Measuring Noble Gases in Coma Samples from Comet Wild 2

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ABSTRACT

Since comets originated during the formation of the solar system, the processes of solar system formation can be better understood through compositional analysis of cometary material. A low density, silicon-based substance called aerogel was used by NASA's Stardust spacecraft to collect coma samples from comet Wild 2. Aerogel not from the spacecraft ("non-flight") was investigated to determine the possibility of measuring noble gases in Stardust samples. Gas evolved from heated, non-flight aerogel was measured initially using a residual gas analyzer, then a high-sensitivity mass spectrometer. Levels of helium and neon isotopes observed from both instruments were sufficiently low that noble gases from Stardust samples were measured using the same technique. Intrinsic helium and neon was not identified in flight aerogel without apparent cometary material. Helium and neon were detected above background in flight samples containing impact particle tracks.

INTRODUCTION

Material not incorporated into the sun formed a protoplanetary disk from which the planets and other solar system bodies condensed. The compositions of these bodies should be similar to that of the sun under the assumption that solar and disk material came from a common source. However, the compositions of bodies throughout the solar system are known to be wildly different from one another. These differences are attributed to physical and chemical processes, but the exact sequence of events that occurred to form the solar system remains unknown.

Studying the evolution of the solar system requires knowing what the early solar system was like compositionally. Elemental abundances and isotopic compositions from numerous planets and satellites have been studied, so constraints can be put on current formation theories by comparison with information from early solar system materials. The inert noble gases are of particular interest because they are good indicators of elemental and isotopic mass fractionations caused by chemical and physical processing (Pepin 2006).

Characterizing noble gases in the protoplanetary disk requires finding a source of material that has remained unchanged since that period. Comets are an important resource in that they are artifacts from the formation of the solar system, and were accordingly the focus of NASA's Stardust mission. This mission was designed mainly to collect samples of material from comet Wild 2 and return them to earth for analysis. Far from the sublimating effect of solar radiation, comet Wild 2 has spent most of its life well-preserved in the outer region of the solar system. A recent interaction with Jupiter decreased Wild 2's perihelion distance, conveniently bringing the comet closer to earth for study. The Stardust spacecraft encountered the nucleus of comet Wild 2 to collect samples of coma gas and dust by impact into aerogel, a very low density, silicon-based material that has been shown to be effective at stopping and preserving high velocity particles. Tracks were observed in the aerogel, revealing the trajectories of the particles as they were captured.

Due to their relative importance as tracers of fractionation and transportation in the solar system, the measurement of noble gas isotopes in Stardust samples was the primary objective in this study. Flight aerogel gas can be classified as either cometary,

intrinsic to the aerogel, or from some other source. Cometary gas was expected to originate from captured particles and particle fragments scattered along the tracks. As an additional cometary gas source, it was anticipated that gas may have been trapped within bubbles formed as particles melted aerogel along the entry path.

EXPERIMENT

The proposed method for obtaining trapped gases from flight samples was to heat the aerogel in a stepwise fashion. The advantage of step heating is that the amount of outgassing in the sample is temperature controlled; more tightly held volatiles are released at higher temperatures. A two-phase approach was undertaken for processing the flight samples: an initial, low temperature heating phase (no more than about 200 °C) to release potential surface gases and a high temperature phase (upwards of 1000 °C) for the release of coma gas. The low temperature phase was important as a means of discarding any contaminating gas that would otherwise compete with the coma gas in the mass spectrometer. Heating has been effective in measuring gas from interplanetary dust particles (some of which are thought to have originated from comets), but it was unknown whether or not noble gas concentrations in non-flight, or blank, aerogel would be low enough to permit detection of gases at the levels anticipated in flight samples.

The feasibility of this technique was first investigated by making measurements of the volatile composition of aerogel block E226-5B with a residual gas analyzer (RGA). This non-flight aerogel was identical to that flown on the Stardust spacecraft. The first sample cut from block E226-5B had an area of ~0.5 cm^2 and was enclosed in a platinum envelope attached to two wire leads on a flange (Figure 1). The flange was attached to a vacuum system and the external leads on the flange were connected to a power supply. The sample was heated from 50 °C to ~1000 °C in eleven steps, each step lasting for one hour. Gas measurements were taken periodically throughout each step with the RGA set to scan over a mass range of 1 to 50 u.

