

# COMPARATIVE SOOT DIAGNOSTICS: PRELIMINARY RESULTS

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## Introduction

The motivation for the CSD experiment lies in the broad practical importance of understanding combustion generated particulate. Depending upon the circumstances, particulate matter can affect the durability and performance of combustion equipment, can be a pollutant, can be used to detect fires and, in the form of soot, can be the dominant source of radiant energy from flames. The non-buoyant structure of most flames of practical interest makes understanding of soot processes in low gravity flames important to our ability to predict fire behavior on earth. These studies also have direct applications to fire safety in human-crew spacecraft, since smoke is the indicator used for automated detection in current spacecraft [1].

In the earliest missions (Mercury, Gemini and Apollo), the crew quarters were so cramped that it was considered reasonable that the astronauts would rapidly detect any fire. The Skylab module, however, included approximately 20 UV-sensing fire detectors. The Space Shuttle has 9 particle-ionization smoke detectors in the mid-deck and flight deck and Spacelab has six additional particle-ionization smoke detectors. The designated detectors for the ISS are laser-diode, forward-scattering, smoke or particulate detectors. Current plans for the ISS call for two detectors in the open area of the module, and detectors in racks that have both cooling air flow and electrical power. Due to the complete absence of data concerning the nature of particulate and radiant emission from incipient and fully developed low-g fires, all three of these detector systems were designed based upon 1-g test data and experience. As planned mission durations and complexity increase and the volume of spacecraft increases, the need for and importance of effective, crew-independent, fire detection grows significantly. To provide this level of protection, more knowledge is needed concerning low-gravity fire phenomena and, in particular, how they might be detected and suppressed.

Prior to CSD, no combustion-generated particulate samples had been collected near the flame zone for well-developed microgravity flames. All of the extant data either came from drop tower tests [2,3] and therefore only corresponded to the early stages of a fire or were collected far from the flame zone [4]. The fuel sources in the drop tower tests were restricted to laminar gas-jet diffusion flames and very rapidly overheated wire insulation [2, 3]. The gas-jet tests indicated, through thermophoretic sampling, [2] that soot primaries and aggregates (groups of primary particles) in low-gravity may be significantly larger than those in normal gravity (1-g). This raises new scientific questions about soot processes as well as practical issues for particulate size sensitivity and detection/alarm threshold levels used in on-orbit smoke detectors. Preliminary tests in the 2.2 second drop tower suggest that particulate generated by overheated wire insulation may be larger in low-g than in 1-g [3]. Transmission Electron Microscope (TEM) grids downstream of the fire region in the Wire Insulation Flammability experiment [4] as well as visual observation of long string-like aggregates, further confirm this suggestion. The combined impact of these limited results and theoretical predictions is that, as opposed to extrapolation from 1-g data, direct knowledge of low-g combustion particulate is needed for more confident design of smoke detectors for spacecraft.

This paper describes the operation and preliminary results of the CSD, a project conceived and developed at NASA Lewis Research Center. The CSD flight experiment was conducted in the Middeck Glovebox Facility (MGBX) on USMP-3. The project is supported by NASA Headquarters Microgravity Science and Applications Division and Code Q. The results presented here are from the microgravity portion of the experiment, including the temporal response of the detectors and average sizes of the primary and aggregate particles captured on the thermophoretic probes. Complete assessment of the microgravity data and its combination with the normal-gravity data are still in process and will be reported later.

## Background/Previous Work

Well-established normal-gravity fires emit small particulate in the size range to which ionization detectors are more sensitive than optical detectors [5]. Less well-established or smoldering fires will produce larger particulate, owing to the large amount of condensed, unoxidized fuel pyrolysis products and the incomplete soot oxidation. For this type of fire, light scattering/obscuration detectors are more appropriate. However, for materials heated slowly as in the very early stages of some fires, the particulate can be very small, and therefore more easily sensed by ionization detectors [6]. This analysis was used by Brunswick Defense in their decision to pursue an ionization detector for the STS. The design consists of a dual-chamber ionization detector that is in the flow path created by a vane pump. This vane pump provides some active sampling capability and also the flow for an inertial separation system which is designed to make the detector insensitive to particulate larger than 1 to 2 microns, depending upon the particle

mass. These advantages are offset by a fairly large power consumption (9 Watts), fan noise, and limited life due to the moving parts. The detector produces a discrete alarm signal and two analog signals related to the detected smoke density. For the tests reported here, the analog voltage was measured. This voltage has a baseline of 11.4 for the detector used here and decreases in the presence of smoke. The alarm value is set at the factory using a fixed offset from the internal detector baseline and consequently the analog output voltage at alarm is different for each detector. The analog voltage at alarm for the detector used in this work was 10.6 volts.

