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HEAT CAPACITY STUDY OF DUST SAMPLES CONTAINING ARSENIC TRIOXIDE

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August 2004

CERL Report 2004-31 (CF)

Work performed for:
Indian and Northern Affairs Canada
Giant Mine Remediation Project Team

Natural Resources Canada | Ressources naturelles Canada

Canada

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Abstract

Dust samples containing arsenic trioxide (As_2O_3) were analyzed using Differential Scanning Calorimetry (DSC) to determine their heat capacity. Measurements were conducted in accordance with ASTM Standard Method E1269-01.

Introduction

Differential scanning calorimetry can be used to provide a fast, simple method for determining the specific heat capacity of materials. The specific heat capacity of dust samples containing As_2O_3 can be determined using a standard test method consisting of heating the dust sample at a controlled rate in a controlled atmosphere through the temperature region of interest. The difference in heat flow between the sample and a reference material due to energy changes in the sample is continuously monitored and recorded.

Experimental

Three different dust samples were provided by SRK Consulting and used without purification.

A TA 5200 Thermal Analysis System with a 2910 DSC module and a Du Pont Instruments Mechanical Cooling Accessory was used to determine the specific heat capacity of the dust samples following ASTM standard test method E 1269-01 [1]. Hermetic Al pans containing samples of the dust were held isothermally at –10 °C for 5 minutes, then heated from –10 to 10 °C at a heating rate of 5 °C min^{-1}. At the end of each run, the sample was held isothermally for an additional 5 minutes before the run was terminated. Sample size varied from 9 mg to 20 mg between samples due to the varying densities of the different samples. For the sample supplied with the label "Mar 0348 2/5 C-212-2(168-189) March 25/04 T38.4" (henceforth referred to as Sample A), 20.0 ± 0.3 mg was used in each run. For the sample labelled "Mar 0348 3/5 old feed T38.4" (Sample B), 8.94 ± 0.02 mg was used in each run. For the third sample, "Mar 0348 5/5 B233-P9 T38.3" (Sample C), 9.6 ± 0.6 mg was used in each run. Sample size was determined such that the sample container held the maximum amount of sample. Each sample was run in duplicate. The DSC was calibrated for heat flow [2] and temperature [3]. Synthetic sapphire was used as a reference material.

Results and Discussion

Originally, attempts were made to determine the specific heat capacity of the samples for the temperature region of –10 to 50 °C. However, in the initial run on Sample C, a large exothermic peak was observed between 40 and 50 °C and a small endothermic peak in the 20 to 25 °C region. It was therefore decided to determine the specific heat capacity only for the temperature region –10 to 10 °C. The DSC thermal curves for the samples are shown in Appendix A.

The thermal curves of the samples were compared with that of the sapphire reference to determine the specific heat capacities of the dust samples. The calculations used for the determination of specific heat capacity can be found in the ASTM method E 1269-01 [1]. The plots of the specific heat capacities obtained for the samples are shown in Figure 1. Error analysis for each sample can be found in Appendix B. Variation between duplicate runs can be attributed to the inhomogeneity of the samples. Variation between samples may be due to the different composition of the samples. The specific heat capacity for Sample A was significantly lower than the heat capacities for Samples B and C. Taking into account the error, there is no significant difference in the specific heat capacity values of Samples B and C.

