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Reducing Adhesion and Agglomeration Within a Cloud of Combustible Particles

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SUMMARY

The study of combustible particle clouds inside flame tubes is of fundamental scientific interest as well as a practical concern. Only the suspended concentration is important to the combustion process, so that assurances must be provided that a minimum of particles are adhered to the tube wall. This paper demonstrates experimentally the ability to minimize adhesion and agglomeration of acoustically-mixed lycopodium particles within a 5 cm diameter Lexan flame tube. The area density of particles (ADP) adhered to the wall of bare Lexan tubes was measured at greater than 100 particles/mm^2. The nature of adhesion was found to be clearly electrostatic, with the ADP level aggravated by increased mixing time, vigor, and the concentration of particles. Increases in the conductivity of the air and the tube wall did not affect ADP levels substantially. However, the observed adhesion was reduced to less than 10 p/mm^2 when the air was ionized by use of an alpha emitter mounted on the inner walls of the flame tube.

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INTRODUCTION

The study of combustible particle clouds is of fundamental scientific interest as well as a practical concern. Such clouds serve to spread fires in underground mining operations as well as contribute to the hazard involving grain bin explosions. The scientific study of such clouds is discussed in reference 1, with the primary issues being that of the effect of the concentration of combustible particles in air on the observed flame speed and overall cloud flammability.

These same issues are the subjects of classical premixed gas combustion studies. Such studies normally involve a quiescent combustible mixture of fuel, oxygen, and nitrogen contained in a "flame tube" of standard dimensions (5 cm diameter by 100 cm length). The tube is simultaneously opened and the mixture ignited at one end. The flame then propagates through the tube, achieving a steady flame speed and shape. The observed speed is a function of the fuel and oxidizer concentrations. As the initial fuel concentration is reduced, a concentration will be reached which will no longer support flame propagation. This concentration is called a flammability limit. Such experiments have been conducted primarily in normal gravity but also in reduced gravity.

A parallel study of premixed solid particle clouds is difficult due to gravitational settling of particles which destroys the requirement of quiescence and of uniform fuel concentration. In order to achieve uniformity, stirring devices or particle feeders (ref. 2) have been employed at the sacrifice of quiescence. As an alternative, a reduced gravity experiment called the Particle Cloud Combustion Experiment (PCCE) was conceived by Berlad which

emulates the characteristics of the classical premixed gas studies (ref. 1). It has been proposed to fly the PCCE on the Space Shuttle to take advantage of the reduced gravity environment it provides. The reduction of gravity minimizes particle settling while maintaining the other features of the classical experiment.

The operation of the PCCE, shown in figure 1, is to be performed in the following stages: (1) fuel particles are mixed into cloud form by acoustic energy from an inline speaker at one end of the tube; (2) when the concentration of particles in the cloud is sufficiently uniform as sensed by four optical detectors and visual observation, the speaker is de-energized and the particle motion allowed to decay toward quiescence; (3) an igniter is energized which burns through a membrane at one end of the tube and ignites the particle cloud; (4) the flame proceeds down the tube length, with its position and shape being photographed by high speed cameras. The desired experimental test conditions and requirements are displayed in table I.

A critical feature of the experiment is the determination of the concentration of the suspended particles. The optical detectors sense concentration by use of an optical attenuation technique (ref. 3). The strength of a collimated light beam is measured by a photocell and the signal strength is a function of the number of particles in the beam path. This oft-used technique does not differentiate between particles which are suspended in the air and particles which are adhered to the tube wall. Particles adhered to the tube walls do not participate in the combustion process and as such do not affect the observed flame speed. Since only the suspended concentration is important to the combustion process and since the optical detectors can not separate the adhered concentration from the suspended concentration, assurances must be provided that a minimum of particles are adhered to the wall. Historically combustion experiments either ignored this effect or assured clean walls by directing an air jet at the wall surfaces in the neighborhood of the detectors (ref. 4); the latter technique is not applicable for the PCCE which requires quiescence.

A secondary problem is the presumed monomeric particle size of the PCCE. The combustible fuel particles are lycopodium which are nominally 28±4 μm diameter spheres (refs. 2 and 5). If individual particles become agglomerated, a longer preheat and vaporization time occurs prior to combustion. The light scattering characteristic is different for large and small particles as well, making the interpretation of the attenuated light signal difficult if the particle size is variable and unknown. Therefore, it is also important for correct understanding of the experimental combustion results that the PCCE employ a known and time-independent size distribution, preferably maintaining its original monomeric nature.

