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Scaling terrestrial thermal analysis data for planetary applications

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Abstract

Thermal analysis instruments have been used on past Mars missions to look for organics as well as to identify minerals from their decomposition temperatures and products [1, 2]. The Sample Analysis at Mars (SAM) instrument on the Mars Science Laboratory (MSL) will conduct similar measurements. Size and mass constraints lead to planetary instruments being operated under different conditions than is typical of terrestrial laboratory studies. These different conditions can have a significant impact on the temperature of thermal decomposition, complicating the analysis of planetary data by making comparisons to existing terrestrial data difficult. This work seeks to establish a relationship between instrument pressure and thermal decomposition temperature that could be used to predict decomposition temperatures under different conditions based on existing terrestrial studies. Preliminary results show that a relationship exists that can be used to make predictions for certain classes of minerals.

1. Introduction

Thermal analysis is a general term used to describe the study of how materials change with temperature. Thermal analysis can be used to identify minerals and organic molecules (among other things) based on thermodynamic phase transitions or decompositions. Many different factors can affect decomposition temperatures such as sample particle size, packing density, the mass of sample analyzed, and the particular instrument used for analysis. The relatively wide range of decomposition temperatures for a given mineral generally require complementary analysis in order to make a confident identification of a sample (Evolved Gas Analysis or X-ray Diffraction are two examples).

Thermal analysis in terrestrial labs is generally carried out under ambient pressure with high carrier gas flow rates, generally between 20 and 100 standard cubic centimeters/minute (sccm). Planetary instruments are operated at much lower pressures and flow rates; for example, the TEGA instrument on the Phoenix lander operated at 12mbar N_2 with 0.04 sccm flow and the SAM instrument will operate at 30mbar He with 1.5 sccm flow rates. The lower pressure in particular can have a large effect on thermal decomposition temperatures, generally resulting in lower decomposition temperatures [3, 4]. This effect complicates the identification of any detected signal as comparison to traditional laboratory data is not possible due to the different instrumental conditions. This work seeks to establish a predictive relationship between instrument pressure and thermal decomposition temperature to enable comparisons of signals obtained on planetary missions with those obtained under standard conditions.

2. Experimental Setup

We used a Netzsch STA 449 F1 Jupiter with thermal gravimetry (TG) and differential scanning calorimetry (DSC) capabilities, with the exhaust gas line coupled to a Pfeiffer ThermoStar GSD 320 quadrupole mass spectrometer for simultaneous thermal and evolved gas analysis (EGA). Samples were run at 12mbar (TEGA), 30mbar (SAM), 150mbar (intermediate value), and 1000mbar (terrestrial standard), using He carrier gas with a 3sccm flow rate. Samples were heated to ~1050°C at a ramp rate of 35°C/minute (the planned ramp rate for the SAM instrument). Each sample is run twice and peak temperatures are averaged. About 10mg of sample is used for each run. Samples of kaolinite, montmorillonite, epsomite, kieserite, magnesite, and calcite have been analyzed, representing clay minerals, carbonates, and sulfates.

3. Results

Table 1 lists the minerals studied in order of increasing thermal decomposition temperature for runs conducted at 1000mbar instrument pressure. The transitions for magnesite and calcite are the decomposition of the carbonate mineral and the subsequent release of CO_2. The remaining peak temperatures reported are for water-loss transitions (sulfate decomposition takes place at temperatures at or above the maximum temperatures used in these runs and, therefore, was not measured).

Table 1: Thermal decomposition temperatures of selected minerals at 1000mbar.

Sample	Peak Temperature at 1000mbar (°C)
Epsomite	313.5
Kieserite	405.6
Kaolinite	523.5
Magnesite	628.6
Montmorillonite	763.9
Calcite	807.0

Figure 1 shows how the change in thermal decomposition temperature varies with the initial decomposition temperature obtained at 1000 mbar. This relationship is plotted for the three different pressures used: 12, 30, and 150 mbar. The data are fit to linear trend lines with the R^2 value for each fit displayed below the legend.

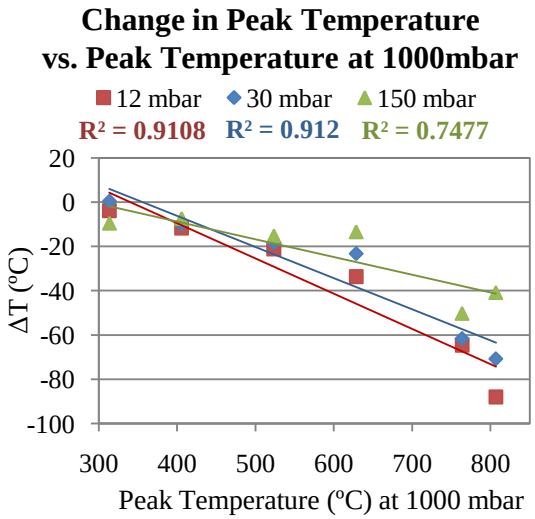

Figure 1: Difference in thermal decomposition temperatures ($T_{lower\ pressure} - T_{1000mbar}$) plotted vs. the temperature at 1000mbar.

4. Summary and Conclusions

These data show that establishing a predictive relationship between instrument pressure and thermal decomposition temperature is possible, at least for certain types of minerals. The nature of thermal analysis is such that in order to identify a mineral with a high degree of confidence, the particular mineral in question should be analyzed under conditions as similar to the flight instrument as possible. However, the predictive relationship established here is useful as a first cut, allowing the existing data conducted under standard terrestrial conditions to be used to narrow down the list of possible minerals that will be run subsequently under flight-like conditions.

This model will be further refined by performing similar analyses on more carbonate, sulfate, and phyllosilicate minerals. Furthermore, peak data from EGA results from these runs will also be analyzed to see if the results match those obtained by DSC. This is important as the SAM instrument on MSL does not have DSC capabilities and relies solely on EGA for mineral analysis.

References

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