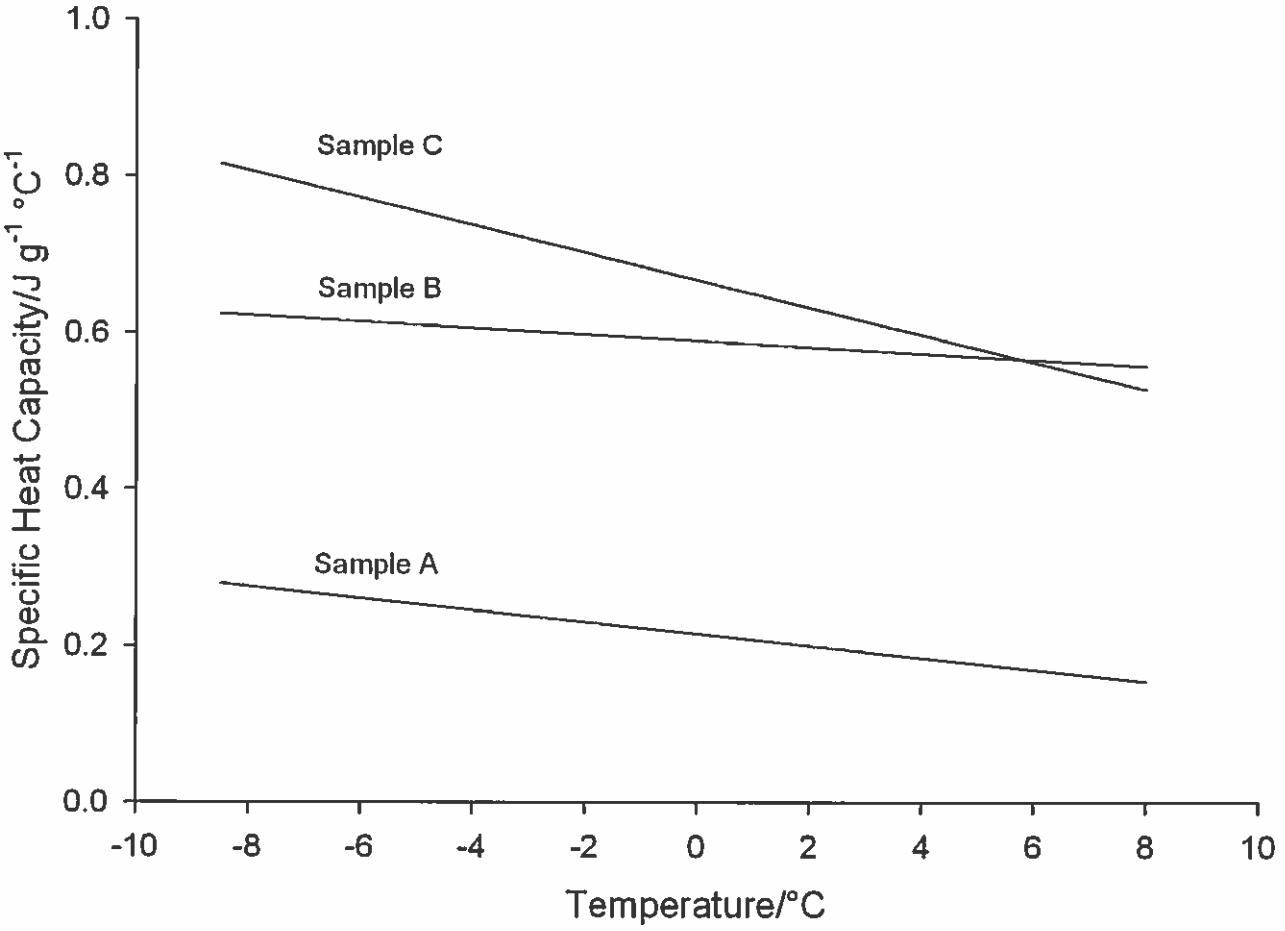
References

1. ASTM E 1269-01, Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry, American Society for Testing and Materials, Philadelphia, PA, U.S.A.

2. ASTM E 968, Standard Practice for Heat Flow Calibration of Differential Scanning Calorimeters, American Society for Testing and Materials, Philadelphia, PA, U.S.A.

