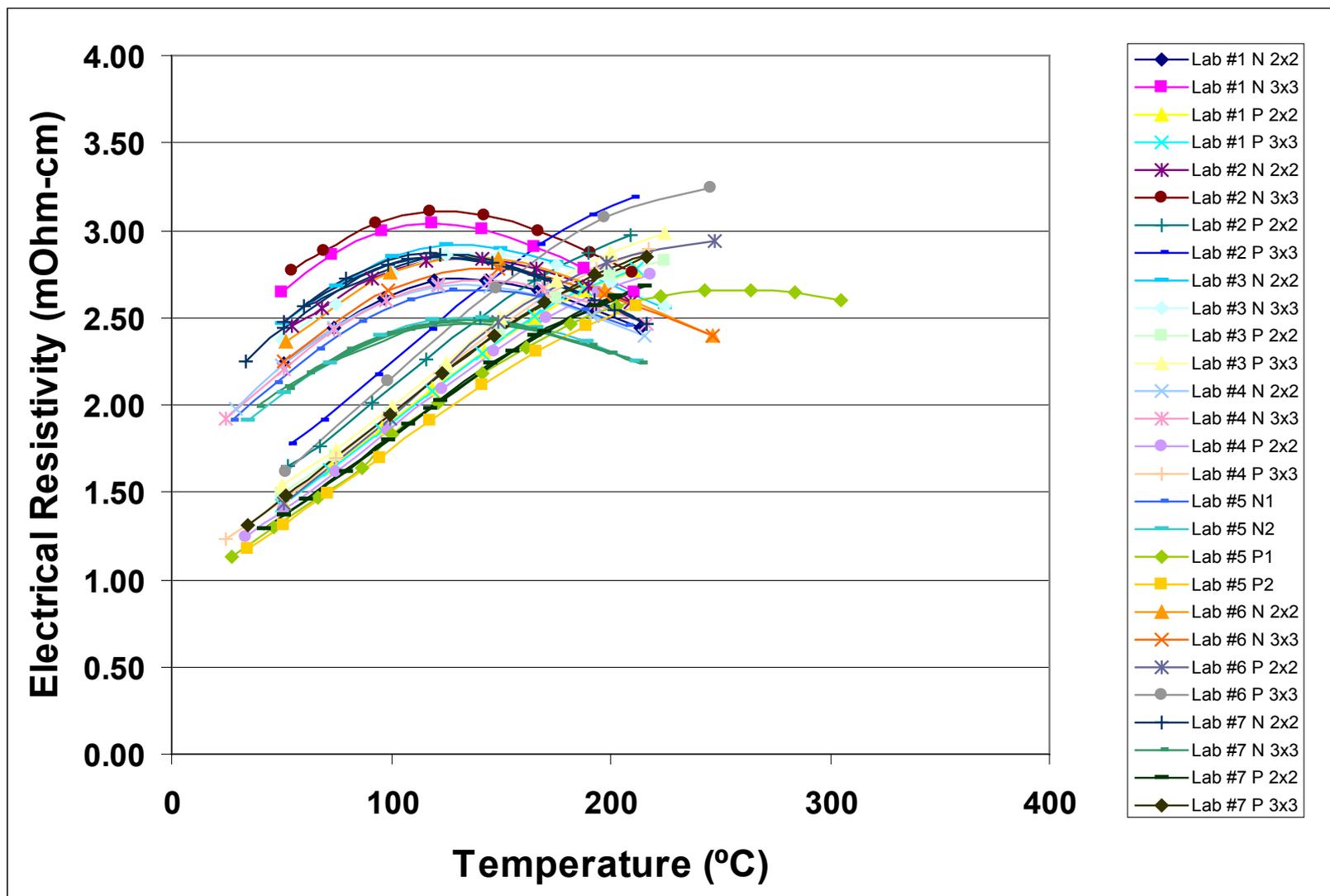


Results from 8 labs

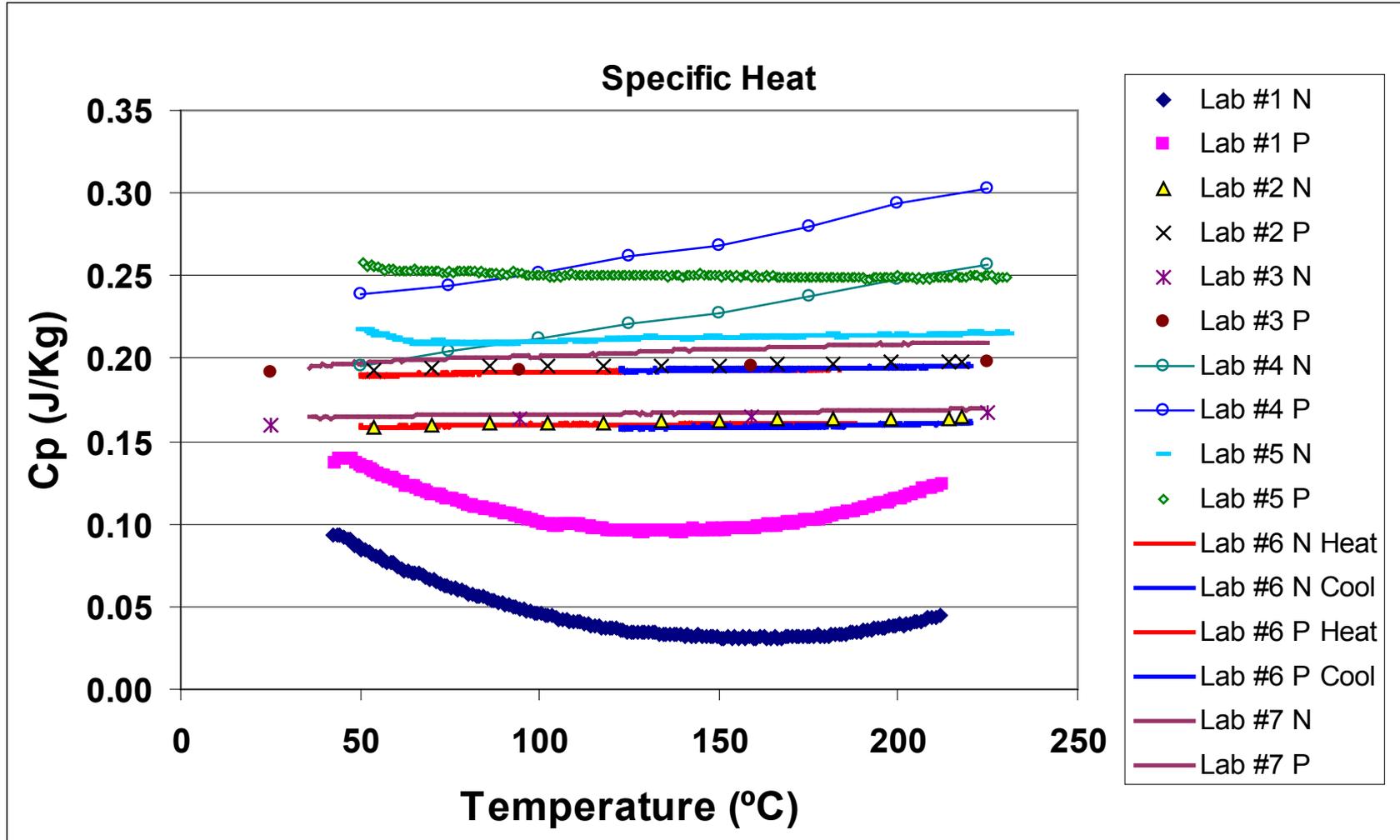
Round-robin 1: Seebeck Coefficient



Round-robin 1: Electrical Resistivity



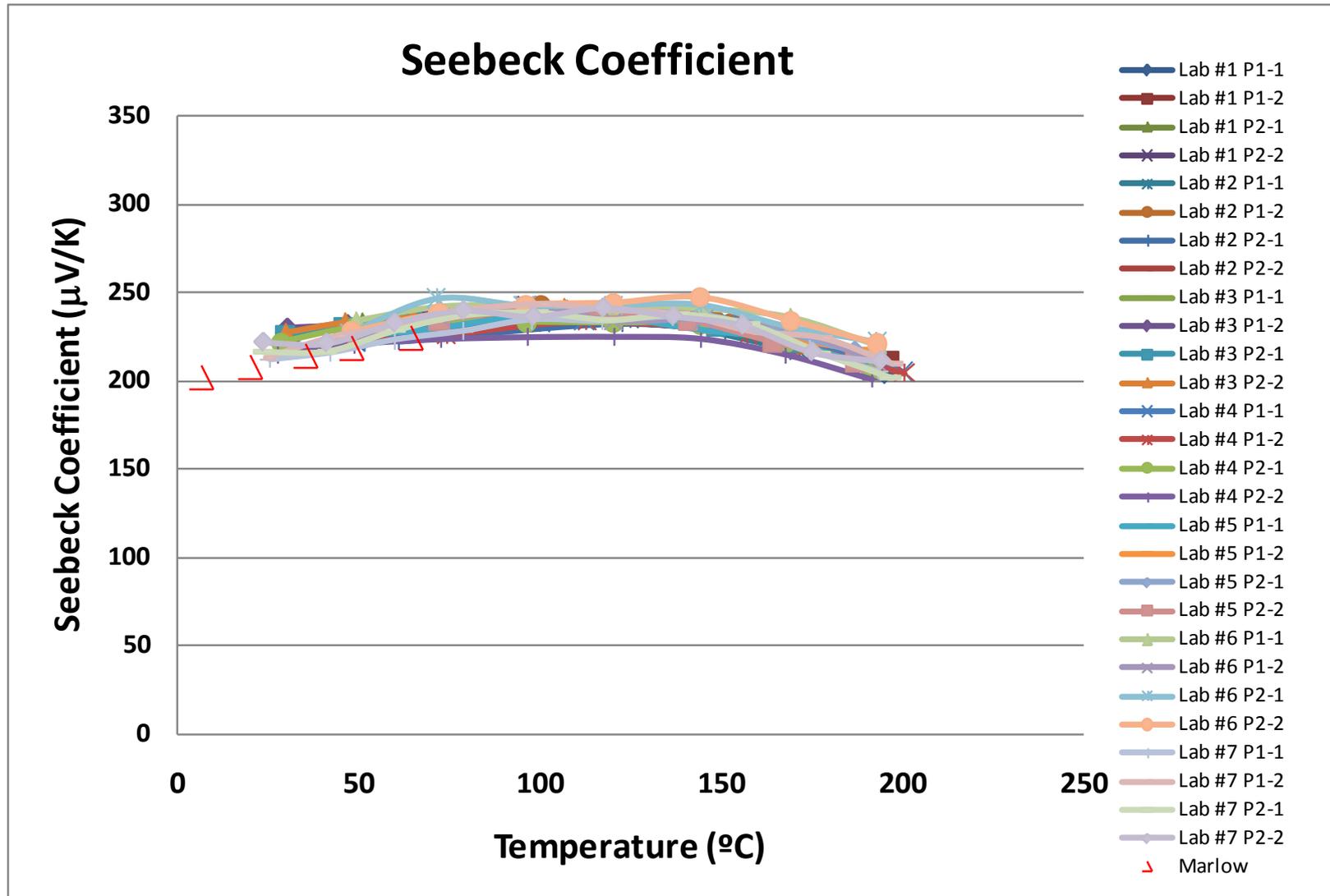
