

## **vSANS Sample Environment**

- 1) Collimation Options → New Sample Sizes
- 2) Sample Holder Options for Complex Fluids
  - a. Commercial (Hellma-type) Quartz Cells
  - b. New sizes of Ti-Cells (Circular and Rectangular)
- 3) New Sample area layout
  - a. Rad. Safety Interlocked Sample Staging area
  - b. No sample chamber → Condensation issue
  - c. Larger available area → Larger sample blocks
- 4) Heat Transfer Calculations / Meas. w Current 10CB
- 5) Possible New Cooling Block Design Features
  - a. Insulate (foam and/or vacuum)
  - b. Double Block/Bath using “Leap-Frog” T-settings
  - c. Reduce Block Volume for Quicker Equil. Response
- 6) Shutdown Fabrication “to do” List

## Sample Aperture Sizes

{ beam current ~ area }

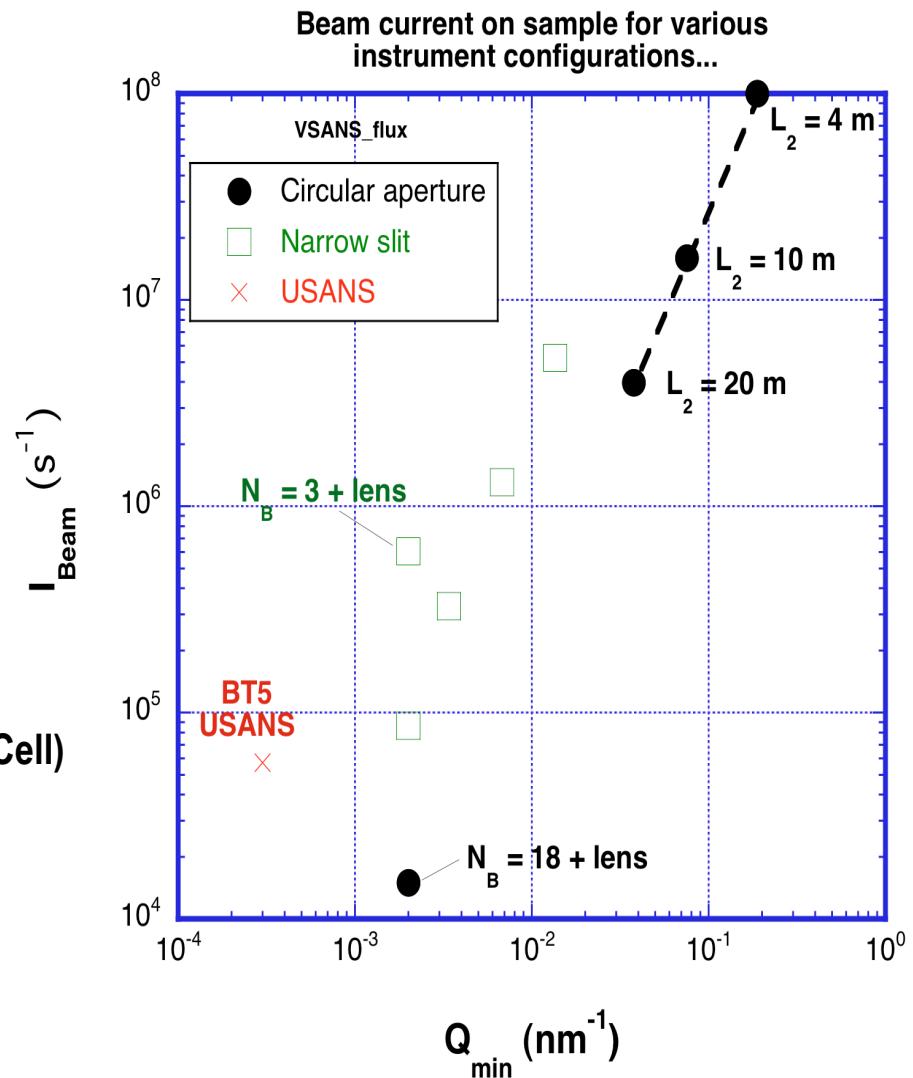
- For circular aperture: **30 mm dia.**
- For Narrow slit  
**1.5 mm or 3 mm x 75 mm**
- For Converging Beams:  
**38 mm x 75 mm**

## USANS Beam Current:

16 mm (5/8") dia. → 28,000 s<sup>-1</sup> (current Ti-Cell)

22 mm (7/8") dia. → 48,600 s<sup>-1</sup> (gain 1.7)

29 mm (9/8") dia. → 67,900 s<sup>-1</sup> (gain 2.4)



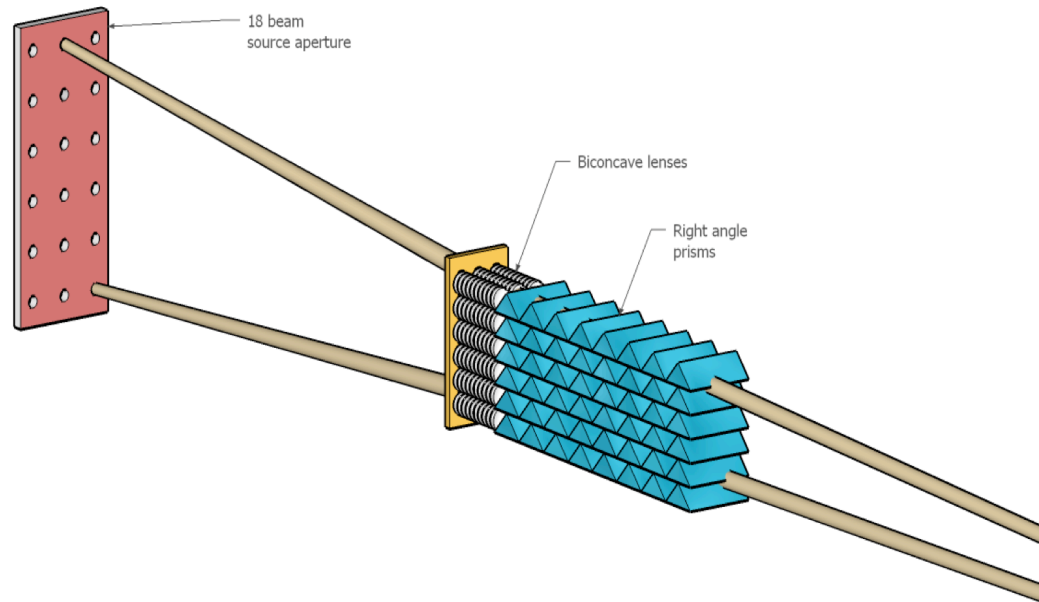
## 18 Converging Beams:

3 x 6 beams using 12.5 mm dia.

Lenses:

Width = 3 x 12.5 mm = **38 mm**

Height = 6 x 12.5 mm = **75 mm**



## Largest Commercial Quartz Cell Options (Hellma)

Hellma – Precision in SpectroOptics Worldwide – Küvetten für Fluoreszenzmessungen

1/6/11 5:10 PM

Hellma – Precision in SpectroOptics Worldwide – Küvetten für Fluoreszenzmessungen

1/6/11 5:01 PM

**Order Number** 120-000-1-40  
**Type** 120-QS

**Material Color Code:** ■ QS ■

**Light Path:** 1 mm

**Volume:** 280 µl

**Outer Dimensions:**

**Height:**

**Width:**

**Depth:** 3,5 mm

**Inner Dimensions:**

**Width:** 19 mm dia.

**Base Thickness:**

**Number of windows:** 2



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**Order Number** 404-1-46  
**Type** 404.000-QX

**Material Color Code:** ■ QX ■

**Light Path:** 1 mm

**Volume:** 700 µl

**Outer Dimensions:**

**Height:** 47,5 mm

**Width:** 23,6 mm

**Depth:** 3,5 mm

**Inner Dimensions:**

**Width:** 18,5 mm

**Base Thickness:** 2,5 mm

**Number of windows:** 2



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**Height: 38 mm**  
**Width: 18.5 mm**

### Disadvantages:

- sometimes hard to clean properly
- Lost of cells through breakage
- Significant cost > \$300 each



## Possible choice for Narrow Slits... → 1.5 mm wide x 75 mm tall: Also quartz capillaries or custom cells

<b>Order Number</b>	<b>110-1-40</b>
<b>Type</b>	<b>110-QS</b>

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Material Color Code: **■ QS ■**

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Light Path: 1 mm

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Volume: 350 µl

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**Outer Dimensions:**

Height: 52 mm  
Width: 12,5 mm  
Depth: 3,5 mm

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**Inner Dimensions:**

Width: 9,5 mm


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Base Thickness: 1,5 mm

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Number of windows: 2

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**Height: 37 mm**  
**Width: 9.5 mm**

<b>Order Number</b>	<b>100-1-40</b>
<b>Type</b>	<b>100-QS</b>

---

Material Color Code: **■ QS ■**

---

Light Path: 1 mm

---

Volume: 350 µl

---

**Outer Dimensions:**

Height: 45 mm  
Width: 12,5 mm  
Depth: 3,5 mm

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**Inner Dimensions:**

Width: 9,5 mm


---

Base Thickness: 1,5 mm

---

Number of windows: 2

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**Height: 37 mm**  
**Width: 9.5 mm**

### Existing Ti-Cell Design:

19 mm dia. Fill volume

25 mm dia. x 1.6 mm a-Quartz windows



### Enlarged Ti-Cell Design → Circular Apertures

32 mm dia. Fill volume

38 mm dia. x 1.6 mm a-Quartz windows

### Other Design Changes:

- Thin body by 2.5 mm
- Retaining plates from **Aluminum**
- Increase max. scat. angle from 25° to **30°**
- Bolts from #8 Low-heads to **#10 Buttonhead**
- Fill Port from #10 to **1/4"-20**