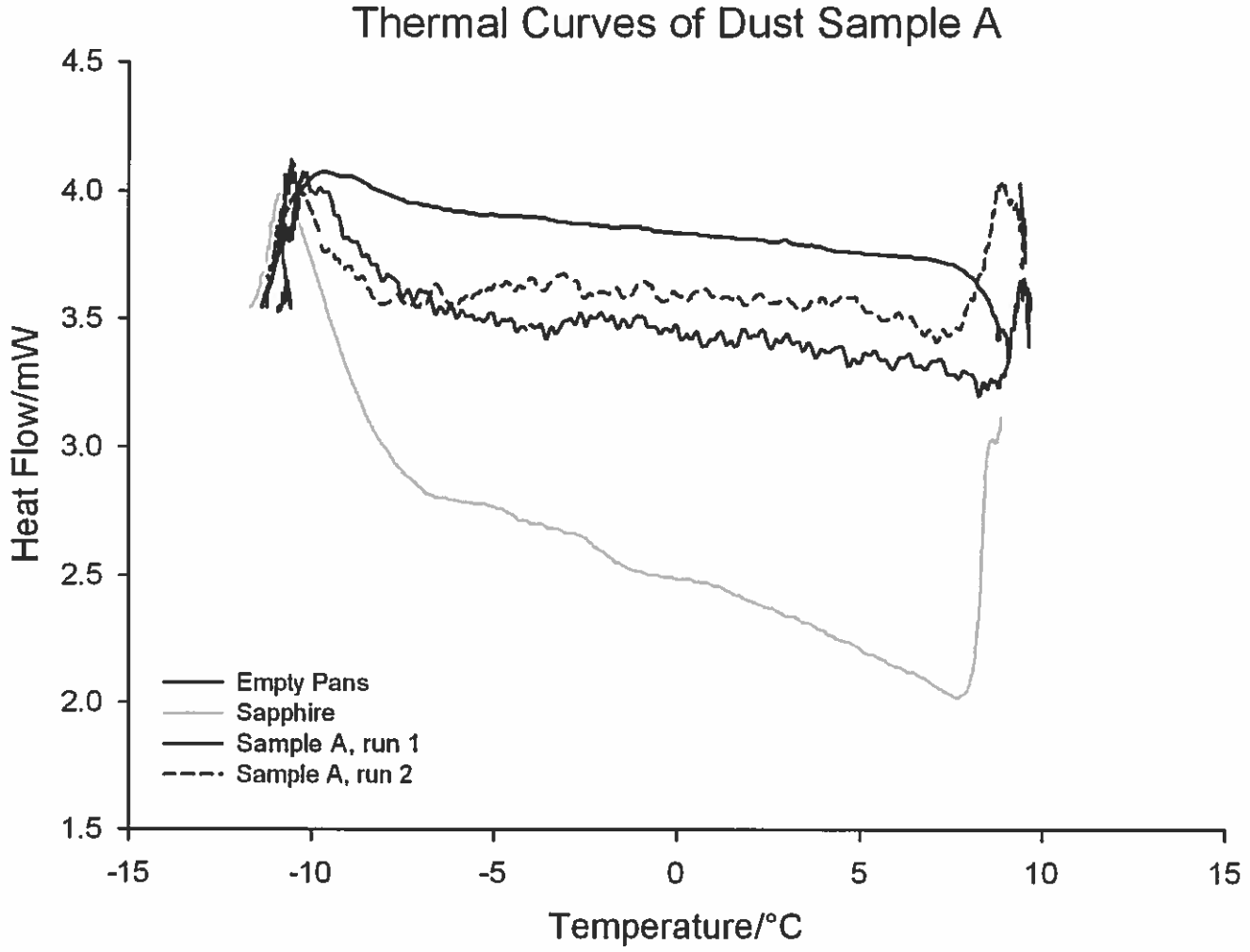
3. ASTM E 967, Standard Practice for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers, American Society for Testing and Materials, Philadelphia, PA, U.S.A.

