Workshop on Measurement and Analysis of Thermochemical and Thermophysical Properties of Molten Salts

Calorimetric Methods for Heat Capacity Determination

Dr. Juliano Schorne-Pinto

Research Professor

Nuclear Engineering Program, Mechanical Engineering Department,
University of South Carolina

Molten Salt Reactor PROGRAM

General Atomics Center
College of Engineering and Computing
UNIVERSITY OF SOUTH CAROLINA

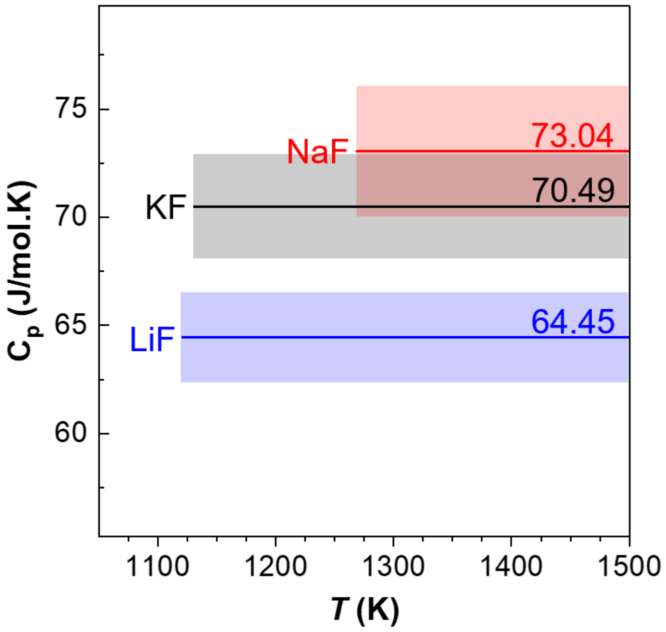
NEAMS
Nuclear Energy Advanced Modeling and Simulation

NEUP Nuclear Energy University Program
U.S. Department of Energy

Outline

- Why Heat Capacity is Important?
- A Bit of History of the MSRE
- Behavior of Heat Capacity for Molten Salts
- Measurement Instruments
  - Drop Calorimeters
  - Differential Scanning Calorimeters
- How to Carry Out Heat Capacity Measurements?
  - Methods and Factors that Affect the Measurements
  - Optimization of Experimental Conditions
  - Measurements, Data treatment and Errors
- Coupling Different Type of Measurements
- Example: Comparing FLiNaK Results with Literature
- Can We Get Closer to the Correct Value?
- Some Key References

Error in the heat capacity of alkali fluorides



* Assessed for the MSTDB-TC (internal work)

Why Heat Capacity is Important?

- Importance of accurate heat capacity:

Material Design
Used in the Calphad optimization to predict the thermal behavior of materials

Process
Optimization of industrial processes involving heat transfer

Energy Storage
Design and performance of energy storage systems

Safety
Heat capacity data helps ensure the safe and reliable operation of equipment and systems

- Heat capacity is the amount of heat needed to raise the temperature of the material by 1 K
  - Molar heat capacity (C_P) in J/mol.K
  - Specific heat capacity (c_P) in J/g.K
- Describes Gibbs energy temperature -dependence

$$\Delta G_i^{\circ}(T) = \Delta H_{298.15}^{\circ}(i) + \int_{298.15}^{T} \boldsymbol{C_{P_i}^{\circ}(T)}\, dT - T\left(S_{298.15}^{\circ}(i) + \int_{298.15}^{T} \frac{\boldsymbol{C_{P_i}^{\circ}(T)}}{T}\, dT\right)$$

A Bit of History

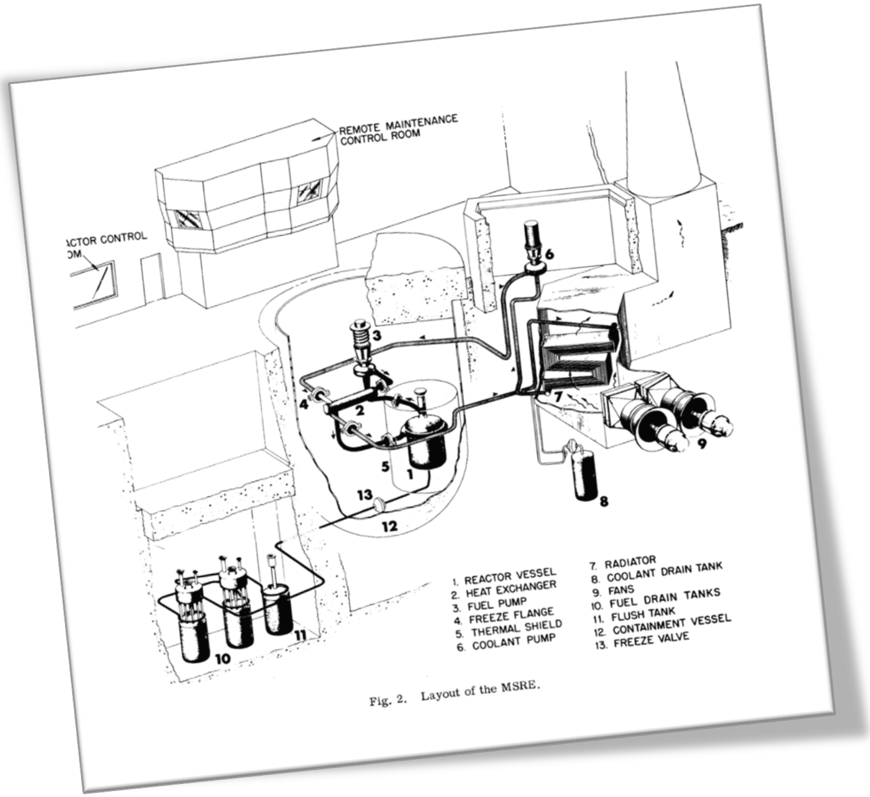
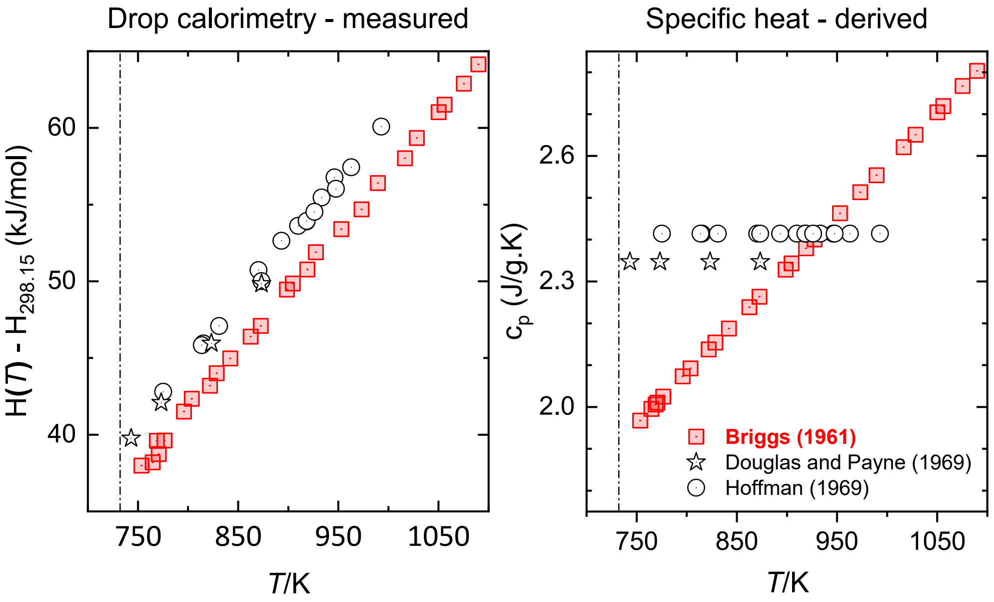


Fig. 2. Layout of the MSRE.

- **MSRE has not always been an 8-MW(th) reactor**

  Heat balance calculations were modified over the years resulting in an increase of 11% of its maximum power [7.2 to 8-MW(th)] because heat capacity was not well known

Specific heat of the coolant



Fuel: ^7LiF-BeF_2-ZrF_4-UF_4 (65.0-29.1-5.0-0.9 mole%)
Coolant salt: ^7LiF-BeF_2 (66-34 mole%)

Reactor operational data suggested that $c_p(T)$ is not temperature dependent

Haubenreich & Engel (1970)
Briggs (1961) – ORNL-3215
Gabbard (1968) – ORNL-MSR-68-80

Behavior of Heat Capacity for Molten Salts

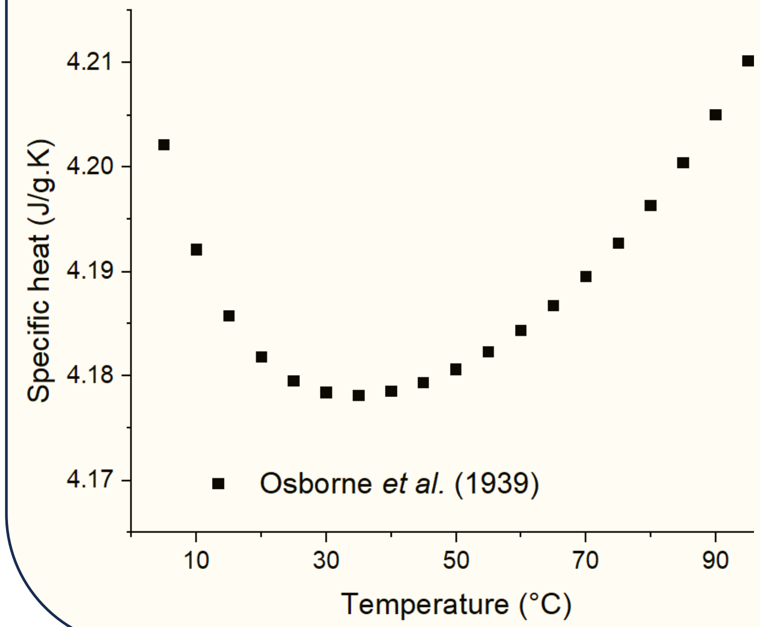
- The heat capacity can be mathematically approximated

$$C_P(T) = \left(\frac{dH}{dT}\right)_P = a_0 + a_1 T + a_2 T^{-2} \dots$$ *Maier-Kelley polynomial*