The results from each temperature step were superimposed on a plot of partial pressure versus mass, as displayed in Figure 2. The partial pressures of He, Ne, and Ar in
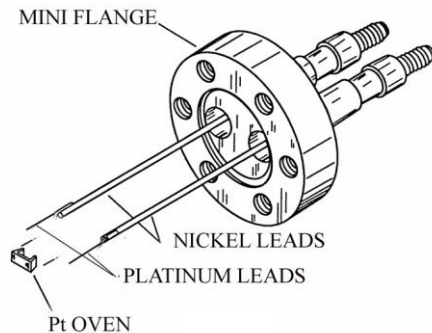
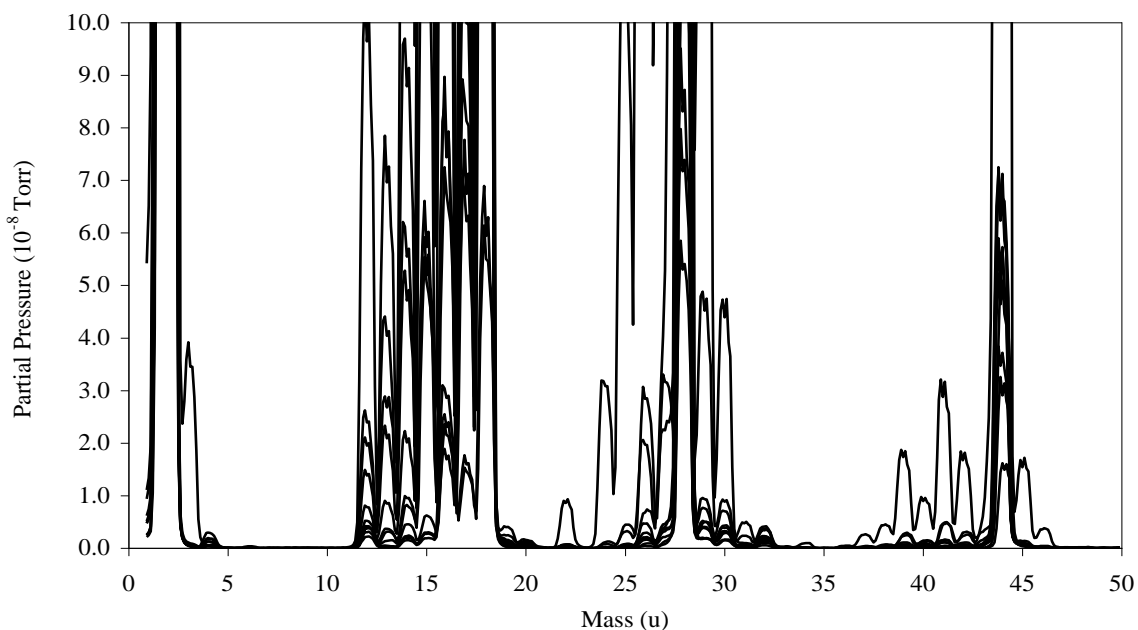Figure 1. The sample holder used during gas extraction.

Figure 2. A composite plot of the RGA partial pressure measurements of gas components in E226-5B for eleven temperature steps from 50 to 1000 °C.

the system never exceeded 10^{-8} Torr at any time during measurement. This amount is negligible relative to the maximum amounts observed for H_2O and CO_2, which were above 10^{-7} Torr, and H_2 and CO, which exceeded 10^{-5} Torr. These results showed that as long as the dominant peaks could be reduced, heating to outgas Stardust samples was worth pursing on a higher sensitivity measuring system.

