A Thermal Review of the Sample Cartridge Assembly (SCA) Gravitational Effect of Distortion in Sintering (GEDS) Experiment Flight Processing

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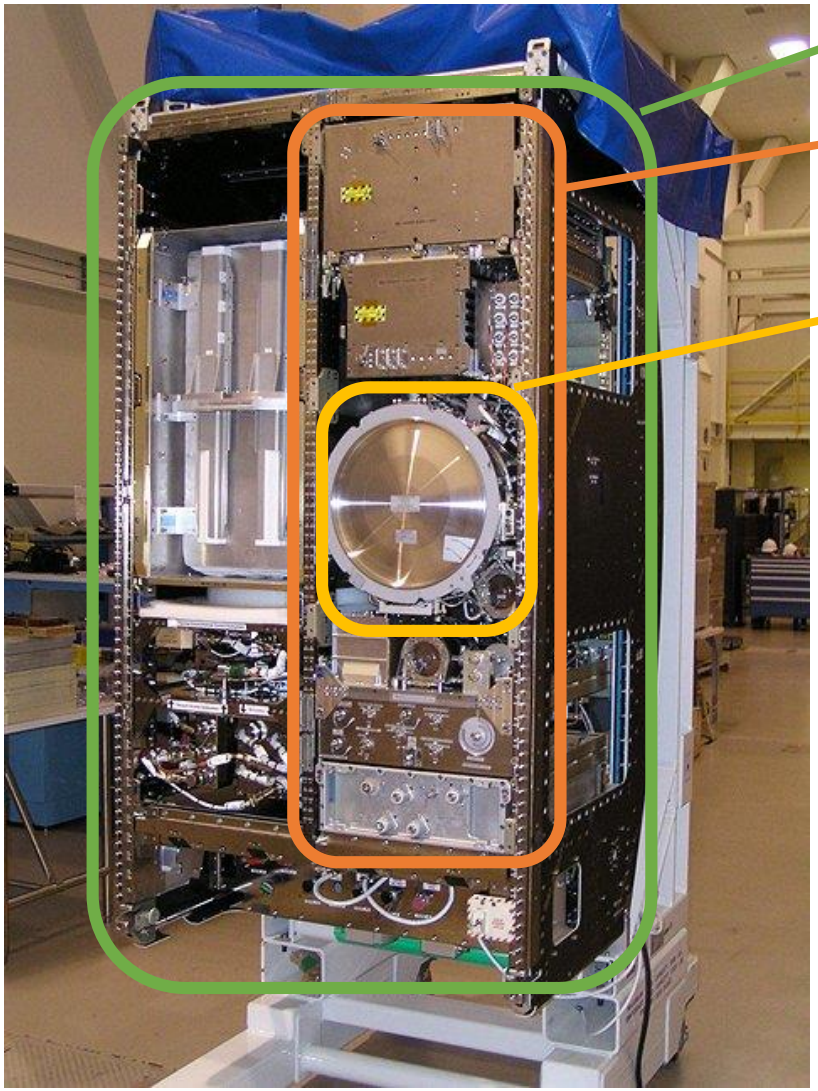
Outline

- What is a SCA?
- Gravitational Effects on Distortion in Sintering (GEDS)
- GEDS Flight Cartridges Layout
- Flight Processing Profile
- Comparison of TC to Sample Transient Predictions
- Discussion of TC Error Impact on Predicted Sample Response
- Forward Work
- Lessons Learned
- Questions

MSRR - LGF

- The SCA is processed in the Low Gradient Furnace (LGF) in the Materials Science Laboratory (MSL)
  - MSL has two different furnace inserts the LGF and the Solidification and Quench Furnace (SQF)
- The MSL is a ESA payload onboard the International Space Station (ISS). It is operated by the Microgravity User Support Center (MUSC) in Cologne, Germany
- MSL is housed in NASA's Materials Science Resource Rack (MSRR) in the U.S. Lab module on ISS

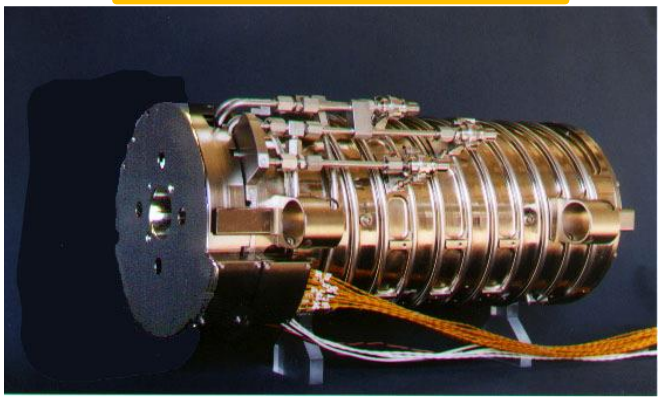
MSRR-LGF

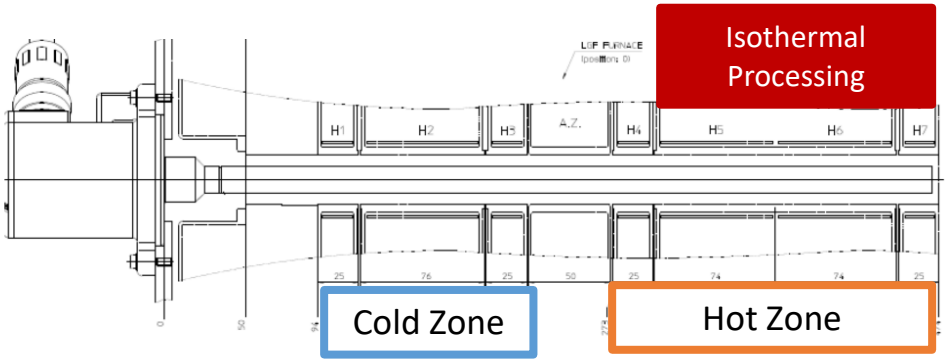


MSRR

MSL

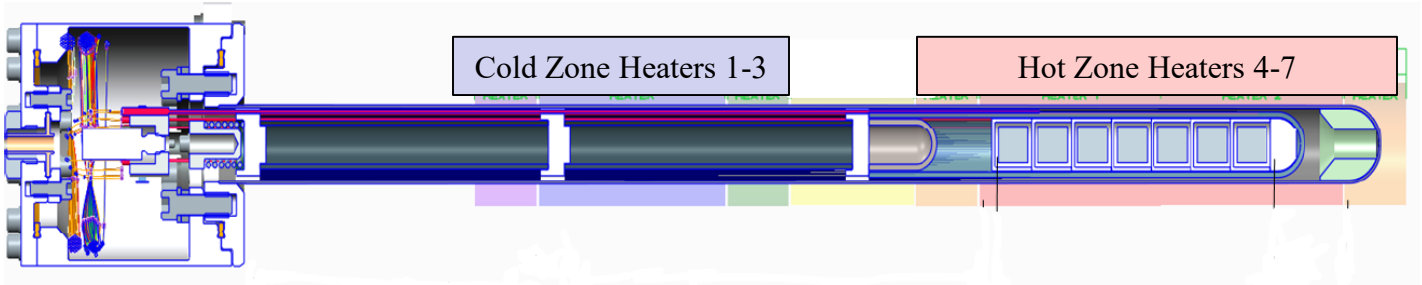
Core Facility or Process Chamber – LGF located inside





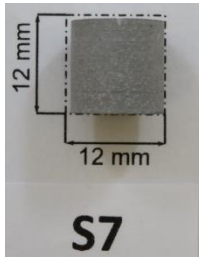
SCA

- SCA – Sample Cartridge Assembly
  - Consists of a plasma sprayed MoRe cartridge with an Alumina liner brazed to a stainless steel head
  - The cartridge holds flight Principle Investigator's (PI) science and provides a level of containment for safety
  - PI's science is contained inside an ampoule held within the furnace heated zones
- The stainless steel SCA head contains a pressure transducer, temperature limiting resistor, and the mounting location for thermocouple transitions