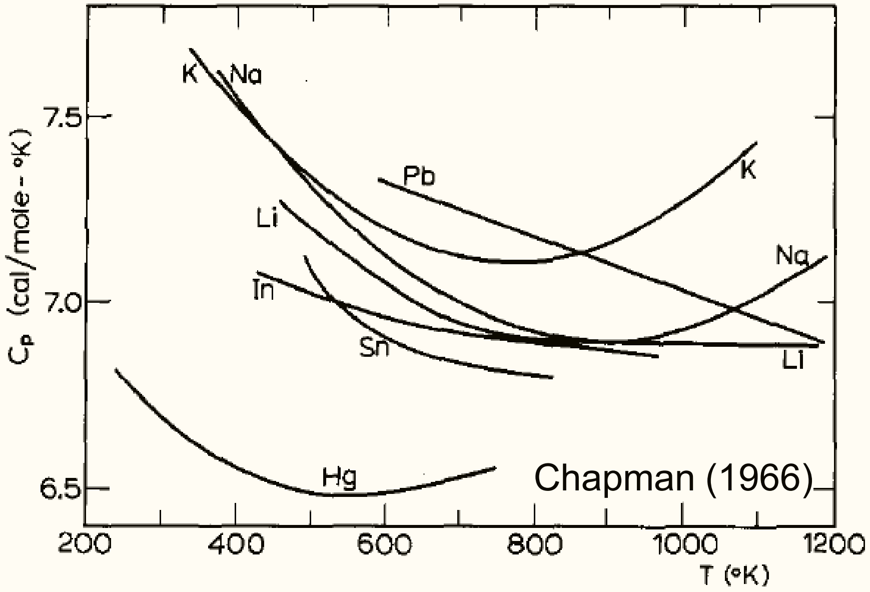
- *$C_P(T)$* of molten salts is assumed to be constant (a_0) or linear ($a_0 + a_1 T$)

$C_p(T)$ of liquids:

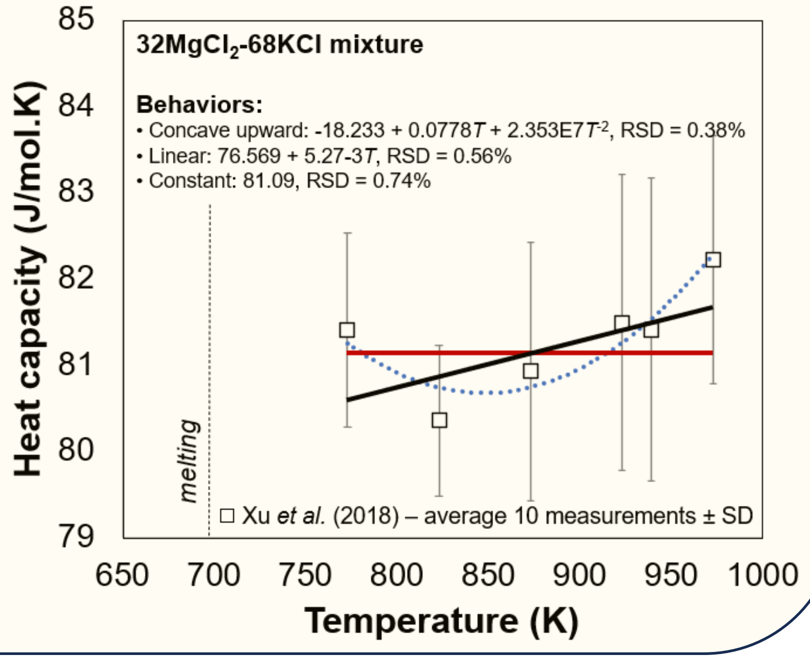
Water



Metals



Molten salts?



Measurement Instruments: Drop Calorimeters

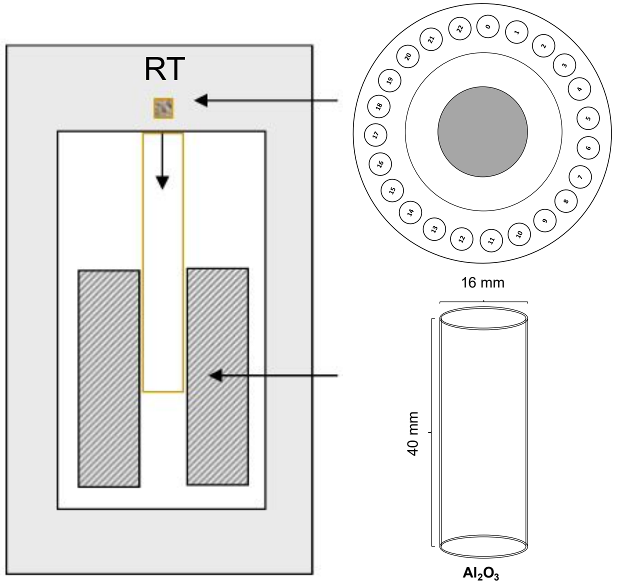
- Custom-Made Calorimeters
- Setaram AlexSys 800/1000 Calvet Calorimeter
- Setaram MHT96 Calorimeter

AlexSys

- Has the highest sensitivity
- 2 cylindrical thermopiles with 128 thermocouples each
- Limited temperature (800/1000 °C)

https://setaramsolutions.com/

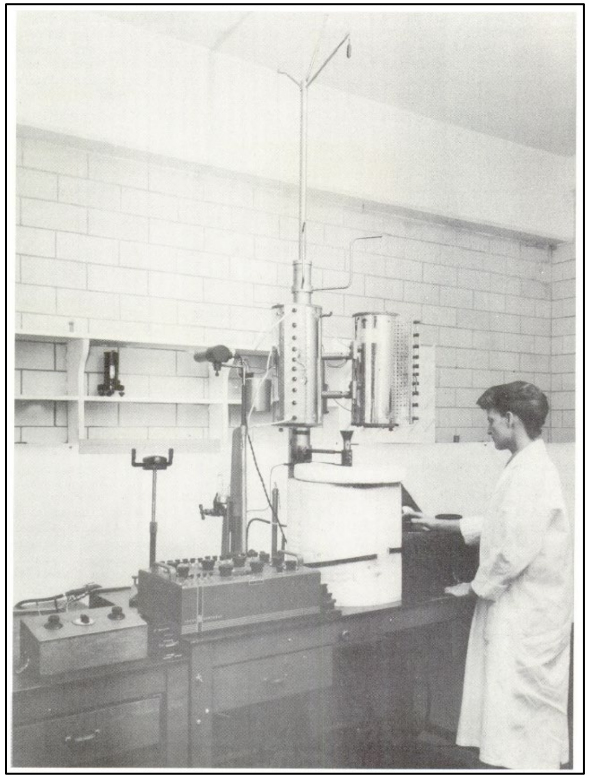
MHT96 Calorimeter



- Less sensible than AlexSys
- Measurements at high-temperature (1500 °C)

Schorne-Pinto et al. (2020) – Adaptation of Ph.D. thesis

Ice Calorimeter at Federal Bureau of Mines



- Legacy instrument

Smith *et al.* (1961) – Construction, Calibration and Operation of Ice Calorimeters

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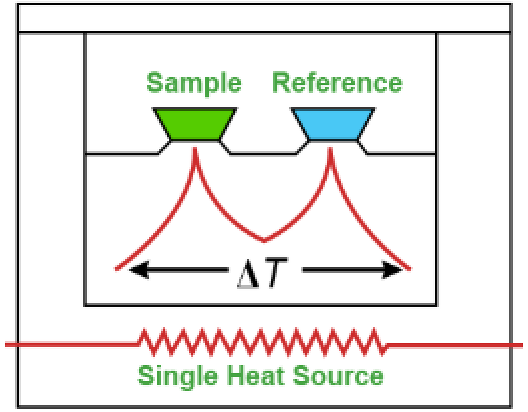
Measurement Instruments: DSCs

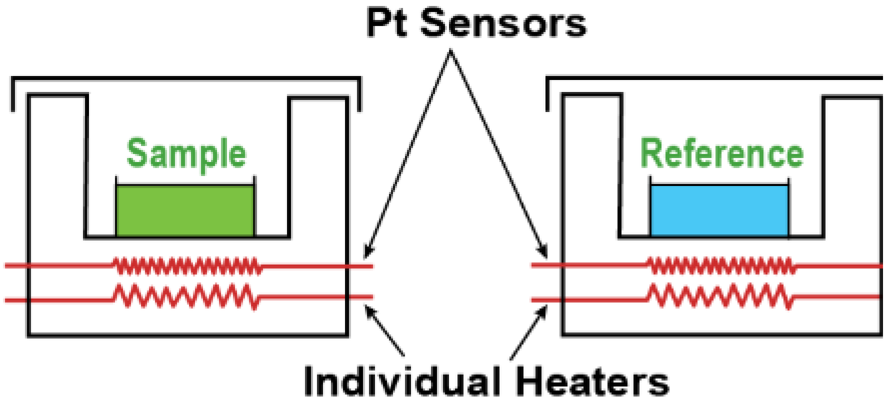
Differential scanning calorimeters can be divided in three categories:

Heat-flux DSC



Keeps heat-flux constant

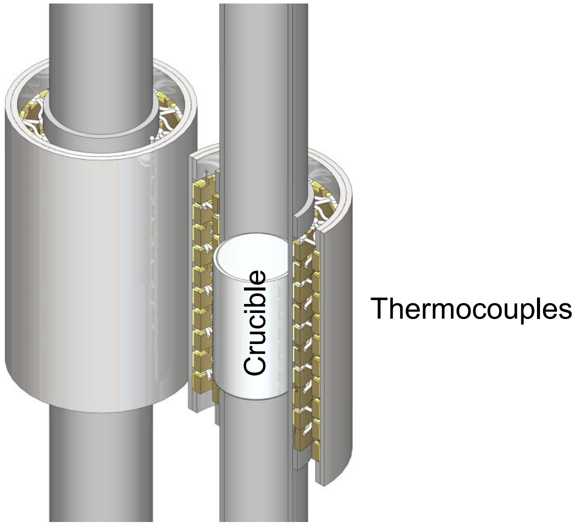
Power-compensated DSC



Keeps power supply constant

Sensitive to heat transfer by conduction only

Tian-Calvet DSC



Similar to the heat-flux DSC, but with a 3D sensor

"Handbook of Differential Scanning Calorimetry – Techniques, Instrumentation, Inorganic, Organic and Pharmaceutical Substances", edited by J. D. Menczel & J. Grebowicz, Elsevier (2023)
K. Parwani (2018) - Thermal Analysis of Cobalt Chloride using DSC
https://setaramsolutions.com/