A second sample was cut from block E226-5B in order to accurately determine the He and Ne composition of blank aerogel. The sample had an approximate area of 1.5 mm^2 and was loaded into a platinum envelope and attached to a vacuum system in the same manner as the first sample. To reduce the amounts of H_2, H_2O, CO, and CO_2, the system was fitted with getters (cartridges of gas-adsorbing alloys), and liquid nitrogen-cooled charcoal. In addition to the adsorption of heavy components, these materials also adsorb Ar. While it was possible to isolate Ar from the charcoal and getters, the amounts of Ar expected were extremely small (if present at all), and in this initial study the focus was He and Ne isotopic compositions.

The sample was heated to 310 °C for 15 seconds, and the evolved gas was purified by the getters and charcoal for 10 minutes. After purification, the gas was analyzed for individual counts of He, Ne, ^{40}Ar, CO_2 and H_2O using a high-sensitivity mass spectrometer. The detection sensitivities of the instrument for ^4He and ^{20}Ne were 4.4 x 10^{-13} cm^3 STP/cps and 1.2 x 10^{-12} cm^3 STP/cps respectively. Eight more steps were performed for temperatures of 640, 840, 970, 1070, 1140, 1200, 1250, and 1330 °C. The gas acquired at each step was allowed to accumulate in the system such that every measurement taken was of a cumulative amount. To determine the amount of indigenous gas in the system that contributed to these measurements, the same piece of aerogel was reheated using the same procedure.

The cumulative amount of He for the heat and reheat runs is plotted as a function of temperature in Figure 3. The amount of He detected during the initial heating was not statistically different from the background (reheat run) for lower temperatures. Helium was detected above background at 1330 °C, suggesting aerogel may have an intrinsic background concentration of He that must be considered when analyzing Stardust samples. The amount of ^{20}Ne measured at each step was corrected for contributions from $H_2^{18}O$ using the ratio $H_2^{18}O/ H_2^{16}O = 2.06$ x 10^{-3}. The water contribution comes from the atmospheric oxygen isotope ratio $^{18}O/^{16}O$. Cumulative Ne is plotted against temperature in Figure 4. The amount of Ne from the initial heating was not statistically different from the background suggesting aerogel does not have an intrinsic Ne concentration at our detection level.
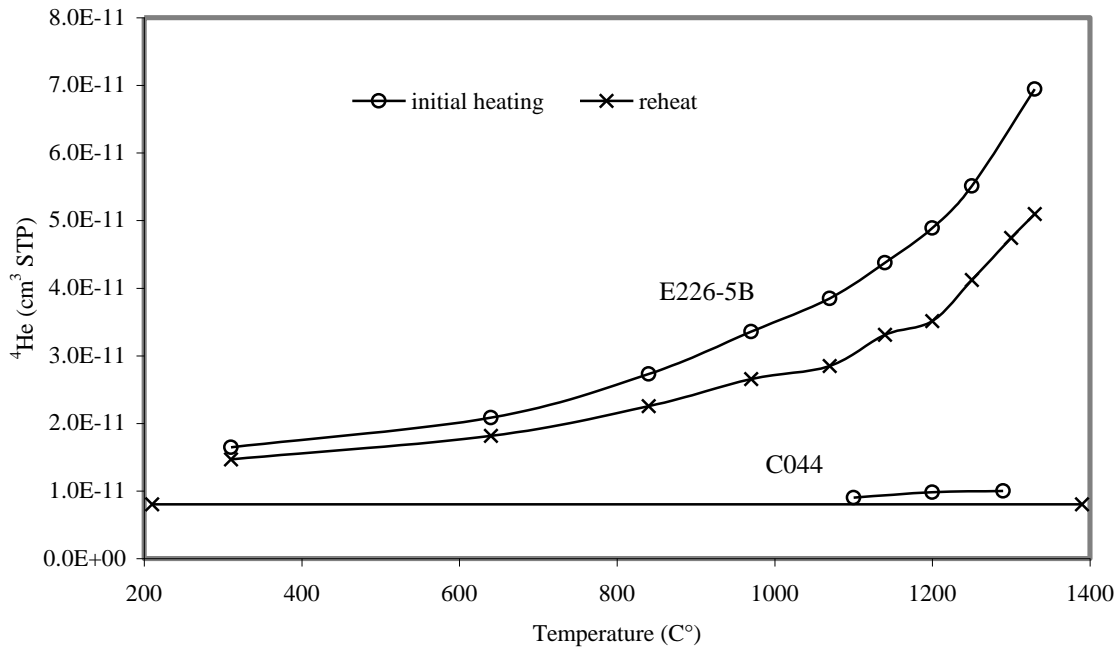
Figure 3. Amounts of ^{4}He obtained from the initial heatings of E226-5B and C044 compared with the respective backgrounds.

