PRELIMINARY DEFINITION PHASE FINAL REPORT

NASA CONTRACT NAS8-39716

TEMPERATURE DEPENDENCE OF DIFFUSIVITIES

Period of Performance
2/3/93 through 7/31/93

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1. Statement of Work

During the six months definition phase of the instrument development program, research personnel at the Center for Microgravity and Materials Research of the UAH were to furnish all of the necessary labor, services, materials, and facilities necessary to provide science requirement definition, initiate hardware development activities, requirements and timetable for integration and experimental accommodation of the GAS payload into the Shuttle cargo bay and an updated ground-based research flight program proposal consistent with the NRA selection letter. These activities were to be accomplished in parallel and consistent with the necessary research and development work toward the accomplishment of the overall objectives of the selected proposal.

2. Work Performed

2.1 Science Requirements Definition

The Draft and Preliminary Science Requirement Documents were completed on time and have been delivered to MSFC personnel. No changes have been suggested to these documents.

2.2 Project Review

The Principal Investigator Status Review/Project Requirements Review presentation was held at MSFC on June 28, 1993.

2. 3 Initiation of Hardware Development Activities

2.3.1 Tasks

The initial hardware development activities included the following tasks:

1) A preliminary conceptual design based on initial dimensional analysis of the optimal diffusion capillary and collimator geometry considering experiment time, spatial resolution and diffusivity data precision, detector sensitivity and minimization of radioactive dose, isotope half-life and collimator/radiation shield absorption characteristics.

2) Specification and acquisition of: **detectors** (minimization of the diameter in interaction with the supplier), specification of integrated preamplifier and discriminator circuits, tests of detector performance (discriminator grade vs. counter grade) at elevated temperatures; **interface boards and software** considering number of data channels, signal amplitude and I/O rates (detectors, thermocouples or thermistors, temperature, and power control); **a heater cartridge** which was to be tested with a copper liner (design goal: maximum temperature 700^{o}C, temperature uniformity ± 0.1-1^{o}C).

2.3.2 Preliminary Conceptual Design and Dimensional Analysis

- ***Estimation of sample temperature nonuniformity for various cell dimensions***

The steady-state sample temperature nonuniformity in a silica-enclosed sample inside a heated gold cell was estimated using an axisymmetric model with conductive heat transfer only. The boundary conditions were: either uniform or non-uniform heat flux input along the outer cylindrical side wall, and heat losses according to published values for a horizontal surface at the two ends. Various values for the outer diameter and length of the gold cell were chosen. The inner diameter and length, i.e., the dimensions of the cell cavity, were fixed at 0.8 and 5.0 cm, respectively. The thermal conductivity of gold was used for the cell. For the cell cavity we assumed conduction according to an averaged conductivity for silica glass and tin. Figure 1 shows a schematic of the cell, together with temperature difference resulting for the various cell dimensions with uniform and "cold spot" heating conditions.

These results indicate that a 0.5 cm wall of a high thermal conductivity material such as gold should lead to sample temperature nonuniformities of $\leq 1^{o}C$.

- ***Estimation of radioactive dose required***

An estimation of the radioactive dose (weight of radioactive material deposited by electroplating) required was made based on geometrical, material (including heat transfer/insulation estimates), and detector considerations. Figures 2a and b schematically show the assumed experimental arrangement. The assumptions were: Sample dimensions of 0.3 cm o.d. $\times$ 3 cm long; thicknesses of the silica cell glass wall, gold cell, heater, and thermal insulation were 0.2, 0.7, 0.2, and 10 cm, respectively; detector diameter of 1 cm; radiation through the collimation hole fills the detector area. Also, the radiation energy absorption was calculated for all materials including self-absorption in the sample for an averaged photon energy. The detector efficiency was assumed as unity. The background count rate was assumed as 20 counts/minute. At the completion of the experiment, when the radiotracer has diffused throughout the sample, we assumed that the count rate will be 1000 counts/minute. Furthermore, we assumed 0.5mCi/mg for the maximum specific activity of the plated material .