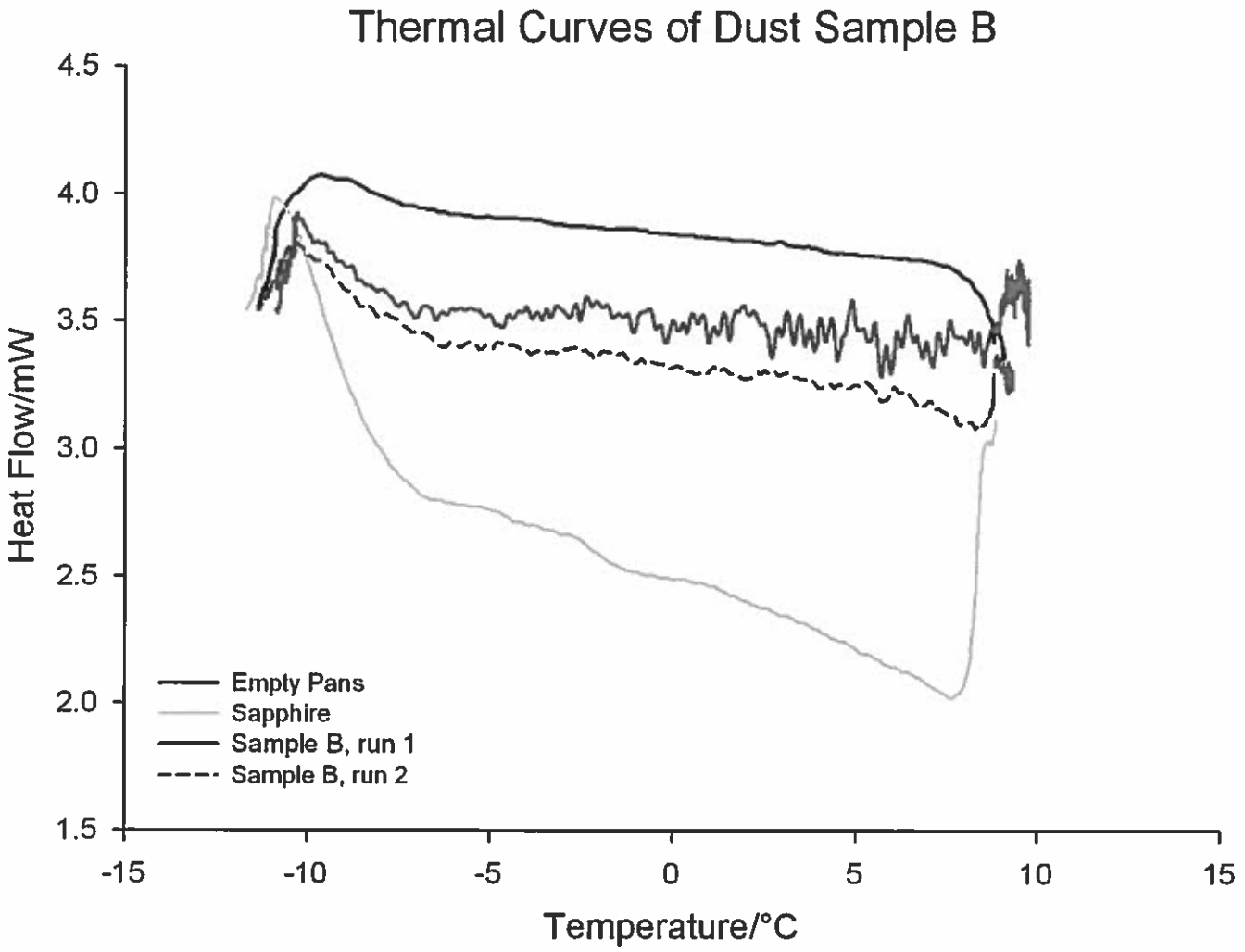
Figure 1. Specific Heat Capacity of Dust Samples containing Arsenic Trioxide