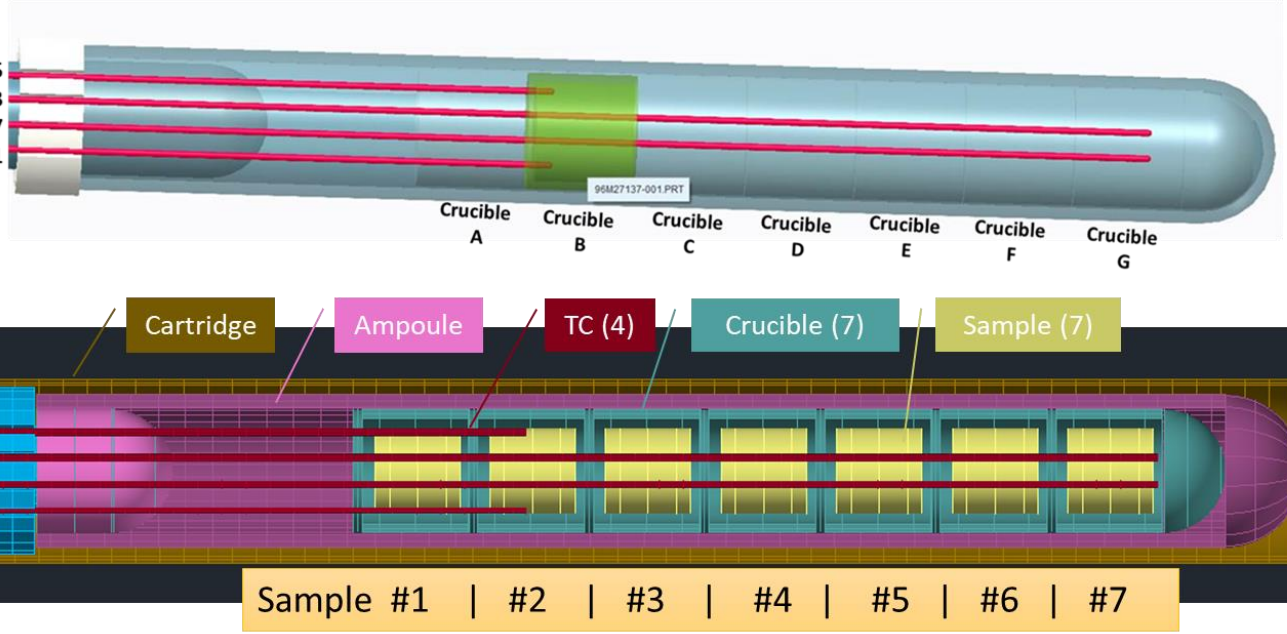
GEDS Experiment

- Dr. Rand German is the PI based of out San Diego State University
- The GEDS experiment focuses on determining the underlying morphology that governs density, size, shape, and properties for liquid phase sintered bodies over a broad range of compositions in Earth-gravity (1g) and microgravity (μg) conditions
- Samples are processed by holding the ampoule at isothermal conditions for a prescribed period of time to obtain a snap shot of liquid phase sintering for various exposure periods



GEDS Experiment Layout

- Each GEDS cartridge is made up of 7 samples contained in crucibles
  - Each sample is primarily Tungsten (W) with varying percentages of Nickel (Ni), Copper (Cu) and Manganese (Mn)
  - The seven GEDS SCAs are identified as C0, C1, … , C6
  - Science goal is isothermally at temperature over exposure time



GEDS TMM Predicted Sample Processing Tables

Sample Time > 1205ºC (Minutes)

GEDS SCA #	LGF Hold	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7
C1	32.5 min	none	6.4	11.3	13.4	14.5	15.1	15.5
C2/C6	35 min	none	7.7	13.9	16.0	17.1	17.7	18.1
C3	42 min	none	15.0	21.1	23.2	24.3	24.9	25.3
C4	57 min	none	30.2	36.2	38.3	39.4	40.0	40.4
C5	91 min	none	63.3	69.3	71.4	72.4	73.0	73.4

Sample Peak Temperature (ºC)

GEDS SCA #	LGF Hold	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7
C1	32.5 min	1202.7	1205.6	1207.5	1208.7	1209.3	1209.6	1209.8
C2/C6	35 min	1203.0	1206.0	1208.0	1209.1	1209.7	1210.1	1210.3
C3	42 min	1203.6	1206.6	1208.6	1209.8	1210.5	1210.9	1211.1
C4	57 min	1203.9	1207.0	1209.1	1210.4	1211.0	1211.4	1211.6
C5	91 min	1204.0	1207.1	1209.2	1210.4	1211.1	1211.5	1211.7

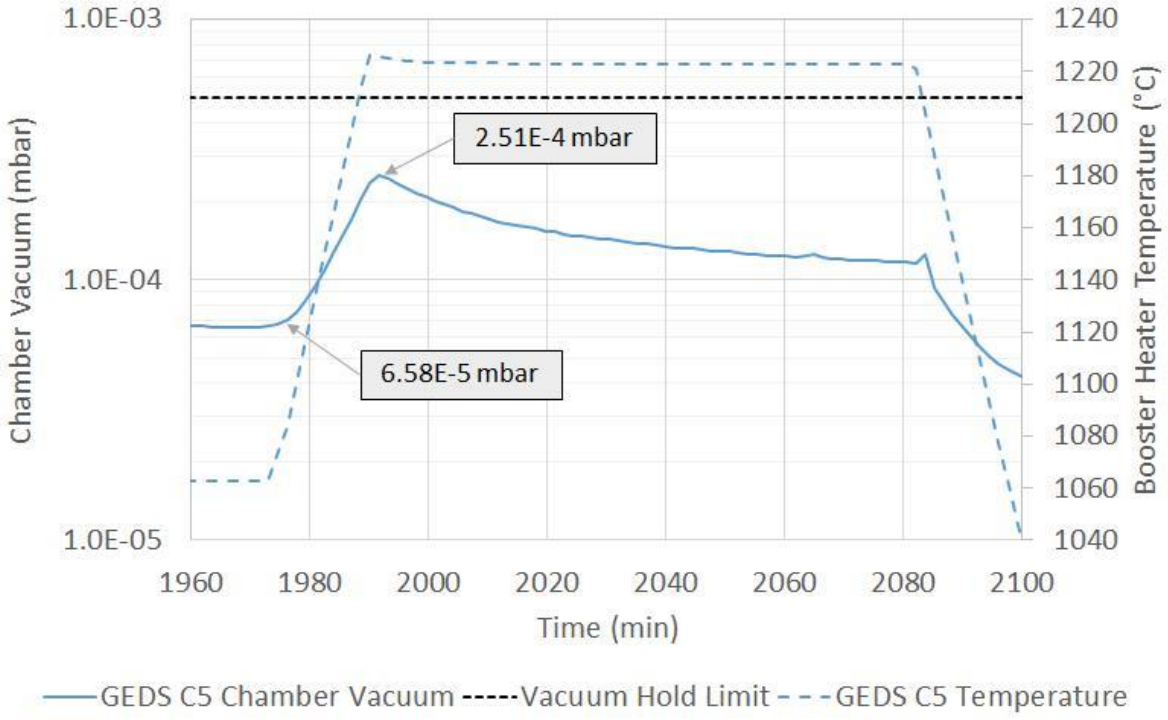
- Analysis model does not simulate sintering but assume homogenous sample properties
- Analysis predictions for the duration above 1205ºC that approximates the sample time above 1200ºC based on ground sample evaluation

GEDS Flight Processing Profile – Processing Hold

- LGF flight calibration cartridge showed the LGF heater set point thermocouples had drifted
  - Δ7ºC to the actual temperature at 1000ºC (i.e. heater set at 1007ºC)
  - Δ10ºC to the actual temperature at 1200ºC (i.e. heater set at 1210ºC)
- Cold and hot zone heaters 1-7 are heated from ambient to 1050ºC at 5ºC/min and allowed to reach steady state. Thermal evaluates heater response
  - Make adjustment to the heater set point in anticipation of final heating
  - For example GEDS C5 flight the pre-processing hold at 1050ºC showed an actual temperature of 1040ºC based on SCA TC steady state values
  - Adjusted hold temperature to 1063ºC such that the hold is at 1053ºC
- The furnace temperature is held constant until an appropriate processing window becomes available (within 30 hours)
  - A low initial chamber vacuum pressure is needed to prevent a pressure recovery hold during final heating
  - Final processing during crew sleep
  - Processing window with 30-45 minutes of uninterrupted data coverage

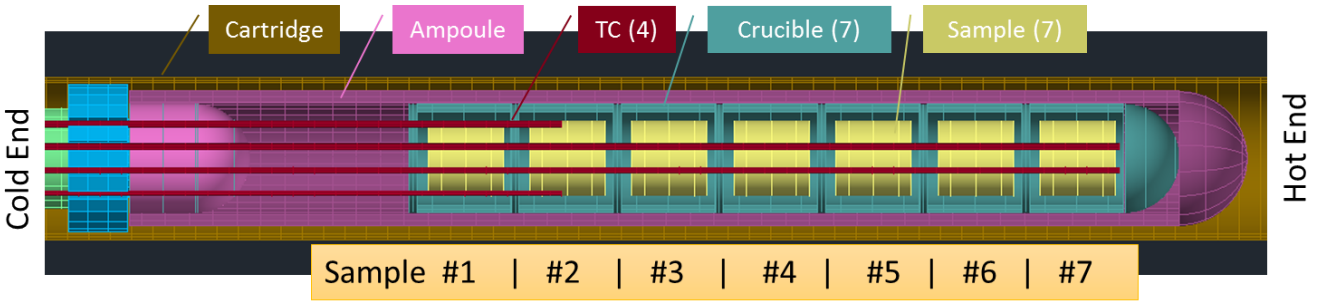
GEDS Flight Processing Profile – Final Heating