Round-robin 1: Specific Heat



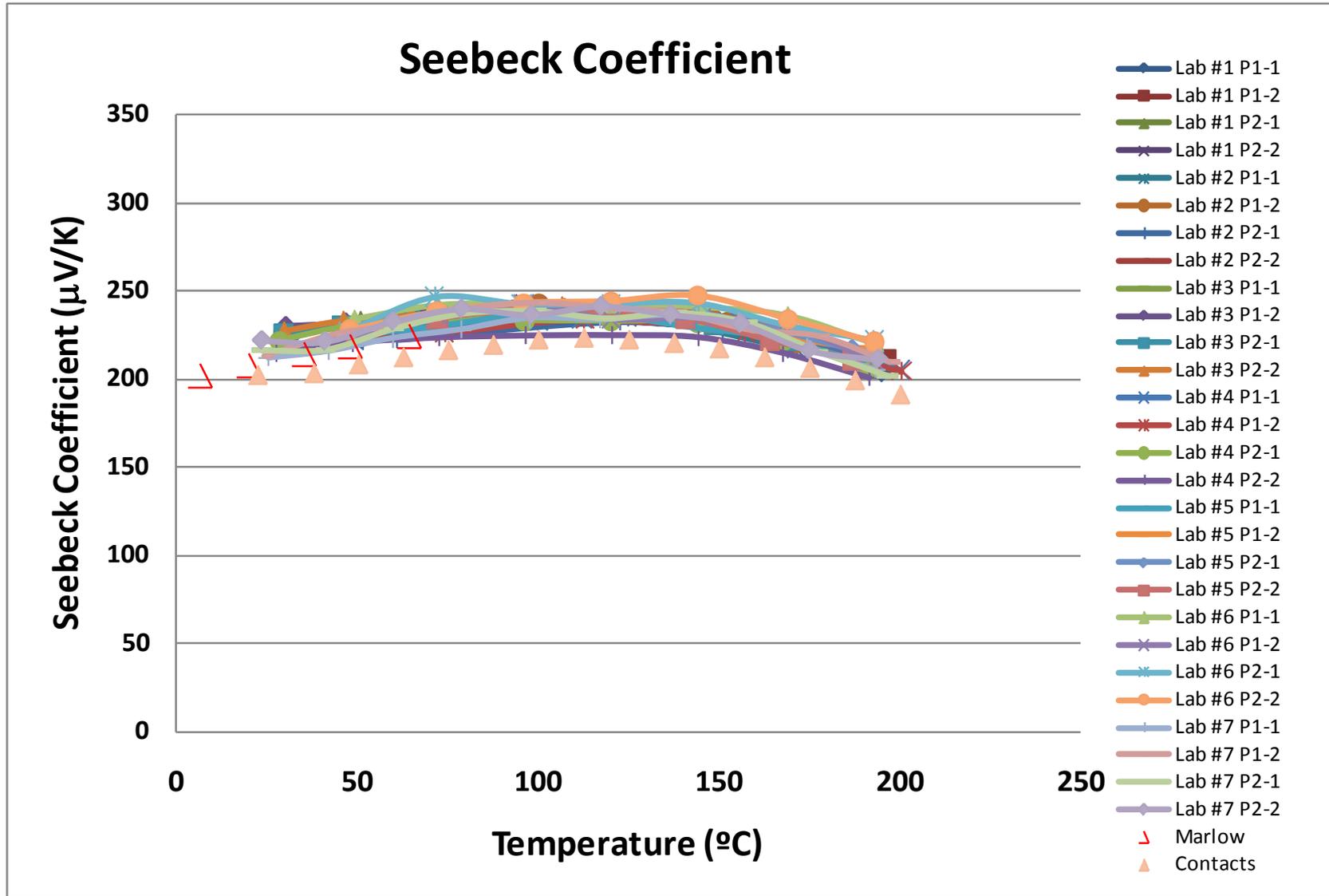
Round-robin #2 Started October 2010

- Procedures for DSC prepared by ORNL
- Two sets of p-type samples
 - Set #1: ORNL -> Clemson-> Corning -> ZT-Plus -> Germany -> China -> Canada
 - Set #2: China -> (Japan) -> Germany -> ORNL -> Clemson-> Corning -> ZT-Plus -> Canada
- Completed in September 2011
- Report to IEA-AMT: October 2011
- IEA-AMT Topical report November 2011

Round-robin 2: Seebeck Coefficient



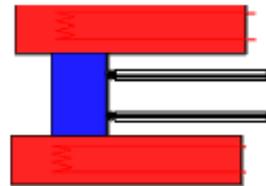
Discussion on Seebeck Measurements



Comments on Seebeck coefficient measurement with ZEM 3 (M10) and IPM-SR1

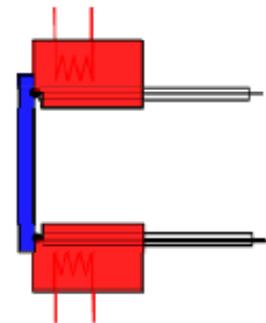
- ZEM-3 (M10)

Configuration of measurement:



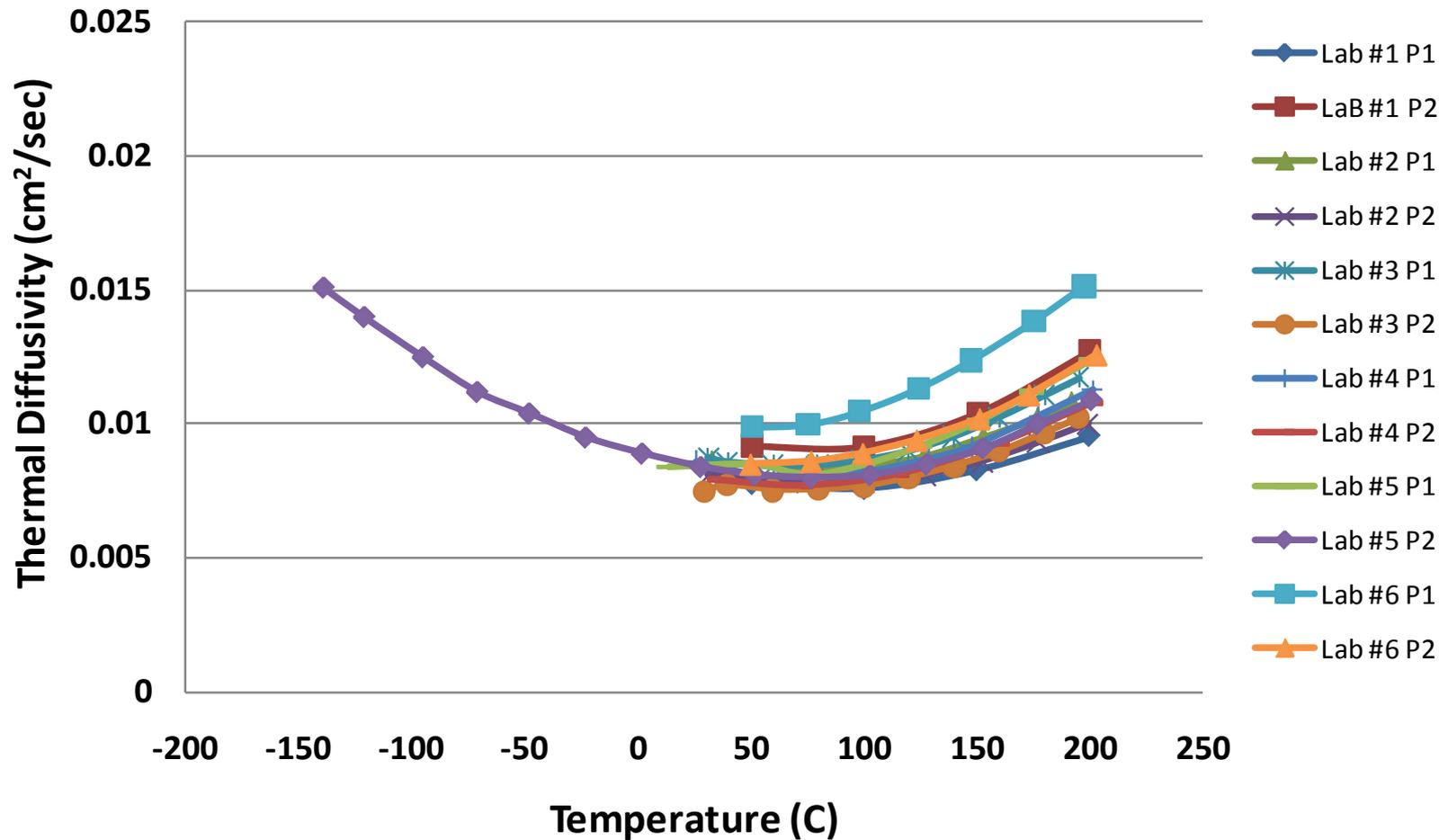
- IPM-SR1

Configuration of measurement:

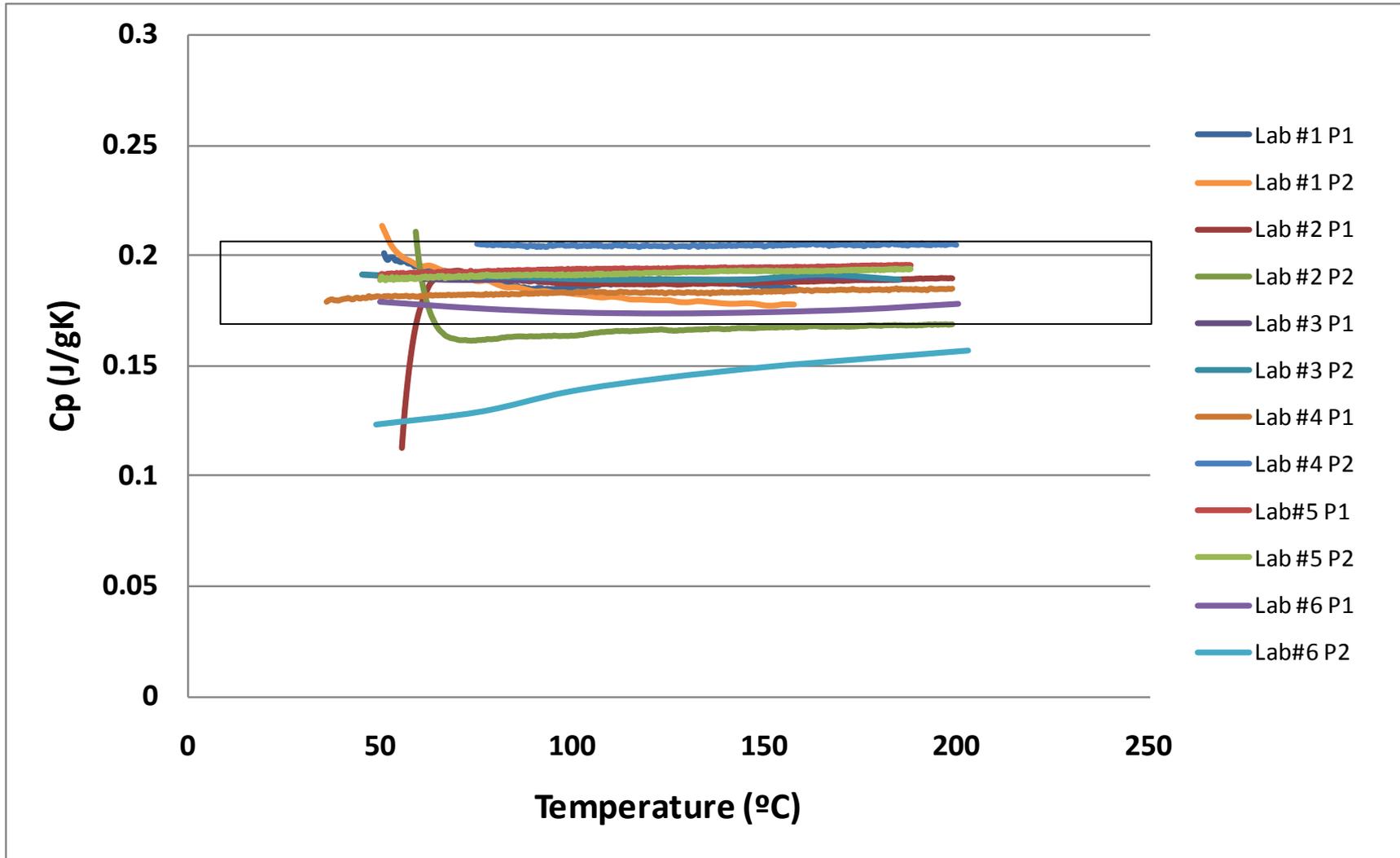


Assuming the real temperature gradient is the same, the temperature gradient measured with the ZEM-3 tends to be smaller than the one measured with the Fraunhofer IPM-SR1 because of the difference of configurations.

Round-robin 2: Thermal Diffusivity



Round-robin 2: Specific Heat



Round Robin 3 (Spring-Summer 2012)

- **Temperature range: RT-500C**
- **Materials n-type: lead telluride**
 - Difficult to machine, especially small disk for DSC
 - Professional cutting for thermoelectrics
 - Back-up materials is n-type skutterudite
- **Test Plan: Germany-> China-> US (ORNL, ZT-Plus, GMZ, Clemson, Corning) -> Canada**
 - Alternate methods: Marlow, ARL



Summary

- **IEA-AMT is addressing the important issue of measurement standardization of thermoelectrics**
- **Significant measurement issues were observed, especially in specific heat and electrical resistivity.**
- **Good agreements in Seebeck coefficient, electrical resistivity**
- **Thermal diffusivity in good agreement expect for one test (data analysis)**
- **Specific heat remains an issue for reliable ZT**
- **Round-robin 3 underway**