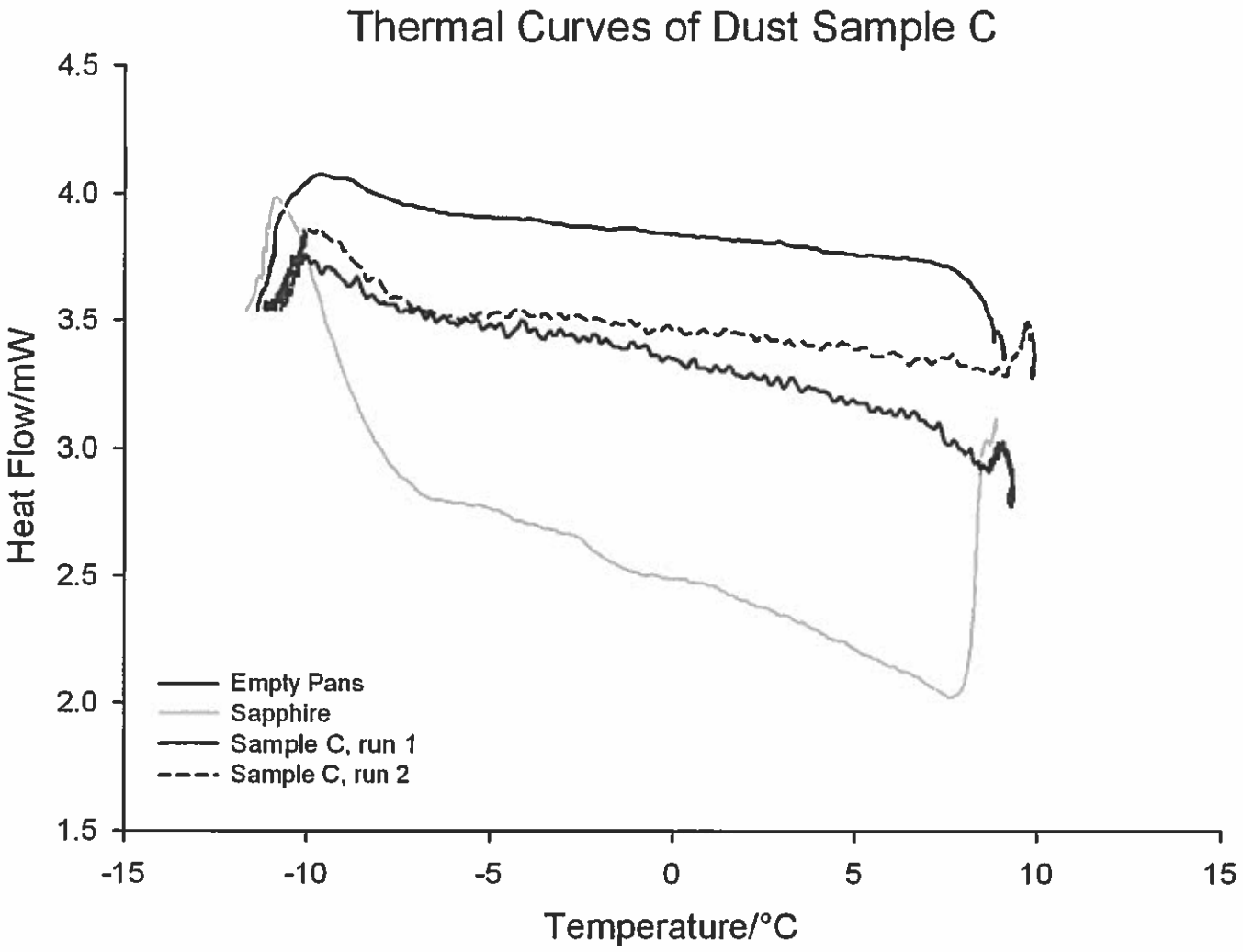


APPENDIX A

Below are the thermal curves for the dust samples containing As_2O_3. Each graph shows the duplicate curves of the sample, as well as the curves for the standard sapphire and the empty sample holders, used to calculate specific heat capacity.

