Final Summary of Research for NAG5-9345
Accretion of Interplanetary Dust: A New Record from $^3$He In Polar Ice Cores

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Activities
This grant funded measurements of extraterrestrial $^3$He in particles extracted from polar ice samples. The overall objective was to develop measurements of $^3$He as tracers of the flux of interplanetary dust particles (IDP's) to the earth. To our knowledge these are the first such measurements, apart from our earlier work.

The project also funded an EPO activity – a climate and global change workshop for high school science teachers.

We had three objectives:

1) **Determine the reproducibility of $^3$He measurements in Antarctic ice by making several replicate measurements from a large late Holocene ice sample from the Vostok ice core site.** The reproducibility of $^3$He measurements in ice is expected to be controlled by sample size because of the small number of IDP's expected in a typical ice sample. Understanding the reproducibility of replicate measurements is a prerequisite for interpreting down-core records of $^3$He flux.

2) **Determine which particle size fraction carries the majority of the $^3$He in ice core samples.** Theory and modeling suggests that small particles (< 1-5 microns) do not carry much of the $^3$He because they are heated sufficiently in atmospheric entry to lose gases. Larger particles are predicted to be less abundant and therefore also carry less of the $^3$He flux. Theory distinguishes between solar wind implanted $^3$He in particle surfaces and primordial $^3$He, with different implications for the sizes of particles that carry $^3$He.

3) **Assuming objective 1 and 2 could be achieved, measure a down core record of $^3$He from samples of the Vostok ice core.**

Our activities during the award period were directed toward these objectives. To address objective 1 we obtained a 3m section of a shallow core at the Vostok ice core (Vostok is ideal for this work because accumulation rates there are low, concentrating the IDP signal). We sectioned this sample into multiple replicates of ~750-1200 g in the clean room at the National Ice Core Laboratory, filtered them on to 0.45 micron silver membrane filters, and made $^3$He measurements in eight of the replicates by melting the filters in the vacuum furnace then analyzing the released gases for helium isotopes at the Woods Hole Oceanographic Institution. Procedural blanks prepared from filtered laboratory de-ionized water were processed in the same fashion as the samples.

To address objective 2 we developed a sequential sieving and filtration technique to separate particles from ice samples into 5 size fractions: > 63 um, 20-63 um, 10-20 um, 5-10 um, and 0.45-5 um. Because we trap samples on filters it is not possible to extract
particles smaller than 0.45 um (the smallest filter we generally use). Tests using 0.22 micron filtration following 0.45 micron filtration suggest that particles smaller than 0.45 microns don’t contribute significantly to the $^3$He flux (Brook et al., Geophysical Research Letters, 27, 3145-3148, 2000). The sequential filtration started with a large ice sample (1.5 kg). The sample was melted in a large stainless steel funnel and passed through a stack of 63, 20, and 10 micron nylon mesh screens. The material collected on each screen was rinsed from the screen into a second filtration funnel and collected on a 0.45-micron filter for analysis. The sample passing through the 10 um sieve was filtered through a 5 micron silver filter, and the final filtrate was filtered through a 0.45-micron filter. Procedural blanks prepared from filtered laboratory de-ionized water were processed before and after the filtration experiment.

Following this sequence of measurements we processed a time series of Vostok ice samples for analysis. Samples were prepared in the clean air freezer at NICL. Over a period of about a week we cut approximately 45 2 kg samples from the Vostok 5G core into duplicate samples. These samples were individually cleaned by shaving the surfaces with a clean stainless steel chisel. Blanks were prepared from filtered laboratory de-ionized water, were transported to NICL, and processed in the same fashion as the samples. We analyzed 13 of these samples in duplicate, and the blanks. While doing these measurements we encountered problems with the blank measurements that had not occurred previously. We suspended sample processing to examine the source of the blank. We conducted extensive tests of pristine filters, as well as blank filters processed in the filtration apparatus immediately after processing samples (to test how much sample was left behind in the filter funnel). The conclusions of these experiments are described in the next section of the progress report.

The EPO activity (Workshop on Climate and Global Change for high school teachers) was conducted in summer 2001, in collaboration with the State of Washington Educational Service District 112. Approximately 10 teachers participated in a one-week, intensive workshop that covered the basics of climate physics, climate history, and future climate change. A one-day field trip to a local mountain glacier was included, where we discussed the use of glacier records in examining climate history. The grant covered all expenses for the teachers, preparation expenses, and paid tuition for continuing education credit for the teachers. The workshop culminated in a formal debate about future climate change. The workshop was very successful and lead to a second workshop (supported by another agency) the second year where we expanded (with a different group of teachers) on the themes developed the first year.

Findings

1) Reproducibility of $^3$He measurements in Polar Ice.

Figure 1 shows the $^3$He concentrations and extraterrestrial $^3$He flux inferred from the Vostok replicate measurements. The samples are from the Vostok BH-5 core and the age of this ice ~3.8-4.0 ka based on the Vostok EGT time scale. Assuming an
accumulation rate of 2 g ice/cm²/yr, the mean $^3$He flux from the eight replicates is 1.1 ± 0.3 x $10^{12}$ cc STP $^3$He/kyr (mean ± 2σ).

Figure 1. $^3$He concentrations (cc STP/g ice) and $^3$He fluxes for Vostok ice core samples (BH-1 core, 112-115 m, ice age 3.8-4.0 kyr).