The design developed for the ISS by Allied Signal consists of a 2 pass laser diode obscuration system that also has a photo-diode positioned to sense forward scattered light (30 degrees) on the return path. The system is designed to alarm based on the magnitude of the scattered light signal. Dust discrimination is based on frequency analysis of the scattered light signal. The system is less sensitive to particles smaller than the wavelength of the laser (near IR) than it is to larger particles. The minimum reported sensitivity is 0.3 microns [7]. It draws relatively little power (1.5 W) and it has a long operational life. The current planned alarm value is 2 volts or 1% obscuration per foot.

The performance of these two detectors has been compared in normal gravity [7, 8]. Consistent with expectations, for cases where large particles were expected (punk smoke) the initial response of both detectors was about the same but the ISS detector reached the alarm threshold more rapidly. For sources of smaller particle-size smoke (over heated wire) the STS detector was quicker both in initial response and in time to alarm. The implementation (alarm threshold selection) of both of these systems in microgravity is hampered by the lack of knowledge of their performance against low-gravity combustion generated particulate.

### **Objectives**

The objectives of CSD are to examine the particulate formation from a variety of particulate-generating sources and to quantify the performance of several particulate-sensing diagnostic techniques. The sources include: a candle tested at three coflow air velocities and four overheated materials (paper, silicone rubber, and teflon and kapton coated wires) each tested at three heating rates. The paper, silicone rubber and wire insulations are materials found in spacecraft crew cabins and candles produce hydrocarbon soot typical of many practical 1-g flames. Four diagnostic techniques were employed: thermophoretic sampling to provide samples of the particulate for size analysis, laser-light extinction measurements near the source to provide total smoke production data, and laser light scattering and ionization detector measurements to provide data concerning the performance of the detectors for these particulate sources.

### **Hardware Description**

The CSD experimental hardware consists of two modules named the Near-Field Module and Far-Field Box as shown in figure 1. The Near-Field Module is installed inside the Glovebox and contains the sample and the near field diagnostics. The Far-Field Box is external to the Glovebox and contains two spacecraft smoke detectors. Products from the near field tests are transported to the Far-Field Box and subsequently back into the Glovebox via teflon hoses which enter the Glovebox through ports in the airlock door. All of the combustion products are contained in either the Glovebox or the Far Field Box; by the time the experiment is completed, all of the products are returned to the Glovebox.

The Near-Field Module consists of a small test chamber fitted with a sample carrier that holds the sample being tested. A small fan blows air from the glovebox into the right side of the chamber past the sample and out the left side where it enters the hose to the Far-Field Module. The sample is ignited or overheated by a resistively heated Kanthal wire. The smoke particulate is sampled by a rake of thermophoretic probes and smoke production is detected by a laser light extinction system.

The Far Field Box contains a duct and a fan to transport the smoke from the Near Field Module to two spacecraft smoke detectors, one matching the STS detector and the other identical to the ISS detector except that its signal is amplified by a factor of 6.6 over the ISS standard. This amplification was added because the engineering model of the ISS detector that was used for developing the test matrix displayed very low signal levels. Since the tests were intended more as a test of each technology rather than specific designs or alarm levels, the signal was amplified in the model used in the flight tests. Post flight calibration of both units by Allied Signal revealed that the engineering model was operating below specification by a factor of 5, voiding the reason for the amplification. However, despite the amplification, sufficient dynamic range exists on the measurement that it is possible to predict when an unamplified unit would show significant detection. The analog signals from the various instruments are displayed by digital readouts on the Far Field Box where they are recorded by a video camera for later transcription.