- Hot zone heaters 4-7 are heated to 1210ºC at 10ºC/min. Thermal watches furnace vacuum pressure and SCA internal pressure
  - With heaters adjusted for drift the furnace set point was typically 1223ºC for a 1211ºC SCA TC reading at steady state
- Heaters are maintained for the duration requested by PI
- The furnace is controlled during cooled down until 800ºC



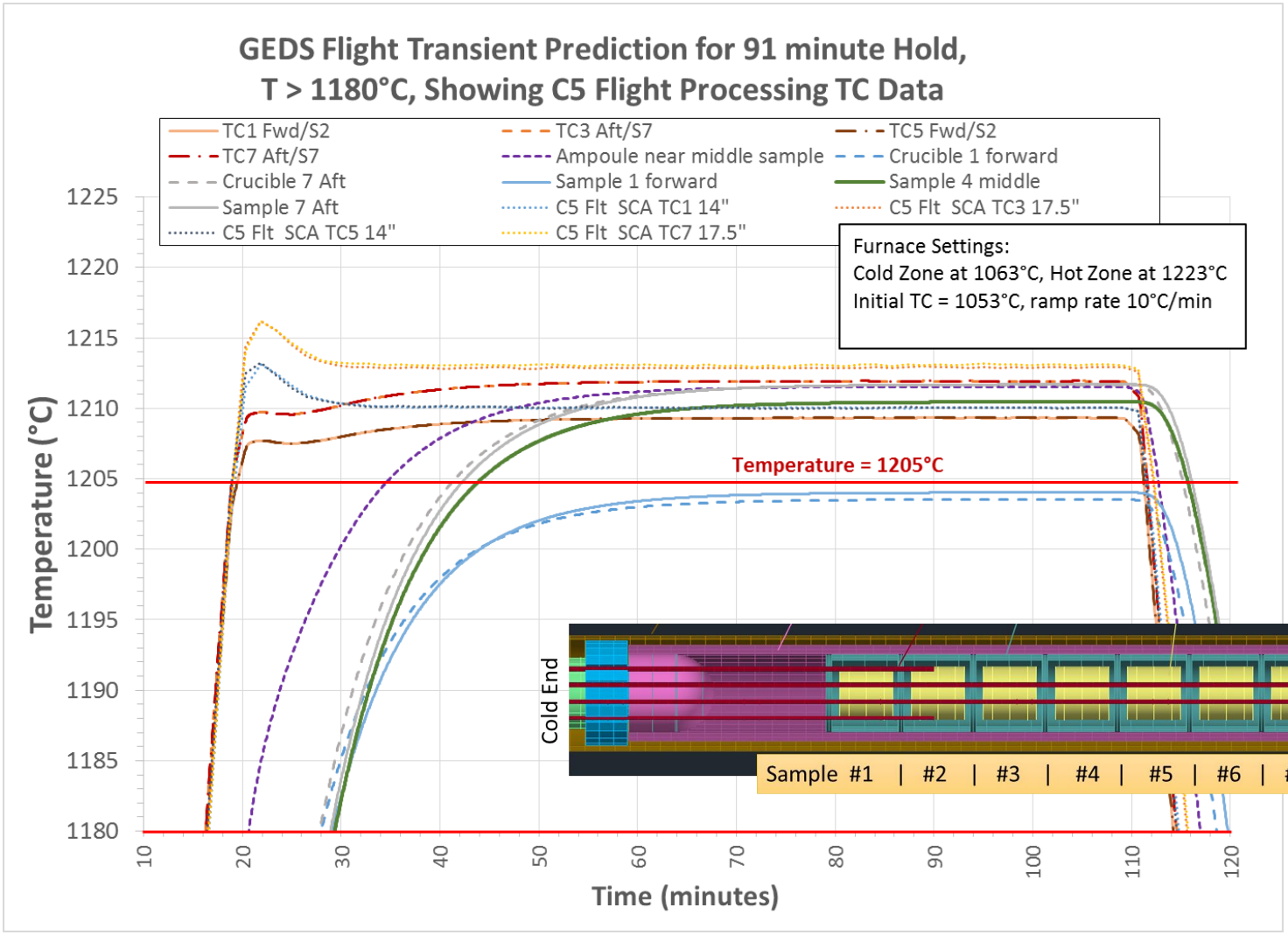
GEDS SCA Sample Transient Response

- A Thermal Mathematical Model (TMM) of the SCA internal to the furnace is needed to predict GEDS SCA sample response
  - GEDS sample response lags behind the SCA TC response
  - TMM simulates the furnace response with transient boundary conditions
  - Due to LGF heater setting drift, SCA TC data instead of actual heater setpoints were used to adjust the LGF heater bore temperatures in the TMM
- TMM used to predict sample transient response
  - Analysis model does not simulate sintering but assumes homogenous sample properties
- Evaluation of SCA TC error is included in evaluation of sample transient response



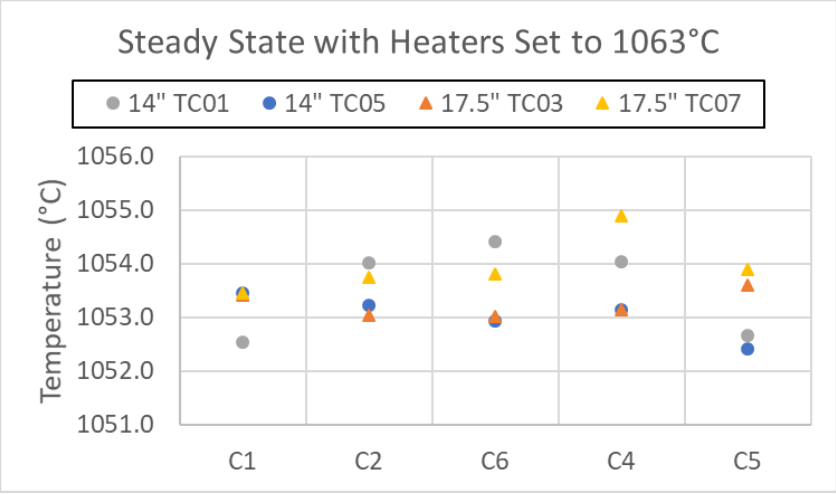
Comparison of SCA TCs and Sample Transients

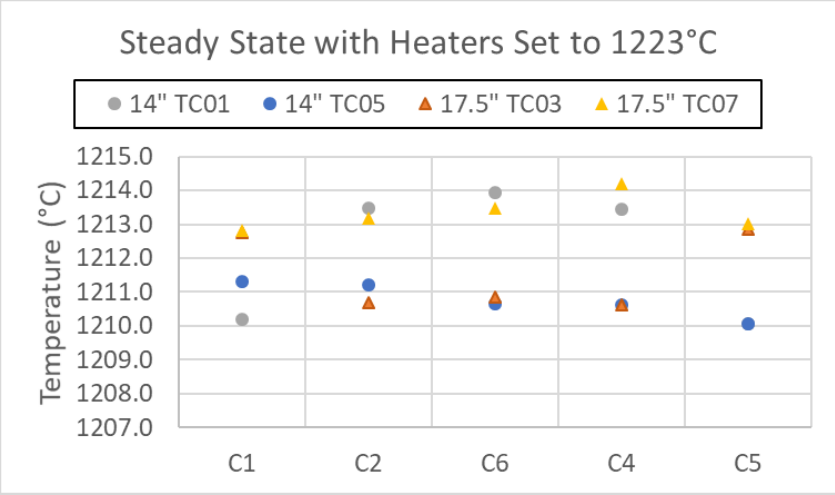
- Samples lag the SCA TCs response



GEDS SCA TC Distribution

- TC measurement uncertainties assess whether science goals are met: isothermally and duration at temperature
- Compared TCs for GEDS C1, C2, C4, C5 and C6
  - 4 TCs at steady state for each GEDS flight processed
- Steady state shown at LGF heater set points of 1063ºC and 1223ºC
- This corresponds to actual SCA TC readings of 1053ºC and 1212ºC
  - TC range Δ2.49ºC preprocessing steady state at 1063ºC
  - TC range Δ4.14ºC during processing steady state at 1223ºC





