Minimizing Pressure In-homogeneities for Large Samples in High Pressure Neutron Scattering Measurements

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NIST Center for Neutron Research

New Directions for High-Pressure Neutron Workshop
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Minimizing Pressure In-homogeneities for Large Samples in High Pressure Neutron Scattering Measurements

- Reasoning
- Apparatus
- Results
- Technique

Reasoning

A. Freezing ($V_{constant}$)

B. Cooling to Freezing Point ($P_{constant}$)

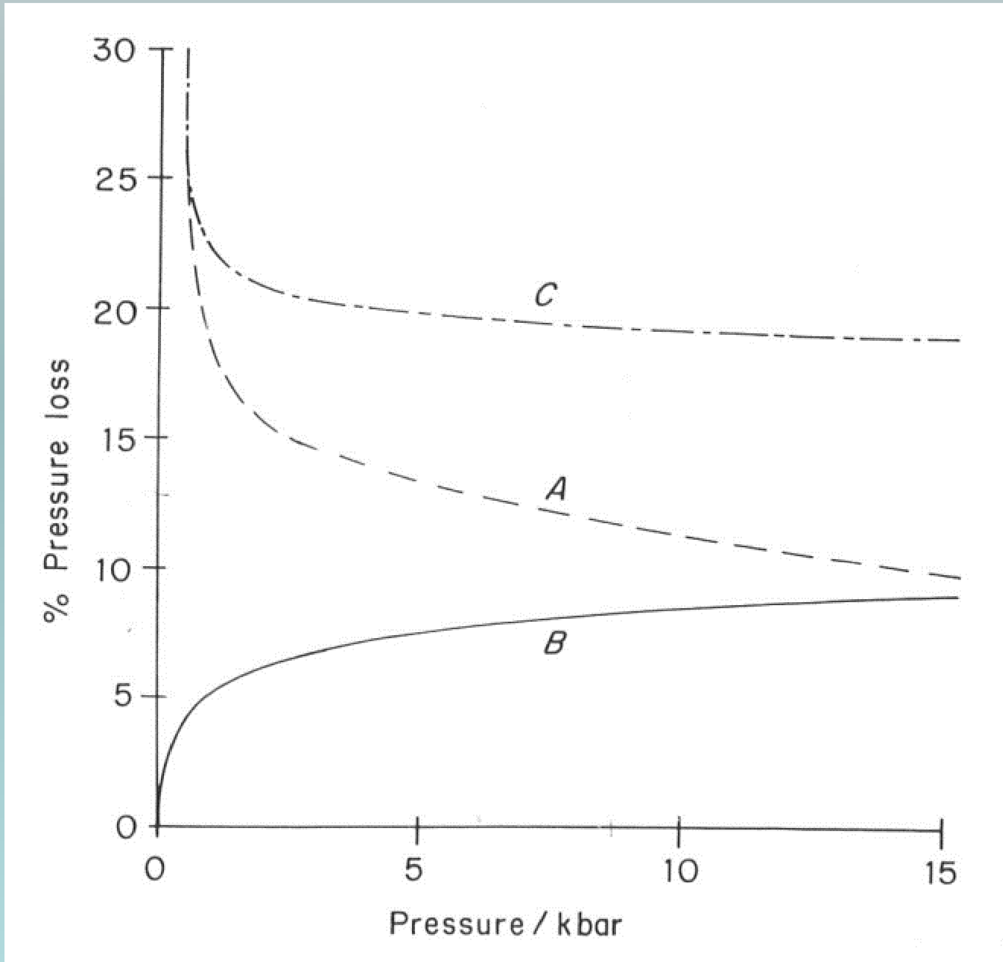
C. Freezing and Cooling ($VP_{constant}$)

W.F. Sherman, A.A. Stadtmuller. *Exp. Tech. in H. Pressure Research*

i. Pressurize above PxT curve

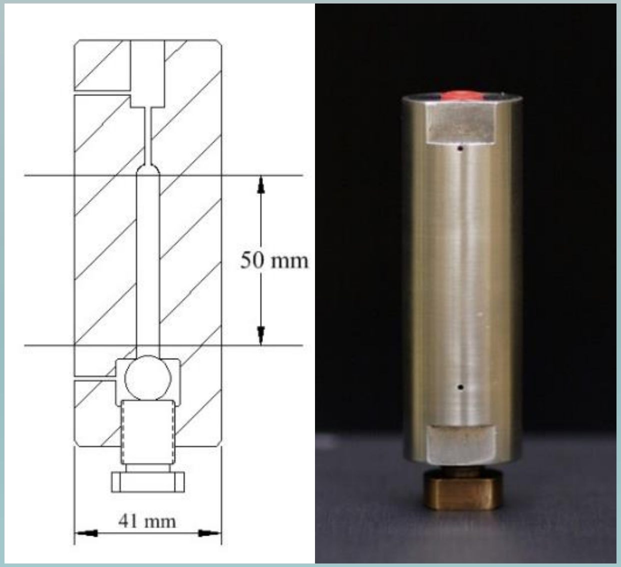
ii. Cool slowly under $P_{constant}$ down to the freezing point