Based on the above conditions the collected/total sample volume is 5×10^{-3} and the emitted/received photon flux is 3×10^{-3}. Combining these losses approximately 15 of every 1,000,000 photons produced are received at the detector. This yields a total required source strength of 0.06mCi or 0.15mg of electroplated material.

- ***Experiment sensitivity analysis***

During this definition phase we have also begun to set up a model to carry out a systematic system performance analysis. In this model we consider 1) differences between the input and the calculated diffusivity value due to inherent errors in the integral equation used for data evaluation; 2) the optimum experiment time for various sample temperatures; 3) the optimum number and spacing the detectors; 4) the measurement error due to misalignment and finite collimation angle of the collimator/detector arrangement; 5) counting errors due to statistical fluctuations in the decay rate; and 6) various levels of background noise. Preliminary results were presented at the Project Review.

2.3.3 *Specification and Acquisition*

All of the components listed above (detectors, radioactive calibration standards, interface boards and software and, heater cartridge) have been specified and ordered. In the following we describe the results of various tests performed with these components.

• **Detectors** and their electronics were checked for statistical noise fluctuations at: different bias voltages, threshold discriminator levels, source energies, counting times, and temperatures. Four different detector materials (of various sizes) and two different electronics packages were obtained from eV products and Aurora Technologies. The four detector materials were: Tl doped-CsI mounted on a low noise silicon photodiode (eV products), High Pressure Bridgman (HPB) CdTe (eV products), Cl-doped traveling heater method CdTe (obtained through eV products) and HPB CdZnTe (Aurora Technologies). The electronics consisted of a preamplifier, amplifier, discriminator, and shaper. The results of the above tests are:

1) At counting rates of ~1000 counts/minute the statistical fluctuation for HPB-CdTe and CdZnTe detectors is 2.5-3% at approximately 25^{o}C. For the same counting rates and temperature conditions the statistical fluctuation for (Tl)CsI-photodiode detector is about 1.5%.

2) (Tl)CsI-photodiode detectors are not usable for photon energies less than 30-40KeV because of the photodiode noise level. Therefore, for the radioisotopes (i.e., Sn^{119m}, Cd^{109}, Se^{80}, and In^{114}) for the 4 diffusion couples chosen, (Tl)CsI-photodiode detectors would be acceptable only for the Se^{80} source.

3) The CdZnTe detectors require approximately twice the bias voltages needed for the HPB-CdTe detectors to reduce the statistical fluctuations to 2.5-3%. At equal bias voltages (i.e., 150V) the statistical fluctuation of the CdZnTe detector was 3.3% versus 2.5% for HPB-CdTe. HPB-CdTe detectors can be used (for the sizes we have tested) at bias voltages down to 80V with no increase in statistical fluctuations. The more expensive (Cl)CdTe detector behaved essentially the same as the HPB-CdTe detector.

4) Increasing the detector temperature from 25 to 40 and 60^{o}C increases the statistical fluctuation from 2.5 to 5 and 10% for both HPB-CdTe and CdZnTe. This can be compensated for only by increasing the discriminator threshold level to ~40KeV which precludes detection of

the photons of interest to us. Therefore, it will probably be necessary to maintain the detector temperature at or below 25°C.

5) Only the eV products electronics have a noise level of below that required for this project.

6) Increasing the temperature of the eV products electronics from 25 to 40°C did not increase the noise level.

HgI_2 detectors are expected to arrive in the next few weeks.

Based on the above results we will continue to work with eV products to miniaturize their electronics. At the present time it appears that the HPB-CdTe material fulfills our requirements. However, we will continue to evaluate various solid-state detectors as they become available during the next phase of this project

• The **interface boards and software** acquired consisted of: a multifunction I/O board, a 10 channel counting/timing I/O board, thermocouple modules and, solid state relay modules; associated connectors were also acquired. No tests could be performed on these components as their host computer was not authorized during this reporting period.