SCA TC Error Evaluation

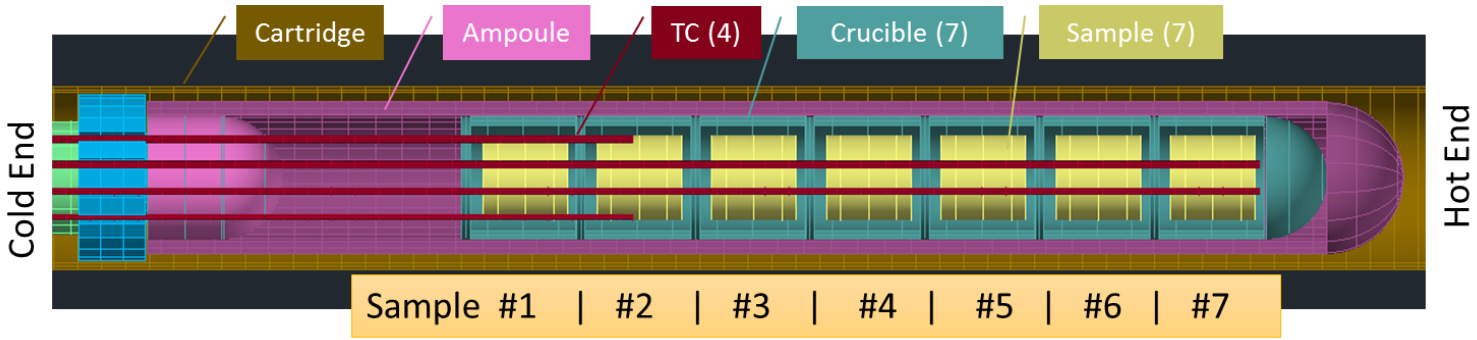
- Thermal Analysis of C5 included evaluation of TC error impact (calculated prior to flight processing)
  - Heater setpoint assumed based on C5 SCA TC at steady state is 1211.5ºC
- The GEDS SCA TC error was applied to the hot zone bore input data used in the GEDS SCA TMM transient analysis
  - The SCA TC error is based on two equal length TCs
  - The two locations resulted in a variation of ± 3.38ºC at each location

Sources of GEDS SCA Platinum Sheath Type S TC Error	
MSL TC Processing Accuracy (±°C)	2.49
MSL RTD Processing Accuracy (±°C)	0.1
Class B RTD (@100 °C) (±°C)	0.8
Type S Special Tolerance (±0.25%) and two TCs at location (±°C)	2.14
Σ RSS for net SCA TC error (±°C)	3.38

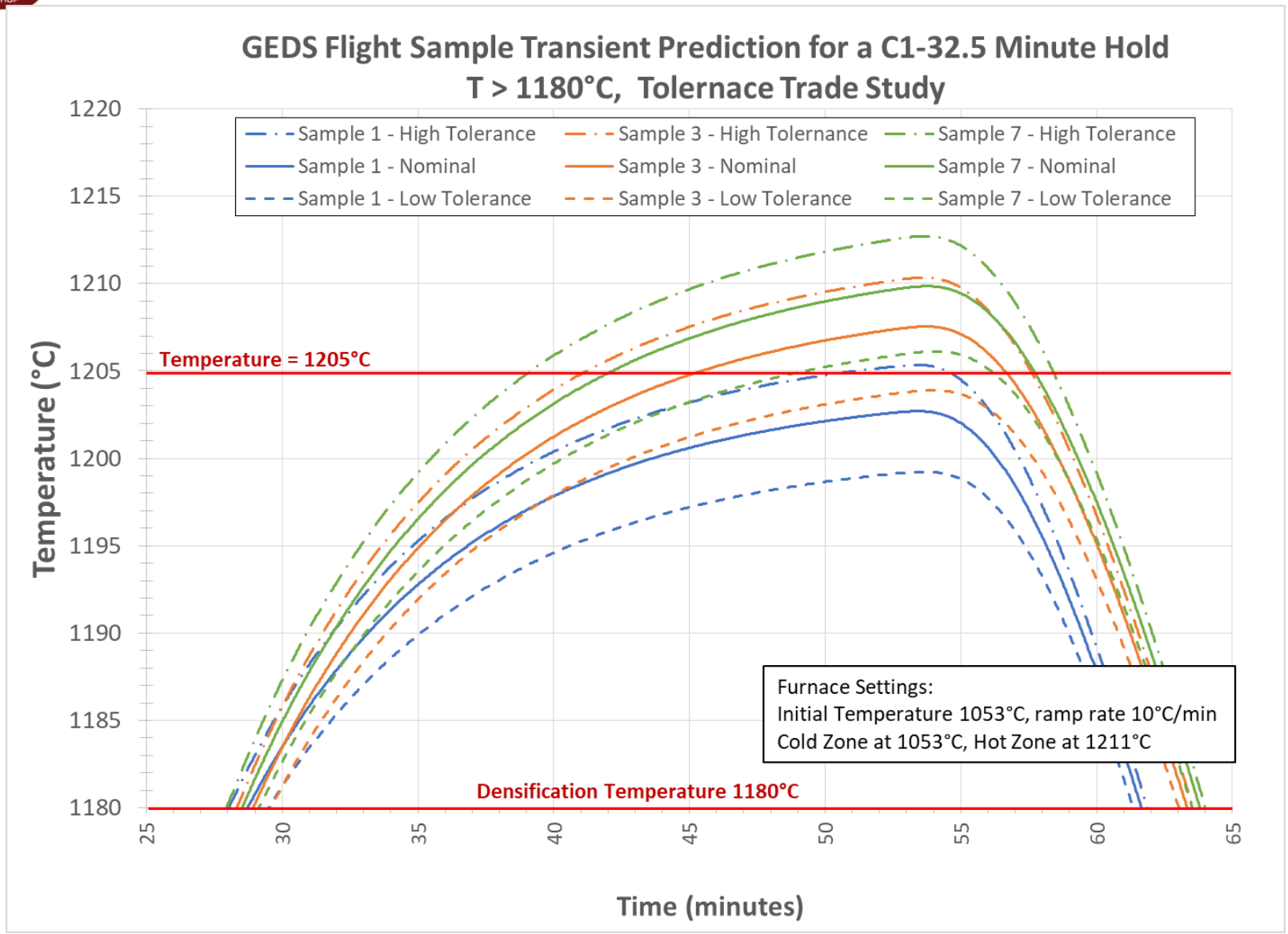
GEDS C5 Sample Response with System Error

- Predicted durations > 1205ºC are nonlinear with application of hot zone temperature error

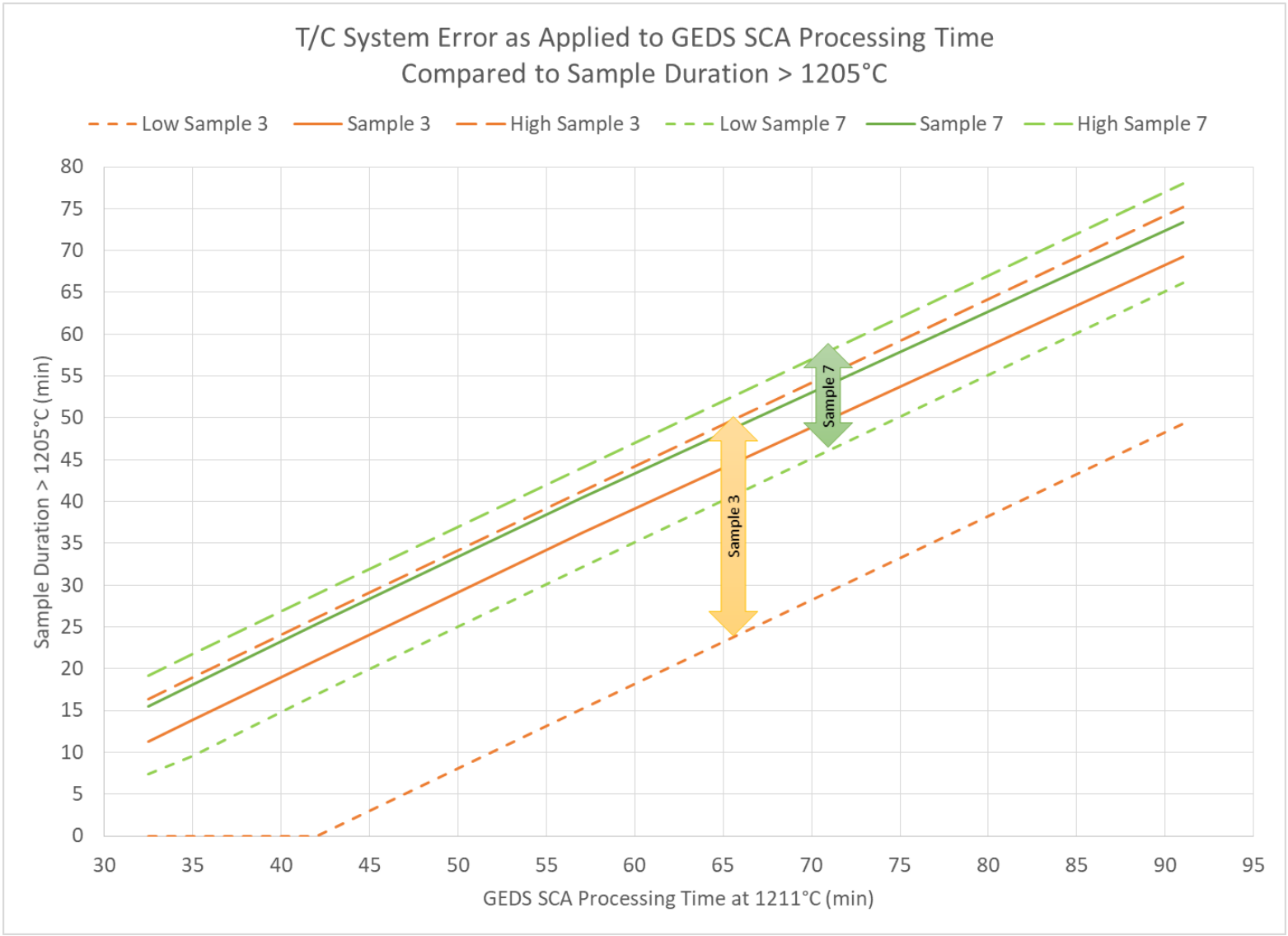
Sample Predictions for C5 with a 91 minute hold	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7
Hot Zone -Δ3.38°C input Sample Tmax (°C)	1200.5	1203.5	1205.4	1206.6	1207.3	1207.6	1207.8
Nominal Hot Zone input Sample Tmax (°C)	1204.0	1207.1	1209.2	1210.4	1211.1	1211.5	1211.7
Hot Zone +Δ3.38°C input Sample Tmax (°C)	1206.7	1209.9	1212.0	1213.3	1214.0	1214.4	1214.6
Hot Zone -Δ3.38°C input Minutes Sample > 1205°C	none	none	49.3	61.0	64.0	65.4	66.2
Nominal Hot Zone input Minutes Sample > 1205°C	none	63.3	69.3	71.4	72.4	73.0	73.4
Hot Zone -Δ3.38°C input Minutes Sample > 1205°C	62.7	72.2	75.2	76.6	77.3	77.7	78.0