**{ Drawing Markups Complete }**

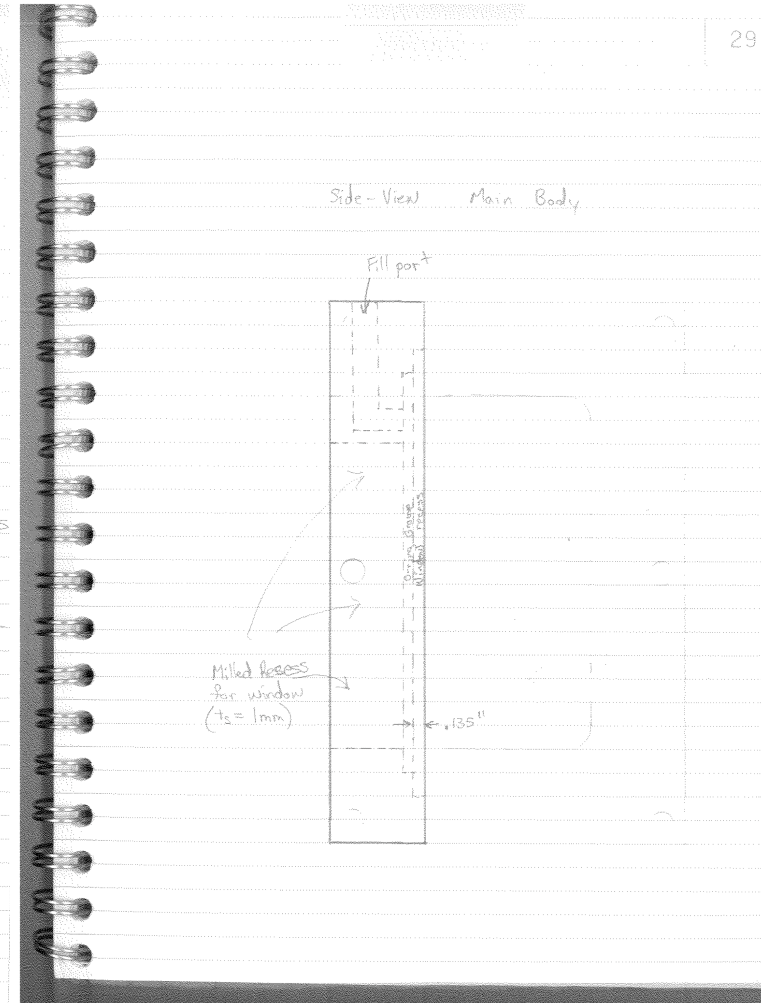
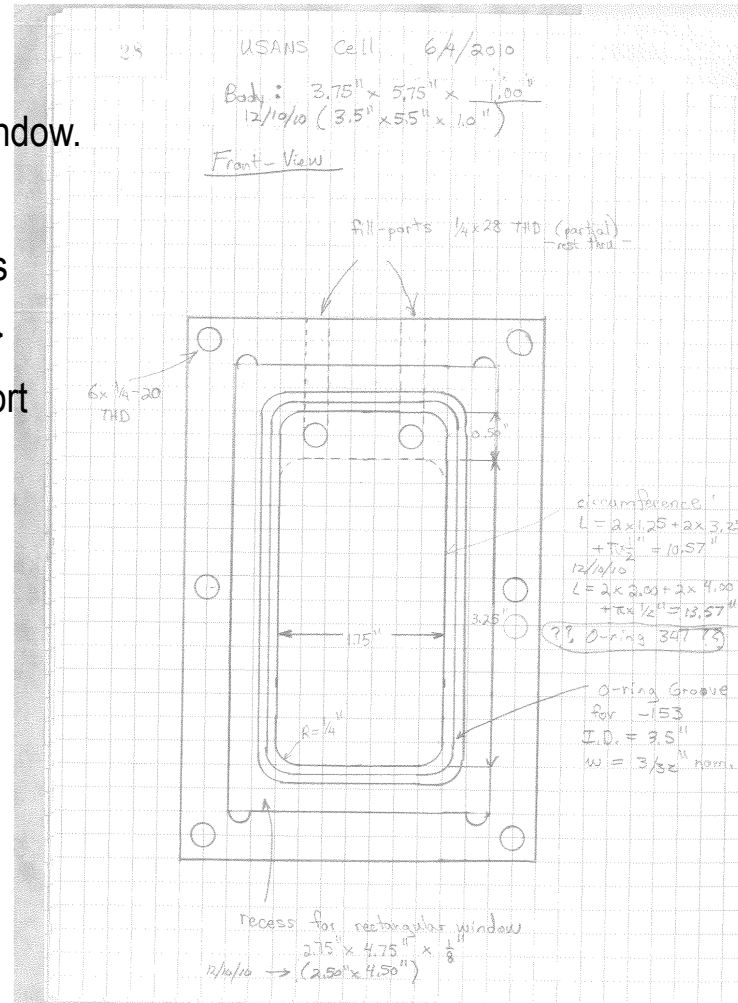
## New Rectangular Style "Large" Ti-Cell

44 mm wide x 95 mm tall fill volume

Window: a-Quartz 70 mm x 120 mm x 3.2 mm

### Notes:

- Grade 5 Ti for low Bgd Window.
- One rectangular window.
- One O-ring sealed to glass  
{ Eliminates excess fill gap }
- **Offsetted** Fill + **Exhaust** Port



MAX	MIN
280	260
212	226
	240

$.248 > .251$  FALSE

4201 BARNES, J. L. 1986.

TRUE

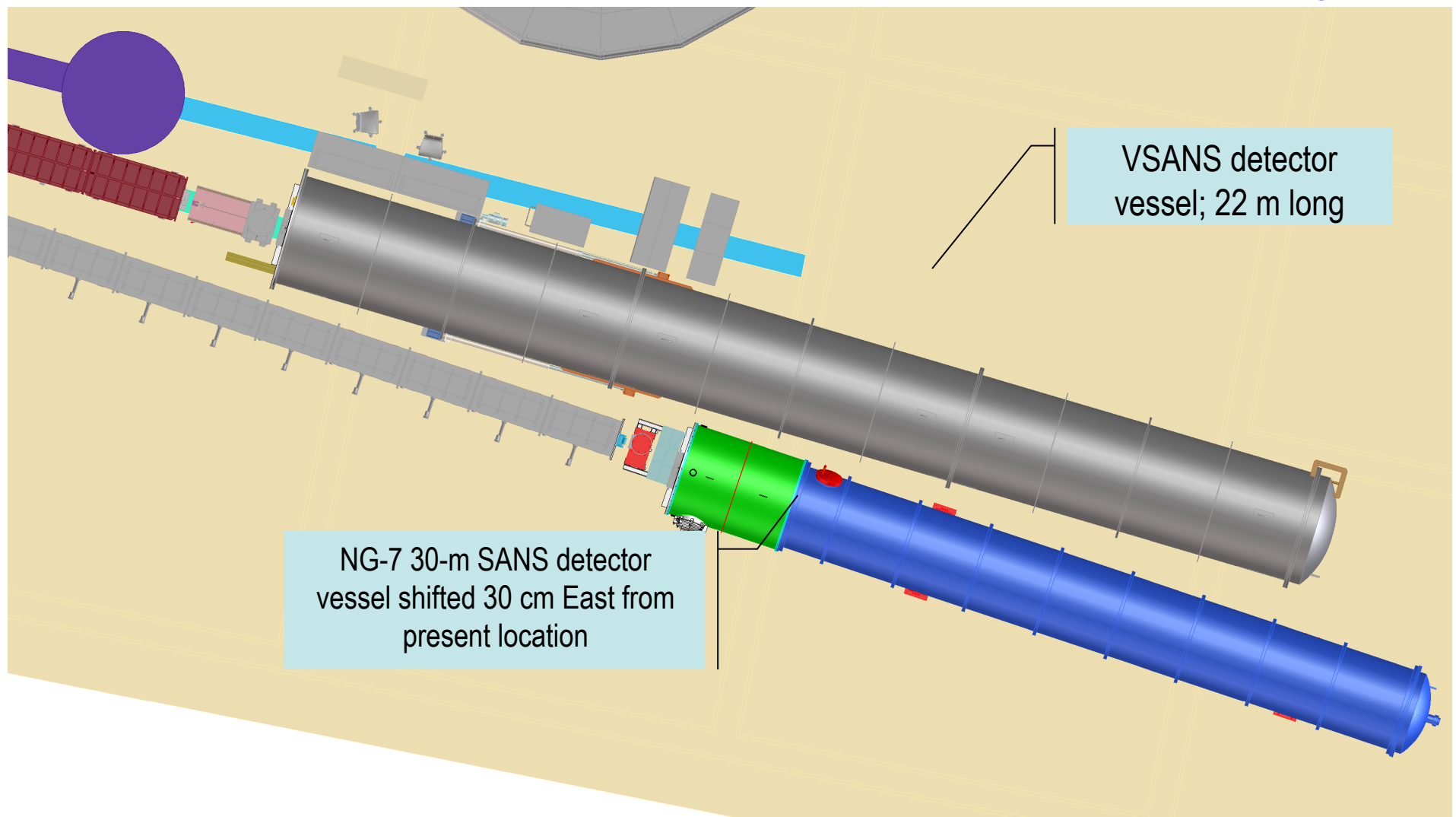
[illegible]

Sample Area space: Center of Beam to Obstruction:

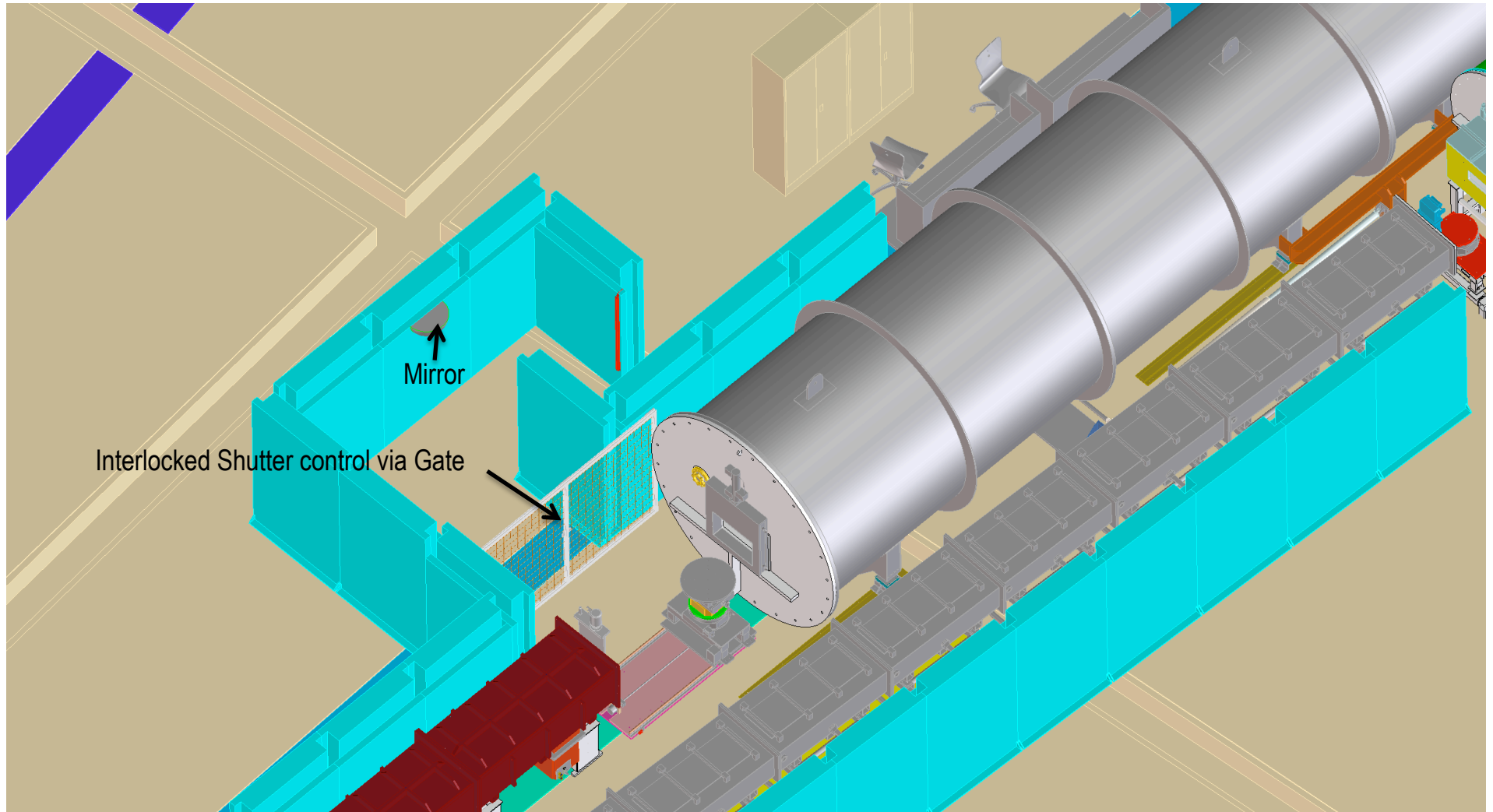
vSANS: 55"