iii. Hope for the best down to base



W.F. Sherman, A.A. Stadtmuller. *Exp. Tech. in H. Pressure Research.*

Apparatus



P_{max} = 7.0 kbar

Working Pressure = 6.5 kbar

Al 7075-T6 Construction

1.5 cm^3 sample volume

69% Neutron transmission at 2Å

Harwood Eng., Inc. 2-Stage Intensifier

Results

i. Pressurize above PxT curve

ii. Cool slowly under $VP_{constant}$ down to the freezing point

iii. Hope for the best down to base

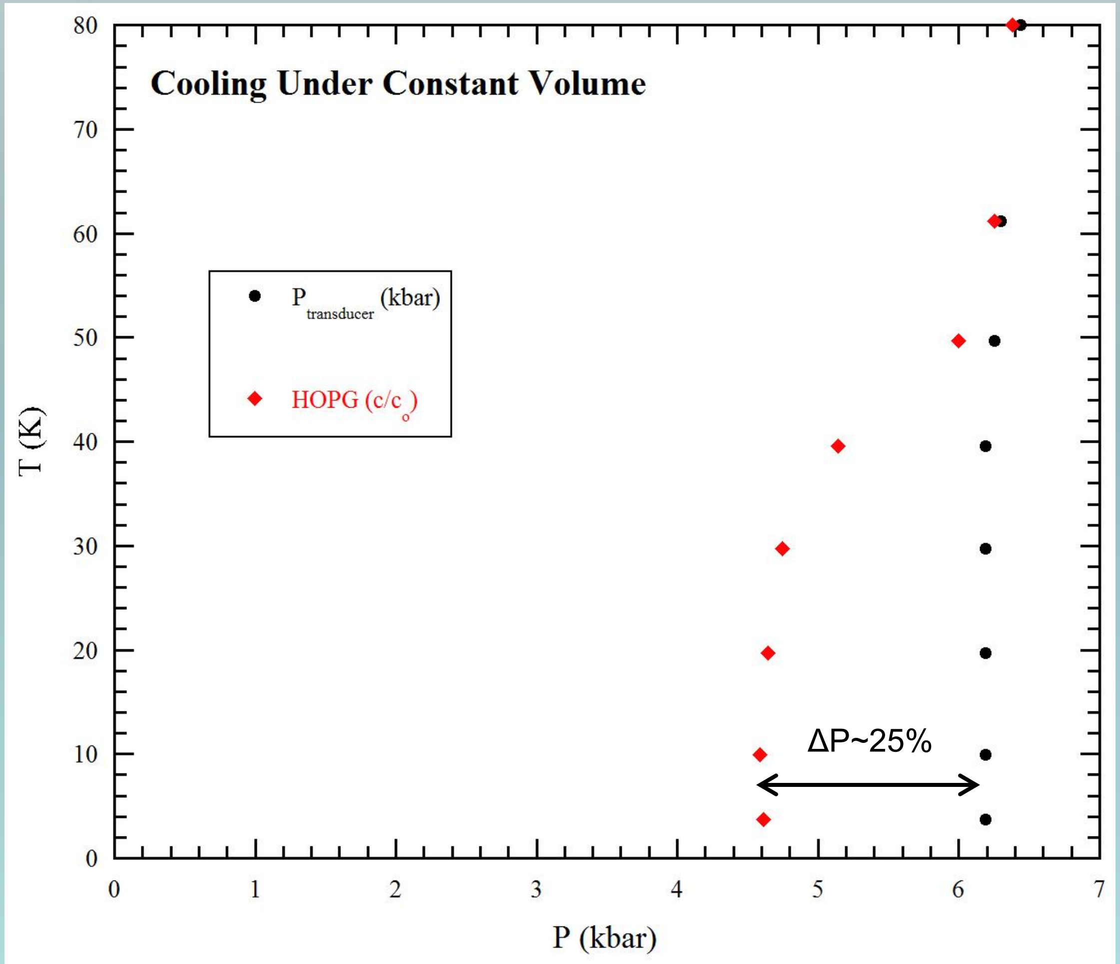
$$P = \left(\frac{\beta_o}{\beta'}\right)\left[\left(\frac{r}{r_o}\right)^{-\beta'} - 1\right]$$

From 1-D Analog to the Murnaghan Equation

$$\beta' = 10.8(9)$$

$$\beta_o^{-1} = -\left(\frac{d \ln r}{dP}\right)_{P=0} = 373^{-1}\ kbar$$

Hanfland, Beister, Syassen. *Phys. Rev. B* ***39****, 1989*

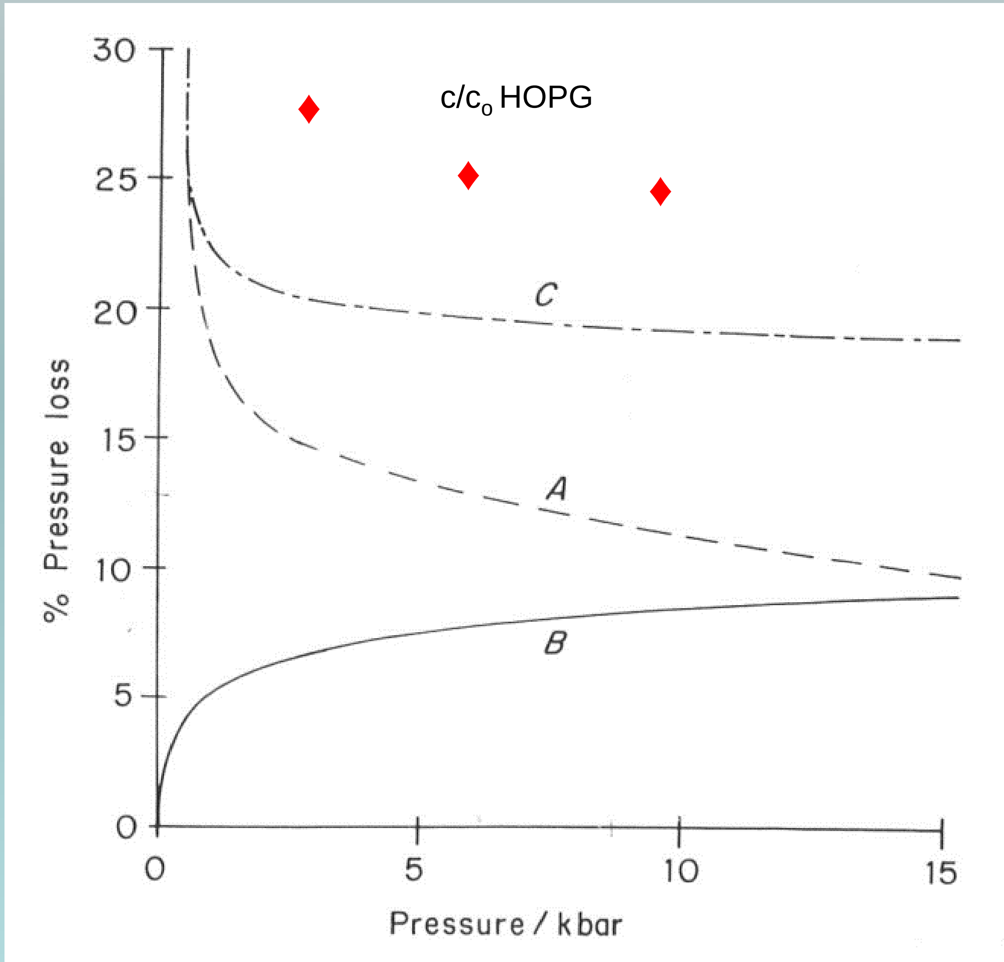


Results

Freezing and Cooling ($VP_{constant}$)

for neutron elastic measurements of

HOPG (c/c_o)