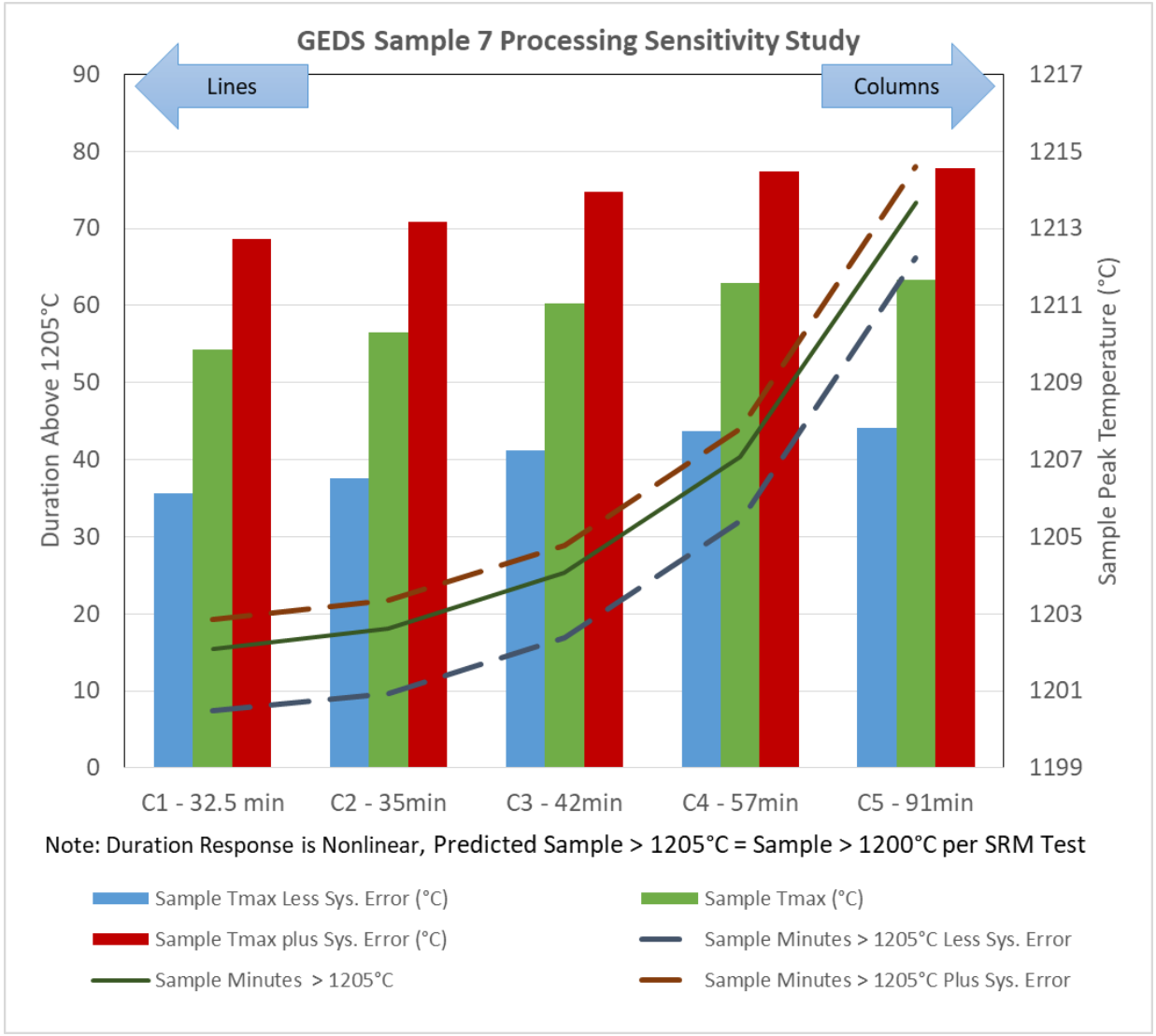
GEDS TC Error Impact on Sample Processing



GEDS TC Error Impact on Sample Processing



Sample 7 (Hot End) Response for Transient



Discussion of TC Error Impact on Predicted Sample Response

- Key parameters for GEDS science
  - All SCAs processed at the same temperature for different durations
  - Analysis showing each samples' temperature time profile for energy integration
- SCA TC error variation
  - Calculated error for a specific GEDS SCA is ± 3.38ºC
  - Actual SCA TC range was better at Δ4.14ºC during processing steady state at 1223ºC
- SCA TC error impacts the predicted sample response
  - For GEDS C5 using the calculated error of ± 3.38ºC GEDS C5 predicted temperature varied as follows:
    - Sample 7 (hot end) is least error at -10% to +6%
    - Sample 4 (middle) error at -15% to +7%
- TC measurement uncertainties assess whether science goals are met: isothermally and duration at temperature
  - Error decreases using GEDS TC actual variation from flight

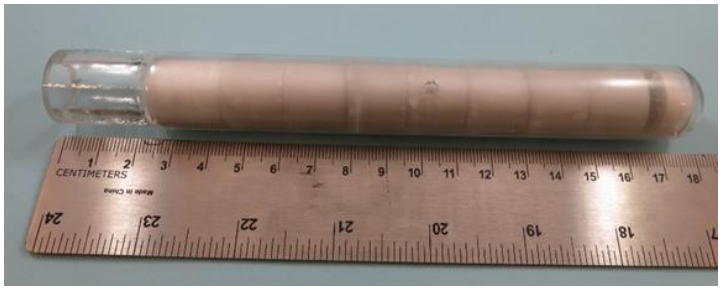
Lessons Learned

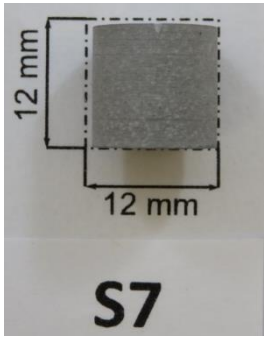
- Short duration experiments such as GEDS require transient analysis to predict sample response-
- Furnace control TC drift has contributed to the uncertainty in transient sample response
  - Response variation for steady state at the same heater settings is manifest as variation in predicting sample transient response
- Additional SCA cartridge out gassing mitigation techniques are required
  - This is being addressed for GTCS and all future PIs with additional cartridge bake-outs prior to assembly