NG7-SANS: 32"

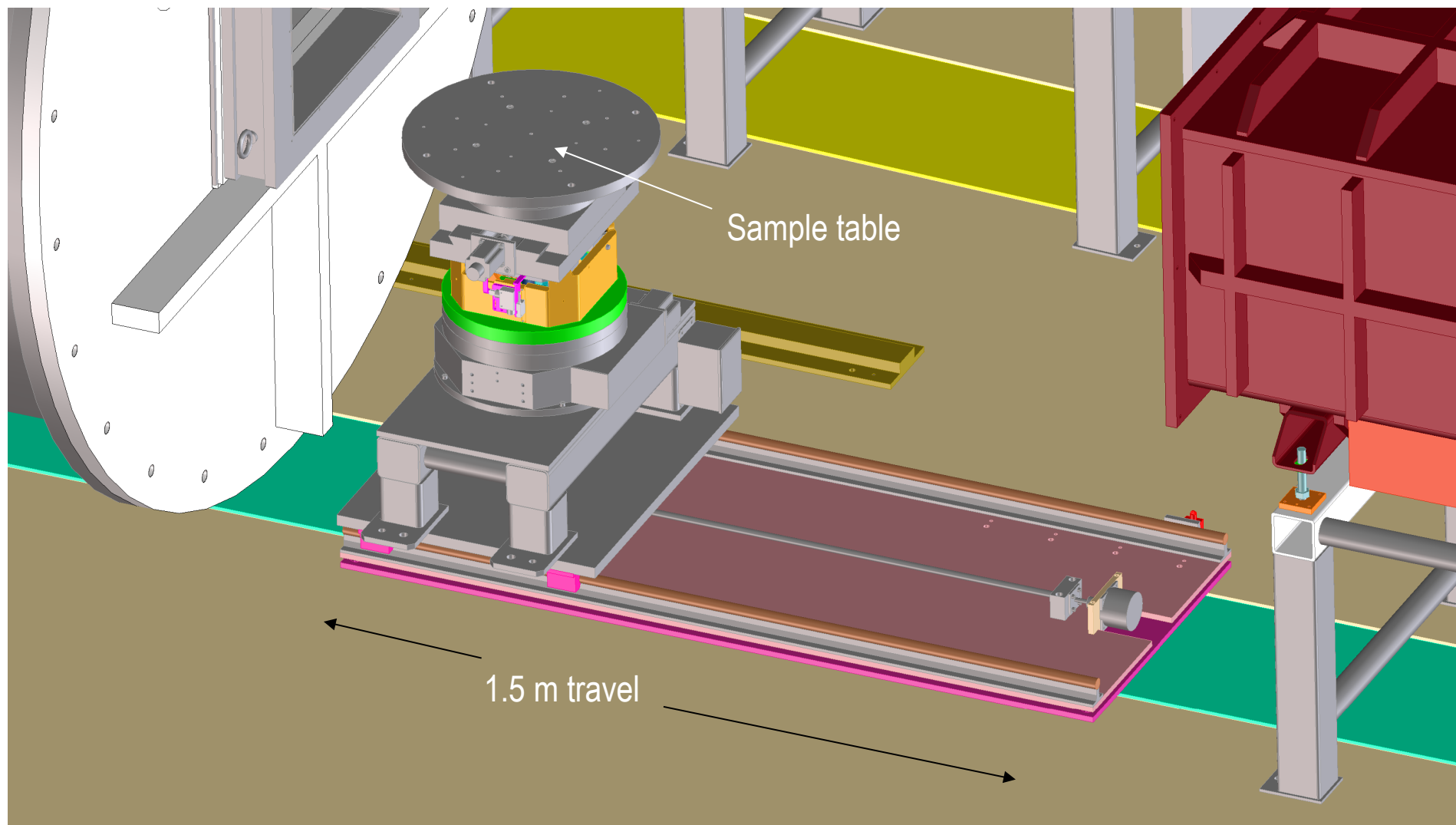
vSANS **Velmex slide: 60" dovetail, 48" travel (2.5x current 30m instr. range)**



Possibility of higher beam current (40 to 500x) and ability to walk thru beam requires:  
1) Wall shields to maintain Low radiation areas + ALARA      2) Interlock shutter

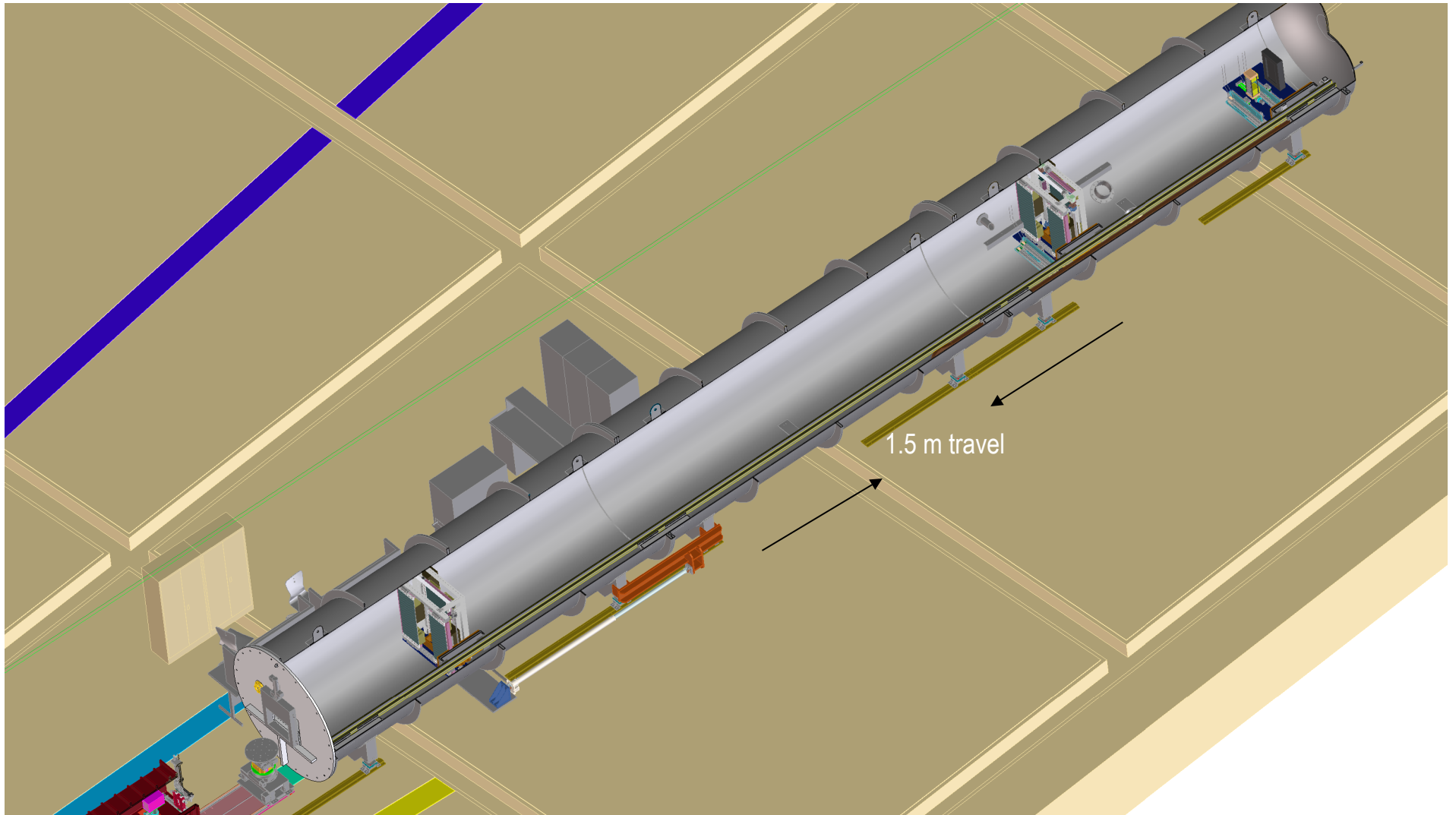


New vSANS sample Area → No Sample Chamber





Cutaway view of detector vessel showing three movable detector carriages





Possible design changes for new **Heating/Cooling Blocks**:  
( Choice of materials, plumbing, insulation, size ... )

### **SANS Thermostat Sample Holder**

Ancillary code: SC-1

**Used on:**

Quokka (SANS)

**Quokka (ANSTO) Cooling Block Designs  
( similar to NCNR's blocks... massive )**

**10 sample positions**

Based on a design at the NIST neutron scattering facility in the USA this 10 position sample changer operates between 20C and 300C.



### **Sample Changer - SANS (twenty position)**

Ancillary code: SC-2

**Used on:**

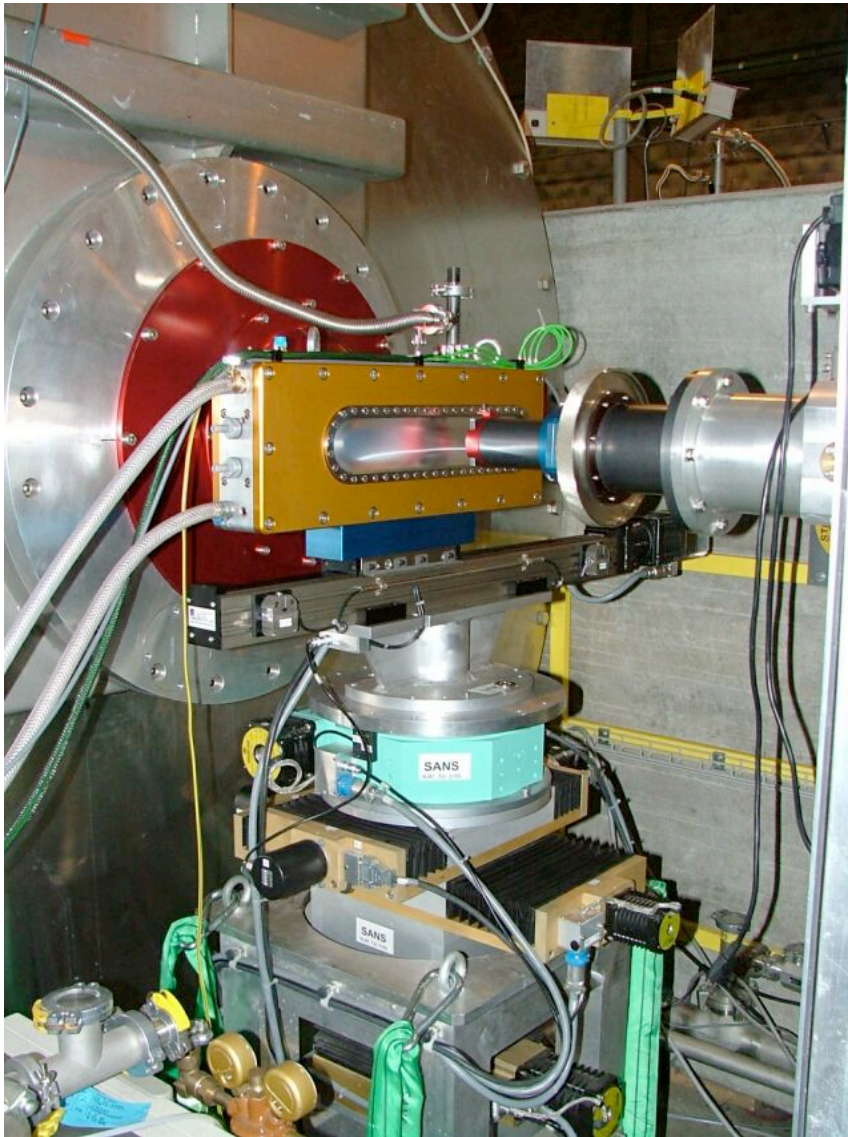
Quokka (SANS)