### **Procedure and Operational Sequence and Data Reduction**

The crew installed the near field hardware in the Glovebox, attached the Far Field Box to the Glovebox, and positioned the video cameras. The operator then ran the self diagnostic procedures on the two smoke detectors and activated the video cameras, turned on the ignitor for a predefined period of time (15 to 60 seconds), and initiated the thermophoretic soot samplers to sample the smoke. The actual duration of each combustion event was typically 2 minutes. After the experiment, the operator stowed and re-loaded the soot samplers, the test sample, and the filters at the end of the return line from the Far Field Box. At this point the operator either stowed the modules or initiated another run. The tests ran very efficiently and after the first two sessions, typically ran ahead

of schedule. The sessions consisted of 2 to 7 runs each. The majority of the tests were run with predefined igniter power levels, however, to examine the response threshold, some of the tests in the final session were conducted repeatedly on the same samples at small increases of power level for each retest. After flight, the particulate collected on the thermophoretic probes were analyzed using a Transmission Electron microscope (TEM) to determine primary and aggregate particle dimensions. The remains of the samples were weighed to determine their mass loss. The digital data was transcribed from the video record and analyzed to determine the response of the detectors. Once the flight mass loss rates were determined, 1-g operation conditions to produce the same loss were determined. These conditions are being used to produce a 1-g comparison data set.

## **Results**

In all, 25 tests were performed and the data from the runs are summarized in table 1, which lists the mass loss rate of the sample, the time it took for the signal to reach a value of 20% of the operating range of the detector and the time and value of the peak signal for both detectors. Since these tests were designed to function as a test of the detection technologies rather than the detectors themselves (or their alarm setpoints) 20% of the dynamic range was selected to tabulate as a consistent detection point for both detection technologies. The selection of the actual alarm level for each detector is more complex than mere detection as false alarm rejection and other issues must be considered. The bulk of the tests were overheated material tests in which the heating level was established to produce a target weight loss rate without causing the sample to develop a sustaining combustion reaction. The intent of these tests was to produce smoke typical of an incipient fire where much of the material is being heated but has not yet ignited. The candle tests and one of the paper tests were combustion tests where the samples were ignited so the combustion products could be observed. Figures 2-5 contain the signal data from two typical candle, one Kapton and one silicone rubber tests respectively. The response of the smoke detectors, the ignitor voltage and the signal for the lasers in the near field module are plotted with respect to time. As plotted, the data for the ISS detector overstates the sensitivity of the flight unit since the unit used here is 6.6 times more sensitive than the flight model. These tests were neither designed nor expected to reach specific alarm levels. Instead the tests were planned to provide a measure of the relative sensitivity of the two detector types to low gravity smoke particulate by providing sufficient particulate to achieve adequate signal levels on the detectors. To explore the effect of heating rate on particle size, the power level was different for each test with each material. The results of the tests are discussed below by sample type.

### **Candle Tests:**

In all tests the detector's signals showed an immediate peak that is attributed to the wax vapor released by the ignition process. This peak was detected more quickly by the ISS detector. Once the candle was ignited, the STS detector showed strong signals for all tests while the ISS detector showed strong signals for tests 1 and 16 (e.g. Fig. 2) and very weak signals (if unamplified) for tests 6 and 11 (e.g. Fig. 3). The cause of this is still under investigation.

### **Paper Tests:**

Runs 5g, 10 and 25e were pyrolysis runs i.e. they did not produce a flame but instead allowed the paper to degrade into pre-combustion or pyrolysis products. Of these, run 10 showed appreciable mass loss and was readily detected by the ISS detector while the STS detector produced marginal signal. In contrast, 1-g comparison tests show strong signals on both detectors. Runs 5g and 25e were the last runs in a series of tests aimed at finding the threshold detection level for the sample. These tests showed slightly higher signals for the ISS detector, however, if scaled to real detector performance (i.e. unamplified) neither detector showed distinct signal. Test 20 was a flaming test of a small paper sample and showed a marginal advantage for the STS detector. Test 15 was intended to be a pyrolysis test but self-heating caused the sample to develop a self-sustaining smolder reaction late in the test. The ISS detector detected both the pyrolysis and flaming intervals while the STS detector only detected the smoldering interval.

### **Teflon Wire Tests:**

Both detectors showed strong signals for tests 3, 13, and 18. On test 8, however, the STS detector showed minimal signal and the ISS had a weak signal (unamplified). For Test 21 the STS detector showed a weak signal compared to no signal (even amplified) for the ISS detector. Test 23b showed no signal for either detector.

### **Silicone Rubber Tests:**

For all of the tests with silicone rubber, the ISS detector showed very strong signals while the STS detector showed at most a weak signal (Fig. 4). This is contrary to 1-g comparison test results where this material also shows a strong signal on the STS detector.