C_P Measurements on Liquids is Challenging for HF-DSCs

Commercially available calorimeters for measuring C_P of liquids:

Vessel type	*Measurement*	*Representative calorimeters*	*Temperature and pressure ranges*	*Relative quality of heat capacity measurement*
Sealed, constant volume, with vapor space, <0.1 cm^3	Approximately C_P (saturated vapor pressure + external pressure) by temperature-scanning/temperature-modulated calorimetry	DSC from various manufacturers (*e.g.*, TA Instruments, Perkin-Elmer, Netzsch, Mettler)	93–2273 K, depending on specific calorimeter; pressure capability depends on ampule	Poor
Sealed, constant volume, with vapor space, 1–10 cm^3	Approximately C_P (saturated vapor pressure + external pressure) by temperature-scanning heat-conduction calorimetry	• TA Instruments MC-DSC (1 cm^3) • Setaram C80 (10 cm^3) • Setaram MicroDSC (1 cm^3)	• 233–473 K, atmospheric to 41 MPa • room to 573 K, atmospheric to 100 MPa • 253–393 K, atmospheric to 70 MPa	Good–better

…

Chapter 2: "**Calorimetric Methods for Measuring Heat Capacities of Liquids and Liquid Solutions**" by L. D. Hansen and D. J. Russell from the book "Heat Capacities – Liquids, Solutions and Vapours", Editors E. Wilhelm & T. M. Letcher, RSC Publishing (2010)

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How to Carry Out Heat Capacity Measurements?

- Three methods can be used for determining the heat capacity
  - Drop method (indirect) – **Drop calorimeter**
  - Three-step method/Sapphire method (ASTM-E1269 & DIN 51007) – **Heat-flux and Calvet DSC**
  - Small temperature steps (Höhne *et al.* & Gaune-Escard) – **Calvet DSC**
- Factors affecting heat capacity of molten salts

Factor	Drop	Heat-Flux	Calvet	Effect
Sample purity	Yes	Yes	Yes	It will affect the C_P of material and corrections should be made (if small and quantified)
Pan Type and Sealing	Yes	Yes	Yes	It should be compatible with the sample and prevent any potential leakage
Sample Size	No	Yes	No	Large samples can give erroneous C_P
Heat Transfer Mechanisms	No	Yes	No	Conduction, convection, and radiation (T > 600 °C) can affect heat flow
Sample Positions	No	Yes	Yes	Bad crucible placement with generate shifts in the C_P data

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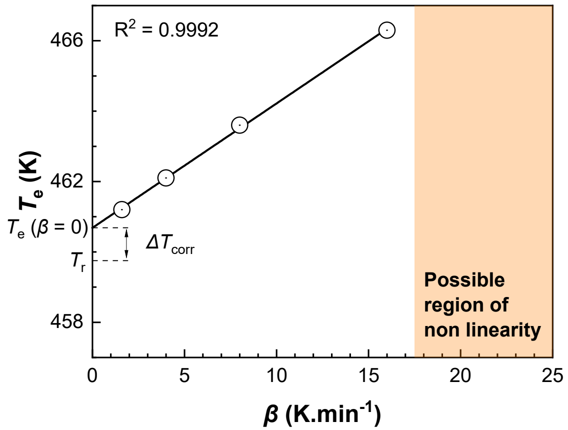
Sensitivity and Temperature Calibration

USC experience:

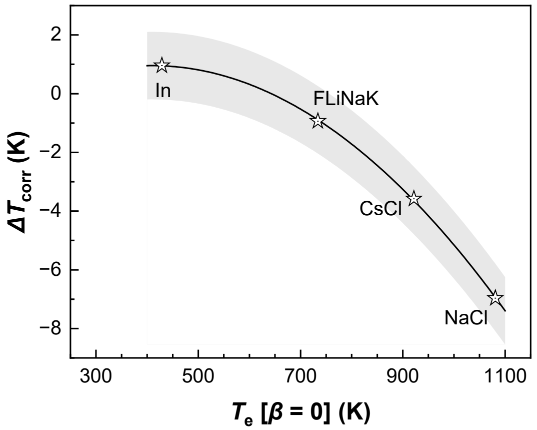
+	Good compatibility
!	Reacts over time
–	Incompatible

- Temperature (IUPAC) – all instruments

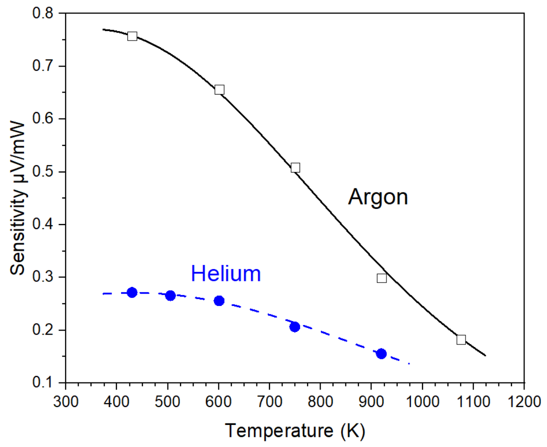
Zero-rate calibration



Correction at β = 0 K.min^{-1}



- Atmosphere – all instruments



STA 449 F1 Jupiter®:
Crucibles: 100 µl SS w Ni liner
Heating rate: 4 K.min^{-1}
Argon: 70 ml.min^{-1}
Helium: 25 ml.min^{-1}

Heat capacity measurements:
Argon: **10-20 ml.min^{-1}**
Helium: **10 ml.min^{-1}**

Metal calibrants – IUPAC Technical Report | **Crucible/liner material**

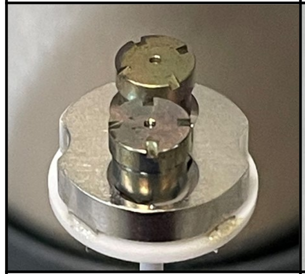
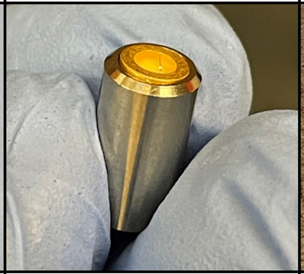
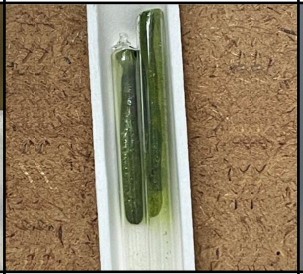
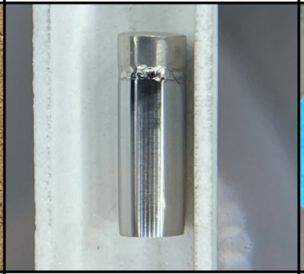
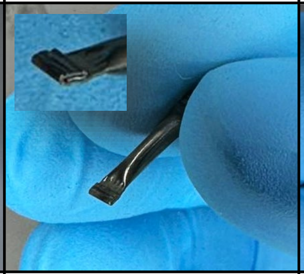
Calibrant	T_{fus}/K	T_{fus}/°C	$\Delta_{fus}h$/J.g^{-1}	Ni	TZM	SiO_2	SS
Gallium	302.915	29.7646	80.07 ± 0.13	+	+	+	+
Indium	429.748	156.5985	28.62 ± 0.04	+	+	+	+
Tin	505.078	231.928	60.38 ± 0.15	+	+	+	+
Bismuth	544.552	271.402	53.18 ± 0.12	+	+	?	+
Lead	600.612	327.462	23.08 ± 0.11	–	+	+	+
Zinc	692.677	419.527	108.09 ± 0.43	–	+	+	–
Antimony	903.778	630.628	162.55 ± 4.91	–	!	+	–
Aluminum	933.473	660.323	399.87 ± 1.33	–	–	–	–
Silver	1234.93	961.78	104.61 ± 2.09	+	+	+	+
Gold	1337.33	1064.18	64.58 ± 1.54	!	!	+	–

Recommended salt calibrants (*evaluated during MSTDB-TC development)

Calibrant	T_{fus}/K	T_{fus}/°C	$\Delta_{fus}h$/J.g^{-1}	Ni	TZM	SiO_2	SS
FLiNaK*	732.9 ± 1	459.8 ± 1	440.3 ± 15.2	+	+	–	–
CsCl	918 ± 1	644.8 ± 1	121.051	+	+	+	+
NaCl	1074 ± 1	800.8 ± 1	481.805	+	+	+	–
NaF*	1269 ± 1	995.8 ± 1	793.94 ± 11.2	+	+	–	–

Preparing Oxygen/Moisture Sensitive Samples for Measurements Outside of Glovebox

- Developed and tested a series of crucibles*

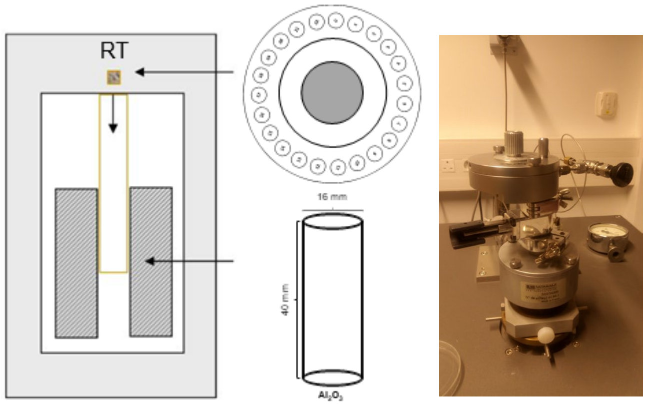
	100 µl SS w Ni liner	27µl SS w Ni liner	130µl SS w Au liner	Sealed quartz	31.5 µl TZM	44 µl Ni folded	44 µl Ni welded
							