**20 sample positions**

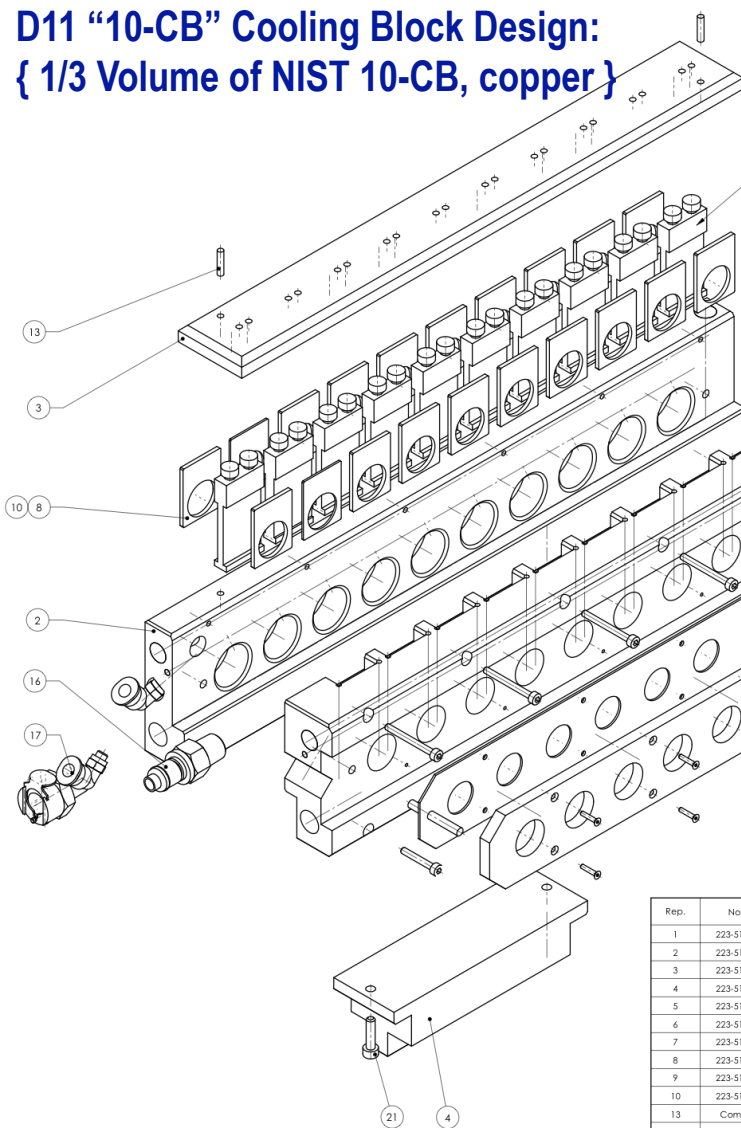
Based on a design at the NIST neutron scattering facility in the USA this 20 position sample changer operates between -10C and 80C.



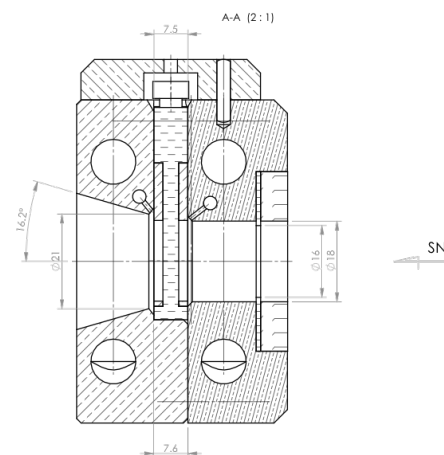
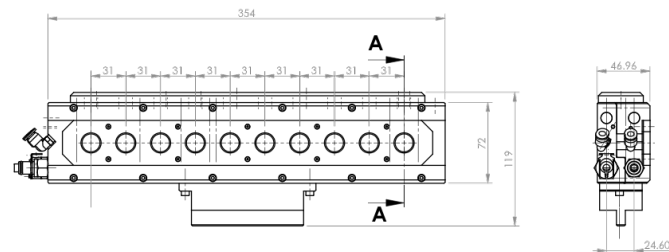
PSI 10-CB: Vacuum (**Thermos**) Insulation, samples **bolted** to copper sink, small **thermal mass**.



# D11 "10-CB" Cooling Block Design: { 1/3 Volume of NIST 10-CB, copper }



Porte echantillon type 404 QS



Rep.	No. Plan	Ind.	Désignation	Matériau	Qté	Fournisseur	Observations
1	223-51-03-001	B	Support échantillon demi bloc	Cuivre	1		
2	223-51-03-002	B	Support échantillon demi bloc	Cuivre	1		
3	223-51-03-003	B	Chapeau	Cuivre	1		
4	223-51-03-004	B	Support inf.	EN AW-5083	1		
5	223-51-03-005	B	Plaque calibrage cadmium	Cadmium	1		
6	223-51-03-006	B	Plaque B4C	B4C souple	1		
7	223-51-03-007	B	Plaque cuivre 34.9x1.98	Cuivre	20		
8	223-51-03-008	B	Plaque cuivre 31.9x1.98	Cuivre	20		
9	223-51-03-010	B	Plaque cuivre 34.9x0.98	Cuivre	20		
10	223-51-03-011	B	Plaque cuivre 31.9x0.98	Cuivre	20		
13	Commerce		Goupille cylindrique d=3-14	Acier	2		
14	Commerce		Goupille cylindrique d=4-32	Acier	2		
15	Commerce		Raccord coudé M5 QSM LMS-6	Matériau «non spécifié»	2	FESTO	
16	Commerce		Connecteur mâle	Matériau «non spécifié»	1	CPC	Ref. PLCD 24004
17	Commerce		Connecteur femelle	Matériau «non spécifié»	1	CPC	Ref. PLCD 10004
18	Viserie		Vis Chc M2-10	Inox	10		
19	Viserie		Vis Chc M3-20	Inox	6		
20	Viserie		Vis Chc M3-30	Inox	6		
21	Viserie		Vis Chc M4-16	Inox	2		

B	01/09/04	CA/SEI		P LINDNER	PLAN REFAIT
A	09/2003	MIGUET			Edition du plan
Ind.	Date	Dessinateur	Vérificateur	Resp. technique	Modifications
Distances générales: Linéaires: x Angulaires: x					
Echelle: 1:1					
Format: A1					
N° de plan 223-51-03-000					
Page: 1/1 Indice: B					

CANAL H15 - D11  
NOUVEAU CHANGEUR ECHANTILLON  
SUPPORT ECHANTILLON  
Ensemble support échantillon type 404 QS

INSTITUT LAUE LANGEVIN  
6, rue Jules Horowitz - BP156  
Grenoble Cedex 9 - France



## Material Choices for Block, cells and windows

Material	Density	Heat Capacity	Thermal Conduc.	Thermal Diffus.	Corrosion Resist.	Activation Resist.	Scattering Bgd
Copper	8.94 g/cm <sup>3</sup>	3.4 J/cm <sup>3</sup> /K	3.94 J/cm/s/K	1.16 cm <sup>2</sup> /s	Poor	Medium	Medium
Aluminum (pure)	2.70	2.43	2.22	0.91	Fair	Excel.	Low
Aluminum (6061)	2.70	2.43	1.67	0.69	Fair	Excel.	High
Titanium Grade 2	4.51	2.35	0.16	0.068	Excel.	Excel.	Medium
Titanium Grade 5	4.44	2.35	.067	0.029	Excel.	Excel.	Low
Stainless Steel 304	8.00	4.0	0.16	0.04	Excel.	Medium	High
Silicon	2.33	1.65	1.49	0.90	Excel.	Excel.	v. Low
a.Quartz	2.20	1.65	0.014	0.0085	Excel.	Excel.	~Low

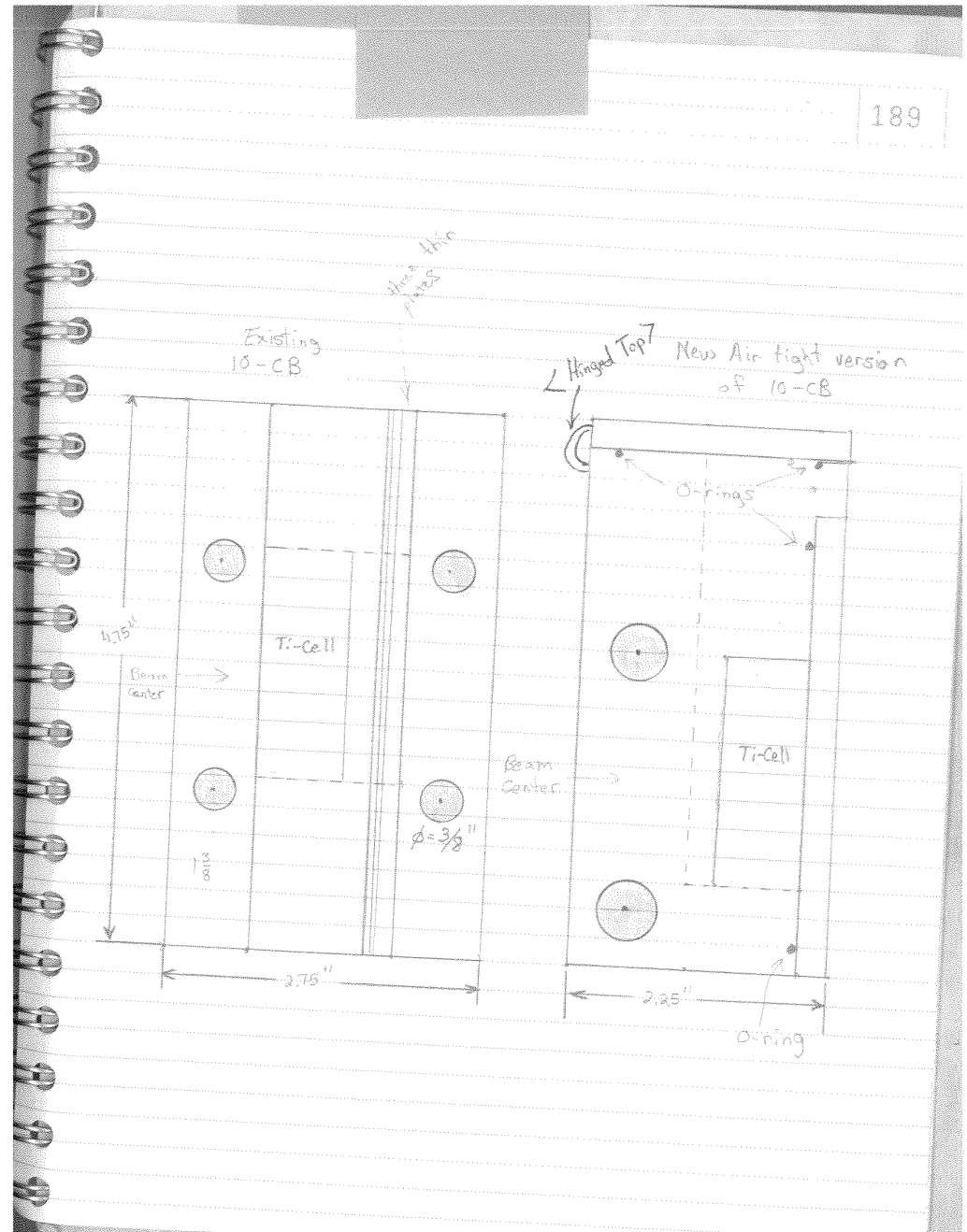
**Block:** → Aluminum 6061 (low weight, easily machined, low activation)