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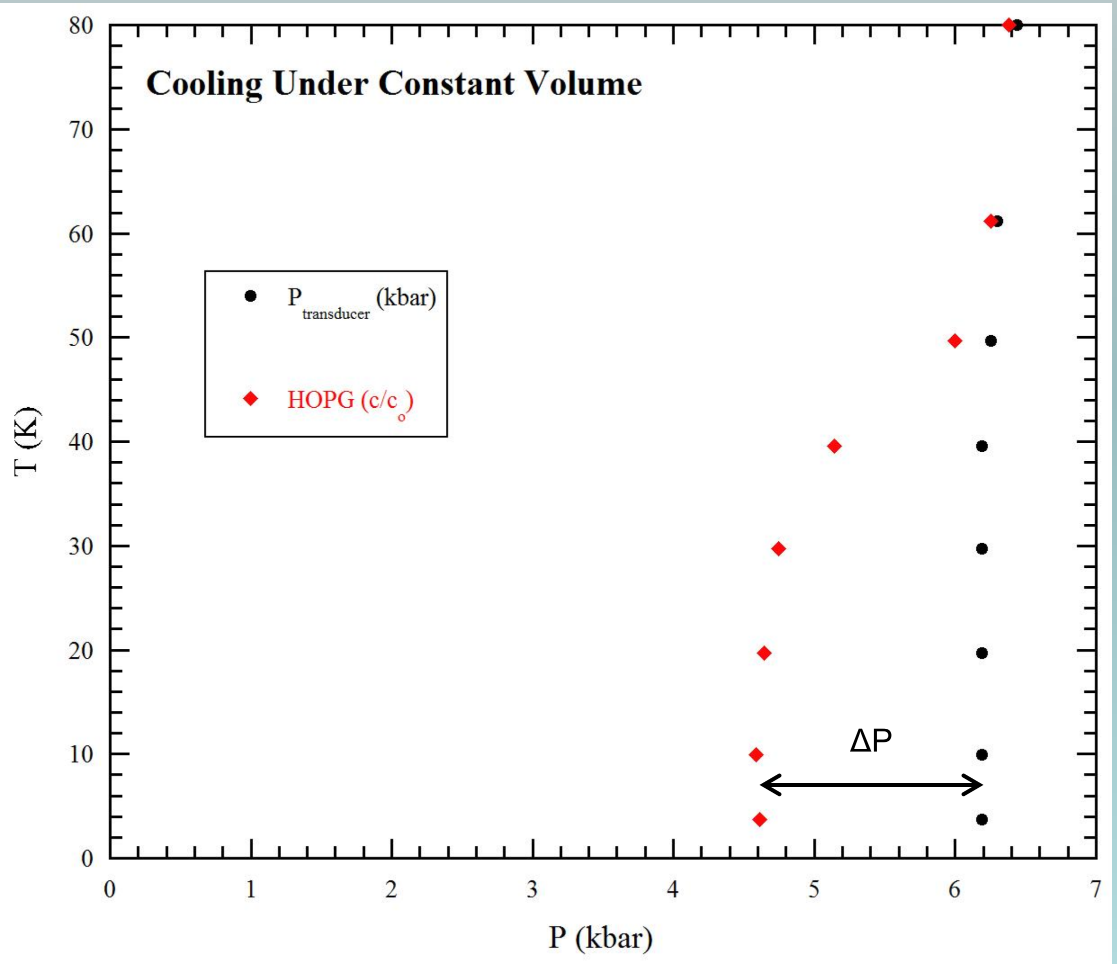
Results

Freezing and Cooling ($VP_{constant}$) → ΔP~25%

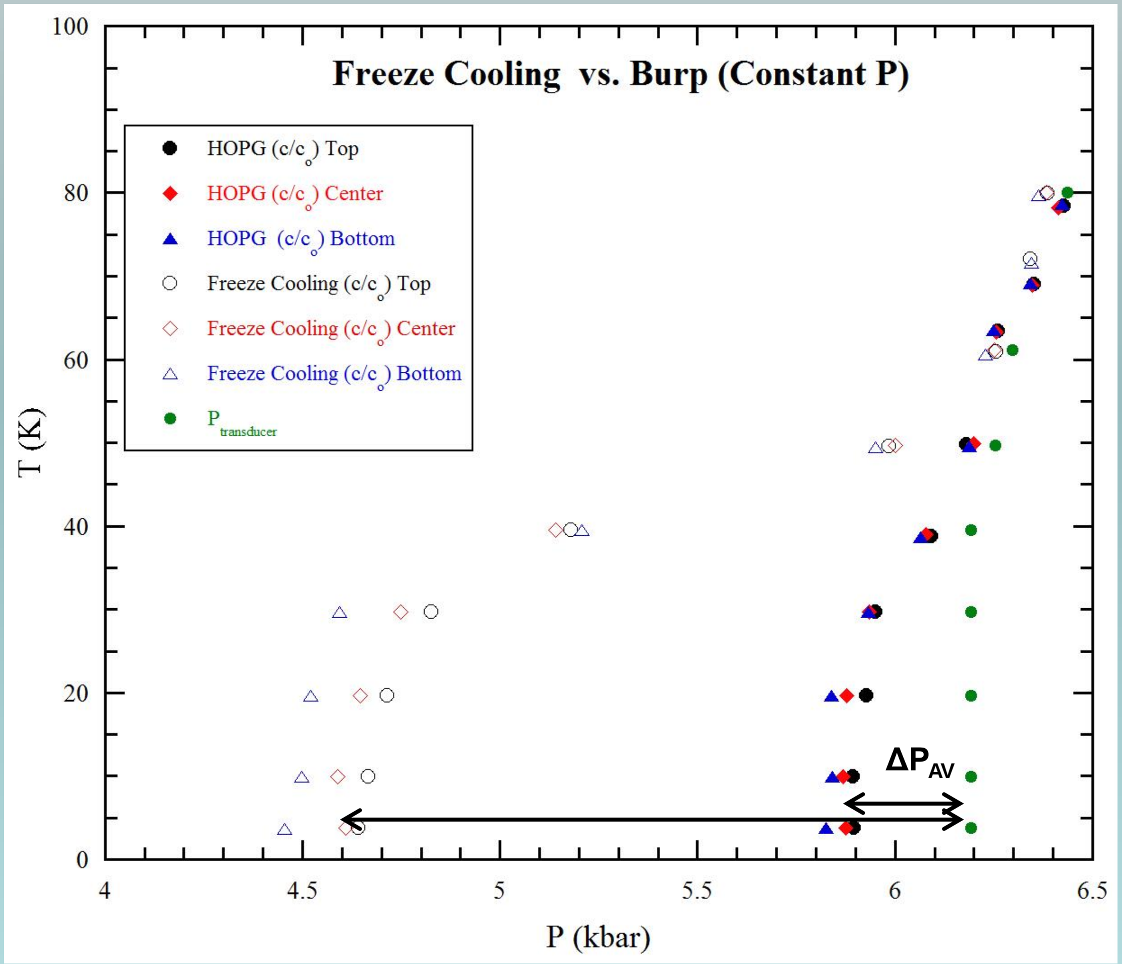


Results

Cooling to Freezing Point ($P_{constant}$)