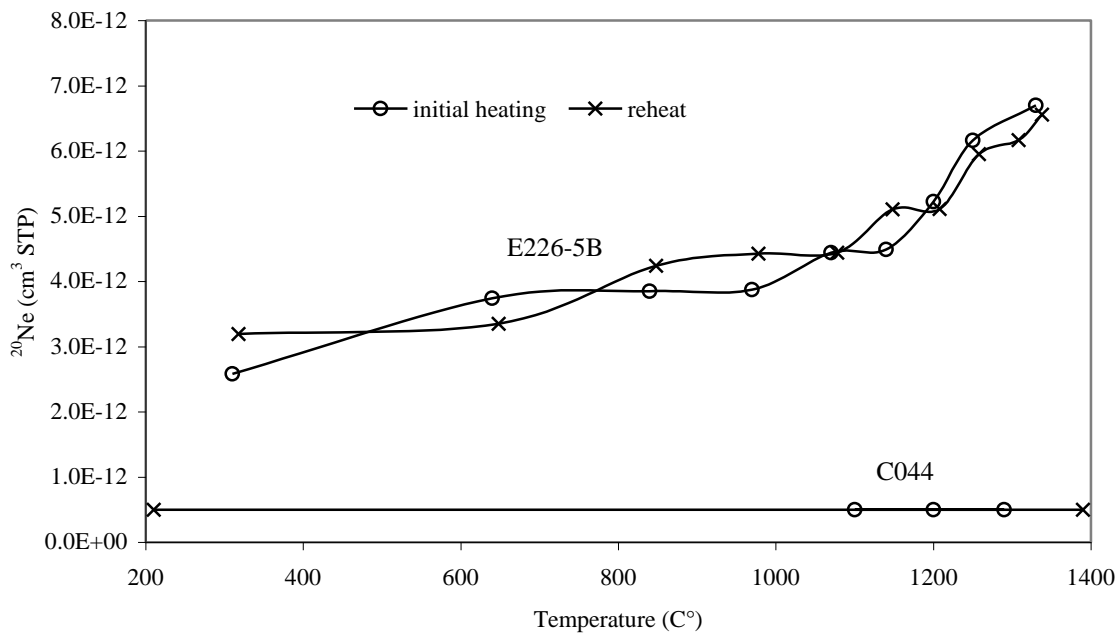Figure 4. Amounts of ^{20}Ne obtained from the initial heatings of E226-5B and C044 compared with the respective backgrounds.

The intrinsic aerogel gas composition was further determined with the analysis of another aerogel sample, Keystone C044. This was flight aerogel, approximately 1.5 mm^3 in volume, and did not contain any apparent cometary material. The sample was initially heated to 200 °C for 20 seconds with 10 minutes exposure of the gas to the getters and charcoal before measurement of the Ne isotopic composition, followed by the same analysis for He. This initial, low temperature heating was done to release any contamination loosely held by the sample and sample holder. The sample gas was liberated in three 15 second heating steps, 1140, 1250, and 1330 °C with a 10 minute purification time after each step. Cumulative gas measurements were taken after the final heating step, in the same order as the low temperature heating. Neon was always measured before He because of the interplay between hydrogen deuteride (HD) and ^{3}He in the system. Hydrogen levels tended to decrease with getter exposure, which was desirable because HD interfered with the measurement of ^{3}He. By making Ne measurements first, the getters had a longer period of time to reduce hydrogen levels, improving the accuracy of ^{3}He analysis for C044.