Source	Netzsch + UOIT & USC	Netzsch + UOIT & USC	Setaram + USC	USC	USC	USC	USC
Mass	~1480 mg	~ 875 mg	~2240 mg	~450 mg	~2435 mg	180 mg	110 mg
Max. sample mass	10-50 mg	10-20 mg	50-80 mg	>100 mg	30-50 mg	30-50 mg	30-60 mg
Sample volume	30-50%	30-50%	30-50%	50-70%	40-50%	40-50%	40-50%
Ratio Wgt_s/Wgt_{cr}	**3.38%**	**2.29%**	**2.23%**	**22.2%**	**2.05%**	**27.78%**	**54.54%**
Leak resistant	Yes	Yes	Yes	Yes	No	No	Yes
Fluorides	Yes	Yes	Yes	No	Yes	Yes	Yes
Chlorides	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Melting point	Yes	Yes	No	Yes	No	No	Yes
Enthalpy of fusion	Yes	Yes	No	No	No	No	Yes
Heat capacity	**Poor**	**Poor**	**Good**	**Bad**	**Bad**	**Good**	**Best**
Calorimeter	HF-DSC	HF-DSC	Calvet only	Calvet only	All DSCs	Calvet & drop	Calvet & drop

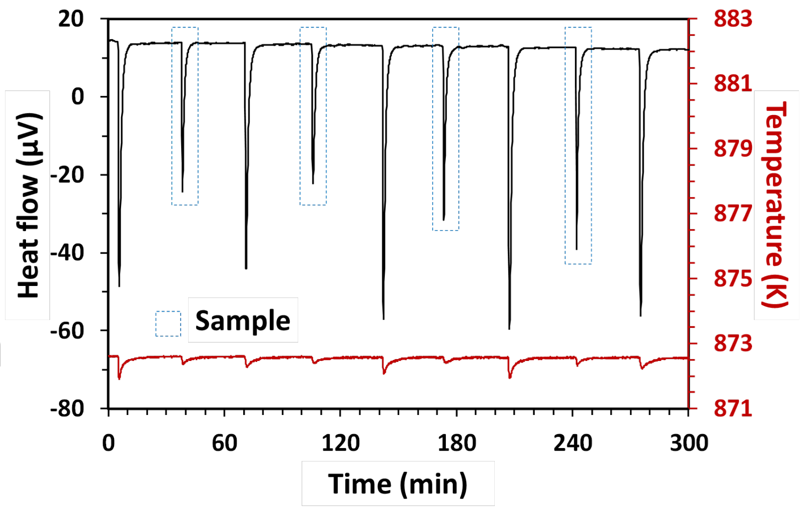
* MSTDB-TC (internal work)

Measurements - Drop Calorimetry

- Provides $H(T) - H_{298.15}$
- Can be used to high-temperatures
- <u>Need multiple drops</u> to get reasonable statistics
- Need <u>equilibrated samples</u>
  - ✓ If A + B = AB, it will be impossible to discriminate $\Delta H_{reaction}$ from $H(T) - H_{298.15}$
- Time-consuming technique
  - ✓ Each temperature takes 4-7h
  - ✓ Machine needs long time to stabilize
- Oxygen/moisture sensitive samples need encapsulation
  - Leak testing is required
  - Need extra corrections for capsule mass

MHT96 – up to 1500 °C





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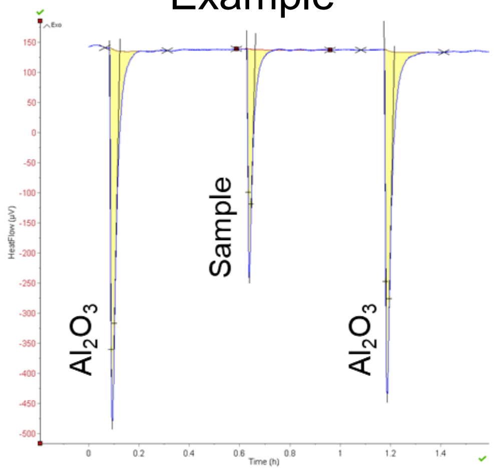
Schorne-Pinto et al. (2020) – Adaptation of Ph.D. thesis

How to Treat Data, Estimate Errors and Report Values

Example



- Treating raw data

$$(H-H_{298})_{Sample}=\frac{m_{Al2O3}}{m_{Sample}}\cdot\frac{\Delta H_{Sample}}{\Delta H_{Al2O3}}\cdot(H-H_{298})_{Al2O3}$$

	Weight (mg)	n (mol)	T_{RT} (°C)	T_{drop} (°C)	Integration (µV.s)	H Al_2O_3 (T_{RT}) (J)	H Al_2O_3 (T_{drop}) (J)	H (T_{drop}) - H (T_{RT}) (J)	H (T_{drop}) - H (T_{RT}) (J/mol)	Average (J/mol)
Al_2O_3	46.1	4.521E-04	26.2	599.47	**6066.4**	-757.60	-729.50	28.09	62131	
Sample	39.5	2.677E-04	26.3	599.47	**3106.4**			**14.38** **13.41**	**53731** **50095**	**51913**
Al_2O_3	40.9	4.011E-04	26.1	599.47	**5773.4**	-672.14	-647.22	24.93	62139	

- Statistical analysis (ideally ≥ 3 samples per temperature)
- Fitting a $H(T)-H_{298}$ equation *assuming $C_P(T)$ constant*

$$H(T)-H_{298}=a+bT$$

$$C_P(T)=\left(\frac{dH}{dT}\right)_P=b$$

	$H(T)$-H_{298} (J.mol^{-1})
Drop#1	51,913.1
Drop#2	53,552.3
Drop#3	54,423.2
Drop#4	57,254.2
Mean	**54,286**
Exp. stand. deviation [SD]	2,235.9
Stand. Uncertainty [u]¥	1,118.0
Student's coverage factor $t_{(n,p)}$*	3.18
Expanded uncertainty [U]	**3,555**
[U] with usual coverage factor k = 2	2,236

¥ u= SD /√N, N = 4
* with ν = N-1 = 3 and p = 95%

- $C_P(T)$ errors for this method is between 5-15% (using CI of the regression)

Evaluation of measurement data — Guide to the expression of uncertainty in measurement (GUM)
Schorne-Pinto et al. (2020) – Adaptation of Ph.D. thesis