ΔP_{AV}~5%

When systematically ensuring that the pressure vessel is completely full of solid He



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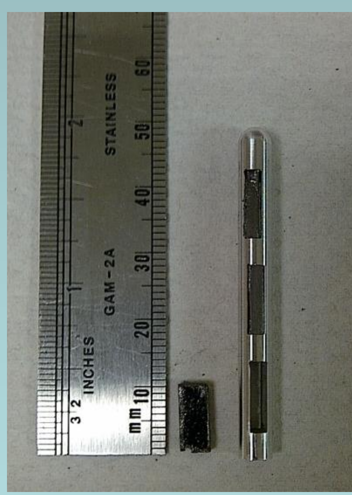
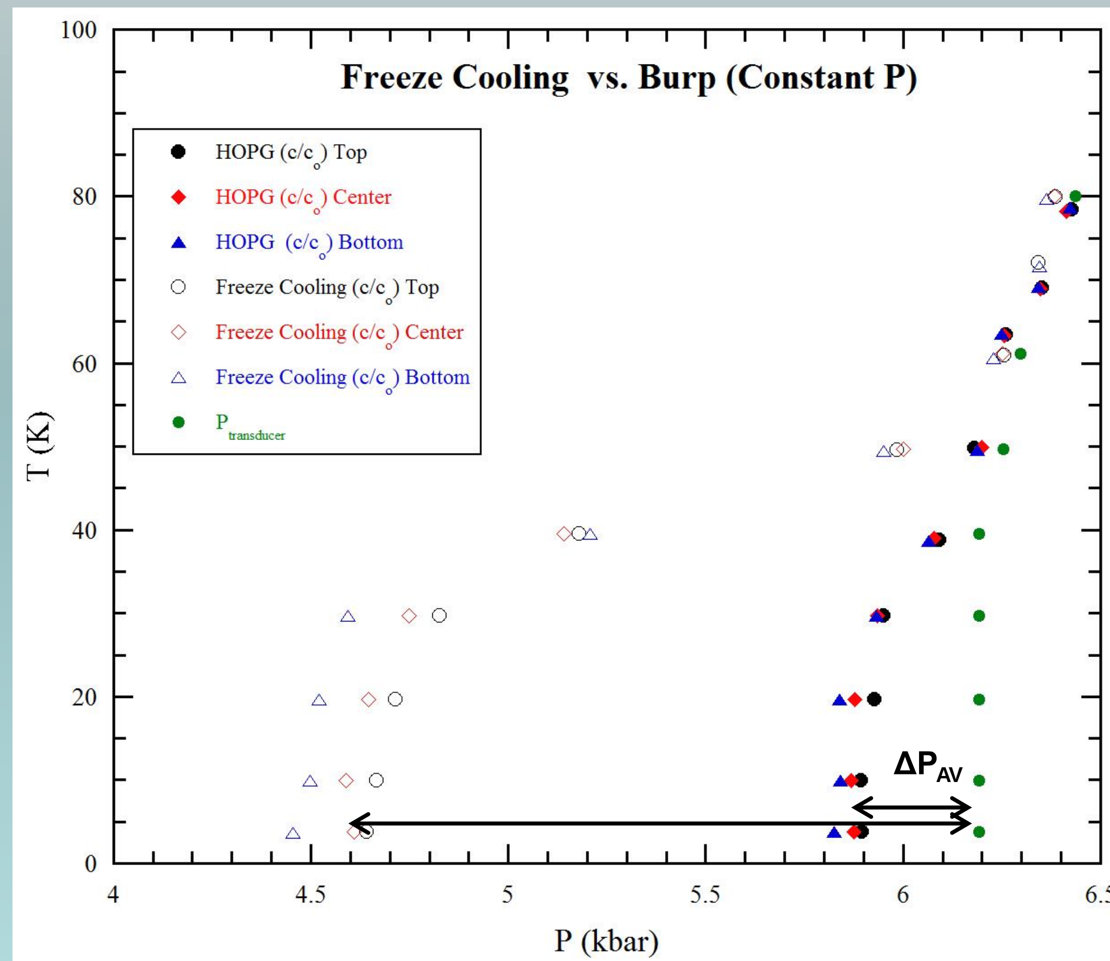


5mm x10mm HOPG Xtals ~10º offset



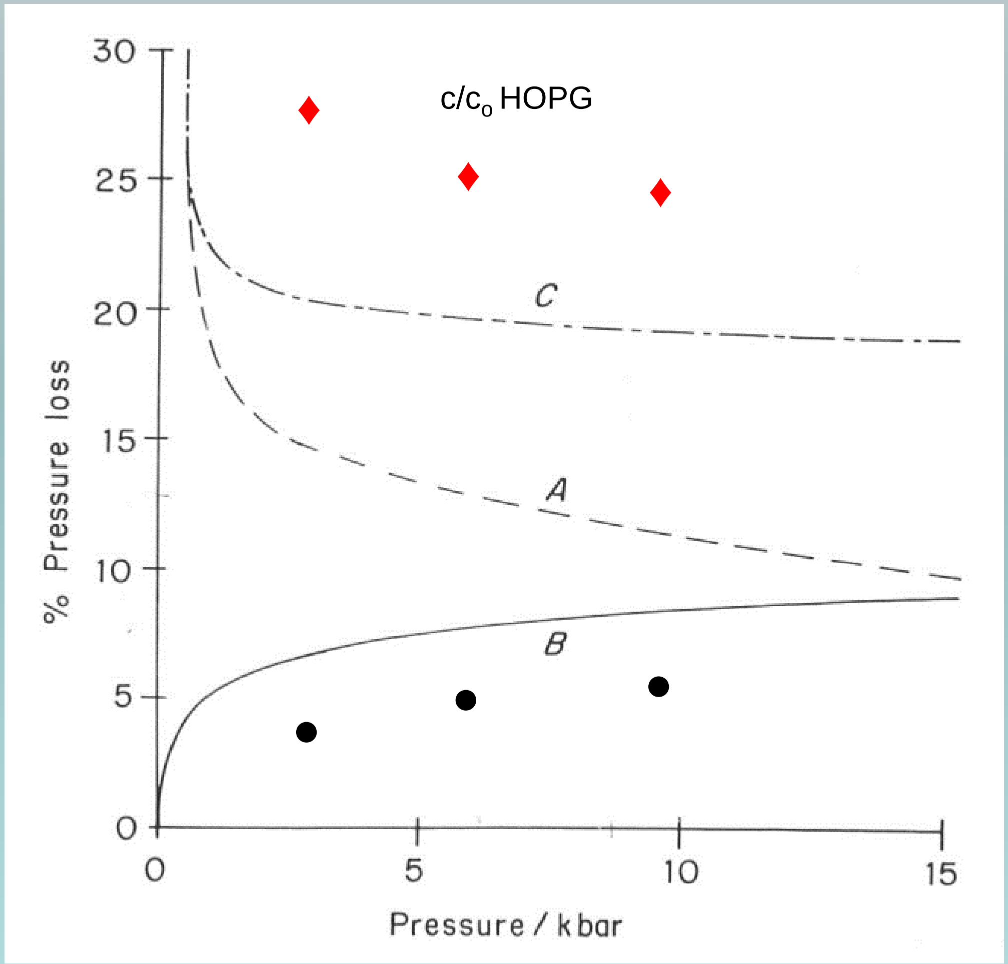
Results

♦ Freezing and Cooling ($VP_{constant}$)