The sample was reheated using the exact same procedure to obtain a system blank. Before reheating, however, a calibration was run that introduced a large amount of gas into the system. This produced a memory effect that was observed as a He buildup in the mass spectrometer. Test runs of the system were done over the course of several days until He levels decreased to an acceptable level. Keystone C044 was reheated a second time, but there were no Ne data acquired using the measurement program designed for Ne isotopes. Also, only the high temperature phase was repeated. The low temperature phase was replaced by one of the test runs from after the calibration. There was no heating involved in the test run, so measurements were taken at room temperature, approximately 25 °C.

The lower portion of Figure 3 shows the amount of ^{4}He measured in keystone C044 compared with hot and cold background levels. The amount of He from keystone C044 is within uncertainty of the background, so in contrast to the non-flight aerogel analyzed earlier, flight aerogel had no He above detection levels. The C044-to-background comparison for Ne is displayed in the lower portion of Figure 4. As before, corrections were made for $H_2^{18}O$ using the ratio $H_2^{18}O/ H_2^{16}O = 2.06 \times 10^{-3}$. Any Ne

released was well within background, indicating that if cometary Ne is indeed trapped within particles in flight aerogel, it should be possible to detect it.

Samples S1, S2, and S3 were fragments of a Stardust particle track wall, containing melted aerogel and potentially fragments of the original impacting particle. These samples were analyzed individually for cometary gas. Samples S1 and S2 were heated first to ~200 °C during bakeout, followed by additional heatings up to ~250 °C as a clean up procedure. Helium and Ne levels for the samples remained within the system blank levels during this low temperature phase. The samples were then heated from ~250 to ~1075 °C in about ten 15-20 second steps. The system blank for each sample was determined by reheating. No He or Ne was released above background for S1 and S2. Recognizing the possibility that sample gas was lost during the bakout phase for these samples, it was decided that the bakeout would be omitted during the analysis of S3. As with S1 and S2, no He or Ne was observed from S3 upon reaching ~1075 °C. These results are shown in Figure 5, where the ranges of ^{3}He, ^{4}He, and ^{20}Ne obtained from the samples are compared to background levels. The samples were then heated from 1100 to ~1400 °C to make sure that they were completely degassed. Sample S1 began releasing gas at ~1250 °C, and gas release for S2 and S3 was initiated at ~1300 °C (Marty et al. 2007). Figure 6 shows the total amounts of ^{3}He, ^{4}He, and ^{20}Ne obtained from each sample compared to background for the high-temperature phase. Concentrations and compositions of He and Ne are given in Table 1.

Table 1. Amounts and compositions of He and Ne in stardust samples S1, S2, and S3.

Sample	^{4}He (x 10^{-11} cm^3 STP)	^{20}Ne (x 10^{-11} cm^3 STP)	^{4}He/^{20}Ne
S1	7.27 ± 0.36	5.34 ± 0.42	1.36 ± 0.13
S2	5.26 ± 0.30	0.94 ± 0.14	5.60 ± 0.89
S3	≤66.9 ± 2.4	1.06 ± 0.15	≤63.1 ± 9.2