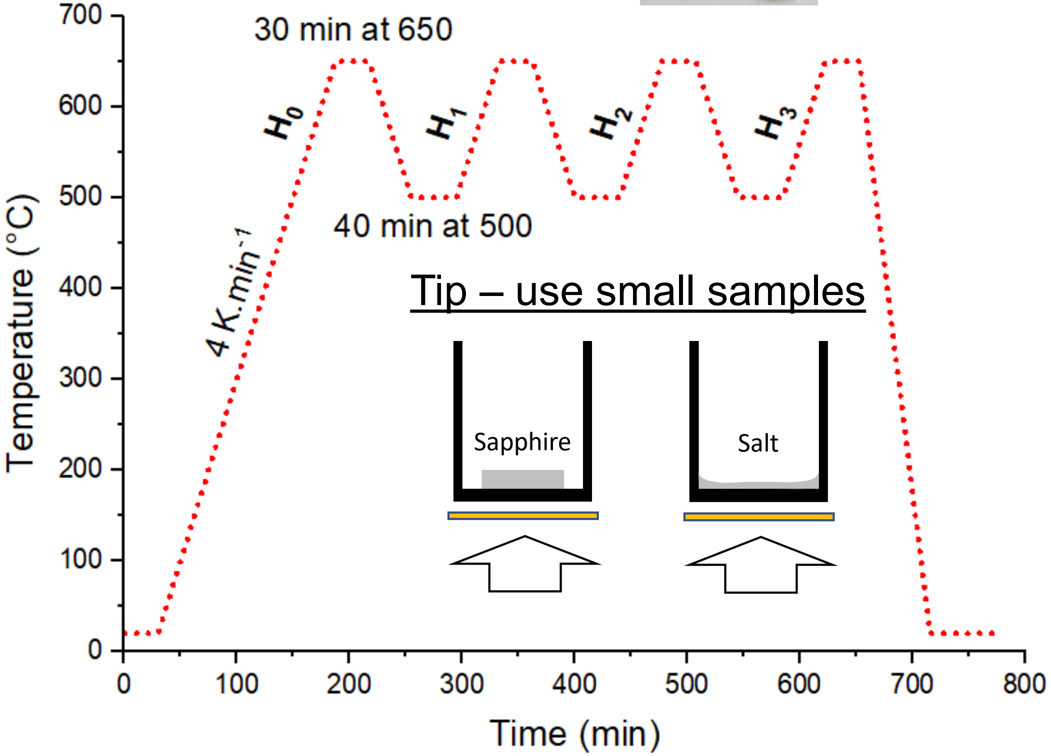
Measurements – HF-DSC

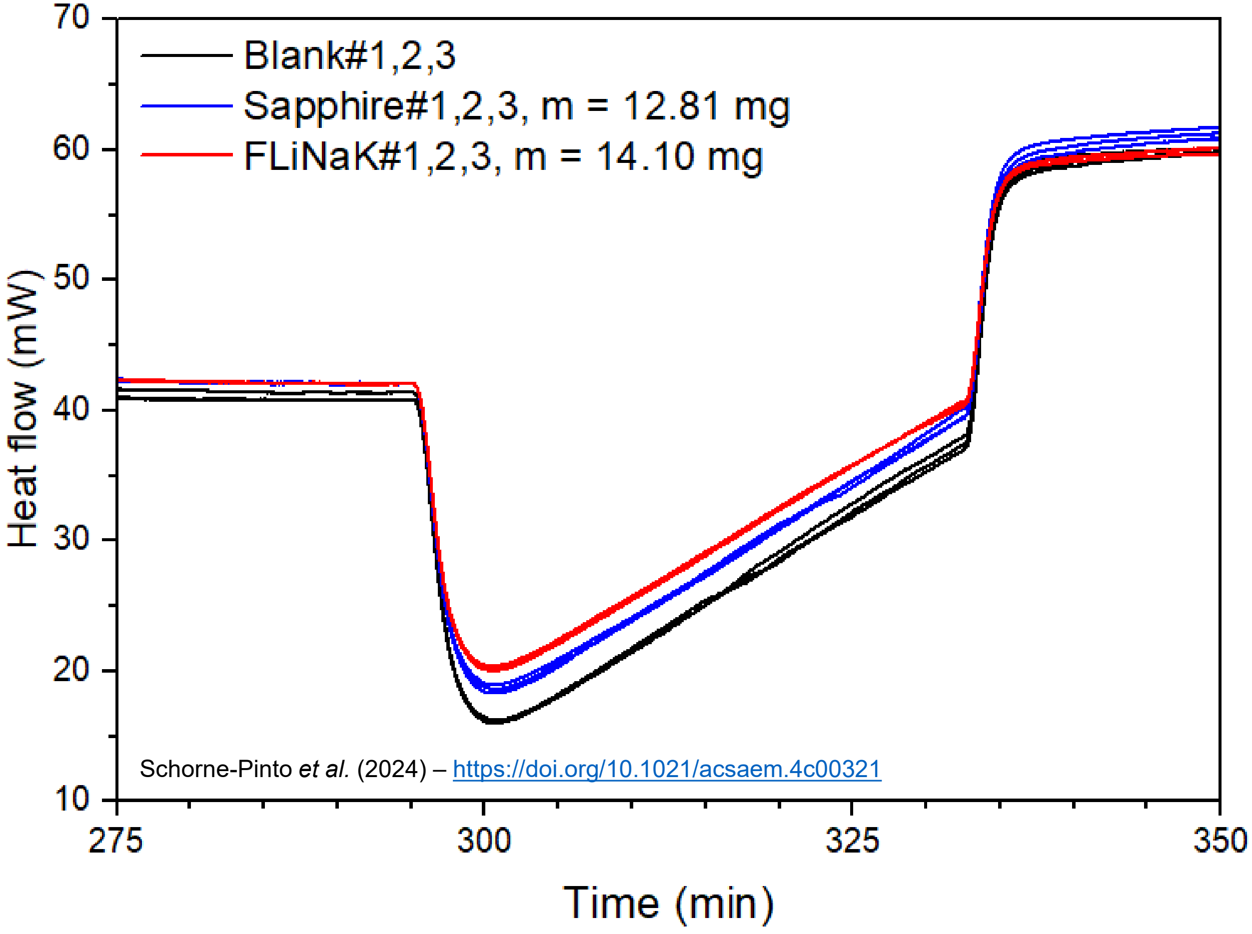
DSC 404 F3 Pegasus® – Netzsch
Sensor Type S (DSC404F1A72)
Crucibles: 100 µl SS w Ni liner
Conditions: 4 $K.min^{-1}$, Ar flow of 20 $ml.min^{-1}$

- **Sapphire/Three-step method**

  Same 100 µl SS w Ni liner was used

  - Blank
  - Sapphire run
  - Sample





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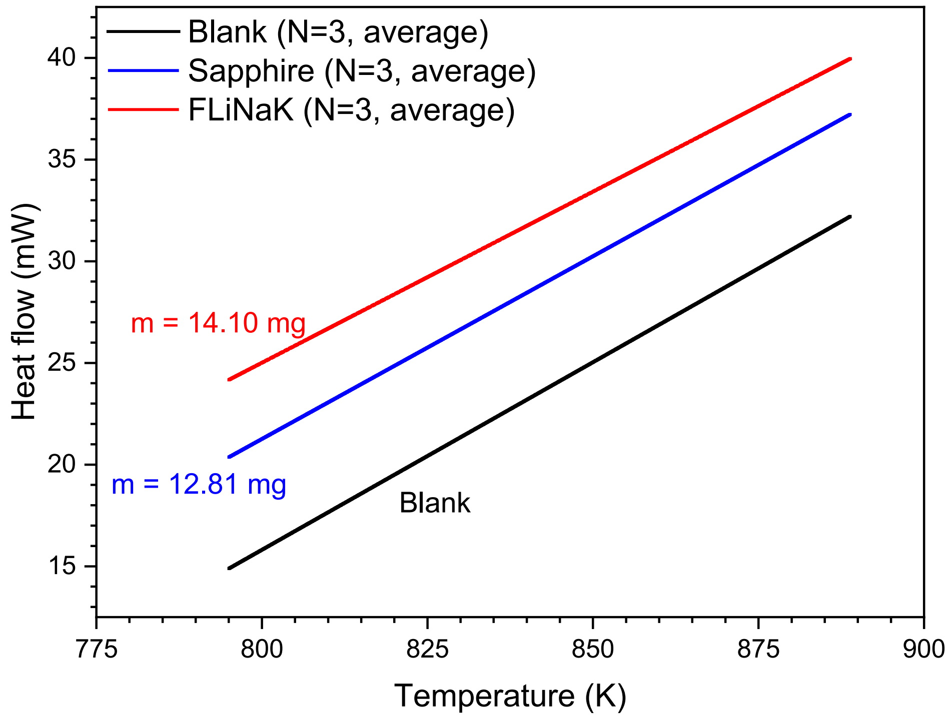
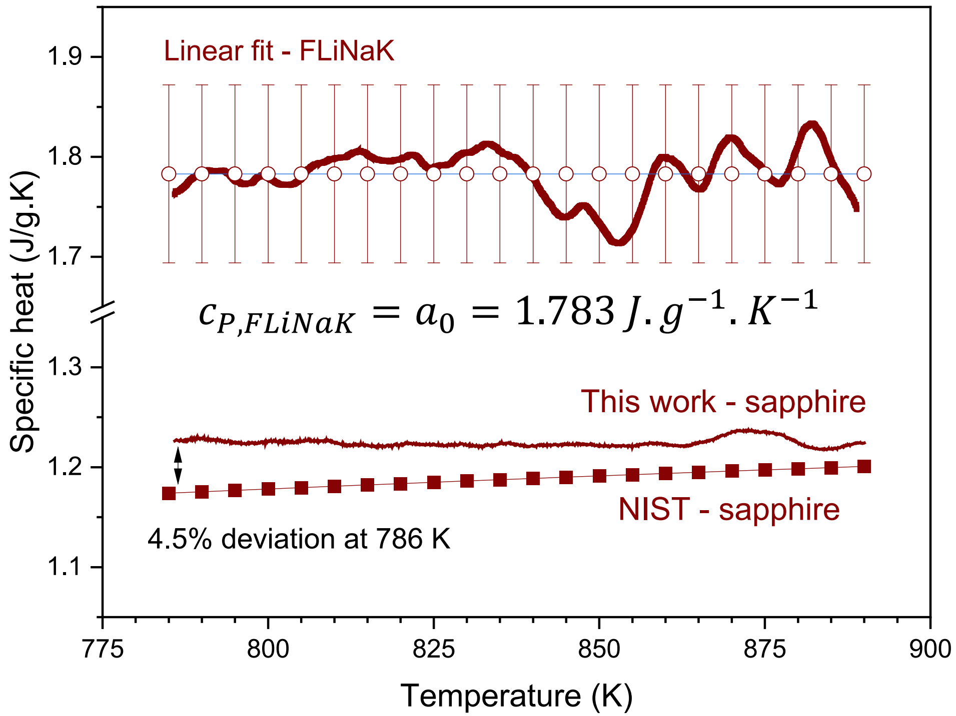
Data treatment and Errors – HF-DSC

- Calculating the specific heat $$c_{P,FLiNaK} = \frac{m_{Sapphire}}{m_{FLiNaK}} \cdot \frac{HF_{FLiNaK} - HF_{Blank}}{HF_{Sapphire} - HF_{Blank}} \cdot c_{P,Sapphire}$$





- Experimental error defined by variation in sapphire reference values (T. Karlsson)

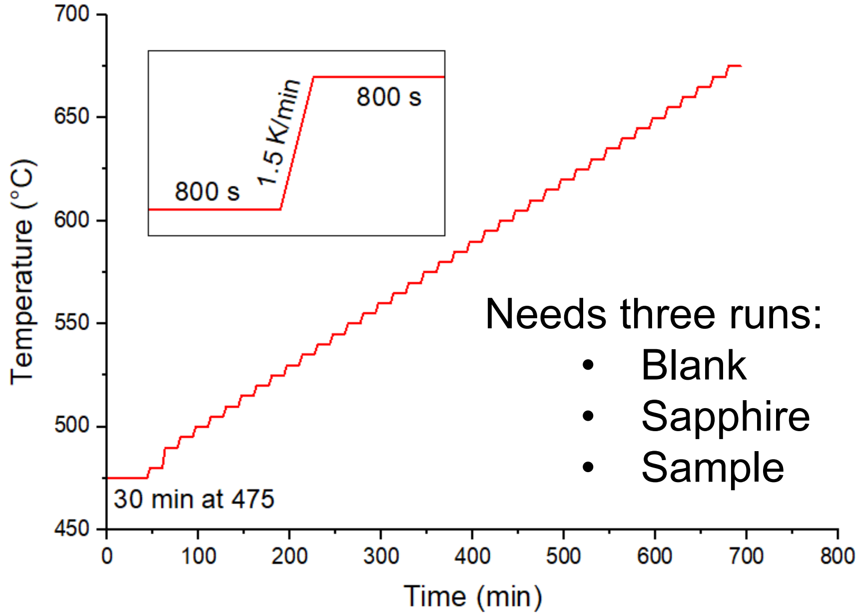
Schorne-Pinto *et al.* (2024) – https://doi.org/10.1021/acsaem.4c00321
Karlsson *et al.* (2023) – https://doi.org/10.1016/j.molliq.2023.122636