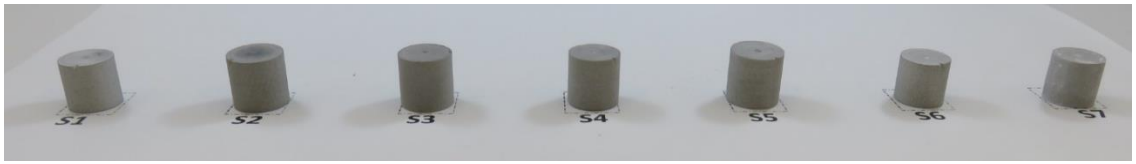
Forward Work

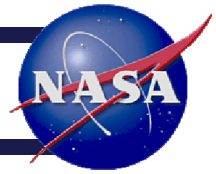
- Revise analysis for each of the GEDS run with the associated flight data to predict sample transients
- Perform analysis considering flight TC variation
- Assess if any drift was experienced by the furnace TCs over the duration of GEDS processing
- Document post flight GEDS analysis by the return of the GEDS SCAs in December of 2020

GEDS C5 – Post-Flight







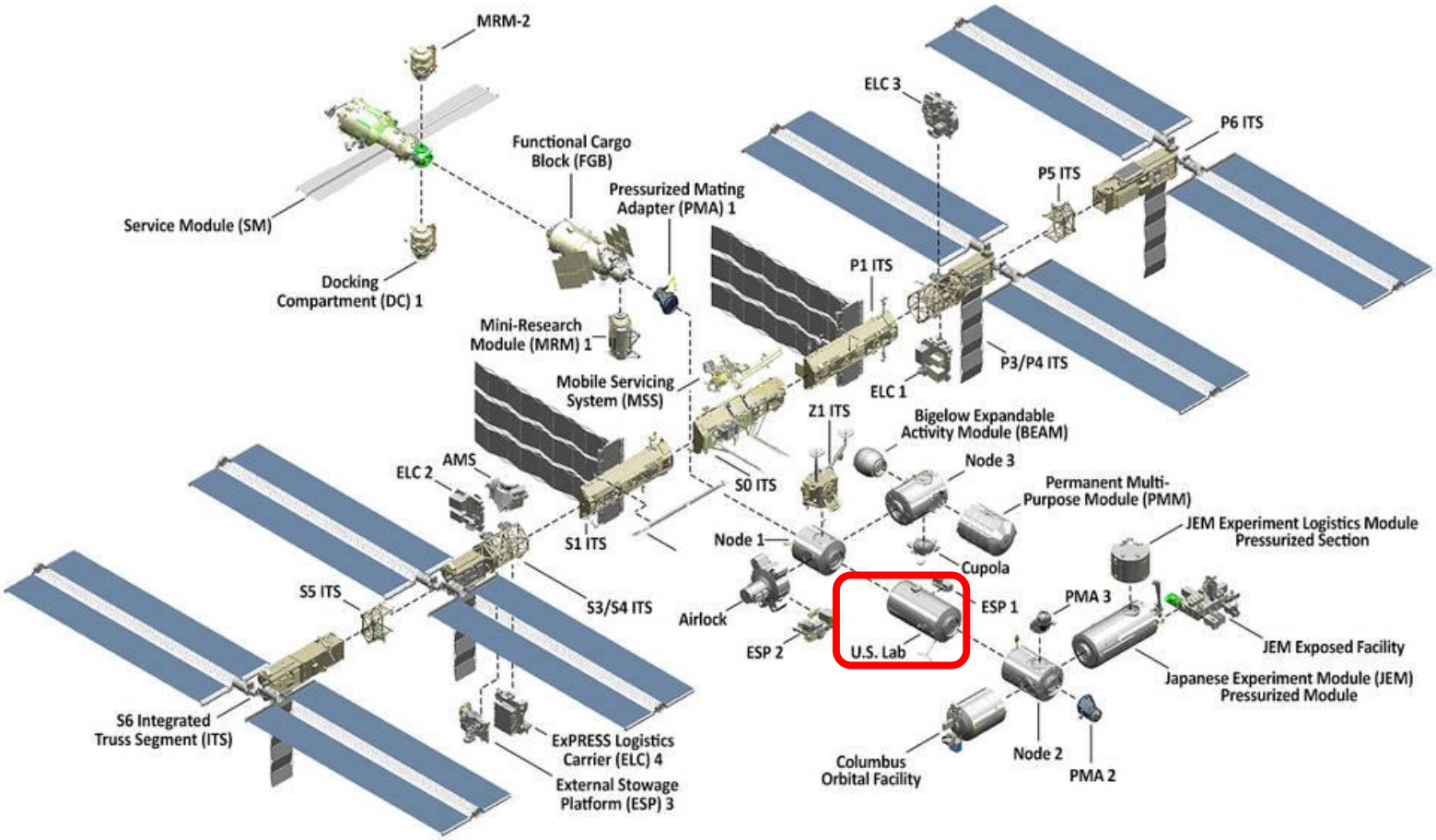


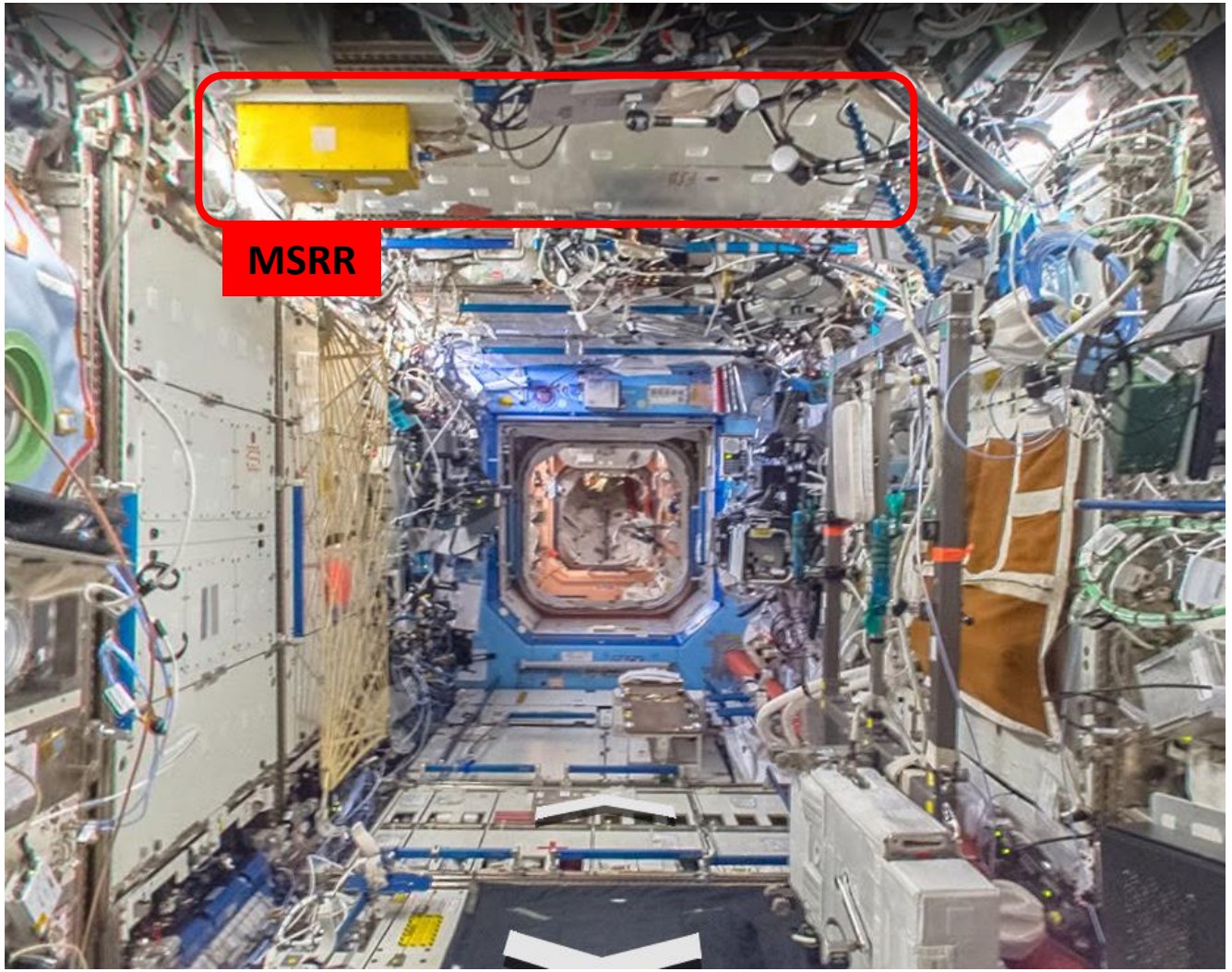
Questions?