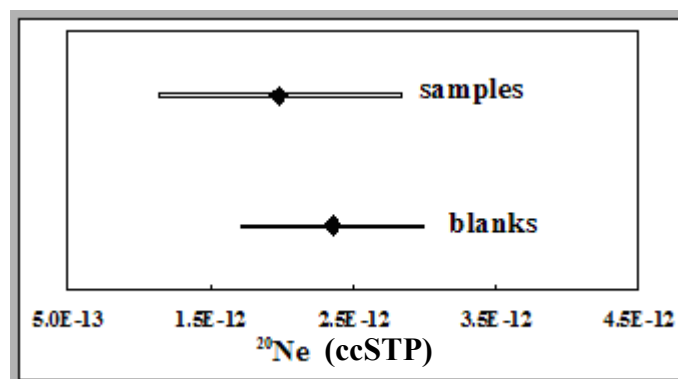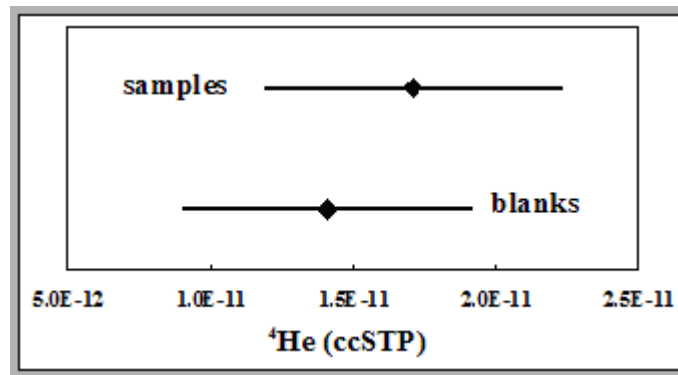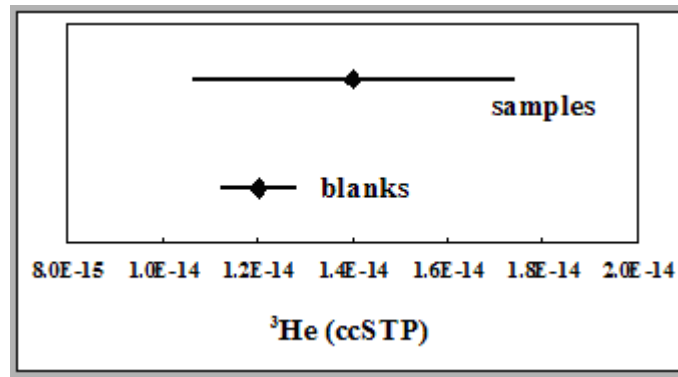| Sample	^{3}He/^{4}He (x 10^{-4})	^{20}Ne/^{22}Ne	^{21}Ne/^{22}Ne
S1	2.92 ± 0.26	10.68 ± 0.35	0.0273 ± 0.0024
S2	2.47 ± 0.34	9.0 ± 1.6	0.039 ± 0.011
S3	≤0.318 ± 0.032	9.3 ± 1.6	0.042 ± 0.018

Figure 5. Amounts of ^{3}He, ^{4}He, and ^{20}Ne from S1, S2, and S3 measured relative to background after heating up to ~1075 °C.
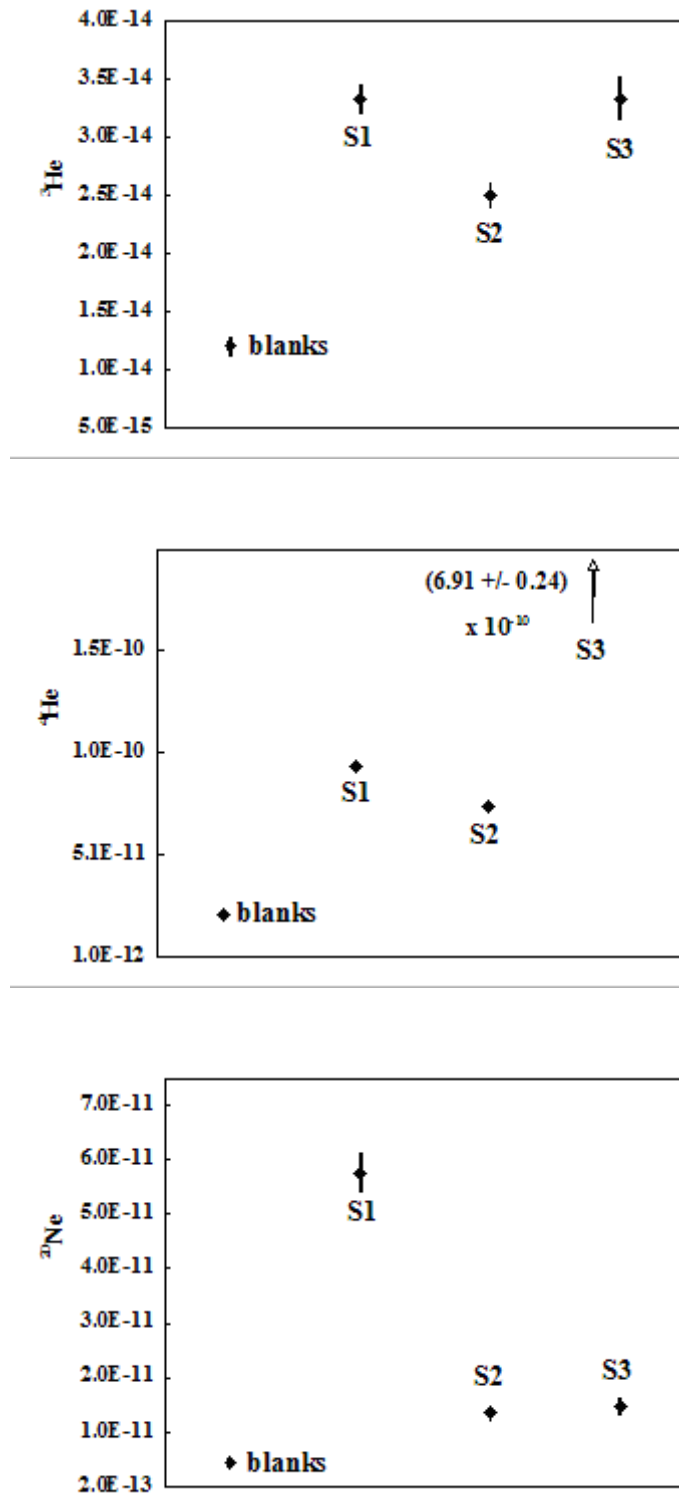
Figure 6. Amounts of ^{3}He, ^{4}He, and ^{20}Ne in ccSTP from S1, S2, and S3 measured relative to background after heating from 1100 °C up to 1400 °C.