### **Kapton Wire Tests:**

Both detectors showed strong signals for 4, 9, 14 and 19 with both producing low signals (STS slightly higher) for test 24 which was a low mass loss test

### **TEM Results:**

Thermophoretic sampling has been used previously in the drop tower for gas jet diffusion flames and for overheated wires [2,3]. For the overheated but not burning materials, a weak thermal gradient was expected and consequently the probe was left in place for 45 seconds while in the case of the much hotter candle flame the probe was left in for less than a second. When the probes were returned to earth, the grids were removed and analyzed in a TEM to determine the primary particle and aggregate size distribution.

Timing of the deployment of the soot sampler required rapid subjective decisions by the crew member. Based upon our video record, they did an excellent job. We encountered less stable flow of the smoke plume than we anticipated and had a high rate of loss of TEM grids due to failure of the adhesive. Consequently particulate samples were not obtained on all tests.

Figures 6 a-c contain TEM images of typical particulate from Teflon, Kapton and Candle tests. The three images are at the same magnification and show the significant variation in the particulate morphology for the three materials. Table 2 summarizes the results for the tests for which good sampling was achieved. Significantly, despite strong smoke levels visible in the video record, no particulate material was found on the TEM grids for overheated paper and silicone rubber tests. The suspected cause of this is that the particulate for these materials is actually liquid droplets which later evaporate or spread out on the grids' surface, rendering them undetectable by the TEM. The primary particle dimensions range from 30 to 40 nm for soot to 60-100 nm for Kapton and 130 to 200 nm for Teflon. The aggregate soot dimensions grow with distance from the candle from 400 nm at 15 mm from the wick to 1200 nm at 35 mm from the wick. The Kapton aggregates range from 220 - 250 nm and the Teflon aggregates range from 600-1100 nm. The dimensions reported in Table 2 are geometric averages (averages based upon a log-normal size distribution). The probe centerlines were 10 mm apart along the flow axis and are numbered sequentially beginning with the probe nearest to the sample. Each probe assembly inserted the 4 probes simultaneously. The rejection criteria for the inertial separation in the STS detector is based upon a 1 to 2 micron particle mass. Since the soot from the candle tests was still very successfully detected, either 1.2 micron soot particles have not reached the rejection mass level or enough particles were below the rejection level to deliver a strong signal. The Teflon and Kapton samples produced large primaries with the largest occurring in Teflon test 13 (195 nm) the resultant aggregates were 1.1 microns.

### **Discussion**

Data analysis and interpretation are well underway but have not yet been completed. The companion 1-g test program is also underway but incomplete. However, the smoke produced in the low-g silicone rubber and paper tests was thick and clearly visible while in comparable 1-g tests, it was only marginally visible for the same mass loss rate. This can be explained by the absence of buoyant flows which (in 1-g) dramatically accelerate the gas velocities in the vicinity of the source, reducing the residence time of the particulate in regions of high concentration. Since particulate sizes for the smoke from these two materials could not be obtained with TEM analysis, the reasons for the lower signal levels from the STS detector can only be inferred. It is likely that these droplets had more opportunities to coalesce into droplets whose size exceeded the maximum size for which the STS detector is sensitive (1-2 microns). For very low mass loss tests for wire insulation materials, the STS detector showed more signal than the ISS model. Once the 1-g comparison tests are complete, further analysis of the results will be possible.

### **Conclusions**

The CSD experiment successfully produced controlled quantities of smoke particulate from a variety of sources. The response of the STS and ISS detectors to these smoke sources was measured and samples of the smoke particulate were successfully obtained from many of the tests. The assembled data provide the first examination of the response of spacecraft smoke detectors to micro-gravity smoke particulate.

The most significant conclusion to be drawn, so far, from this work is that sensitivity to the smoke from an individual material in 1-g does not indicate strong sensitivity to the smoke from the same material in low-g. Despite the presence of a forced flow (provided by a fan) in the low-g tests the absence of buoyant flows apparently changed the particle size distribution. We have not yet determined if the change is sufficiently systematic that correlations can be obtained. Both detectors showed responsiveness for the majority of materials; the STS detector showed limited sensitivity to pyrolyzing materials that produce liquid particulate. The particle dimensions covered a wide range from 30 to 200 nm for primary particles and from 220 to 1200 nm for aggregates.