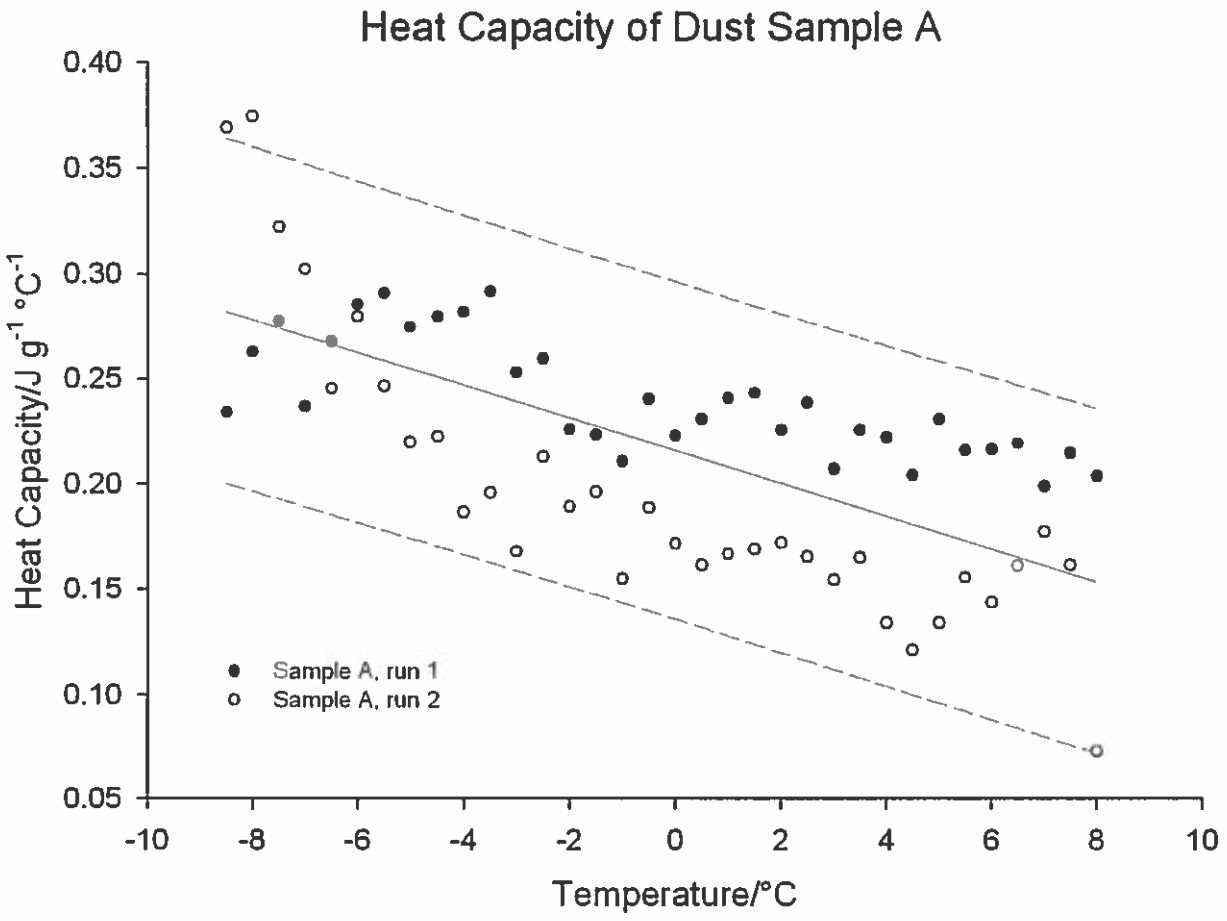


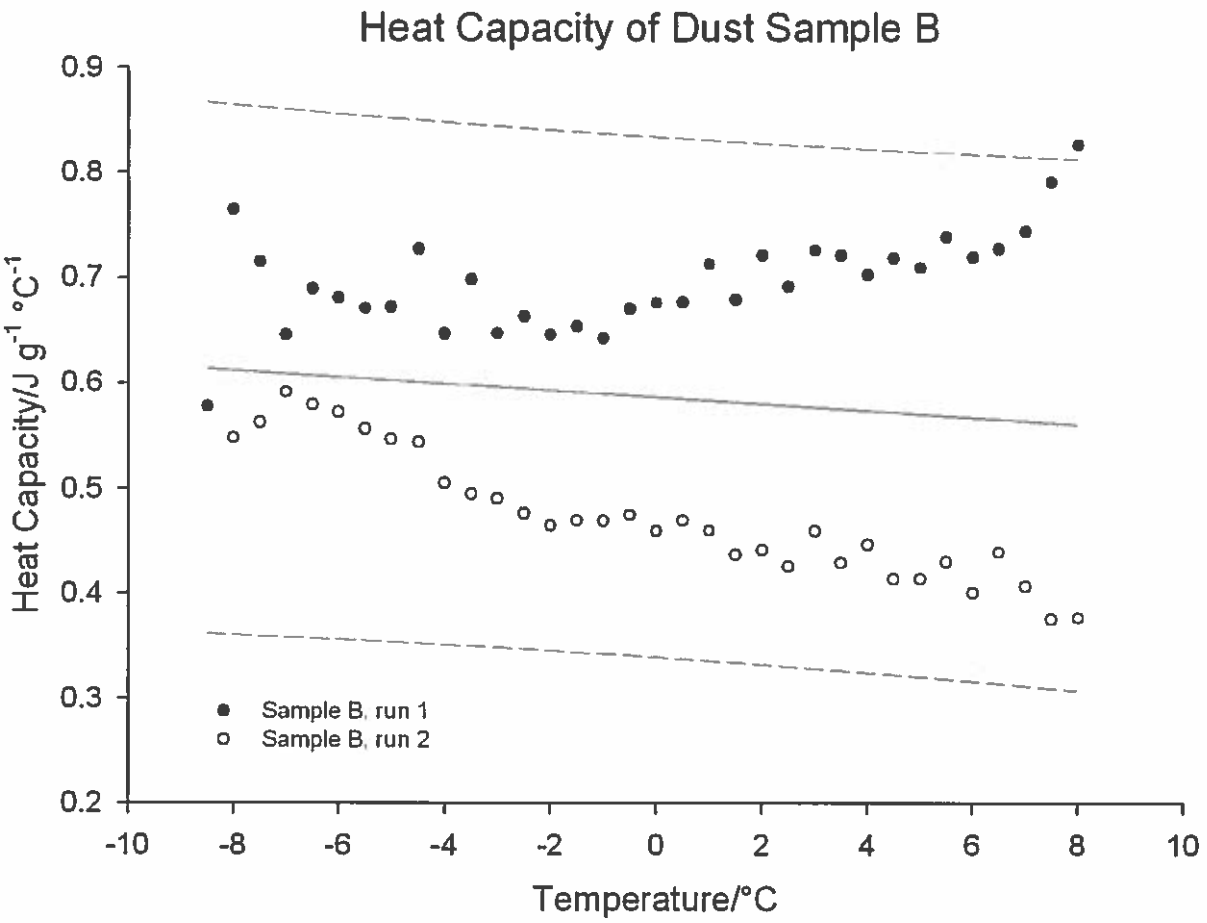

Thermal Curves of Dust Sample B
Heat Flow/mW
4.5
4.0
3.5
3.0
2.5
2.0
1.5
-15
-10
-5
0
5
10
15
Temperature/°C
Empty Pans
Sapphire
Sample B, run 1
Sample B, run 2