→ $\Delta P \sim 25\%$

● Freezing and Cooling ($P_{constant}$) for neutron elastic measurements of HOPG (c/c_o)

→ $\Delta P_{AV} \sim 5.5\%$



W.F. Sherman, A.A. Stadtmuller. *Exp. Tech. in H. Pressure Research.*

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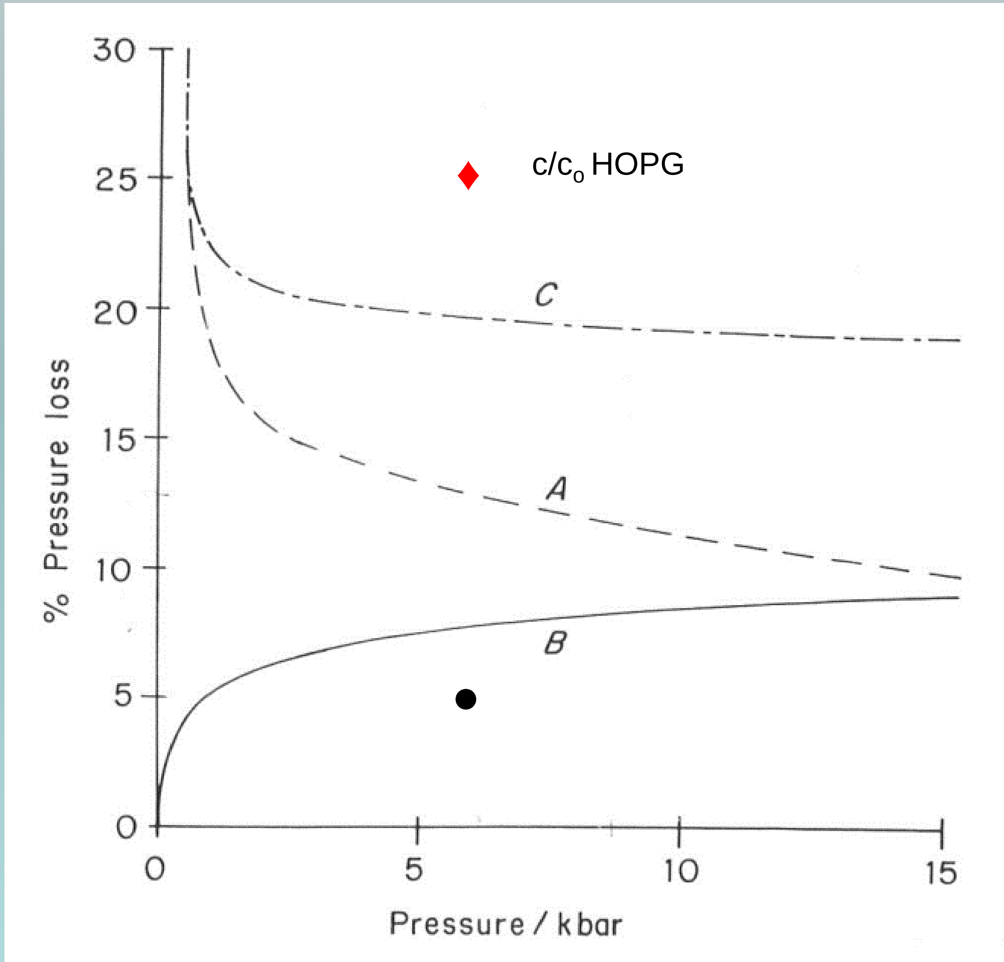