DISCUSSION

That He and Ne were not observed from S1, S2, and S3 until relatively high temperatures indicates that the gas was sited in particles, not aerogel bubbles. At temperatures of ~1200-1250 °C, the He diffusion coefficients for vitreous silica (Marty et al. 2007) are large enough for glassy aerogel with dimensions on the order of 1 mm to completely degass within ~10 seconds. For the ≤100 μm size scale associated with melted aerogel along the particle tracks, the degassing process would only take ~1 second or less. Additionally, typical gas release temperatures for interplanetary dust particles are around 500 °C, which is much lower than ~1250 to ~1300 °C observed for the Stardust samples. The particles in Stardust samples are therefore distinctly different from interplanetary dust particles in that they must have formed at higher temperatures. This is a surprising result as comets are typically thought to have formed in the cooler, outer solar system, but is consistent with the dominant refractory grain mineralogy of the Stardust particles (Brownlee et al. 2006). At least in the case of the particles captured from Wild 2, material originating closer to the sun must have migrated outward by some means to be incorporated into comets.

The results in Figures 3 and 4 support prior investigation (Marty et al. 2006) that blank aerogel does not contain detectable He and Ne above background. Terrestrial ^{3}He abundances are consistently lower than extraterrestrial, so the measurement of excess ^{3}He signifies that cometary gases are in fact present in Stardust samples. The ^{3}He diffusion coefficient for Stardust particles is more than 15% larger than that of ^{4}He at the maximum step-heating temperature. However, excess ^{3}He was not measured for repeat maximum temperature heatings making an experimental perturbation in the relative abundance of ^{3}He to ^{4}He unlikely (Marty et al. 2007). Production rates of spallogenic ^{3}He and ^{21}Ne from galactic cosmic ray irradiation at the surface to a depth of 1 m are correspondingly ~5-7 x 10^{-9} cm^3 STP/g per Ma and ~2-3 x 10^{-9} cm^3 STP/g per Ma. The minimum amounts of ^{3}He and ^{21}Ne in particles from S1, S2, and S3 were estimated at ~6 x 10^{-5} and ~3 x 10^{-4} cm^3 STP/g respectively. It would require ~1000 Ma to produce just 10% and <2% of the estimated ^{3}He and ^{21}Ne by means of spallation due to galactic cosmic rays (Marty et al. 2007). The amount of mass lost from Wild 2 since its last