Measurements – Calvet

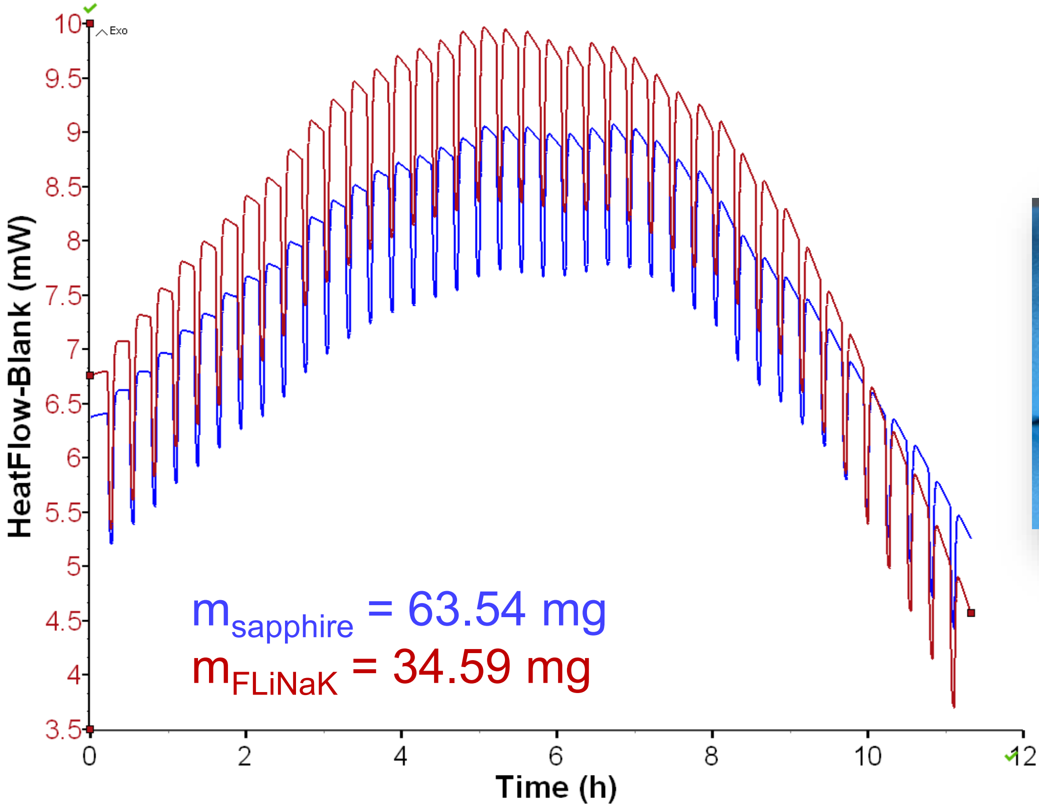
- **Accuracy maximized by using small temperature steps**

Calvet Pro – Setaram

Calvet type (240 thermocouples)
Crucibles: 44 µl Ni welded
Conditions: 5 K step, 1.5 K.min^{-1}, stabilization time of 800s, Ar flow of 10 ml.min^{-1}

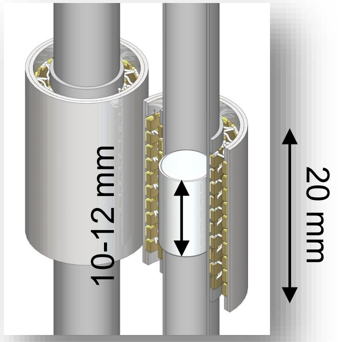


Needs three runs:
- Blank
- Sapphire
- Sample

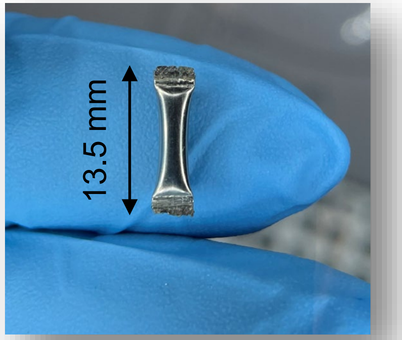


Limited length crucibles required

Thermopiles



44 µl Ni welded



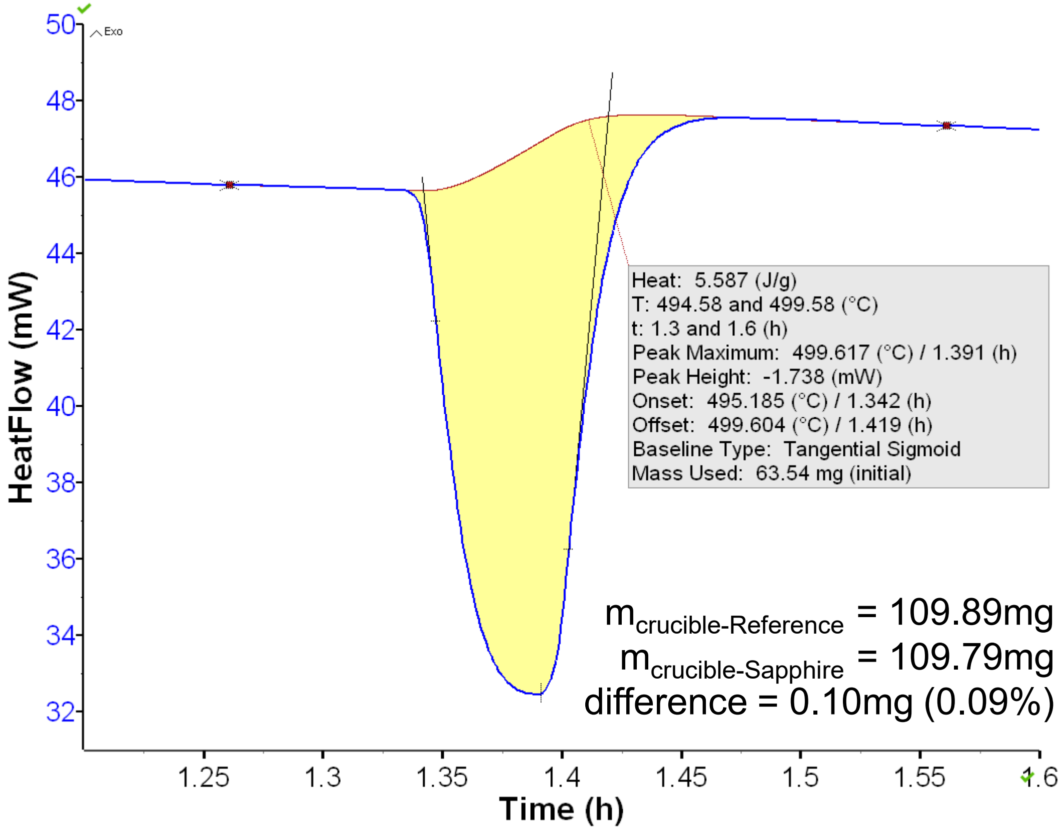
Schorne-Pinto *et al.* (2024) – unpublished
Schorne-Pinto *et al.* (2019) – https://doi.org/10.1016/j.tca.2019.178345

Sensitivity calibration – Calvet

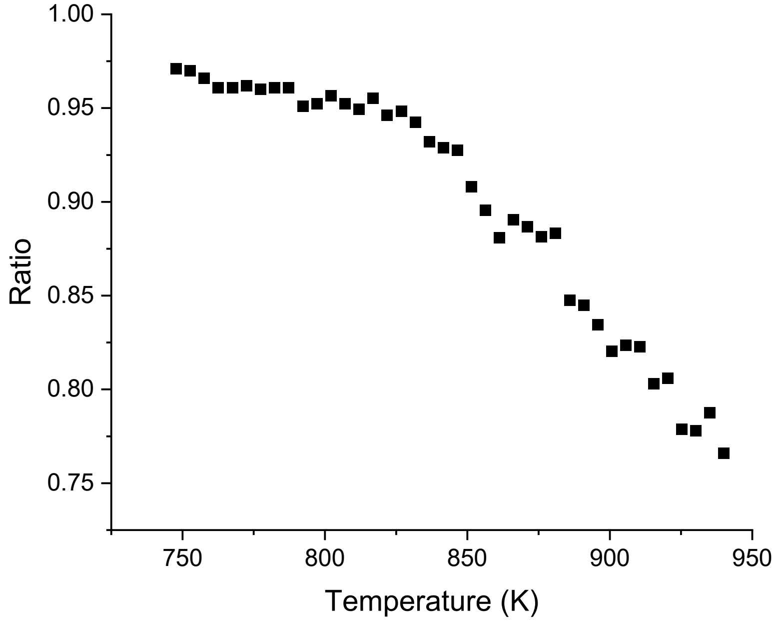
$$Ratio\ (Sensitivity) = \frac{c_{P,Sapphire-measured}}{c_{P,Sapphire-NIST}}$$

- Creating a sensitivity curve

Tinitial (K)	Tfinal (K)	Corrected (K)	ΔT	Enthalpy (J/g)	Raw-cp (J/g.K)	Al2O3-NIST (J/g)	Ratio
747.72	752.73	747.69	4.96	5.598	1.129	1.163	0.971
752.73	757.73	752.64	4.95	5.588	1.129	1.164	0.970
757.73	762.73	757.59	4.95	5.571	1.126	1.166	0.966
762.73	767.72	762.53	4.94	5.538	1.122	1.167	0.961
767.72	**772.73**	**767.47**	**4.96**	**5.587**	**1.128**	**1.169**	**0.965**
772.73	777.72	772.42	4.93	5.556	1.126	1.170	0.962
777.72	782.72	777.36	4.94	5.562	1.125	1.172	0.960
782.72	787.73	782.30	4.95	5.583	1.127	1.173	0.961
787.73	792.73	787.25	4.94	5.578	1.129	1.175	0.961
792.73	797.73	792.19	4.94	5.526	1.118	1.176	0.951
797.73	802.73	797.13	4.94	5.540	1.121	1.177	0.952



$m_{crucible\text{-}Reference}$ = 109.89mg
$m_{crucible\text{-}Sapphire}$ = 109.79mg
difference = 0.10mg (0.09%)



$$c_{P,Sapphire-measured} = \frac{H}{\Delta T}$$

Schorne-Pinto *et al.* (2024) – unpublished
Schorne-Pinto *et al.* (2019) – https://doi.org/10.1016/j.tca.2019.178345