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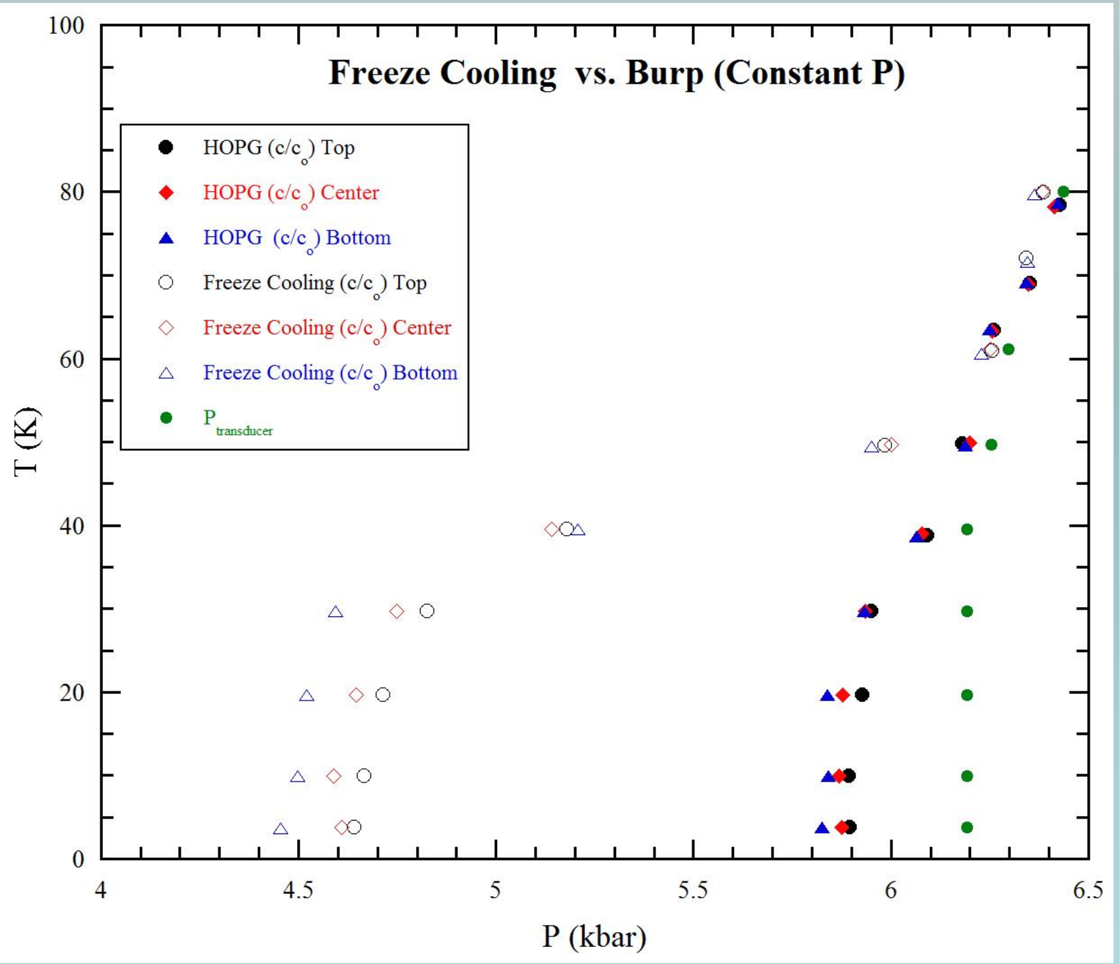
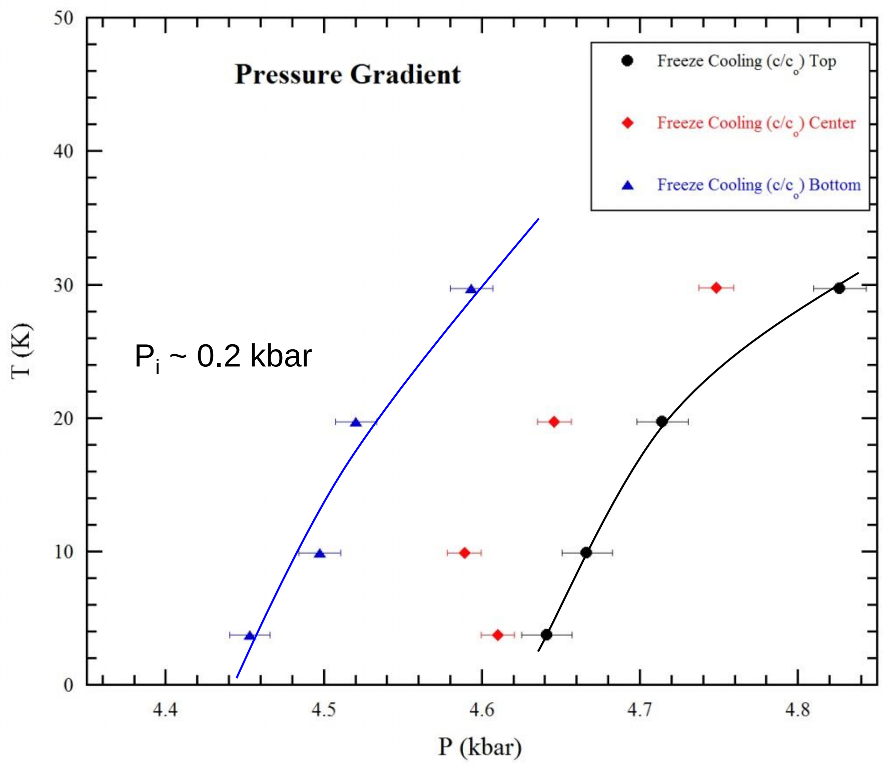
Bonus:
Sample pressure in-homogeneities are minimized



Results

Sample Pressure in-homogeneities (P_i) Comparison



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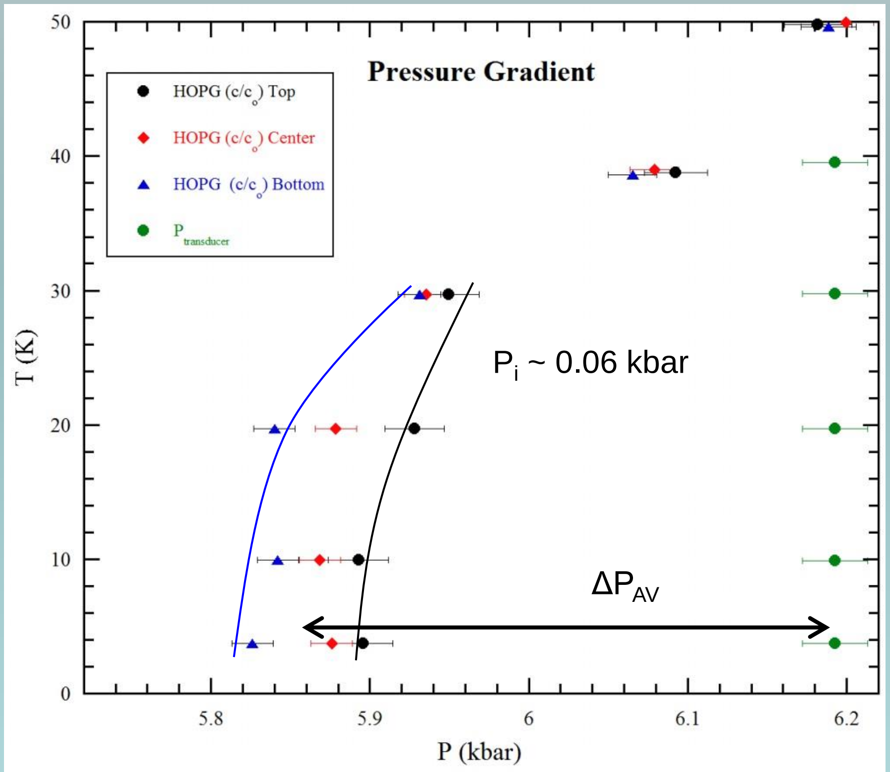
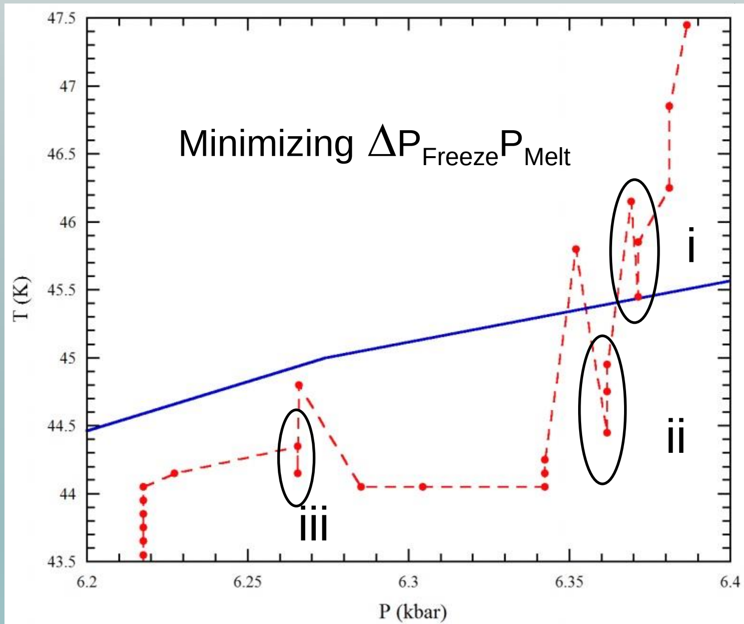