### **Acknowledgments**

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### **References**

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**Table 1 Detector Signals from CSD Flight tests (ISS values are scaled to flight)**

| Test Point     | ISS Detector       |                             |                         |                      |     | STS Detector       |                             |                         |                      |  | mass loss rate<br>(micro-gm/s) |
|----------------|--------------------|-----------------------------|-------------------------|----------------------|-----|--------------------|-----------------------------|-------------------------|----------------------|--|--------------------------------|
|                | Time to<br>20% (s) | Time to<br>Maxi-<br>mum (s) | Voltage at maxi-<br>mum | % FS at Maxi-<br>mum |     | Time to<br>20% (s) | Time to<br>Maxi-<br>mum (s) | Voltage at Maxi-<br>mum | % FS at Maxi-<br>mum |  |                                |
| Paper          |                    |                             |                         |                      |     |                    |                             |                         |                      |  |                                |
| 10             | 31                 | 33                          | 7.22                    | 29.6                 | --- | ---                | ---                         | ---                     | ---                  |  | 43.4                           |
| 15             | 20                 | 21                          | 7.21                    | 29.5                 | 54  | 61                 |                             | 9.2                     | 91.7                 |  | consumed                       |
| 20             | ---                | 24                          | 1.56                    | 6.1                  | --- | 18                 |                             | 11.27                   | 5.8                  |  | consumed                       |
| 25e            | ---                | 73                          | 0.75                    | 0.2                  | --- | 89                 |                             | 11.34                   | 4.1                  |  | multiple tests                 |
| 5g             | ---                | 71                          | 0.74                    | 2.1                  | --- | 70                 |                             | 11.37                   | 1.3                  |  | multiple tests                 |
| Candle         |                    |                             |                         |                      |     |                    |                             |                         |                      |  |                                |
| 1              | 61                 | 62                          | 7.2                     | 29.6                 | --- | 82                 |                             | 9.65                    | 73.4                 |  | consumed                       |
| 6              | ---                | 70                          | 2.3                     | 9.2                  | --- | 67                 |                             | 10.55                   | 36.0                 |  | consumed                       |
| 11             | 69                 | 69                          | 6.88                    | 28.3                 | 35  | 75                 |                             | 9.9                     | 9.9                  |  | consumed                       |
| 16             | 45                 | 49                          | 7.21                    | 29.2                 | 47  | 108                |                             | 9.53                    | 9.5                  |  | consumed                       |
| Silicon Rubber |                    |                             |                         |                      |     |                    |                             |                         |                      |  |                                |
| 2              | 20                 | 20                          | 7.22                    | 30.1                 | --- | 82                 |                             | 11.14                   | 11.6                 |  | 107                            |
| 7              | 18                 | 19                          | 7.22                    | 30.1                 | --- | 44                 |                             | 11.24                   | 7.4                  |  | 58.7                           |
| 12             | 21                 | 23                          | 7.23                    | 30.1                 | --- | 31                 |                             | 11.39                   | 1.2                  |  | 130                            |
| 17             | 16                 | 17                          | 7.22                    | 30.1                 | --- | 39                 |                             | 11.21                   | 8.7                  |  | 128                            |
| 22             | 32                 | 34                          | 7.24                    | 30.2                 | --- | 52                 |                             | 11.28                   | 6.9                  |  | 15.2                           |
| Teflon         |                    |                             |                         |                      |     |                    |                             |                         |                      |  |                                |
| 3              | 55                 | 62                          | 7.23                    | 34.5                 | 72  | 76                 |                             | 10.88                   | 22.6                 |  | 86.2                           |
| 8              | ---                | 61                          | 4.18                    | 16.9                 | --- | 67                 |                             | 11.27                   | 6.6                  |  | 141                            |
| 13             | 27                 | 27                          | 7.21                    | 29.6                 | --- | 35                 |                             | 11.17                   | 10.0                 |  | 155                            |
| 18             | 31                 | 32                          | 7.22                    | 29.6                 | 39  | 42                 |                             | 10.92                   | 20.3                 |  | 143                            |
| 21             | ---                | 78                          | 0.29                    | 0.8                  | --- | 81                 |                             | 11.29                   | 6.5                  |  | 47.9                           |
| 23a            | ---                | 46                          | 0.51                    | 1.7                  | --- | 59                 |                             | 11.36                   | 2.5                  |  | multiple tests                 |
| Kapton         |                    |                             |                         |                      |     |                    |                             |                         |                      |  |                                |
| 4              | 14                 | 15                          | 7.21                    | 29.6                 | 25  | 37                 |                             | 10.1                    | 54.4                 |  | 37.5                           |
| 9              | 14                 | 23                          | 7.23                    | 29.7                 | 28  | 37                 |                             | 10.64                   | 32.5                 |  | 56.5                           |
| 14             | 13                 | 14                          | 7.21                    | 29.6                 | 24  | 33                 |                             | 10.19                   | 50.8                 |  | 106                            |
| 19             | 10                 | 10                          | 7.21                    | 29.6                 | 25  | 30                 |                             | 10.36                   | 43.6                 |  | 99.4                           |
| 24             | ---                | 28                          | 0.88                    | 3.3                  | --- | 74                 |                             | 11.09                   | 14.3                 |  | 4.29                           |