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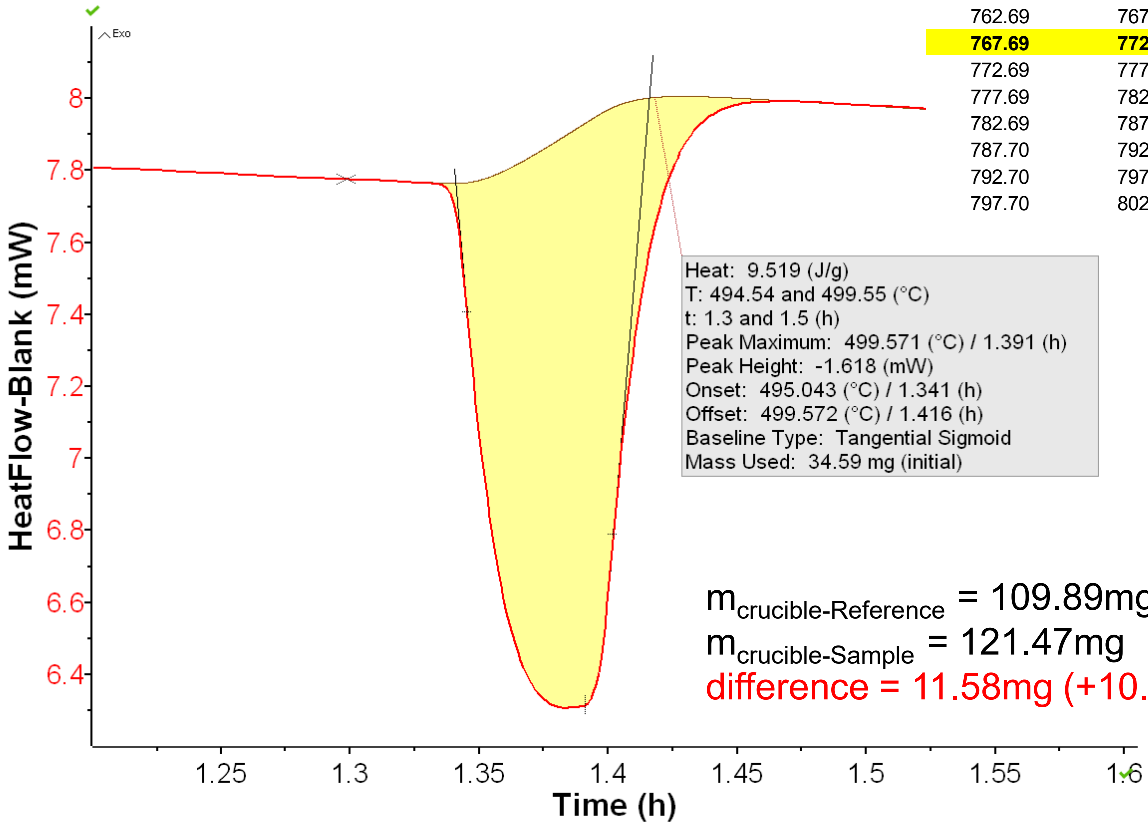
Data treatment – Calvet

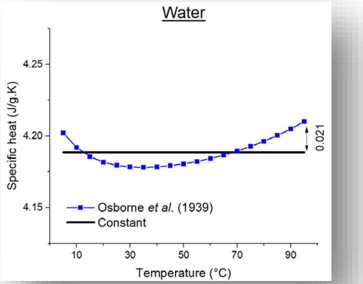
$$c_{P,FLiNaK} = \frac{H_{FLiNaK}/\Delta T_{FLiNaK}}{Ratio} + \frac{\left(\frac{m_{Cr,Ref}-m_{Cr,FLiNaK}}{m_{FLiNaK}}\cdot c_{P,Nickel}\right)}{Ratio}$$

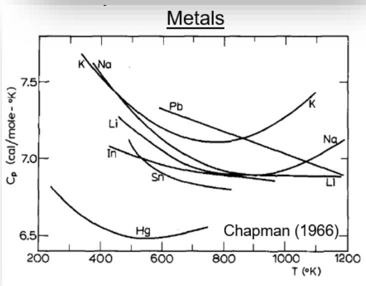
- Results for FLiNaK

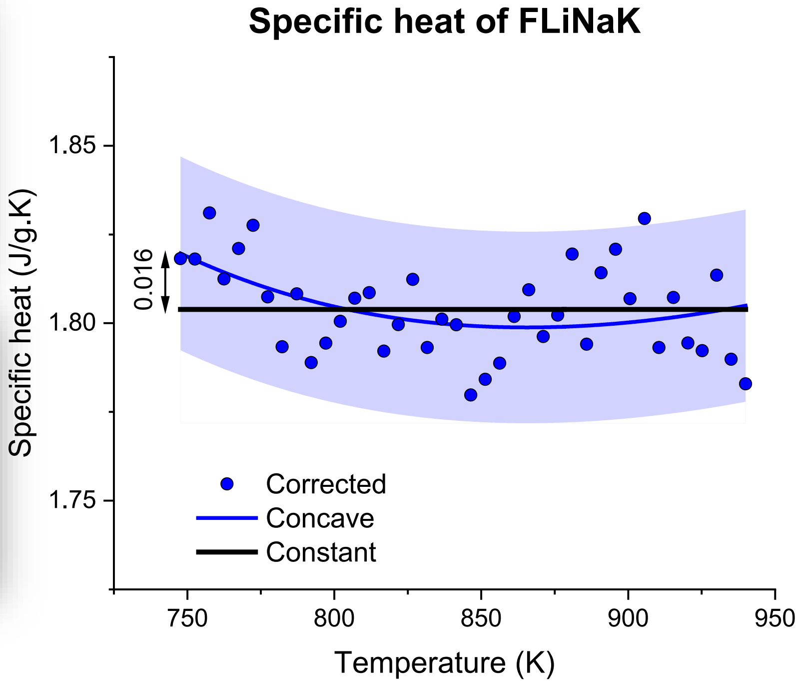
Tinitial (°C)	Tfinal (°C)	Corrected (K)	ΔT	Enthalpy (J/g)	Raw-cp (J/g.K)	Ratio	cp - FLiNaK (uncorrected)	cp-Nickel (J/g.K)	cp - FLiNaK (corrected)
747.69	752.70	747.66	4.96	9.622	1.940	0.971	**1.999**	0.523	**1.818**
752.70	757.69	752.60	4.94	9.573	1.939	0.970	**1.999**	0.523	**1.818**
757.69	762.69	757.55	4.95	9.616	1.944	0.966	**2.012**	0.523	**1.831**
762.69	767.69	762.49	4.95	9.482	1.917	0.961	**1.995**	0.523	**1.812**
767.69	**772.70**	**767.44**	**4.96**	**9.519**	**1.921**	**0.965**	**1.992**	0.523	**1.810**
772.69	777.69	772.38	4.94	9.560	1.933	0.962	**2.010**	0.524	**1.828**
777.69	782.69	777.33	4.94	9.445	1.911	0.960	**1.990**	0.524	**1.807**
782.69	787.70	782.28	4.95	9.402	1.898	0.961	**1.976**	0.524	**1.793**
787.70	792.70	787.22	4.94	9.454	1.913	0.961	**1.991**	0.524	**1.808**
792.70	797.70	792.16	4.94	9.273	1.877	0.951	**1.973**	0.524	**1.789**
797.70	802.70	797.10	4.94	9.310	1.885	0.952	**1.979**	0.525	**1.794**



$m_{crucible-Reference}$ = 109.89mg
$m_{crucible-Sample}$ = 121.47mg
difference = 11.58mg (+10.54%)







Schorne-Pinto *et al.* (2024) – unpublished
Schorne-Pinto *et al.* (2019) – https://doi.org/10.1016/j.tca.2019.178345

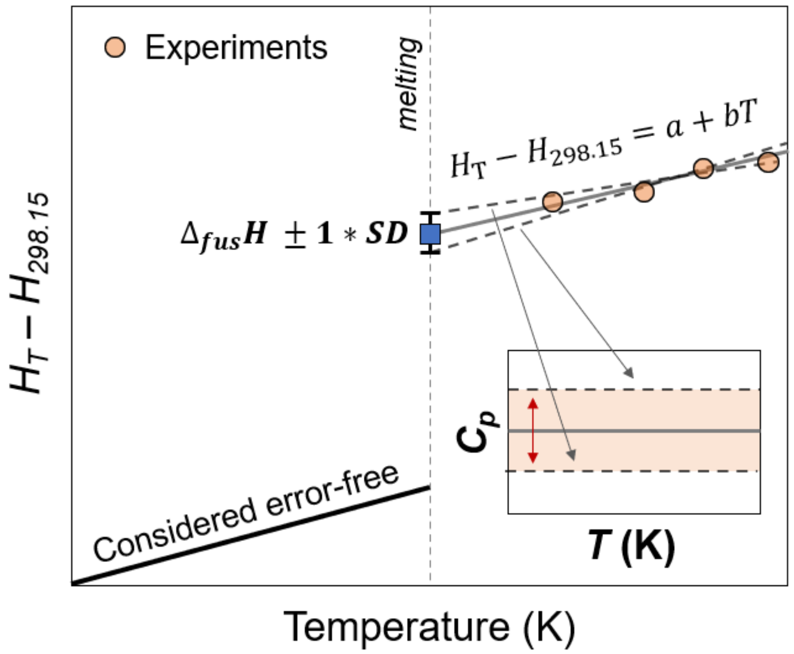
Coupling Different Type of Measurements to Obtain a Complete Understanding of Molten Salt Behavior

Calphad method requiring self-consistency results in accurate representations of thermodynamic values