This choice of using counter/timer I/O boards allows for simultaneous data acquisition of all radiation detectors while permitting independent temperature monitoring and control, and data reduction.

• A **heater cartridge** (2 cm i.d.) which consists of a graphite heater enclosed in pyrolytic boron nitride support was tested with a copper liner (~ 2 cm o.d. × 7 cm long). The copper liner was bored out to 7 mm i.d. and 6 cm deep to accommodate a 7 mm o.d. × 5 cm long silica tube. A sliding thermocouple was placed inside the silica tube. A hollow copper plug sealed the open end of the liner. This arrangement was heated to 300, 400, 500, 600 and 700°C, respectively. The temperature nonuniformity remained ≤ 0.3°C between the bottom and the half-height of the silica tube. Thus the design goal of ≤ 1°C temperature nonuniformity was achieved.

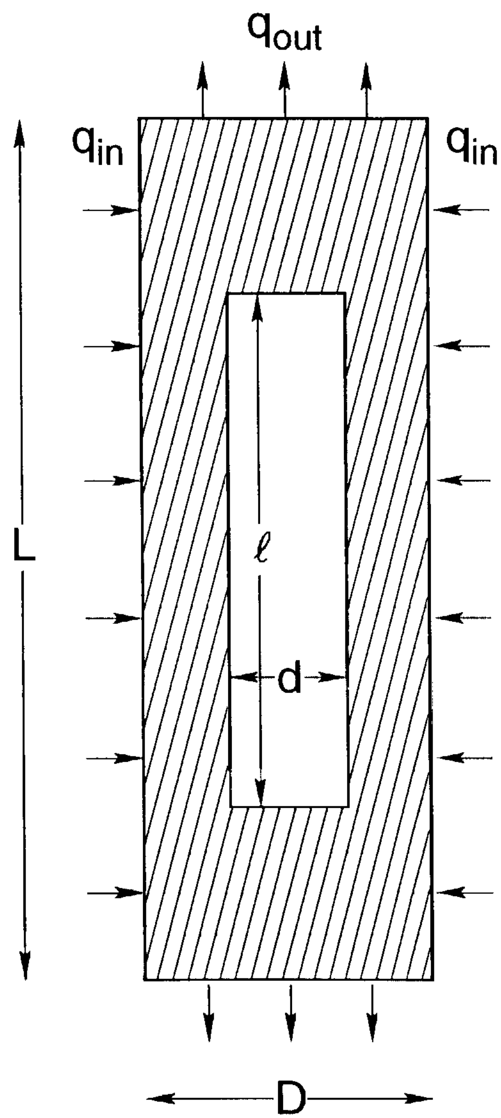
As has been previously discussed we would also like to evaluate Kanthal APM tube material as a possible heater. However, we have still not been able to obtain a sample of this material.

2.4 Integration

The milestones obtained from GSFC have been worked into our schedule of activities. These dates begin with the completion of the Payload Accommodations Requirements (PAR) and go through the receiving of the post-flight data. In addition, the milestone list includes several safety data requirements and reviews (see the Updated Ground-based Research Flight Program Proposal).

2.5 Updated Ground-based Research Flight Program Proposal

The Updated Ground-based Research and Flight Program Proposal was completed and submitted to MSFC personnel (i.e. Bud Yates, Linda Jeter and Patton Downey). Changes or improvements from the original proposal included: 1) specification of the diffusion couples to be investigated during the ground based research and space flight; 2) a (high energy density) silver oxide battery (due to the unavailability of NASA supplied batteries); 3) increase in the number of diffusion cells to be flown from one to three and; 4) increase in the time commitments of the various investigators while an originally planned payload manager position was removed. Unfortunately, this more realistic proposal lead to a considerable increase in the funding required for this project.