**Table 2 TEM results**

| Test Point        | Probe Number | Primary Diameter (nm) | Aggregate diameter (nm) |
|-------------------|--------------|-----------------------|-------------------------|
| 1 (Candle)        | 1            | 44                    | 417                     |
|                   | 2            | 33                    | 742                     |
|                   | 3            | 39                    | 1185                    |
| 16(Candle)        | 2            | 38                    | 710                     |
|                   | 4            | 32                    | 1252                    |
| 8(Teflon)         | 1            | 136                   | 608                     |
|                   | 4            | 134                   | ---                     |
| 13(Teflon)        | 1            | 195                   | 1084                    |
|                   | 2            | 154                   | 669                     |
| 9 (Kapton)        | 1            | 79                    | 234                     |
|                   | 4            | 95                    | 253                     |
| 19(Kapton)        | 3            | 74                    | 119                     |
|                   | 4            | 85                    | 184                     |
| 20(Flaming Paper) | 1            | 29                    | 406                     |
|                   | 2            | 26                    | 425                     |
|                   | 3            | 24                    | 301                     |

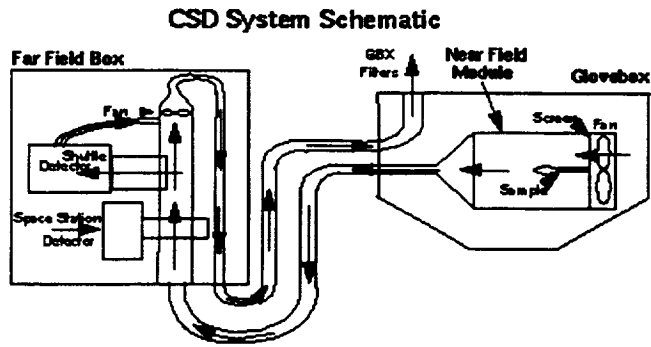


Figure 1. Schematic showing installation of the CSD Near Field Module inside the Glovebox and Far Field Module outside of the Glovebox

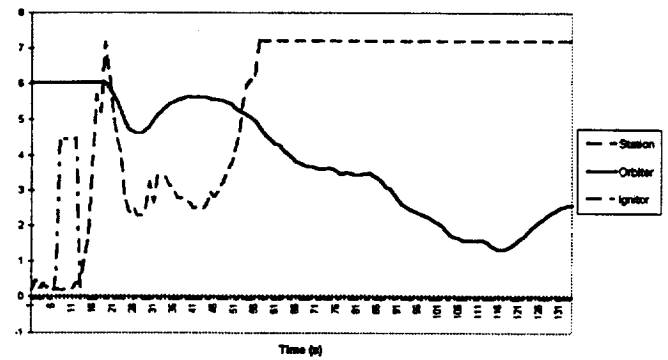


Figure 2. Traces of smoke detector signals as a function of time for a candle test (#16). Initial peak on both detectors is wax vapor released by the ignition process. The units on the vertical axis are arbitrary, the smoke detector data have been scaled so their dynamic ranges have similar vertical extent.