It is the objective of this study to demonstrate experimentally the ability to minimize particle adhesion and agglomeration of acoustically-mixed particles of lycopodium within a 5 cm diameter Lexan flame tube. The goal was to reduce the area density of (wall-adhered) particles (ADP) below 10 particles/mm^2; this value of ADP ensured the uncertainty in the suspended concentration of particles would be less than 5 percent, neglecting instrumentation error. Early in the PCCE development test program, it was determined that this requirement was not being met since ADP levels exceeded a countable value, set nominally at 100 p/mm^2. Furthermore, it was observed that adhesion was multilayered, i.e., particles adhered not only to the wall, but to each other.

Berlad and Joshi postulated that the adhesion was electrostatic in nature (ref. 6). The particles, air, and tube wall materials were dielectric; the motion of the particles relative to the air and tube walls produced triboelectric forces which charged the particles. They performed an experimental study in which various conductive surface coatings were applied to acrylic materials. It was hoped that the addition of a conductive path along the tube wall would reduce ADP levels to an acceptable value. Their experimental technique is shown on figure 2. Particles were loaded into a tube which was closed on both ends. Though the amounts of lycopodium particles were unmeasured in these tests, it was approximated later to be about 500 mg (±250 mg). The top wall was the test surface which had different surface coatings applied. The tube was shaken by hand for 10 to 60 sec to simulate the mixing process employed in the PCCE. The tube was then tapped gently three times; they observed that particles fell from the top surface as a result of this tapping. The test surface was then removed from the tube and examined under a microscope; the ADP level was measured through a gridded reticule of the microscope at six or seven locations of 1 mm^2 area. Figure 3 displays their results. In general, the ADP level diminshed with increased surface electrical conductivity. They therefore recommended that the PCCE Lexan flame tube be coated similarly with conductive material to reduce the ADP level.

BASELINE EXPERIMENTS

This author performed a series of experiments in 15 and 25 cm length Lexan tubes with the tube inner walls coated with conductive material. The conductive material was gold which had been vapor deposited on the inner surface at a thickness on the order of 100 to 400Å. The measured resistance of the coating ranged from 5 to 20 Ω per square for different tubes.

Figure 4 displays the test section. Conductive endcaps were used to contain the powder inside the tubes; copper tape or silvered paint were used to connect the gold-coated tube wall to the endcap, and then to ground potential. These endcaps and connectors provided an additional resistance to ground on the order of 1 to 2 Ω. The lycopodium powder, weighing between 200 and 500 mg, was placed on one endcap prior to the test. The tube was then held at both endcaps, and shaken vigorously by hand for 5 to 10 sec (±1 sec), turned 180° (top to bottom), and shaken again for 5 to 10 sec. The general tube appearance was observed for patches of adhered lycopodium. When a patch was clearly visible, the ADP was always greater than 100 p/mm^2. In tests where few or no patches were observed, the tube was examined under a microscope. The ADP was measured at 6 or 7 spots, each of area equal to 6 mm^2. Most tests were performed inside a glove box maintained at room temperature and less than 5 percent humidity; some tests were performed at room humidity. In some cases the tube was held vertically; in other cases the particles were initially spread along the horizontally oriented tube wall. In several tests the ground path resistance was varied. Appendix 1 contains a complete test description and representative test results, from which the following general observations were made:

(1) ADP levels were unacceptably high regardless of the surface coating properties, local humidity, operator handling procedures, or the electrical resistance to ground potential.

(2) ADP levels increased with mixing vigor, mixing time, and the concentration of lycopodium particles.

(3) The ADP level diminshed somewhat when the tube was again tapped a day or two later, suggesting the electrostatic charge dissipated substantially in this time frame.

(4) Multilayered adhesion occurred readily with no apparent limit to the number of layers which formed.

(5) The number of layers increased with increased mixing time and the number of lycopodium particles employed.

In a few tests, the Lexan tube was replaced by a copper tube. Despite the copper's high electrical conductivity, the ADP level was uncountably high. From this test it was concluded that the addition of a conductive wall could not dissipate with sufficient rapidity the electrostatic charge induced on the particles by the mixing process.