Backup

Backup Charts

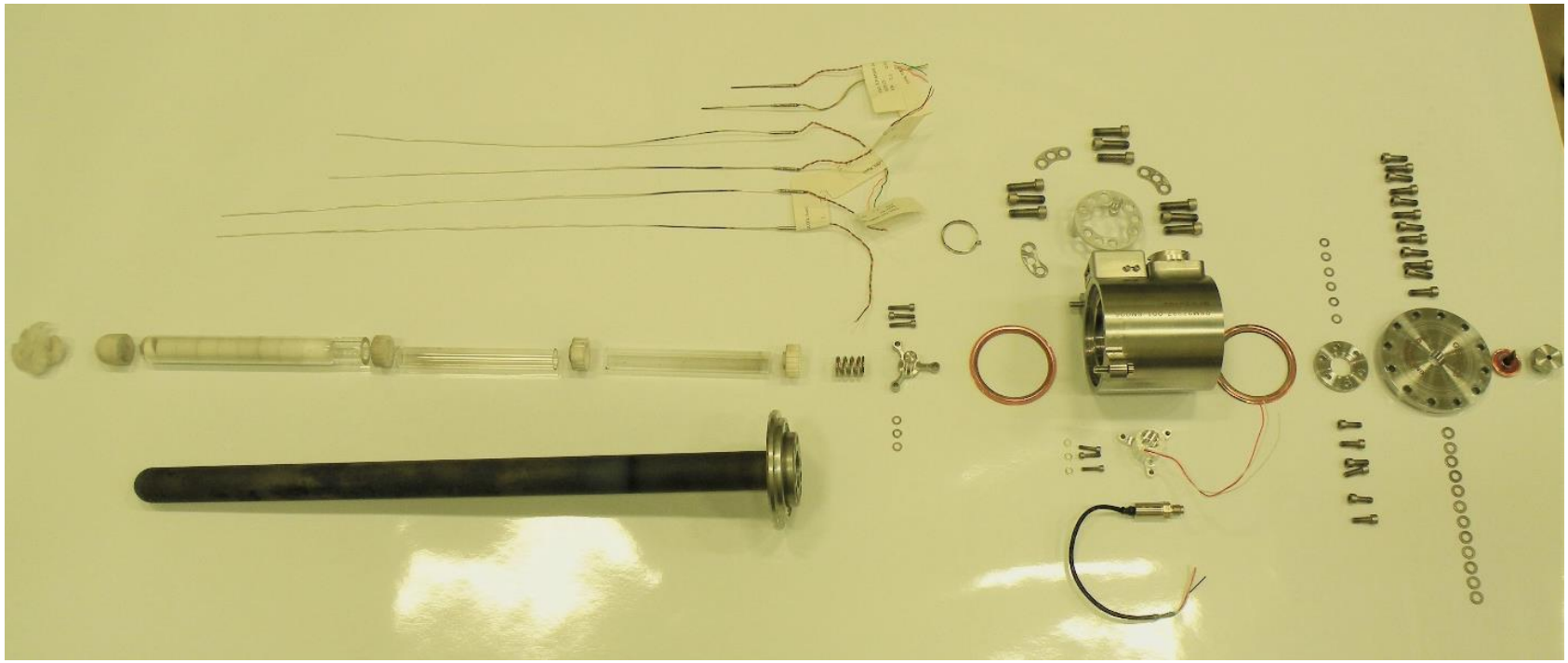
MSRR, MSL, and the Furnaces Location





This is am image inside the U.S. Lab. MSRR is outlined in Red to show its location. The image was taken from ISS on Google Earth. Note that the bicycle is located near MSRR (bottom right of image).

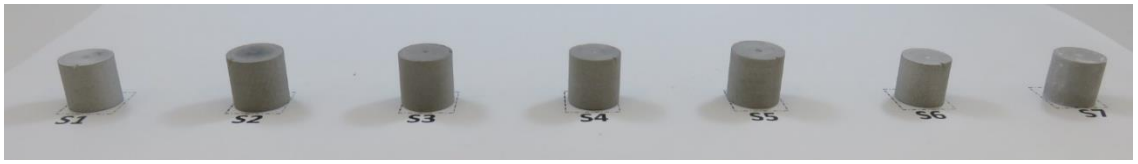
GEDS C5 Post Flight SCA Disassembly

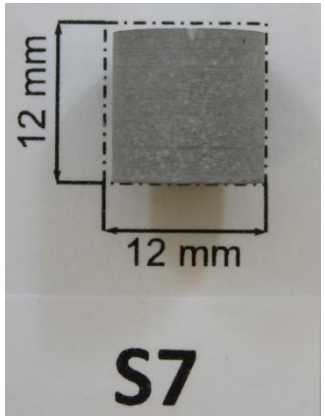


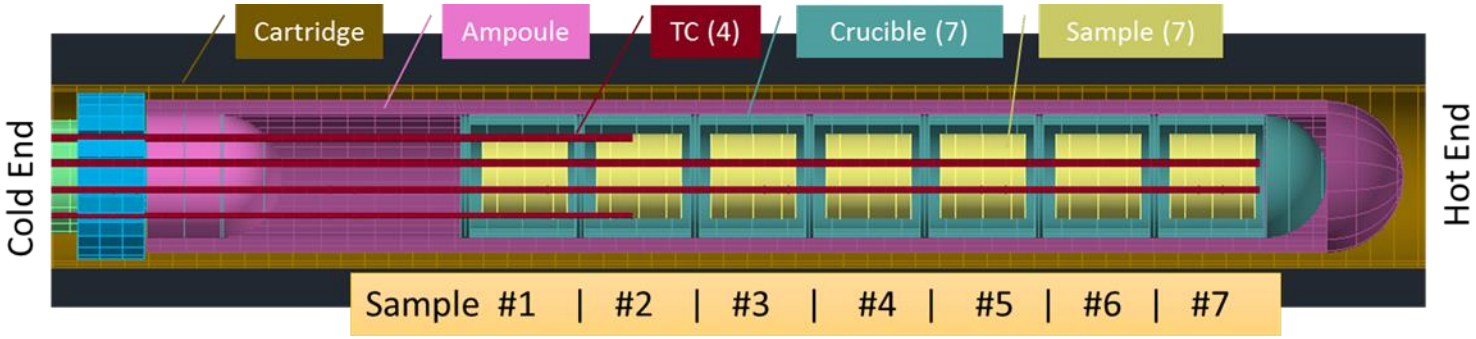
- This is the disassembly/exploded view of the GEDS C5 SCA post-flight