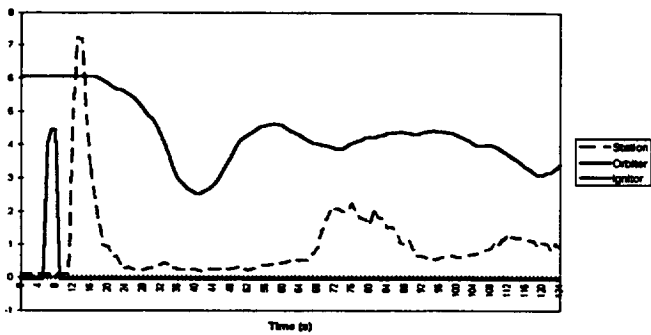


Figure 3. Traces of smoke detector signals as a function of time for a candle test (#6). Initial peak on both detectors is wax vapor released by the ignition process. The units on the vertical axis are arbitrary, the smoke detector data have been scaled so their dynamic ranges have similar vertical extent.

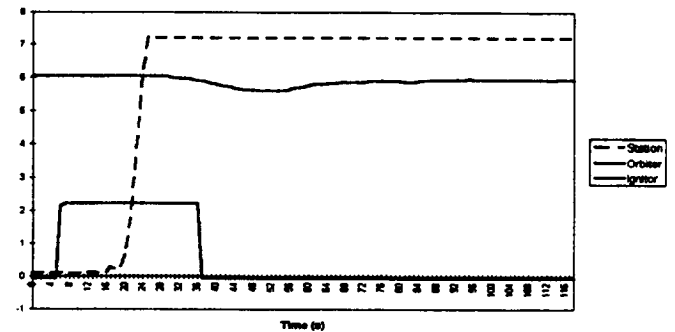


Figure 4. Traces of smoke detector signals as a function of time for a silicone rubber test (#7). The units on the vertical axis are arbitrary, the smoke detector data have been scaled so their dynamic ranges have similar vertical extent.

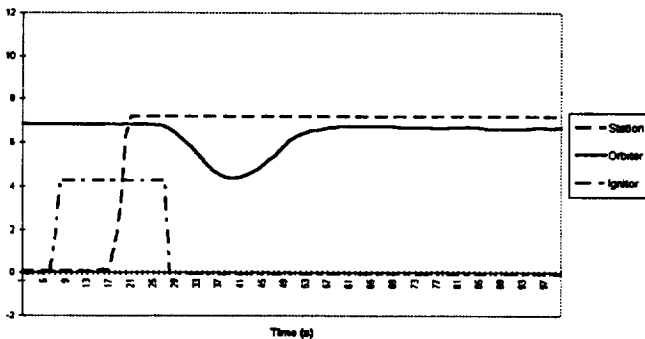


Figure 5. Traces of smoke detector signals as a function of time for a Kapton test (#14). The units on the vertical axis are arbitrary, the smoke detector data have been scaled so their dynamic ranges have similar vertical extent.

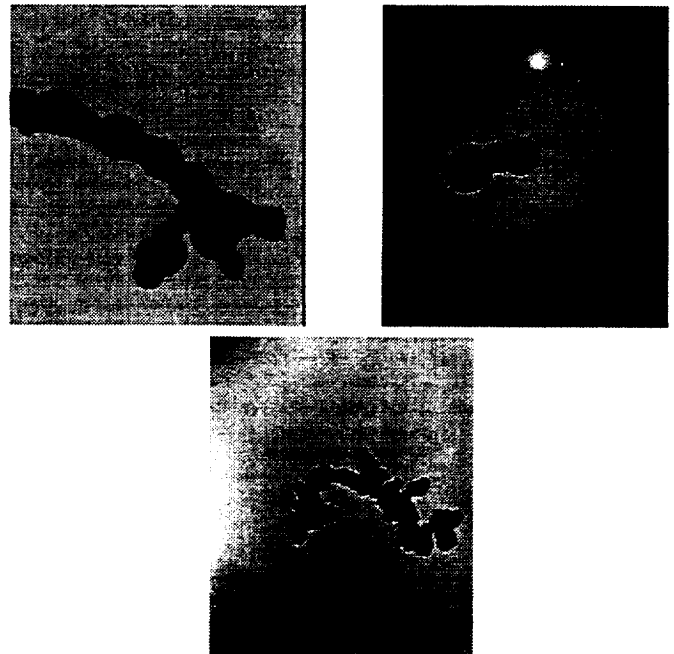


Figure 6. From top left, images of microgravity particulate from overheated Teflon and Kapton and Candle soot.