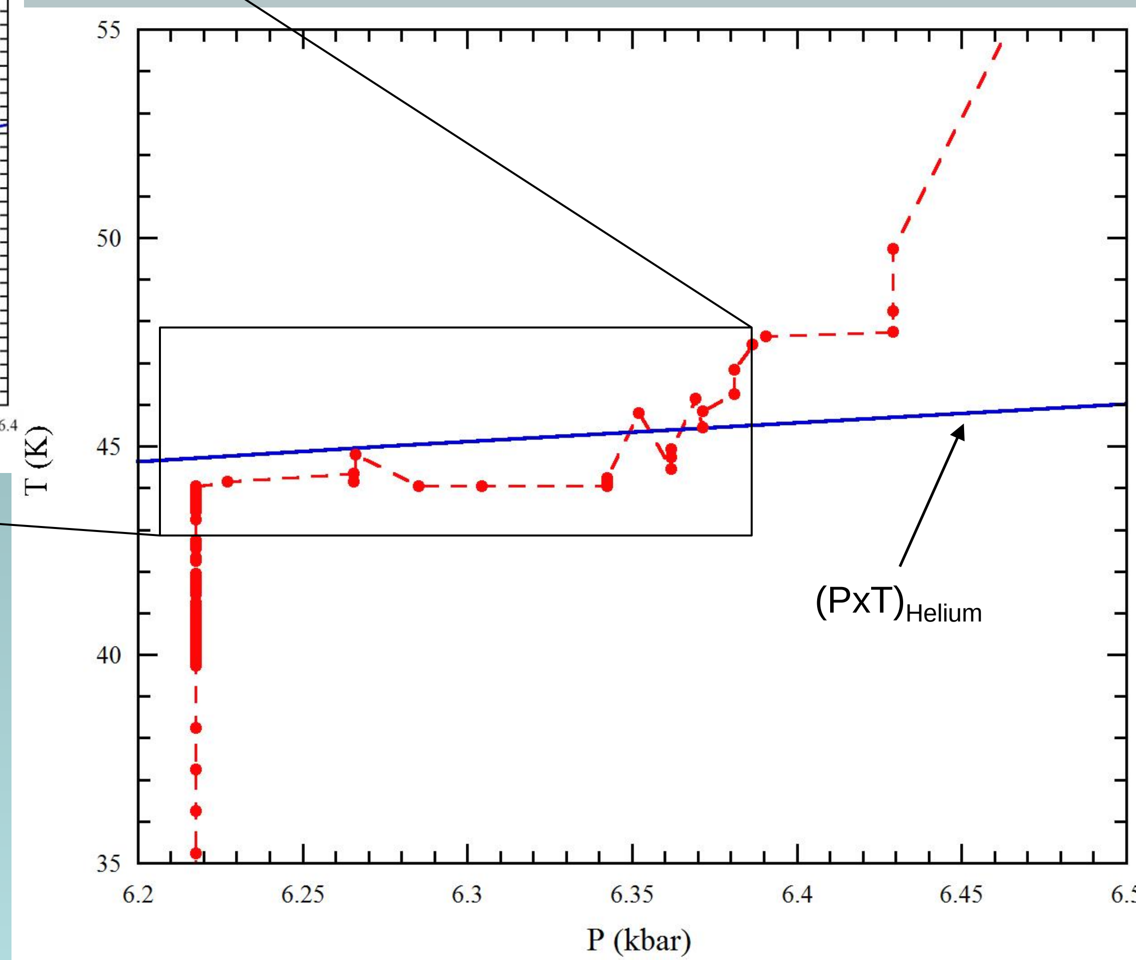
Burp Freezing and Cooling ($P_{constant}$) → ΔP~5%

Technique

- Use He as pressure media
- Monitor pressure line temperature
- Control line temperature well above desired PxT
- Cool down to a few Kelvin of PxT curve
- Apply pressure while heating line
- Begin ramp-cooling through the PxT curve
- Systematically heat line upon transducer pressure “freeze” providing enough power to counteract the cooling of the cell as noted in the sample stick sensor
- Once transducer pressure “melts” reduce line heater power to once again “freeze” the transducer reading
- Repeat until $P_{Freeze} \sim P_{Melt}$
- Continue cooling to base while still heating line
- Begin reducing line heater power when the cryostat cooling bottoms out
- Turn off line heater when $T_{Line} < T_{Freeze}$