This mean flux is consistent with, though slightly higher than, our previous measurements (Brook et al., Geophysical Research Letters, 27, 3145-3148, 2000) of $^3$He flux from smaller Vostok samples at 110 m in this core (0.77 ±0.25 cc STP $^3$He/cm²/kyr). It is also very similar to Holocene fluxes from marine sediments. The slightly higher flux from these samples vs. our previous results may be due to the larger sample sizes. Our previous work (Brook et al., 2000) employed samples of ~250 g, whereas these samples ranged from 750-1200 g. Farley et al. (Geochimica et Cosmochimica Acta, 61, 2309-2316, 1997) predicted on statistical grounds that small samples will under sample the true $^3$He flux. Short-term variations in $^3$He flux are another possible explanation for the difference. The uncertainty in our measured flux is much smaller than would be predicted based on the number of particles expected in samples of this size (Farley et al., 1997).

We believe these results are significant for our understanding of the IDP flux. The mean flux we measure is for an interval of approximately 200 years (3 m of core). The agreement of this value with fluxes measured from marine sediments, which in many cases average fluxes over several thousand years (due to bioturbation), suggests that $^3$He fluxes are relatively constant on time scales of hundreds to thousands of years. If verified with further work this conclusion strengthens the case for using $^3$He as a constant-flux proxy to determine ice accumulation rates, one of the motivations of our work.
2) $^3$He and Particle Sizes

Figure 2 shows the results of the size fraction experiment we conducted. These data show that the majority (~75%) of $^3$He resides in a restricted size fraction of 5-10 microns. This result is important because it confirms model predictions that $^3$He is carried in larger sized particles. The peak in $^3$He at 5-10 microns is also consistent with models that suggest that most of the $^3$He in IDP's is implanted in particle surfaces. Entry heating models for IDP's that assume that $^3$He flux correlates with particle mass predict that a maximum at about 20 microns (Farley et al., 1997). From a more practical perspective these results suggest that filtration at 0.45 microns is adequate to retain most of the extraterrestrial $^3$He in an ice sample.

3) Variations in the $^3$He flux with time?

Results from our replicate experiments suggest a 2-sigma variability of about 30% in inferring the flux from $^3$He measurements in Vostok samples of about 1 kg. This result places limits on how well the flux can be constrained by discrete sampling. Using the sampling protocol we developed (multiple 1 kg samples at each depth) one would expect, based on a simple Monte Carlo model, to constrain the flux at about 60% at the 95% confidence interval. True variations in $^3$He flux must be larger than this to be reliably detected.

Processing large numbers of large samples turned out to be logistically challenging because filtration times are very long (several days). Near the end of the award period we tested a different type of filter (an "Anodisk" alumina filter with small holes rather than a membrane) which may decrease filtration times and has a low $^3$He blank. We will probably use this filter in any future work.
In addition, after processing several large samples from the time series we ran into a serious blank problem that we had not encountered prior to this series of measurements. We had prepared blanks from filtered deionized water, as done in prior work. Blanks processed with the Vostok time series had levels of $^4$He that in some cases were as high as the sample values, and, more troubling, also had high levels of $^3$He. When we recognized the blank problem we had processed and analyzed 13 samples in duplicate. We suspended sample processing at this point and spent a significant amount of time trying to understand the origin of the blank. We are fairly confident that it is not contamination from the laboratory air (dust) because filters left in the apparatus for several days with no sample at all have very low levels of $^3$He and $^4$He. Two other possible sources of contamination exist. One is carryover from previous samples, which might be more significant in these samples because most of them were from the glacial section of Vostok, which has higher dust content. The samples are melted in large stainless steel filter funnels. Though these are extensively cleaned after each use it is possible that particles are retained on the steel. To test how much material might be left behind we installed new filters in funnels previously used for four ice samples without cleaning them, and rinsed the funnels extensively, trapping the rinsate on the filters. Analysis of these filters showed that an amount of $^3$He equivalent to 20-98% of the amount originally measured in the sample is was apparently retained on the apparatus. Although normally much of this material is rinsed on to the original filter this experiment suggests that retention of particles on the apparatus is an important issue.

A second possibility is that the water used to make the blank measurements is contaminated with $^3$H (decays to $^3$He) or particulate $^3$He. Because the water used for the blanks is filtered at 0.22 micron it seems unlikely that a particulate contaminant exists. However, tritium in the laboratory water supply might produce $^3$He in the filters. To test this hypothesis we processed varying amounts of laboratory water through several filters to look for a relationship between the blank and the quantity of water added. Due to a fire in the WHO1 laboratory building that houses the noble gas lab, and some equipment problems, these have not been analyzed yet.

Although we have some reservations about the results from the 13 Vostok replicates for the time series due to the blank problem the data still have value. Figure 3 shows the results plotted as $^3$He flux vs. depth, with ages from the Vostok GT4 time scale indicated on the figure. The shaded bar shows the mean flux from the late Holocene samples. The chief conclusion we can reach now is that the flux during the latter part of the glacial period appears to have been lower than the late Holocene value. This is a tentative conclusion because, given the discussion above, it is possible that some of the particles were lost during cleaning the filtration apparatus.

We believe that the blank and sample handling issues for the time series measurements can be overcome with further experimentation, which we plan to do prior to requesting any additional funding. We are currently writing a paper on the results described in this report.
Figure 3. Apparent $^3$He flux as a function of depth from Vostok. Blue bar shows range (2 sigma) of flux estimate based on late Holocene samples described in section 1.

Publications


Brook, E.J., M.D. Kurz and J. Curtice, in prep, Size fractionation and flux of extraterrestrial $^3$He in IDP's: results from polar ice.