D [cm]	L [cm]	ΔT [K]	
1.8	7.0	0.32	
2.8	7.0	0.30	
3.8	7.0	0.30	
4.8	7.0	0.26	
1.1	6.0	0.64	
1.4	6.0	0.45	
1.8	6.0	0.30	
1.8	6.0	0.32	$q = 0.6\ q_0$
1.8	6.0	0.30	$q = 0.2\ q_0$ for 20% of L

$d = 0.8$ cm, $\ell = 5.0$ cm

Figure 1. Geometrical sketch and results for numerical heat transfer model.

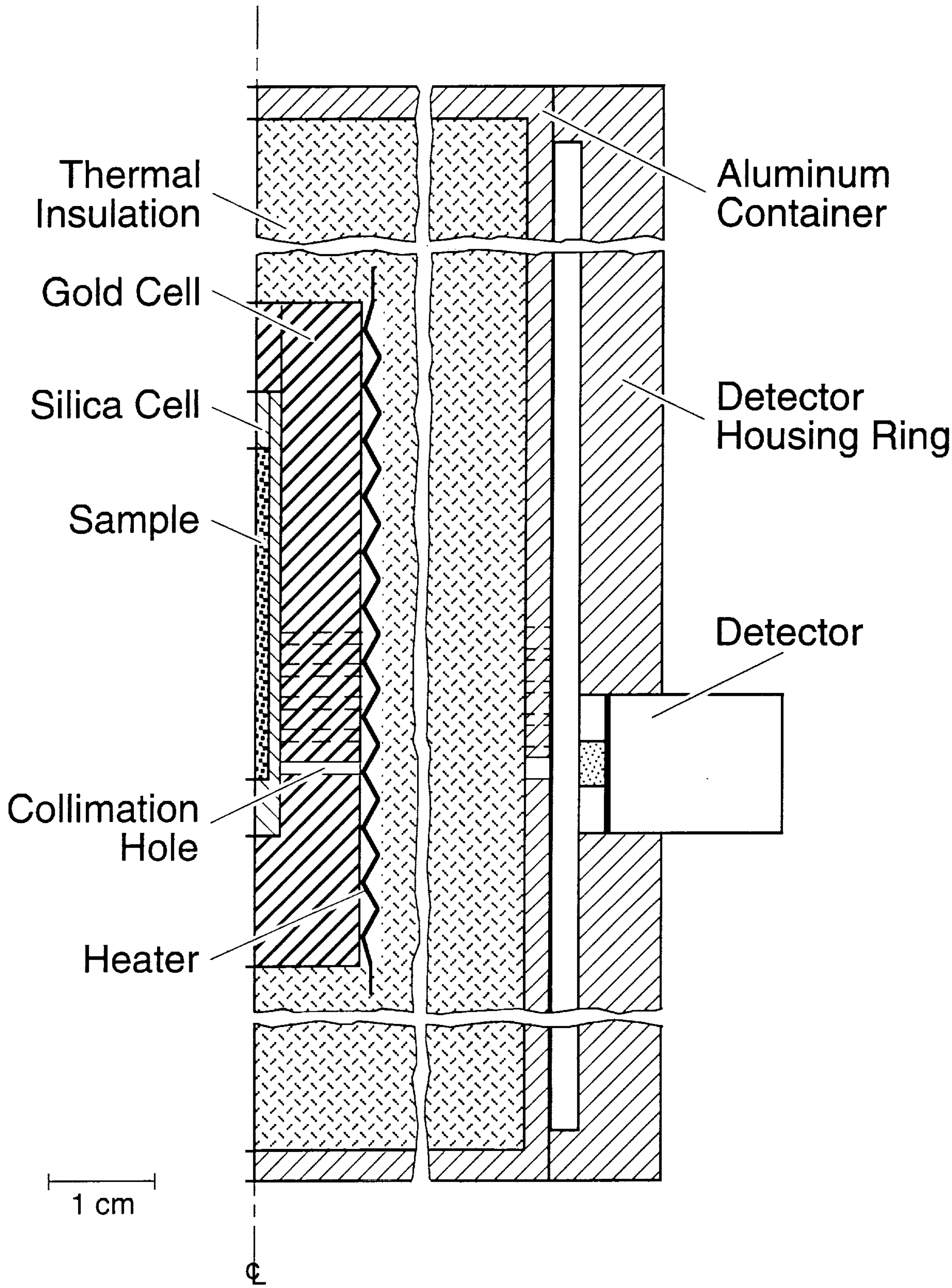


Figure 2a. Schematic diagram of Liquid Metal Diffusion Apparatus.