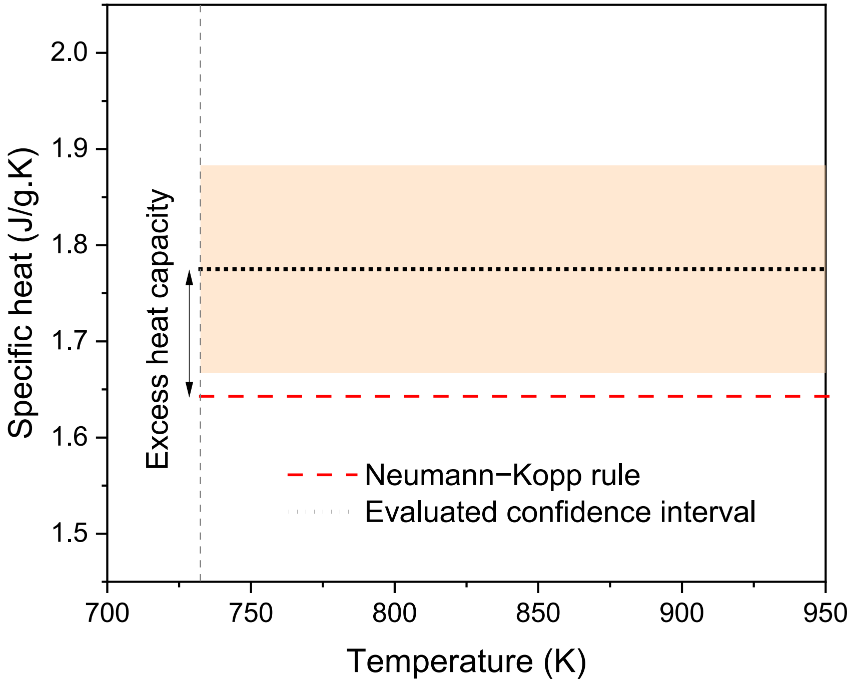
Melting point end enthalpy of fusion

T_{fus} K	$\Delta_{fus}H$ J.mol^{-1}	Error %	N°	Reference
735	16,646	2	27	R (1982)
730.6	16,318	1	3	Y (2016)
737.6	18,275	N/A	1	K (2009)
727	16,483	5	-	P (1956)
725	17,099	-	-	K (1982)
730.8	15,980	N/A	1	W (2006)
735	16,673	3	14	K (2011)
739	17,610	0.8	3	C (2017)
736	18,268	N/A	1	H (1986)
733.8	18,180	3.5%	8	S-P (2024)
	17,153	**± 868 J**		**Average**

Drop calorimetry



Evaluated C_p range vs Neumann-Koop rule



Schorne-Pinto *et al.* (2024) – https://doi.org/10.1021/acsaem.4c00321

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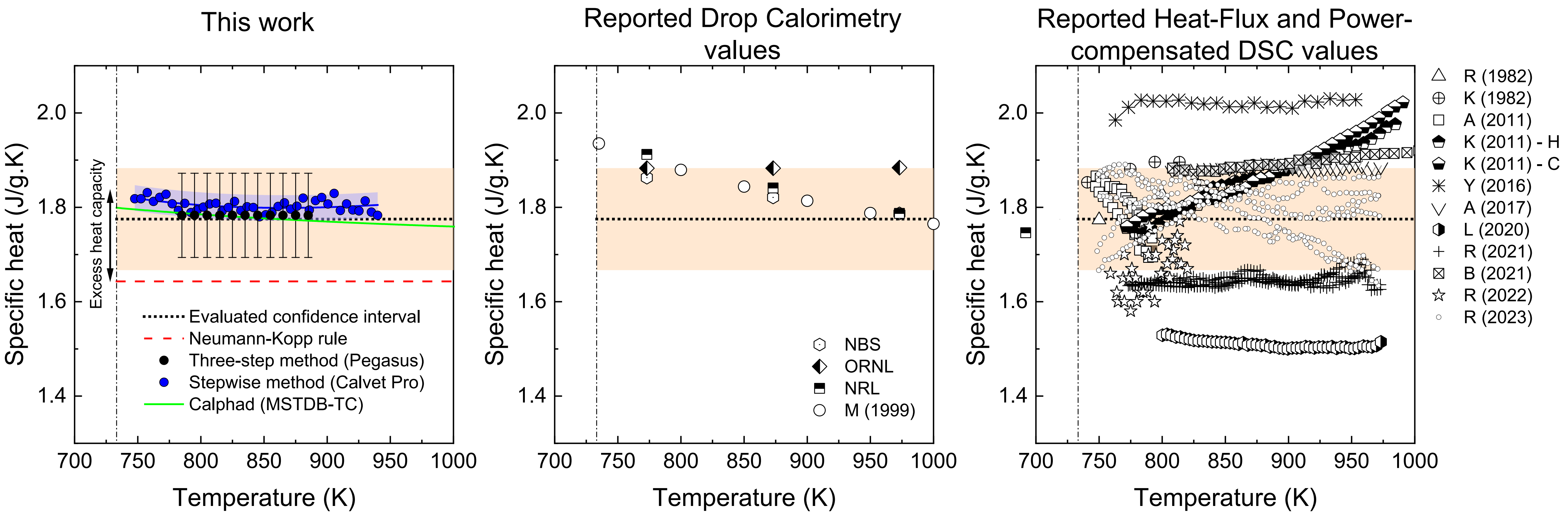
Example: Comparing FLiNaK Results with Literature

- Need to couple experiments with modeling to assure capture of realistic behavior



- “Consistent with the literature” – Any measurement within the range of 1.5 to 2.03 J.g^{-1}.K^{-1} is

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Schorne-Pinto *et al.* (2024) – https://doi.org/10.1021/acsaem.4c00321

Can We Get Closer to the Correct Value?

- Dealing with disparate data
  - Averaging all the data would be a mistake – large datasets would dominate results
  - Bias by source of data
  - A value recommended by committee could still be incorrect
- In an ideal world with infinite time and money
  - Well characterized samples before and after measurements
  - Compare different methods
  - Prioritize the use of instruments with highest sensitivity
  - Confirm agreement and reproducibility by changing conditions (e.g., mass, atmosphere, crucibles, etc.)
  - Analyze each factor of error
  - Average of multiple consistent measurements

Some Key References

Books:

- “**Differential Scanning Calorimetry**” by Höhne *et al.*, Springer-Verlag Berlin Heidelberg (2003)
- “**Heat Capacities – Liquids, Solutions and Vapours**”, edited by E. Wilhelm & T. M. Letcher, RSC Publishing (2010)
- “**Handbook of Differential Scanning Calorimetry – Techniques, Instrumentation, Inorganic, Organic and Pharmaceutical Substances**”, edited by J. D. Menczel & J. Grebowicz, Elsevier (2023)

Norms:

- **ASTM - E1269**: Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry
- **DIN 51007**: Thermal analysis – Differential thermal analysis (DTA) and differential scanning calorimetry (DSC) –General Principles

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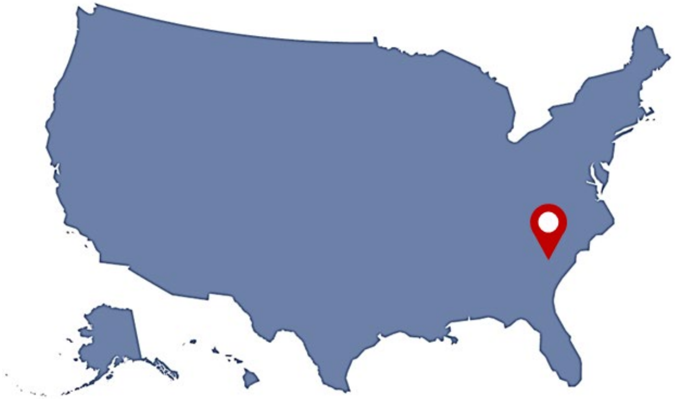
Acknowledgements

Molten Salt Reactor
PROGRAM

Experimental work & MSTDB-TC development effort under the Molten Salt Reactor Program



USC team

Director:


Prof. T. Besmann

Research Professors:


J. Schorne-Pinto


A. M. Mofrad

Postdoc fellows:


J. Paz Soldan Palma


R. Booth


J. Wilson


PM Aiswarya

Students:


C. M. Dixon
Ph.D. candidate


R. McManus
Master's candidate


Z. Gardiner
Undergraduate Researcher

Alumni who work with molten salts:


M. Aziziha
Intel


J. A. Yingling
INL


M. S. Christian
SNL


K. Johnson
Kairos


J. Ard
Boeing

Questions

julianos@mailbox.sc.edu

Radiological Facility at USC (U + limited Pu work)

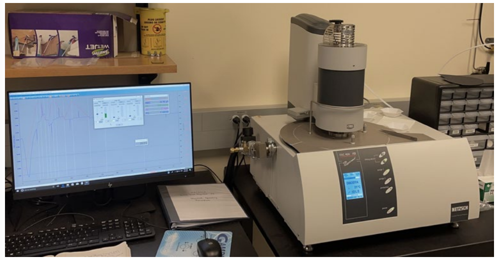
Two 750 sq. ft. laboratories:

- Gloveboxes

> 50 salts (chlorides, fluorides and iodides)
Purification furnace system inside glovebox

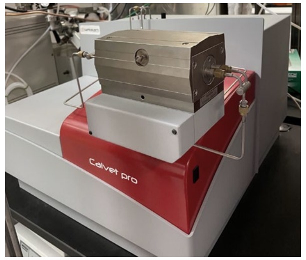
- Calorimeters

DSC 404 F3 Pegasus® - Netzsch



STA 449 Jupiter® - Netzsch

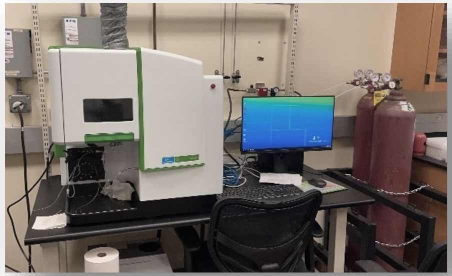
Calvet Pro - Setaram



Melting points (±3 K), enthalpy of fusion (±3% for HF DSC, ± 1% Calvet), C_p (1% solid and 1.5% liquid using the Calvet)

- Chemical analysis

Chemical analysis by ICP-OES



Elementrac for O_2 and H_2

- XRD (+3 other units) - Prof. zur Loye's lab

Rigaku Smartlab XRD (RT-1100 °C)

- Furnaces for thermal treatment and synthesis

Tube furnace (RT-1700 °C)

Muffle - Glovebox

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