**Sample Cell Bodies** → Titanium Grade 2 (Corrosion resistance, easily machined)

**Windows** → a-Quartz, Silicon, Titanium Grade 5 (low Bgd, low cost)

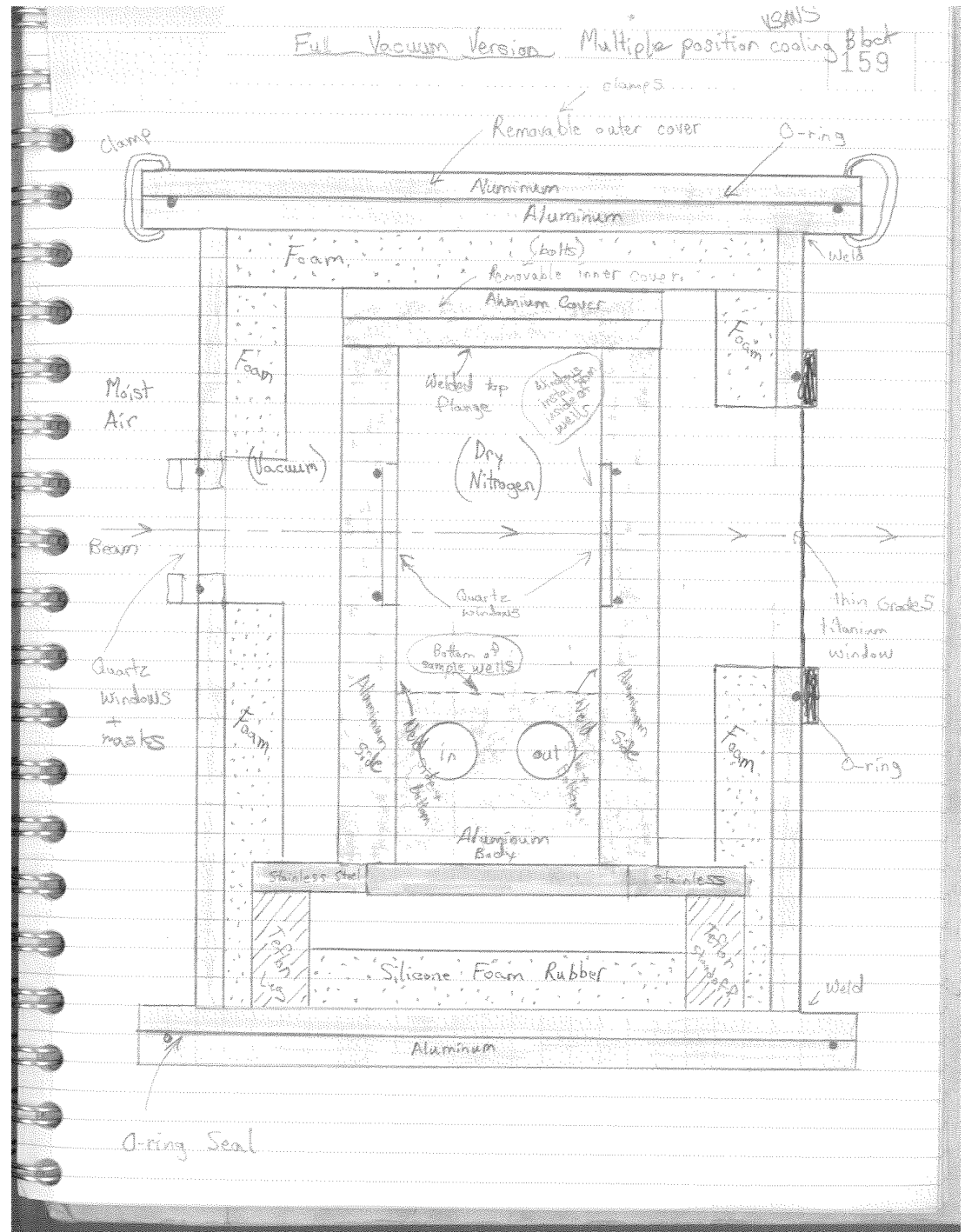
### Suggested changes over current 10-CB

- Two vs. Four flow channels
- Reduce block volume (thermal mass..)
- Increase scat. Angle from  $30^\circ$  to  $45^\circ$ .
- Vacuum tight design (thermos).



## Comments:

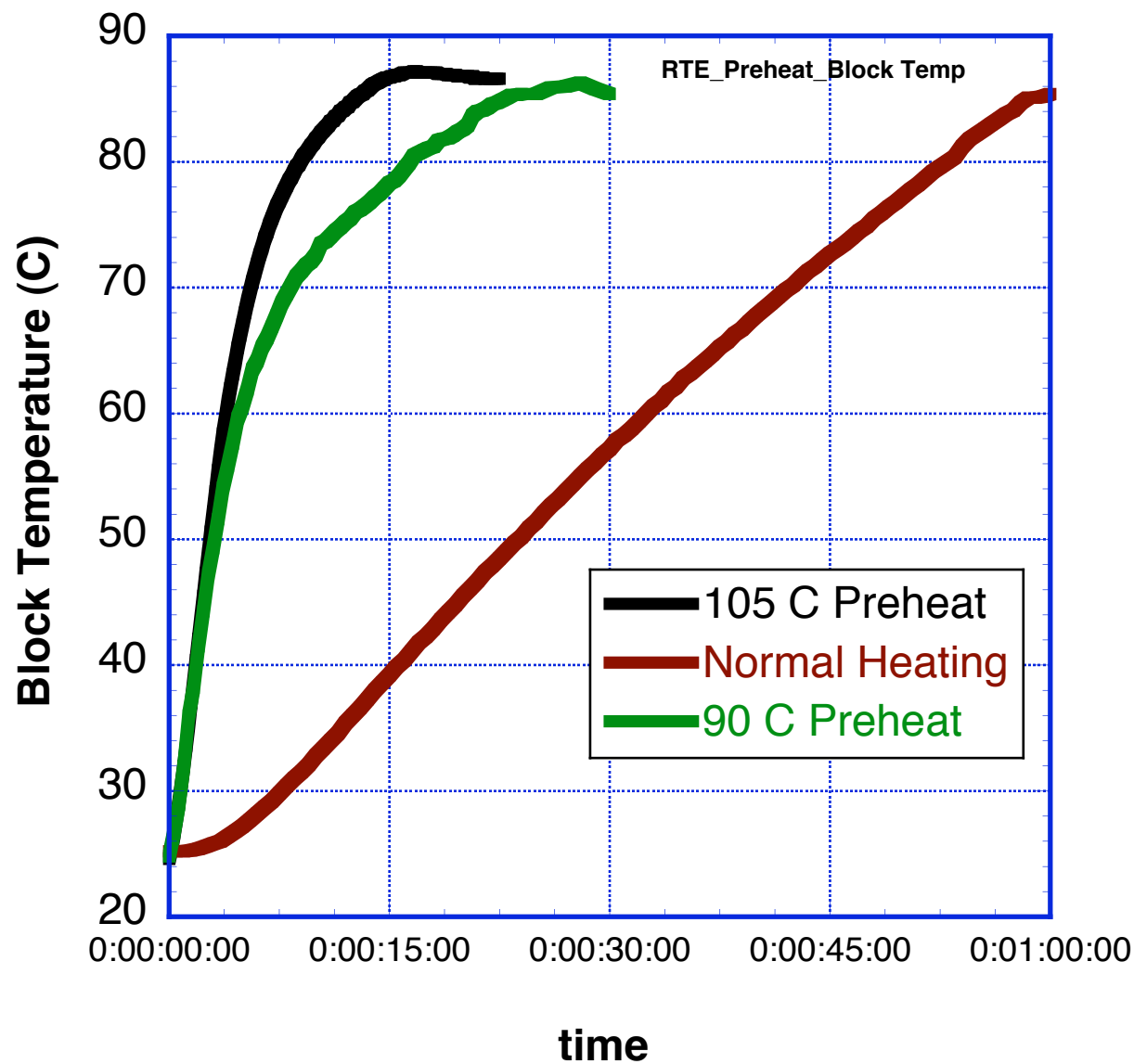
- Sample (inner block) in **dry Nitrogen**
- Outer under Vacuum (**Thermos**)
- Quartz and Ti (grade 5) Windows.
- Only Two-cooling bath channels in base.