Next, tests were performed where the above procedure was followed exactly, except the endcaps were aluminized Mylar and the mixing method was acoustic to reflect the actual mixing process planned for the PCCE. Figure 5 displays the test apparatus. The short 15 cm tubes were used to allow mixing to occur throughout the entire tube in normal gravity. The selected frequencies were those which produced by visual observation the smallest void spaces in the particle cloud. In these tests described in table II, the speaker was energized for various time periods at various power settings. In all cases, patches of adhered lycopodium were observed in the tube. In some tests, a solenoid-operated tapper was energized at a rate of 1.7 Hz during the mixing process; however this simultaneous tapping failed to prevent excessive adhesion. In the long duration tests, the particles were observed to agglomerate in suspension as well as to adhere to the wall. The agglomerated masses remained so indefinately. They were then examined under a scanning electron microscope to determine if: (1) they had been damaged due to acoustic mixing; and (2) if they appeared interlocked, such that the agglomeration was mechanical. Figure 6 displays typical SEM results at a magnification of 2000. No structural damage to the particles was observed. Contact between particles appeared to be edge-to-edge, rather than interlocked, suggesting the particle-particle agglomeration was not mechanical.[1]

A final series of baseline tests were conducted in reduced gravity aboard an aircraft flying parabolic trajectories. In one series of tests in a full length flame tube, excessive ADP levels were observed (ref. 7). Also a simple test was conducted in which a 1-in. plastic bottle containing lycopodium was shaken manually during a trajectory. Particles were observed to agglomerate into a ball, and then drift toward the wall of the bottle (Private communication with M. Brace of NASA Lewis). These tests verified the problem persisted in reduced gravity.

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[1]As further assurance that the particle-wall adhesion was not mechanical in nature, a tube cleaning procedure development program had been instituted previously. See Appendix 1.

METHODS TO REDUCE ADHESION

Some methods available to reduce electrostatic adhesion and agglomeration are described by Loeb (ref. 8) and include: (a) increase the conductivity of the dielectric materials, i.e., in practical terms increase the humidity of the air and the tube surface conductivity; and (b) ionize the air by (1) corona discharge from high voltage wires; (2) alpha, beta, or gamma emission from radioactive materials; and (3) ultraviolet sources. The ionized air serves to neutralize the surface charge built up on the particles by triboelectric forces.

The tests described above revealed that method (a) alone was insuffficient to reduce ADP levels to an acceptable value. Furthermore, increased humidity would tamper with the PCCE combustion modelling effort. Most of the other methods appeared to be inapplicable to the PCCE. A corona discharge might ignite the particles at an unwanted time and also would serve only to reduce adhesion in the neighborhood of the discharge, i.e., within an inch or so of the discharge location. Beta and gamma emitters present a containment and shielding hazard. Ultraviolet sources require an electrical power and physical volume which impinged on the limited available resources allotted to the PCCE.

Instead, an alpha emitter, polonium-210, was selected as the most feasible technique due to the following features: (1) it is a high energy emitter (5.3 keV) which is virtually absorbed within 0.001 in. of gold, and would not penetrate the Lexan tube; (2) it has a range slightly more than 1 in. in air, and thus if mounted at appropriate positions inside the Lexan tube could reach all adhered particles; (3) it is a passive system requiring no electrical power and no additional physical volume outside the flame tube; (4) it is available commercially; and (5) it has been used widely in industrial settings, such as paper mills and photographic film processing plants, in order to reduce electrostatic adhesion and accidential corona discharge.

Polonium-210 is sold commerically in a "sealed source" form as shown on figure 7. An understanding of the manufacturing process involved in the commerical product is helpful to understand the potential hazard of using an alpha emitter (the use of any alpha emitter demands extra safety precautions; these are described later in the paper). Polonium-210 is bonded into the fissures of a ceramic microsphere (approximate size of 60 μm). Several microspheres are stirred in an adhesive solution; the solution is then sprayed onto aluminum strips. A thin epoxy coating(s) is then applied over the entire strip to provide extra assurance that the microspheres will remain attached to the aluminum. When sold in "sealed source" form, the strips are mounted to an aluminum housing and a wire screen placed over the strip to prevent direct contact. The number of attached microspheres and epoxy overlays vary depending on the desired source strength of the strip. Table III displays the dimension and strengths of standard products.