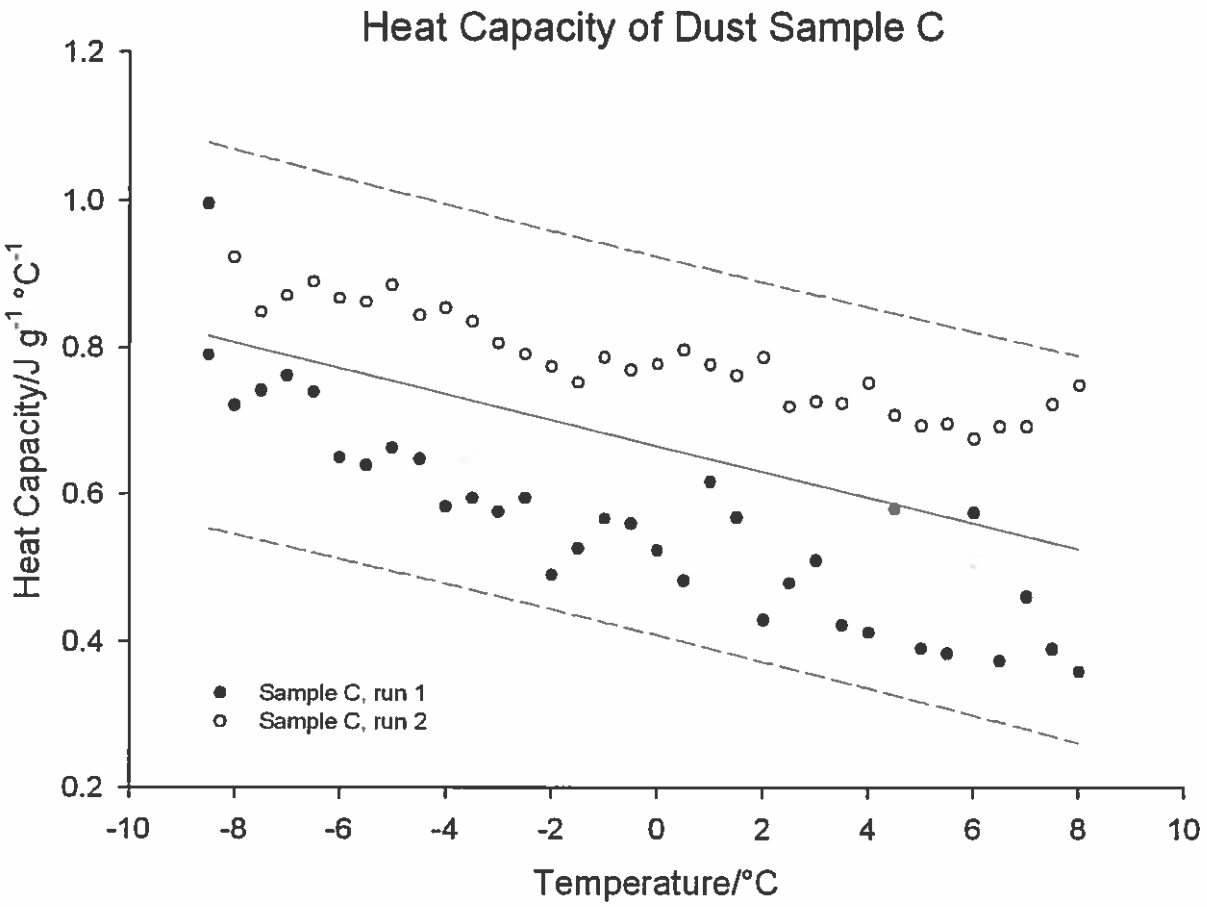

Thermal Curves of Dust Sample C
Heat Flow/mW
4.5
4.0
3.5
3.0
2.5
2.0
1.5
-15
-10
-5
0
5
10
15
Temperature/°C
Empty Pans
Sapphire
Sample C, run 1
Sample C, run 2

APPENDIX B

Below are the individual plots of specific heat capacity for the different samples. The dotted lines indicate 95% prediction limits for the linear regression, represented by the solid line.




Heat Capacity of Dust Sample B
Heat Capacity/J g-1 °C-1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
-10
-8
-6
-4
-2
0
2
4
6
8
10
Temperature/°C
Sample B, run 1
Sample B, run 2


Heat Capacity of Dust Sample C
Heat Capacity/J g-1 °C-1
1.2
1.0
0.8
0.6
0.4
0.2
-10
-8
-6
-4
-2
0
2
4
6
8
10
Temperature/°C
Sample C, run 1
Sample C, run 2