Heating of 10-CB from 25 C° to 90 C° with Neslab RTE-7  
{ Normal ramp, bath preheated to 90 C° and 105 C° }

**Comments:**

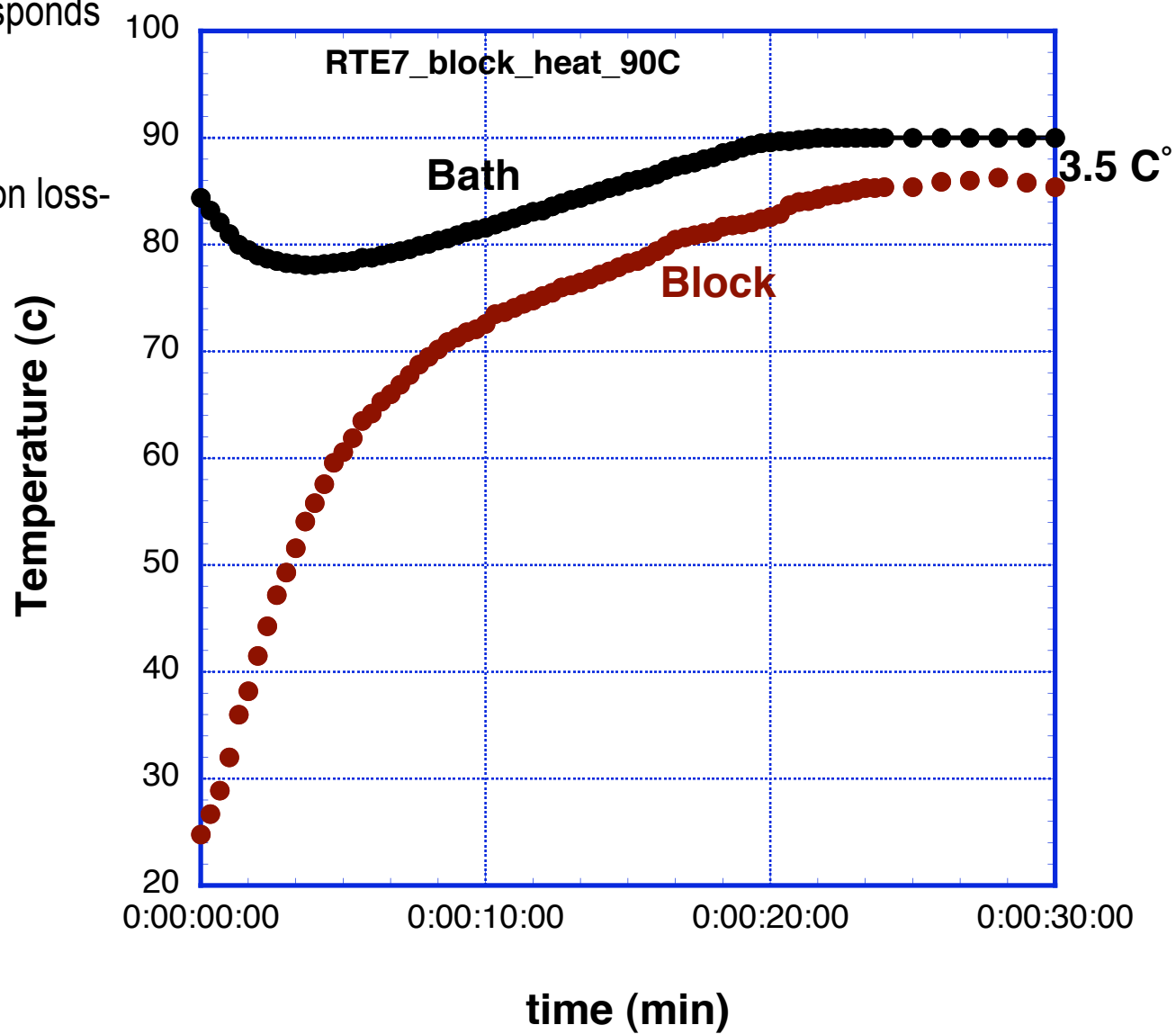
- Slow bath heat rate
- Large T undershoot (-3.5 C°)
- Julabo Bath is much faster!



Temperature Offset Corresponds  
to **2.5 Ltr/min** pump rate.

Cooling rate with pump off

Yields **-100 Watt** convection loss-  
rate

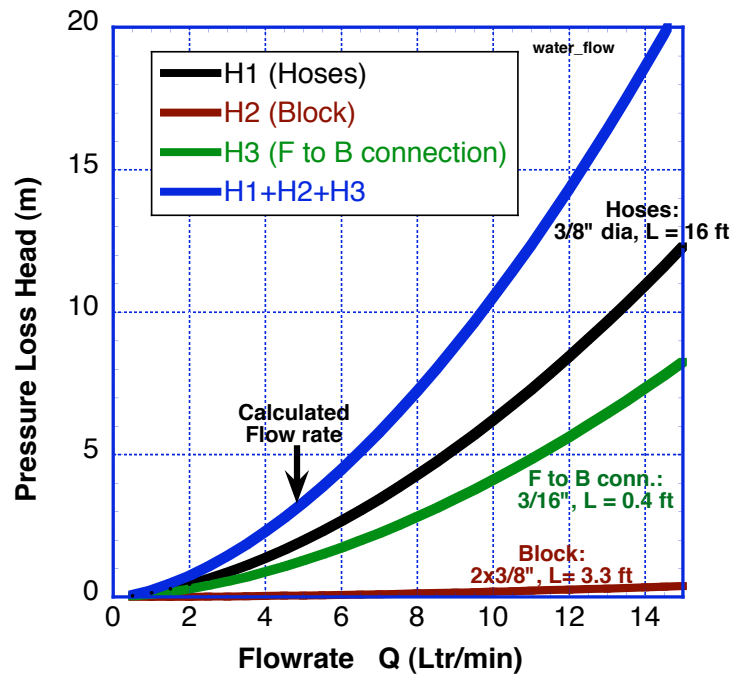




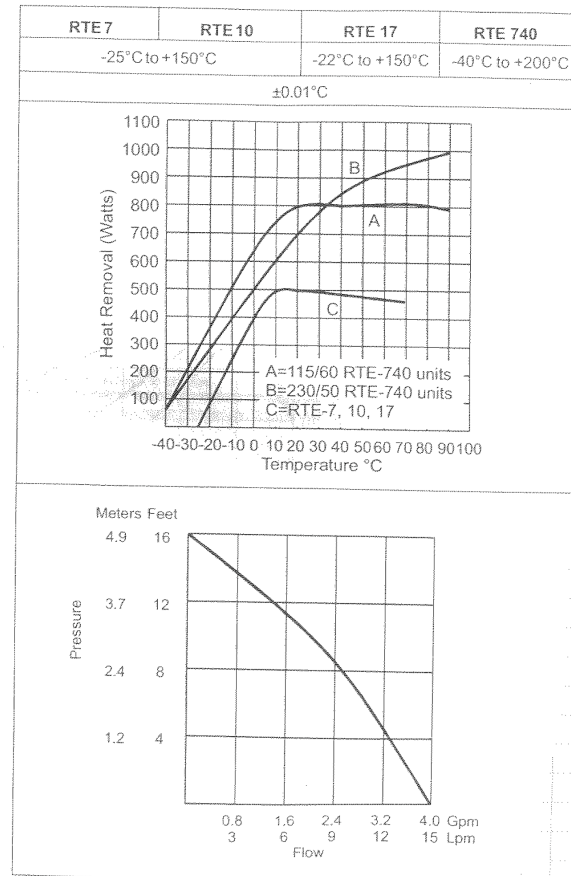
## RTE-7 Bath Flowrate:

Calculated: **5 Ltr/min**

Measured from Temperature Curves: **2.5 Ltr/min**

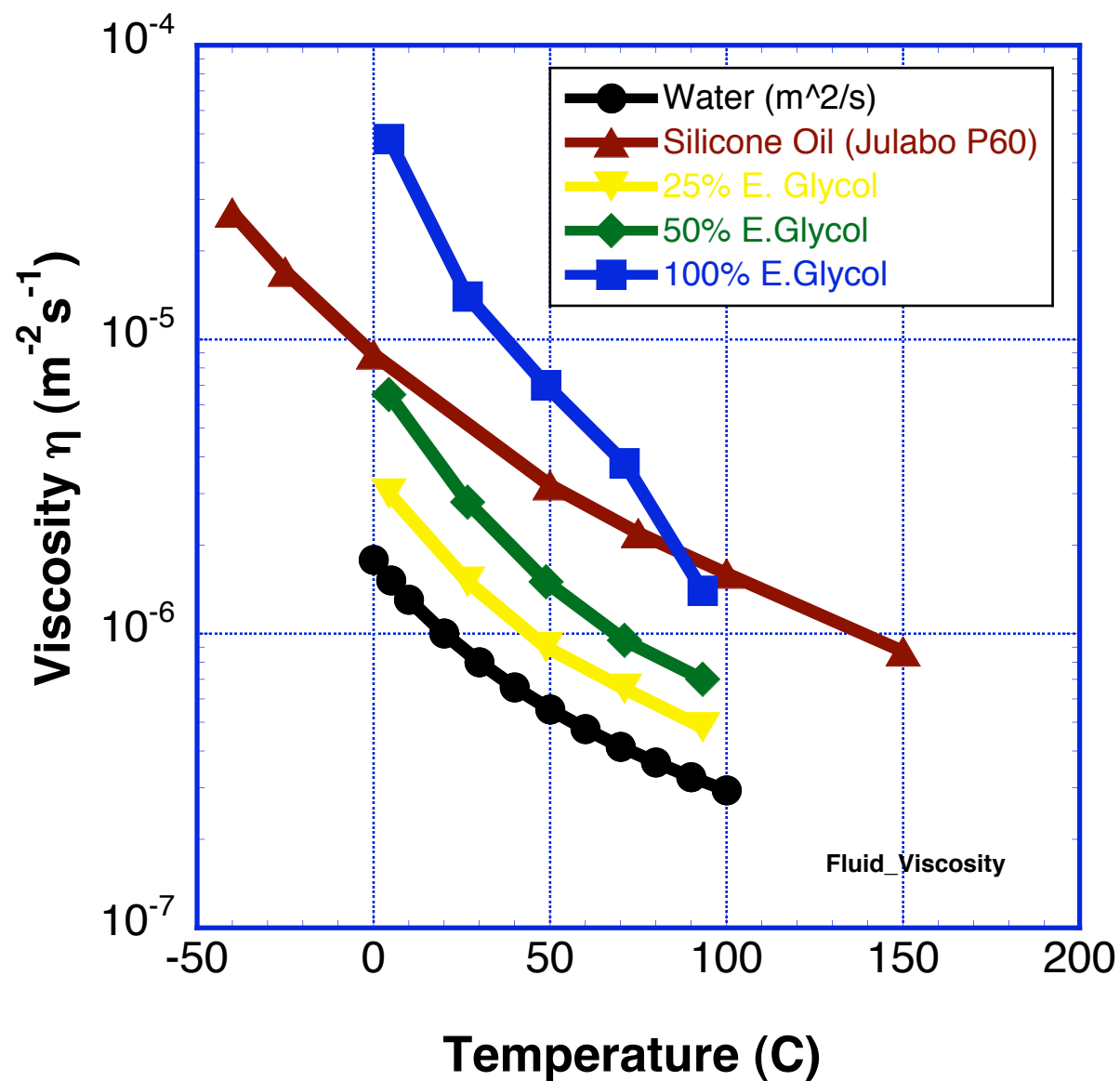


$$h_L = 2C_f \frac{L}{D} \frac{V_x^2}{g}$$



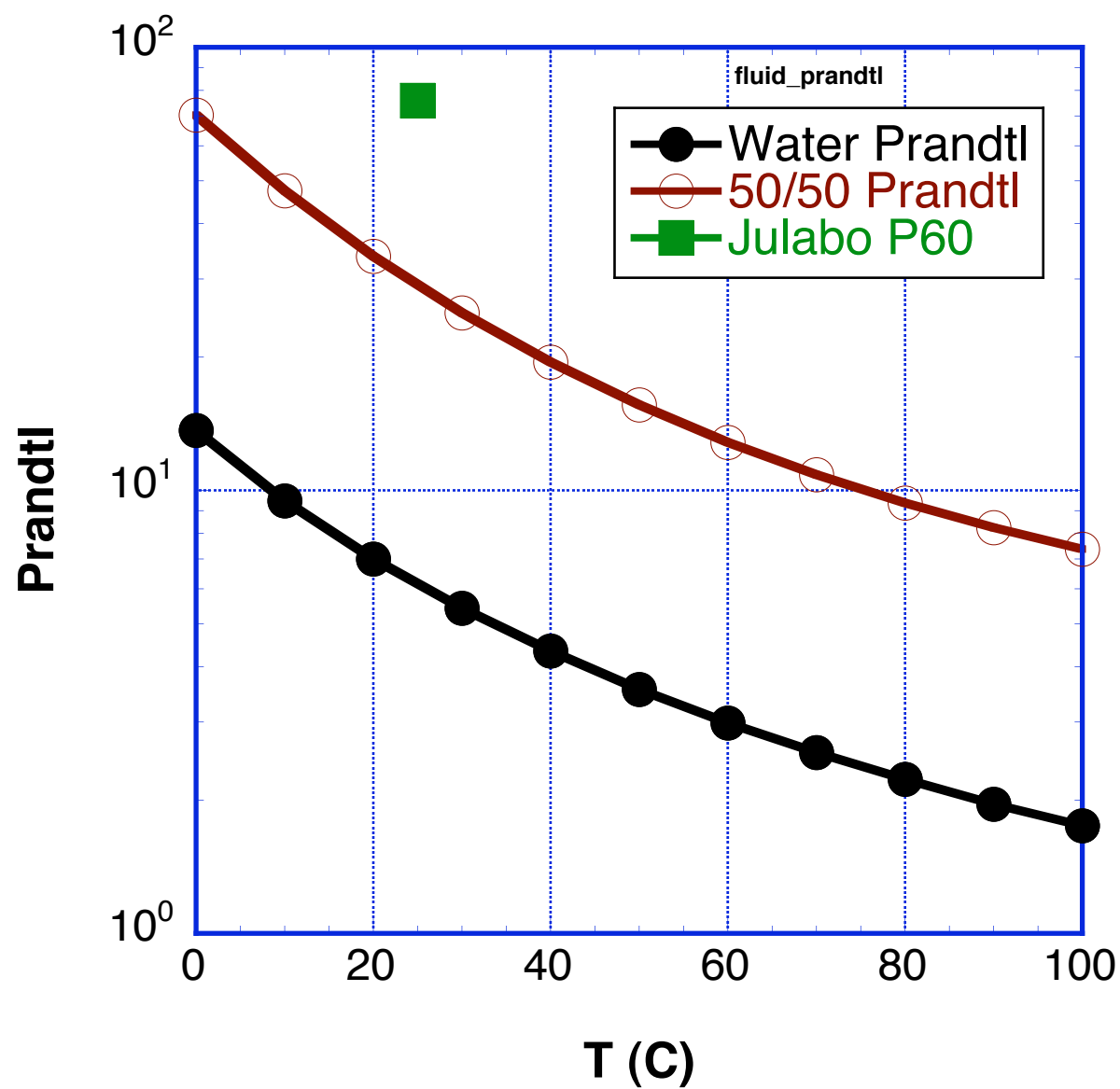
$$v_x = \frac{dS/dt}{\frac{\pi}{4} D^2}$$

FLUID	HEAT CAPACITY
Water	4.2 J/ cm <sup>3</sup> /K
50% Water 50% E. Glycol	3.2
100% E. Glycol	2.2
Silicone Oil	1.6



Prandtl Number for various Heat Transfer (bath) fluids

$$P_r \equiv \frac{\nu C_P}{k}$$



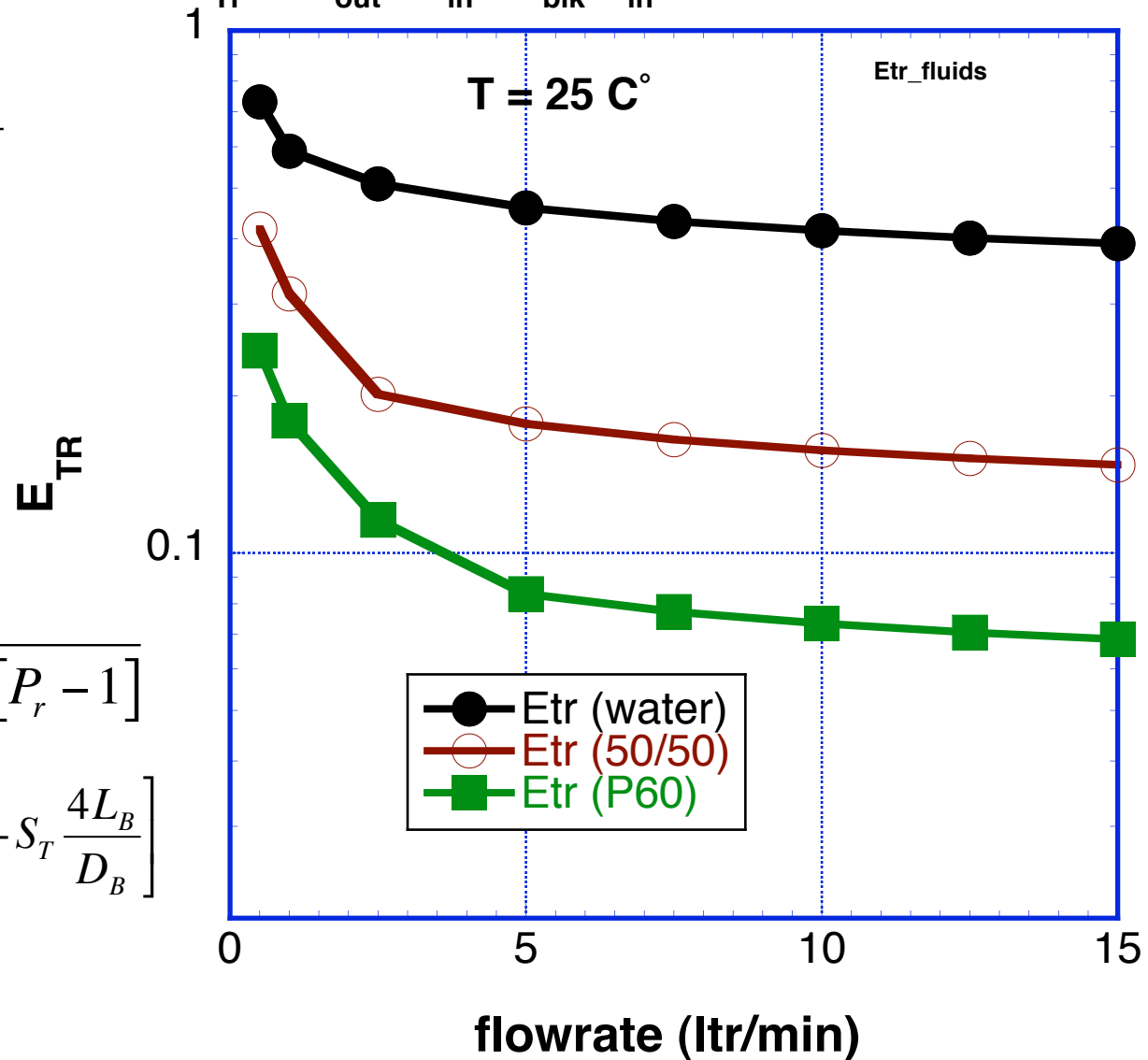
$$R_e = \frac{v_x D}{\nu}$$

$$C_F \cong \frac{0.331}{\left[ \ln \left( \frac{5.74}{R_e^{0.9}} \right) \right]^2}$$

$$S_T = \frac{C_F/2}{1 + 5\sqrt{C_F/2}[P_r - 1]}$$

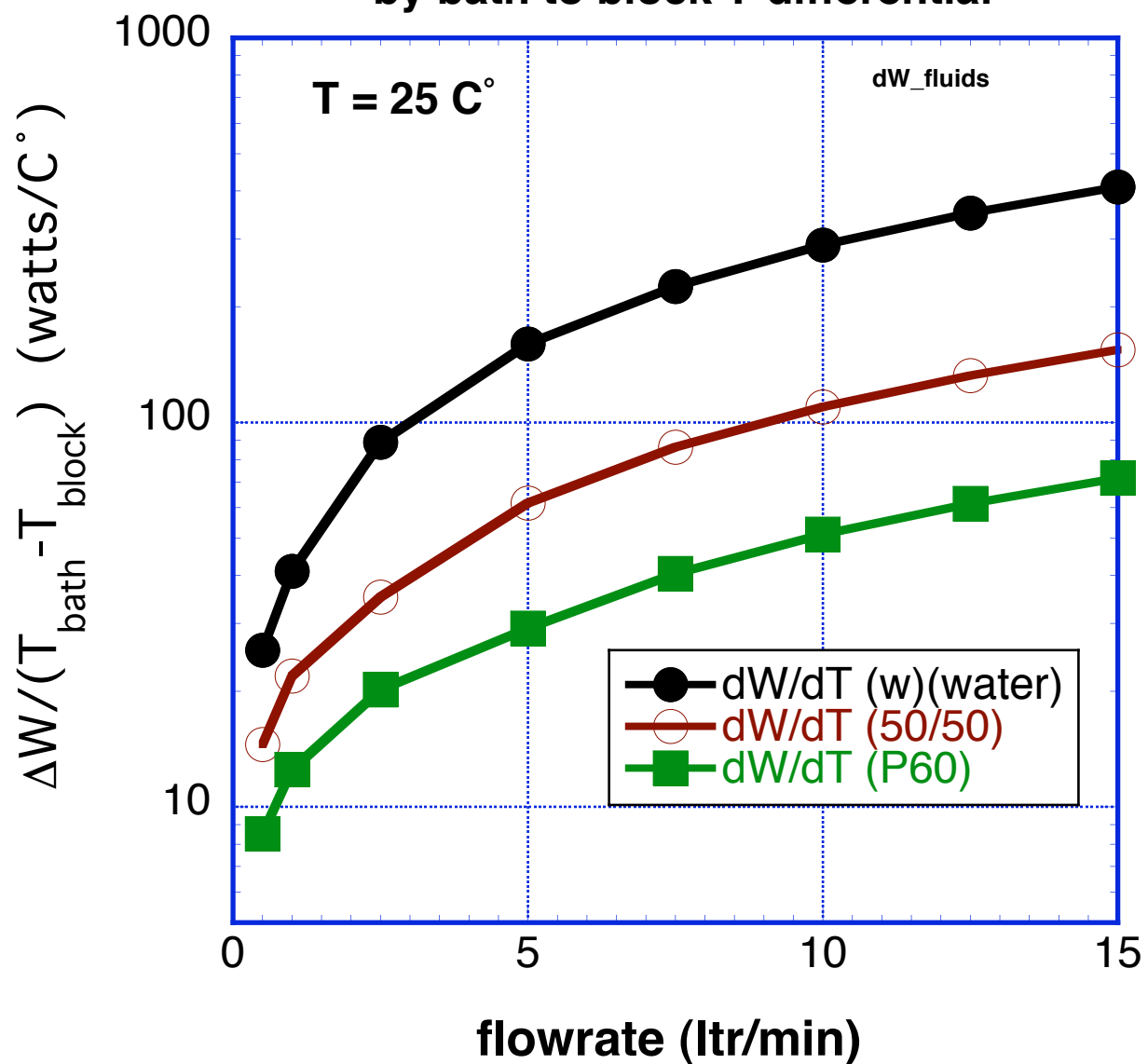
$$E_{TR} \equiv \frac{T_L - T_S}{T_O - T_S} = \exp \left[ -S_T \frac{4L_B}{D_B} \right]$$

Efficiency of Heat Transfer from Bath to block:  
 $E_{Tr} = (T_{out} - T_{in}) / (T_{blk} - T_{in})$  using Prandtl Analogy...