EXPERIMENTS TO REDUCE ADHESION

The first set of experiments, listed in table IV, was designed to determine qualitatively if previously adhered particles could be removed more readily from the tube wall by exposure to the alpha emitter. In these tests, particles were mixed in a 6-in. gold-coated tube via the acoustic method described above

for a period of 10 to 60 sec. The top endcap was then removed, and a "sealed source" was inserted into the tube. Portions of the tube wall were obscured from the alpha emitter. Also its distance from the tube wall was varied. The time of exposure of the source to the tube wall was varied from seconds to several minutes. In most tests, a 15 cm long strip was inserted with a nominal source strength of 2 μCi/2.5 cm; in some cases, a circular spot source three times stronger was employed. After removal of the source strip, the tube was tapped three times. The observations from these tests were:

(1) Patches of heavily adhered lycopodium were removed completely (rendered invisible to the naked eye) after exposure to the source; these same patches could not be removed by tapping alone.

(2) The adhered lycopodium behind the source was unaffected by the source, i.e., there was a definite view factor attributable to the source; in one test, the circular spot source removed a circular patch of wall-adhered lycopodium.

(3) Multilayered adhesion could be removed, but only after repeated cycles of exposure to the source, and tapping of the tube walls.

(4) Adhered particles were removed more easily when the source was located close to the tube wall and/or when the exposure time was increased.

(5) Tapping was required to remove the particles from the tube wall; the sources served to reduce the force with which the particles were adhered, but did not actually remove the particles from the wall.

As a result of these tests, a second set of experiments was performed to determine if adhesion could be prevented completely. In these tests, source strips were removed from their aluminum housing, and epoxied to the inner walls of the tube, as shown in figure 8.[2] The strips were mounted along the full axial length of 15 cm Lexan tubes, and located at 90° intervals around the inner circumferance of the tube. In this way, virtually the entire tube surface was exposed to the sources. Near the tube bottom, where acoustic mixing was observed previously to be most vigorous, extra strip segments were added. The solenoid-operated tapper was employed to provide a reproducible impact on the tube wall. It was operated in parallel with the speaker used for mixing, tapping at a rate of about 1.7 Hz throughout the time period the speaker was energized, except as noted on table V. After each test, the tube was disassembled, and the end pieces replaced with a vinyl tape cover. The tube was then hand carried to a nearby lab table where ADP measurements at 12 tube locations were recorded; the measurement locations are shown schematically on figure 9 and were the same in each test. Photographs were taken through the microscope objective at a x70 magnification; the photographed area was about 12 mm^2. The particles within the photographed areas were later counted, and an average ADP level recorded (as shown in table V).

Parametric tests were performed to determine the effects of: (1) source strength; (2) mixing time; (2) inner wall conductivity; and (4) number of

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[2]Due to the potential hazard and licensing requirements, these tests were performed at the source strip manufacturer's production facility and with assistance from their trained personnel.

particles employed. In order to determine if an improved inner wall conductivity augmented the ability of the sources, tests were conducted in bare Lexan tubes and ones which included a vapor-deposited gold coating. In order to ensure an accurate estimate of the source strength, source strips manufactured one week before the test period were employed. It should be noted that these tests were performed in room humidity under a fume hood. The safety precautions which were followed are described in the next section of the paper.

Table V summarizes the test variables and results. Figure 10 displays a comparison of bare Lexan tubes with and without sources. The amount of lycopodium adhered to the wall in the tube without sources or tapping was sufficient to render the wall opaque to the naked eye. When the sources are employed, the visibility through the tube walls remained unchanged before or after mixing. Figure 11 shows typical photographic records of the ADP levels at a few sites. In general the following observations are made:

(1) Strips with source strengths of 0.1 μCi/cm or greater maintained ADP levels to less than 10 particles/mm^2 on average with mixing times of 180 sec and a total powder loading of 250 mg. Adhesion levels were worst in the lower section of the tubes.

(2) The inclusion or exclusion of a gold coating had no discernible effect on the ADP level.

(3) Heavy adhesion was observed in the lowest portion of the tube with a continuous mixing time of 300 sec and 500 mg of lycopodium powder.