Technique





$\Delta P_F P_M(i) < \Delta P_F P_M(ii) < \Delta P_F P_M(iii)$

Results

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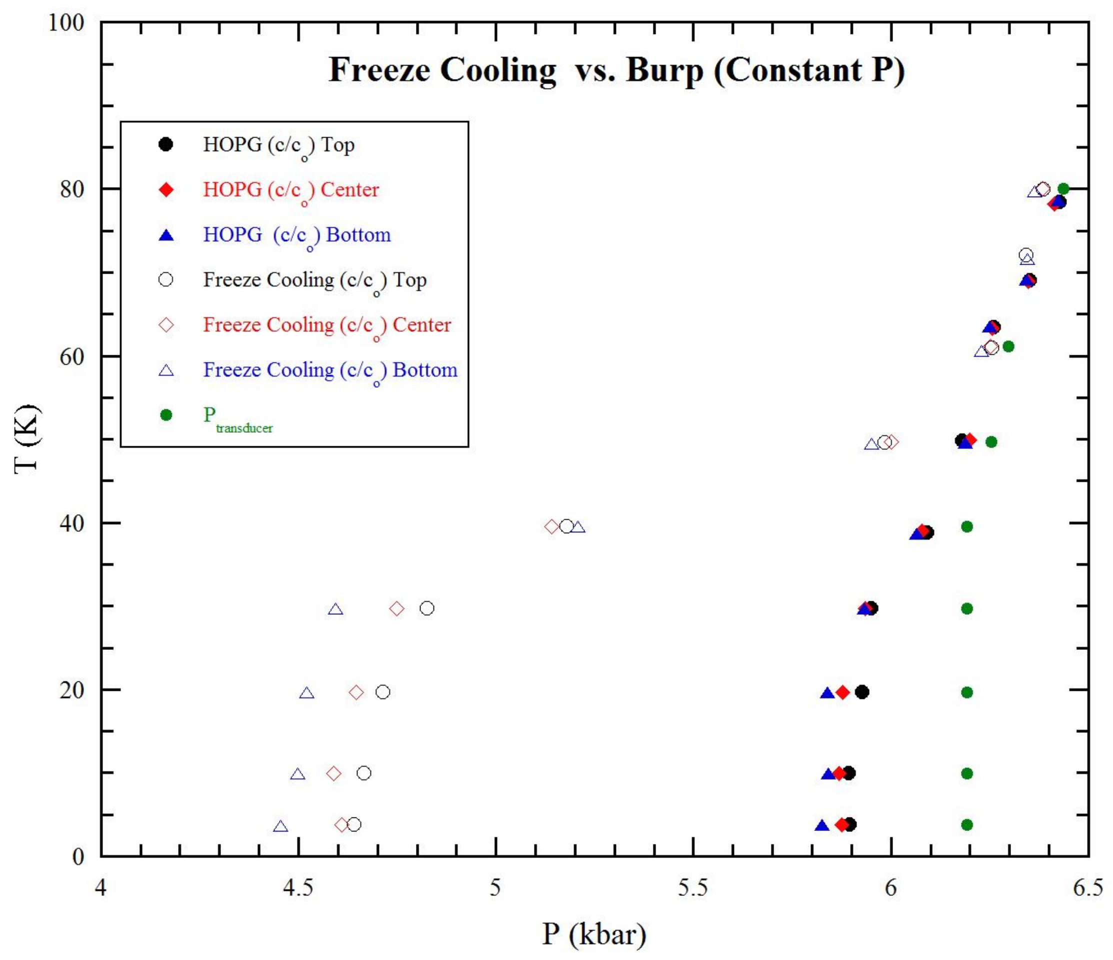

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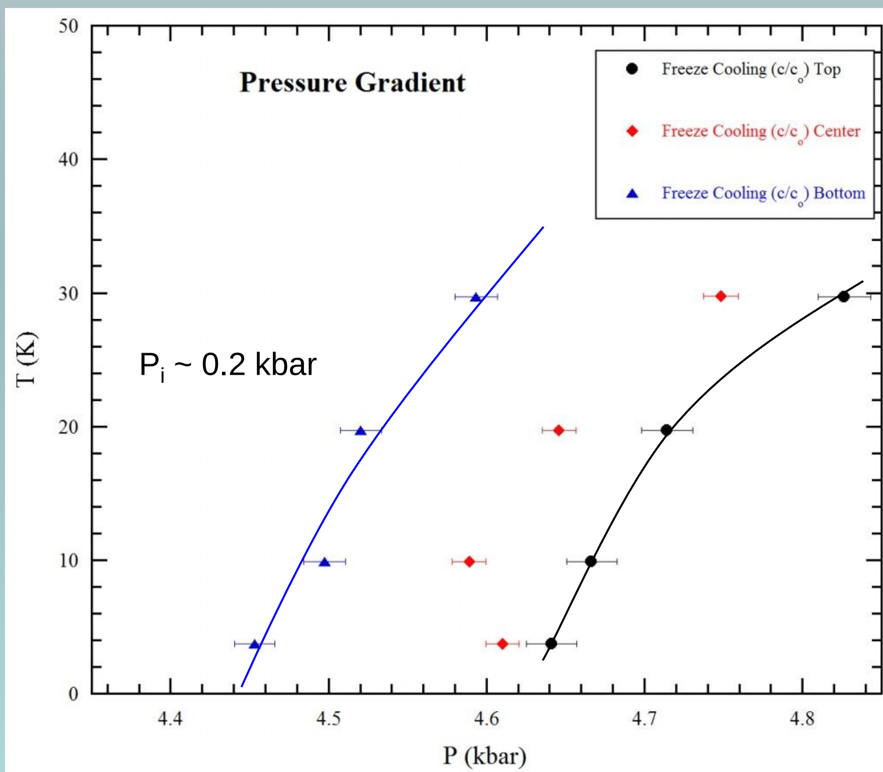

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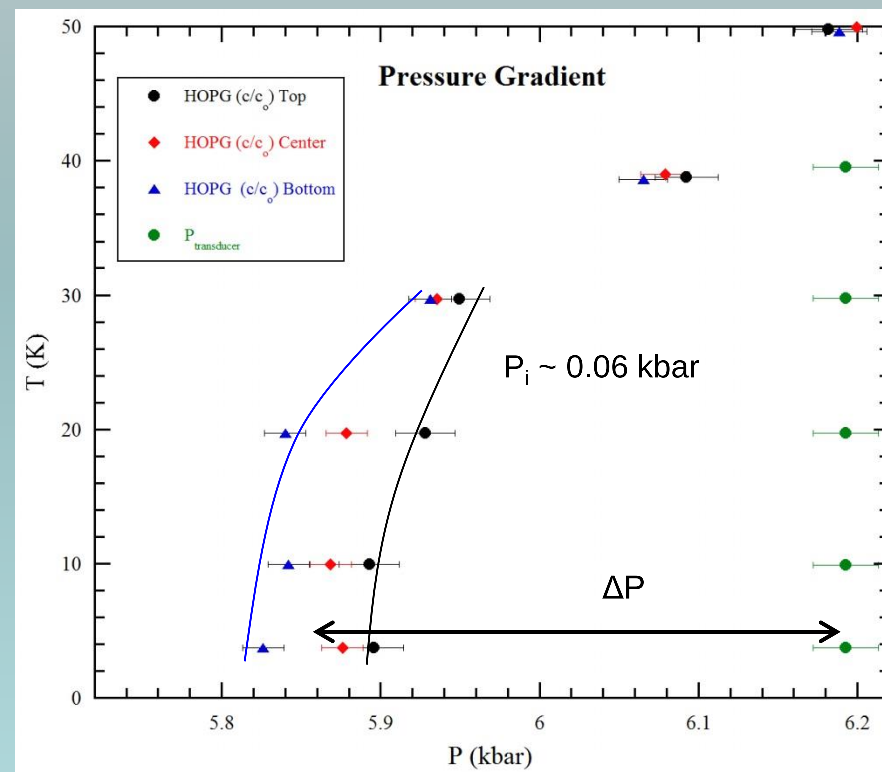

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Thank You!