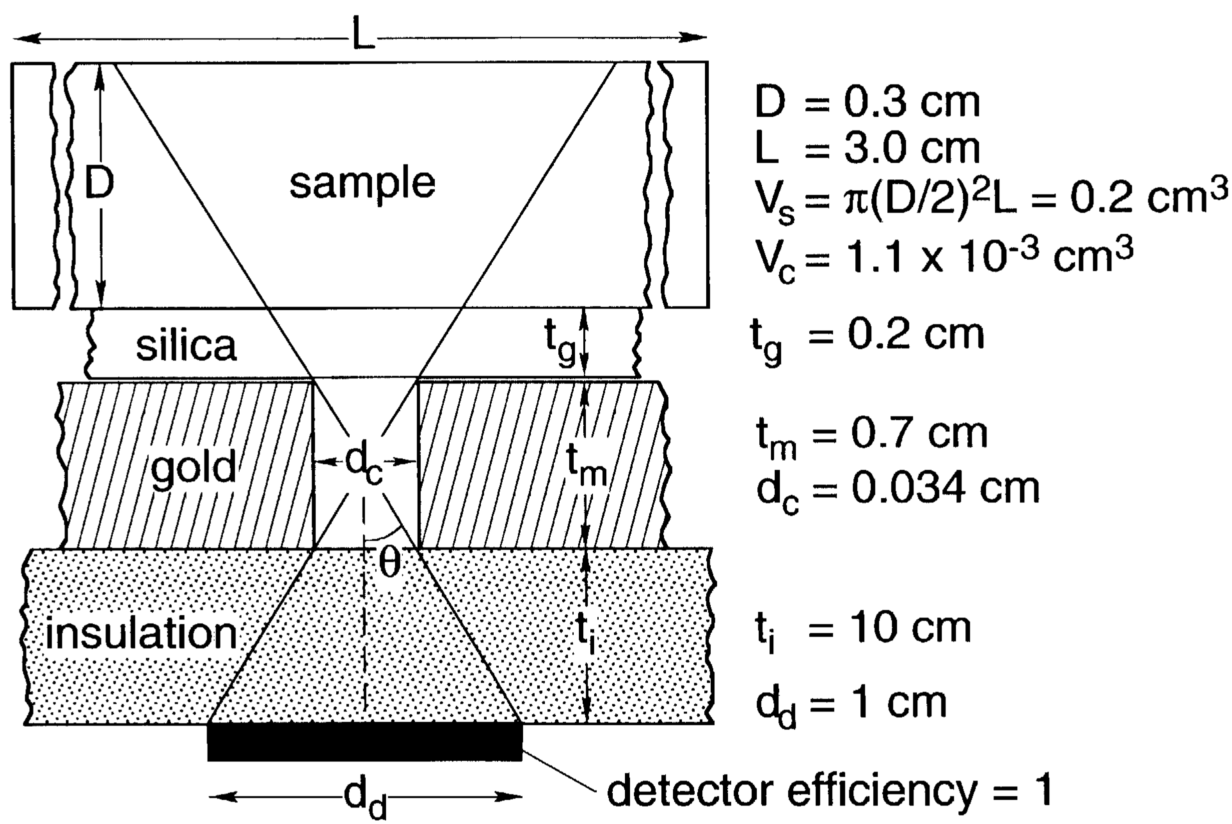


Figure 2b. Schematic diagram of sample to detector geometry and materials.