$$\Delta w = \frac{dS}{dt} C_{P,fluid} (T_{bath} - T_{block}) E_{TR}$$

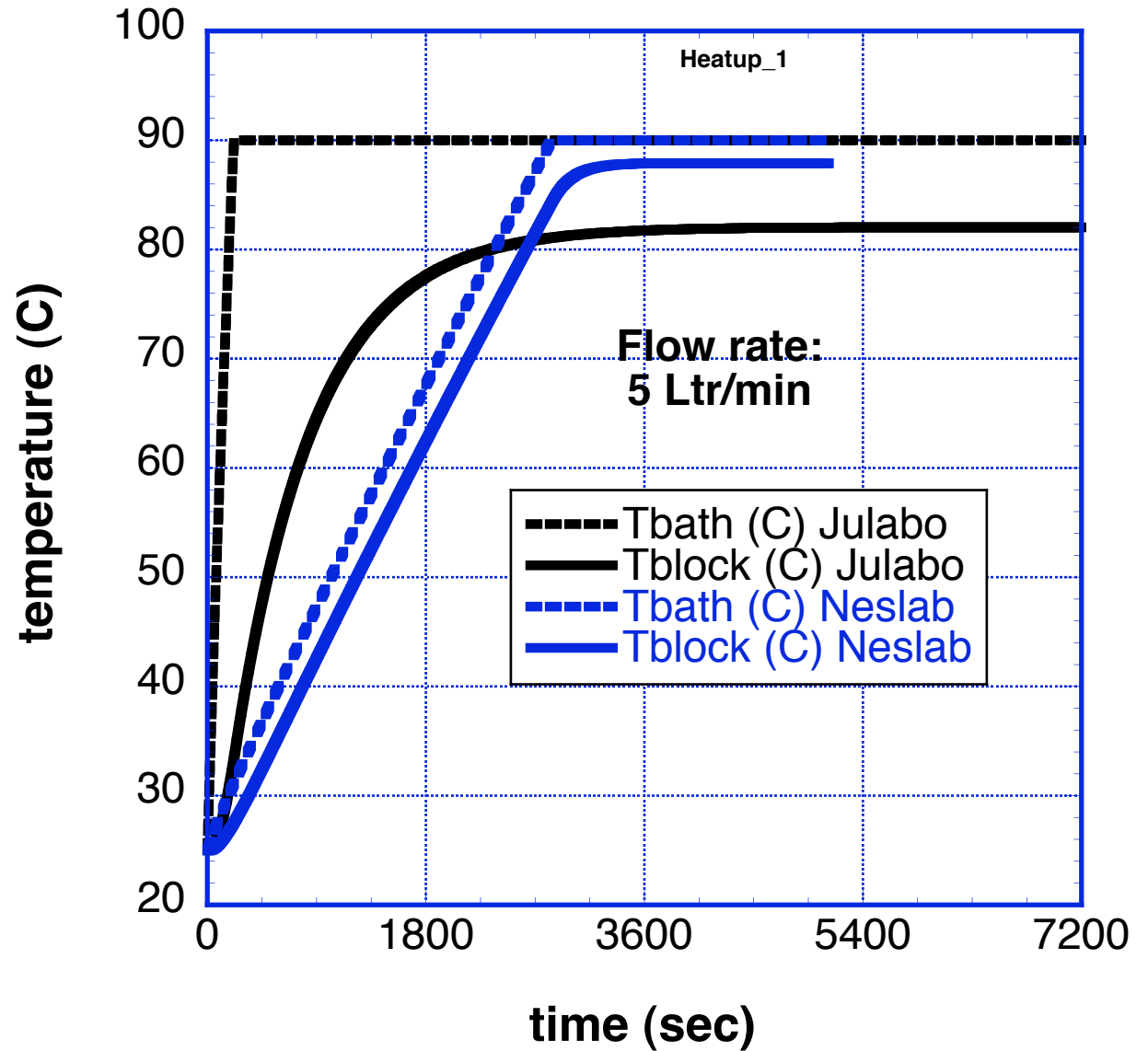
Heat Transfer Rate normalized  
by bath to block T-differential

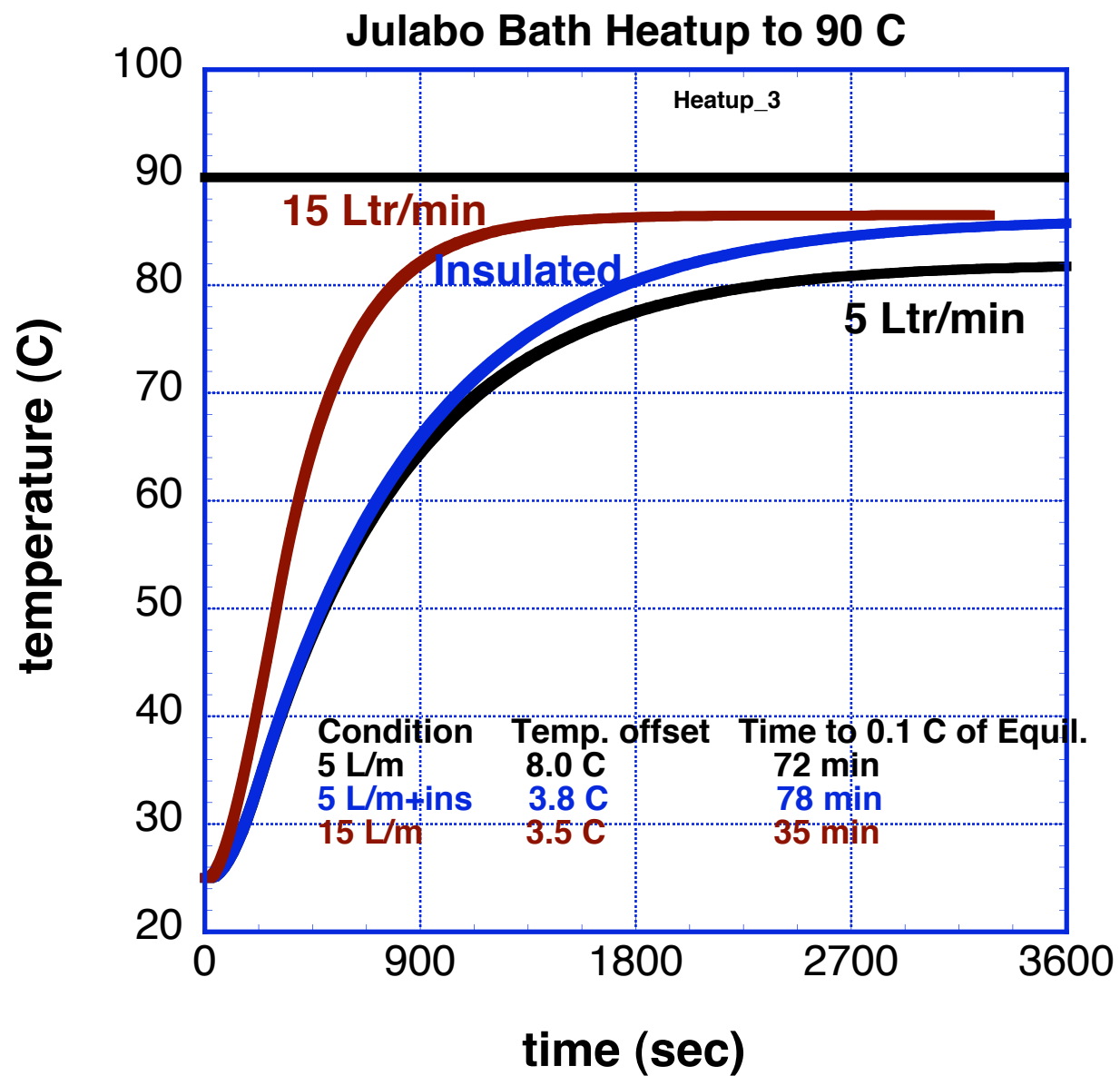


## Bath Specs Comparison

Spec.	Neslab RTE-7	Julabo LH45	Huber "Tango"
Bath Volume	7.2 Ltr	2.5 Ltr	2 Ltr
Pump (max) pressure	0.5 Bar	1.6 Bar	0.9 Bar
<b>Max. Pump rate</b>	<b>15 Ltr/min</b>	<b>24-33 Ltr/min</b>	<b>33 Ltr/min</b>
Fluid	Water+ E. Glycol	Silicone Oil	Silicone Oil
Heat Capacity (T=25 C)	3.24 J/cm <sup>3</sup> /K	1.63 J/cm <sup>3</sup> /K	1.63 J/cm <sup>3</sup> /K
Heater	800 Watts	1800 Watts	1500 Watts
Cooler (max)	500 Watts	1200 Watts	450 Watts
<b>Heating rate (pump+10 CB)</b>	<b>1.4 C°/min</b>	<b>8.0 C°/min</b>	<b>6.9 C°/min</b>
Cooling rate (pump+10 CB)	-0.9 C°/min	-5.3 C°/min	-2.1 C°/min
Temp Range	-25 C° to 150 C°	<b>-40 to 250 C</b>	<b>-40 to 200 C</b>

Item	Julabo	Neslab
Undershoot (C°)	8.0 C	2.1 C
Time to 0.1 C eq.	72 min	57 min







### New Cooling Block Design Choices:

- 1) Two blocks+baths in one allows one block to be at Temperature while other equilibrates (“**Leap Frog**”). Will require ICE to command two separate baths.
- 2) **Insulate** with **Silicone foam** insulation + outer shell. Reduces temperature gradients and temperature undershoot by **factor of two**, and eliminates condensation.
- 3) **Insulate** with **vacuum**. Removes condensation and reduces gradients and undershooting by **factor > 20x**. Inner sample block must be air/vacuum tight.
- 4) **Reduce** volume (**thermal mass**) of block. Reduces equilibration time and undershoot. (Similar to PSI or D11 design). May involve fewer sample positions.
- 5) Increase efficiency of heat transfer  $E_{TR}$  by using serial vs split-parallel flow...
- 6) Increase pump rate  $dS/dt$  by using larger ID tubing and fittings.

## Priority List and Budget for new Ti-Cell Sizes

	Description	Cost
2011	1) Velmex Slide: 48" travel	\$3,189
	2) 32 mm diameter fill cells (Qty: 50 x 1mm, 50 x 2 mm, 30 x 4 mm)	\$13,000
	3) 5-position room temperature holder for BT-5 USANS	\$2,000
	4) 20-position room temperature holder for VSANS and 30m SANS	\$6,000
	5) Two 9-position cooling blocks ( $-25\text{ C} \leq T \leq 130\text{ C}$ ) for vSANS + 30m to be used in tandem.	\$10,000
	6) 4-position cooling block ( $-25\text{ C} \leq T \leq 130\text{ C}$ ) for BT-5 USANS	\$4,000
2012	7) 44 mm x 95 mm Ti Cells for 18 conv. Beams on vSANS (Qty: 30x 1mm, 30 x 2 mm, 20 x 4 mm)	\$16,000
	8) 12-position room temperature holder for 18 beams on vSANS	\$6,000
	9) Two 5-position cooling block for 18 beams on vSANS	\$10,000
	<b>Total</b>	<b>\$70,189</b>