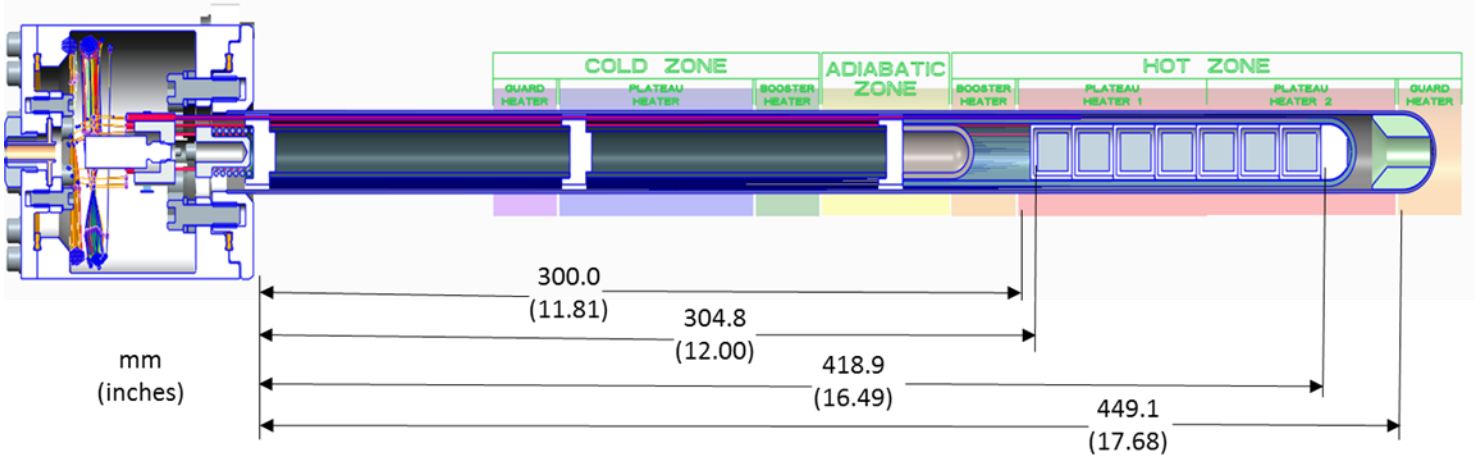
GEDS C5 SCA – Ampoule and Samples

GEDS C5 –
Post-Flight





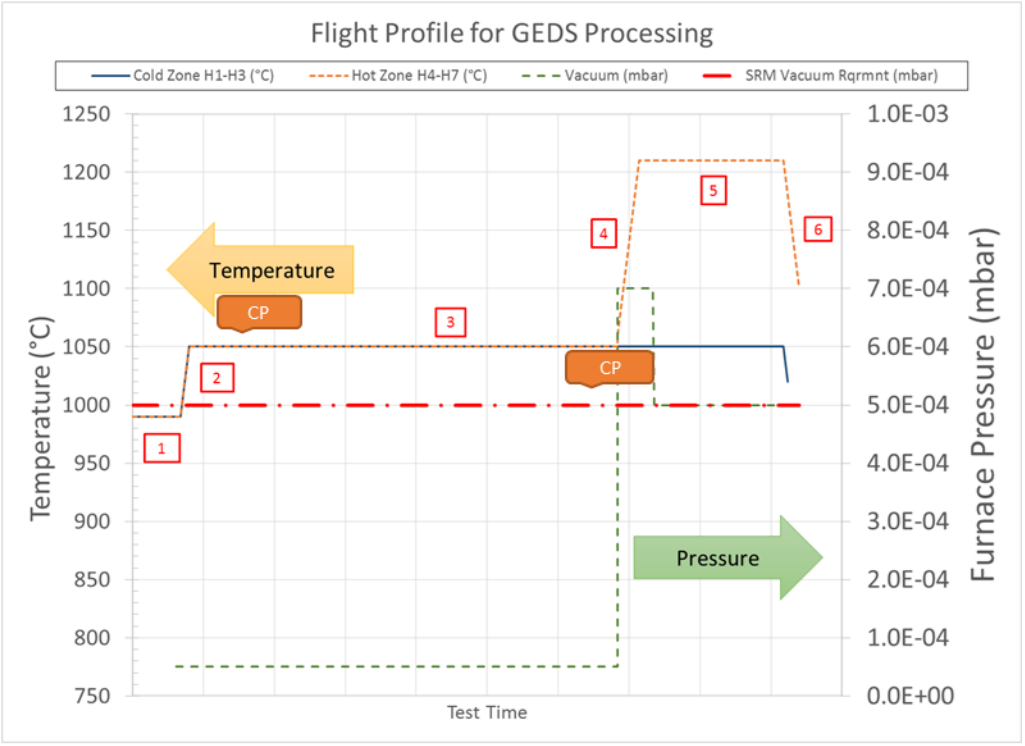

Cartridge
Ampoule
TC (4)
Crucible (7)
Sample (7)
Cold End
Hot End
Sample #1 | #2 | #3 | #4 | #5 | #6 | #7


COLD ZONE
GUARD HEATER
PLATEAU HEATER
BOOSTER HEATER
ADIABATIC ZONE
HOT ZONE
BOOSTER HEATER
PLATEAU HEATER 1
PLATEAU HEATER 2
GUARD HEATER
300.0
(11.81)
304.8
(12.00)
418.9
(16.49)
449.1
(17.68)
mm
(inches)

Actual GEDS C5 Processing Profile November 2019

1. Heating ramp to 990°C at 5°C/min. Hold at 990°C for furnace temperature stability ± 10°C + 10 minutes
2. Ramp hot zone to 1050°C at 5°C/min, increased to 1063°C based SCA TC readings
3. Hold until LGF pressure = 6.3 10^{-5} mbar
4. Ramp hot zone to 1223°C at 10°C/min
5. Hold at 1223°C for 91 minutes for GEDS C5 where LGF pressure remained < 5 x 10^{-4} mbar
6. Ramp down hot zone to 1100°C at 10°C/min

Note: Control temperatures were higher than SCA steady state TC temperatures and were adjusted to allow the SCA TCs to reach the 1210°C set point at thermal direction

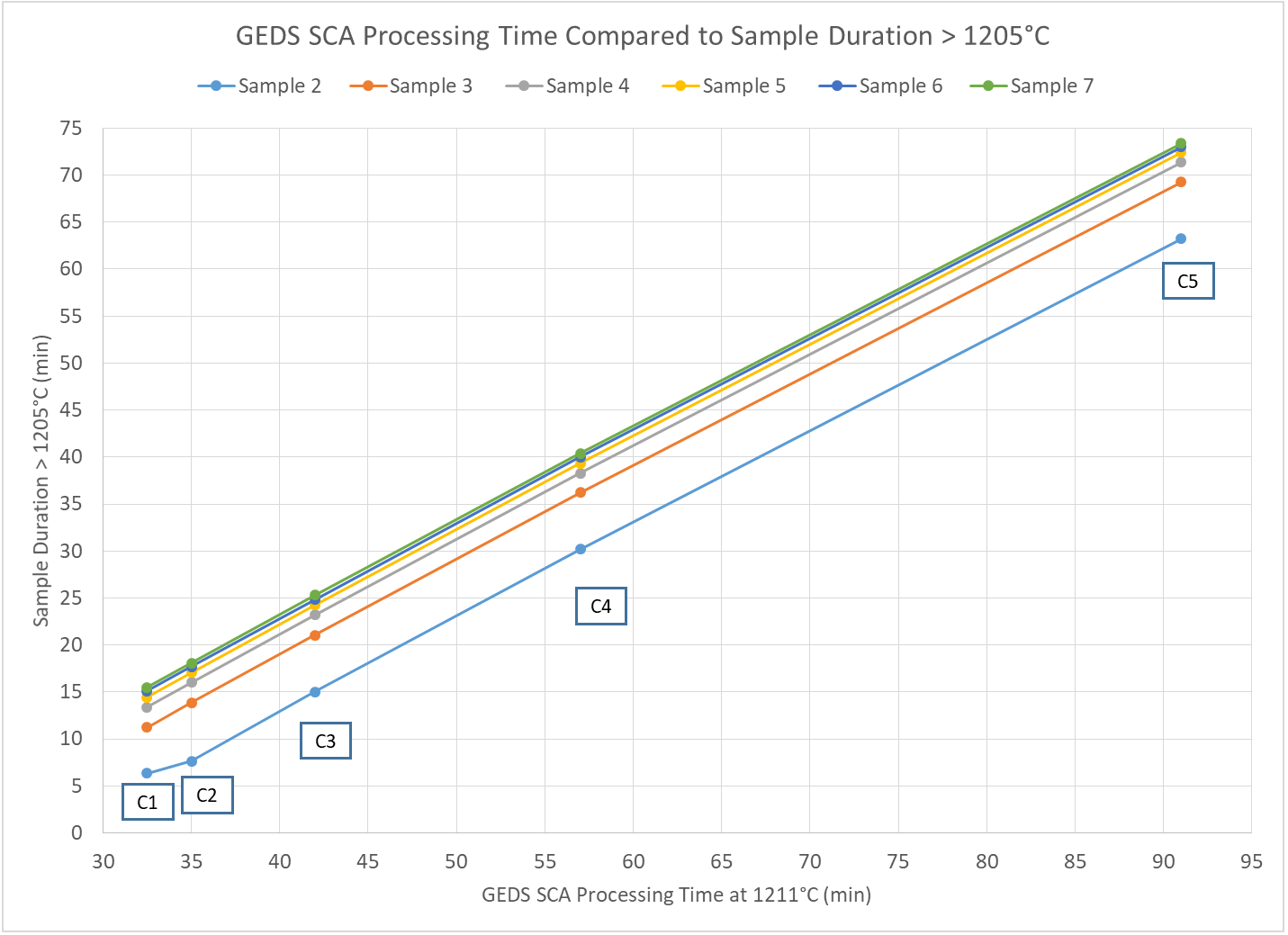


CP = Control Point: Evaluation by GEDS team

LGF Vacuum Requirement: If LGF Pressure = 5 x 10^{-4} mbar no furnace heating is allowed until LGF Pressure < 3 x 10^{-4} mbar

GEDS TMM Predicted Sample Processing Comparison

- Analysis predictions where the duration above 1205°C that approximates the sample time above 1200°C based on ground sample evaluation
- Analysis model does not simulate sintering but assume homogenous sample properties



Calculation of GEDS SCA TC Error

- SCA TC error is based on two TC readings per location
  - Based on TC data fro C5 hold steady state at 1211.5˚ C (average)

$$\sigma_N = \sqrt{\frac{\Sigma(x-\mu)^2}{n}}$$

Where:

- x-μ=1211.5˚ C*0.25%
- n = 2

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MSL RTD Processing Accuracy (±°C)	0.1
Class B RTD (@100 °C) (±°C)	0.8
Type S Special Tolerance (±0.25%) and two TCs at location (±°C)	2.14
Σ RSS for net SCA TC error (±°C)	3.38

- The SCA TC error is based on two equal length TCs. The two locations resulted in a variation of ± 3.38˚ C at each location

Reference Appendix A of "Sample Cartridge Assembly (SCA) Gravitational Effects on Distortion in Sintering (GEDS) Pre-Flight Ground Test G2 and Flight Thermal Analysis Predictions," Deborah Hernandez, ES22-17-110, September 28, 2017

Calculated Error Analysis for GEDS C5