apparition in 1974 has been estimated by Brownlee et al. (2004) to be ~1 m, so Stardust particles were probably buried until recently where irradiation by galactic cosmic rays would have been negligible. Although long-term irradiation on an ancient surface is a possibility, no more than ~50% of the ^{3}He and 5% of the ^{21}Ne measured in Stardust samples could be spallogenic according to the above estimate of minimum gas quantities present, given 4500 Ma for the age of the solar system (Marty et al. 2007). It is therefore unlikely that spallation from galactic cosmic ray irradiation has contributed any significant ^{3}He and ^{21}Ne to the cometary gas in Stardust samples.

Accurately determining the cometary He and Ne isotopic ratios is critical in discerning when and from what source comets acquired noble gases. The average ^{3}He/^{4}He ratio was measured to be ~2.7 x 10^{-4}. This is between the value for the atmosphere of Jupiter, (1.66 ± 0.05) x 10^{-4}, and the respective values for the sun in its post-deuterium burning phase and the current solar wind, (3.65 ± 0.38) x 10^{-4} and (4.82 ± 0.04) x 10^{-4}. The ^{20}Ne/^{22}Ne isotopic ratios most accurately determined between this study and the analysis of samples from the same particle track at the Centre de Recherches Petrographiques et Geochimiques in France are within the range accociated with primitive meterorites, 10.1 to 10.7 (Marty et al. 2007). Stardust Ne isotopic ratios are in contrast very different from current solar wind values, eliminating solar wind as an exclusive Ne source. The observed Ne ratios could be alternatively explained by mixing a Ne composition similar to either the solar wind or primitive meteorites with a terrestrial Ne contaminant. As stated by Marty et al. (2007), however, the ^{21}Ne/^{22}Ne and ^{4}He/^{20}Ne ratios measured in some samples are too low to be consistent with the mixing model. A second alternative is that the cometary Ne composition is unique from other volatile sources in the solar system. Further research must be done so that cometary He and Ne isotopic ratios may be compared with those of specific noble gas sources.

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Author biography: Jacob Simones is a senior undergraduate student of physics and astronomy at Minnesota State University, Mankato. Upon completing his B.S., he plans to earn a PhD in either Physics or Astrophysics. He is interested in both teaching at a university and conducting research of his own, most likely astrophysical. Jacob is an officer for the newly founded Physics club and has been president of the Shotokan Karate club at MSU since 2005. When Jacob is not studying, he can always be found playing his guitar or running outdoors.

Faculty mentor biography: Dr. Russell Palma graduated magna cum laude with a B.S. in Astrophysics from Indiana University in 1974, then earned a Ph.D. in Space Physics and Astronomy in 1981 at Rice University. He was a Professor in the Department of Physics at Sam Houston State University in Huntsville, Texas, from 1980-2004, and was Chairman of the department from 1991-98. He became a Professor in the Department of Physics and Astronomy at Minnesota State University, Mankato in 2004 and is also an Adjunct Professor of Physics at the University of Minnesota. Dr. Palma has used ultra high vacuum mass spectrometers at Rice University, NASA-Johnson Space Center, University of California San Diego, Texas A&M University, and University of Minnesota for over 30 years to measure noble gases and nitrogen in meteorites, lunar materials, interplanetary dust particles, tektites and spacecraft flown materials. Current research interests include: (a) NASA/JPL's Stardust Mission and helium and neon isotopic compositions; (b) NASA/JPL's Genesis Mission and the solar wind nitrogen isotopic composition; (c) Helium, neon and argon isotopic compositions in interplanetary dust particles; (d) solar wind noble gas isotopic compositions as recorded in lunar soil grains, with particular focus on the $^{36}Ar/^{38}Ar$ ratio; (e) meteoritic solar cosmic ray history, with emphasis on the neon isotopic composition. He has supported and mentored 4 undergraduates and 1 graduate student during the past 10 years and procured 7 grants for his work during this period.