(4) ADP levels were acceptable with 500 mg powder and the mixing sequence operated as follows: 90 sec mixing and tapping; 10 sec of tapping; 90 more seconds of mixing and tapping, and 3 sec of tapping.

(5) A 60 sec test without tapping produced patches of heavy adhesion in the bottom portion of the tube. Three taps following the test removed most of the visible powder in these patches.

One problem that occurred during testing was that small amounts of lycopodium became lodged under the aluminum strips, as shown on figure 12. The gluing of the strips to the tube wall was imperfect, thus allowing the lycopodium access under the strips. The problem can be eliminated in future tests with an improved fabrication procedure discussed later in the paper.

A final series of tests determined that weaker strips would still be effective, even in lower humidity. Table VI displays the test conditions and results done in a glove box maintained at 5 to 20 percent relative humidity. When the alpha emitters viewed less than 100 percent of the tube area, patches of adhesion were observed away from the emitters. Also, when mixing was done without simultaneous tapping some excessive adhesion occurred. This adhesion was removed by tapping after the mixing was over. When both mixing and tapping were simultaneous, the ADP levels were acceptable (i.e., <10 p/mm^2). The source strips in the final series of tests were used 6 to 8 weeks after their manufacture, and thus had a calculated source strength of about 0.08 μCi/cm/strip, weaker than those previously used.

SAFETY PRECAUTIONS AND HAZARD ASSESSMENT

The testing room at the manufacturing facility was divided into three zones, representing likely levels of hazard; each zone was outfitted with alpha monitors. In the zone in which the reported testing took place, personnel wore goggles as well as plastic lab coats, globes, and shoe covers. All tests in this zone were conducted inside a fume hood with a face velocity of 300 cm/min. When personnel left the most hazardous zone, they monitored themselves for contaimination, first with and then without the plastic clothing.

At each subsequent zone, they monitored themselves again. In no cases were personnel found to be contaminated above background levels. On returning, the process was reversed, with new plastic clothing being employed each time.

At the end of each mixing test, the tube assembly was dismantled and the lycopodium particles along with each component were measured for contamination by a calibrated alpha monitor. Instrument error was estimated at ±5 percent. The measurements were taken by a trained radiation safety technician at the manufacturing facility.

As noted on table V, the first test involved the strongest sources, each strip being rated at 2.4 μCi/cm. This strength is accomplished by increasing the microsphere density and omitting the epoxy overlays from the manufacturing process described earlier. Contamination levels in the particles and on the end pieces were in the range of 5000 to 10 000 counts/min. This level is considered quite hazardous, and all future tests with this type of source strip were cancelled. The end pieces were scrubbed and retested by the radiation safety technician until no trace of contamination could be measured. These pieces were then reused in subsequent tests.

Subsequent tests used strips with two different source strengths; 0.8 μCi/2.54 cm and 0.1 μCi/2.54 cm. In the one test with the 0.8 μCi/2.54 cm, contamination levels were measured in the range of 5000 counts/min. Future use of these strips was again cancelled.

The results with the weaker strips were markedly different. Up to a total mixing time of 300 sec in a series of tests involving 250 mg of lycopodium, the mixing in tubes containing the weaker strips produced no measurable counts in the components or the powder. After an additional test with one tube mixing 500 mg of lycopodium for 300 sec, contamination levels were measured in the range of 500 to 1000 counts/min. As a result of these tests, it was concluded that the tube components remained free of contamination for at least 300 sec but less than 600 sec of mixing time.

It should be noted that in one baseline test involving a tube without sources, a mylar diaphragm fatigued and tore from the acoustic cycling. It was fortuitous that this test involved a tube with no source strips. If the failure had occurred in a test when source material had become dislodged from the strip, some microspheres may have become airborne and possibly been inhaled. This potential problem was minimized by performing the tests under a fume hood.

After all tests in table V were completed, standard "wipe" tests were performed on the inner walls of the tubes. Smear measurements were taken at four wall locations in a spiral down the tube and at three locations on the source

strips themselves. Table VII displays the results of these tests. In each case, the lowest portion of the tube yielded the highest level of contamination. It is in this region that the mixing was observed to be most vigorous. As expected, the contamination levels for the most powerful sources were the largest. The contamination in the tubes containing the weaker sources reached their levels because the tubes in general were used until they failed due to "contamination," defined as an alpha count above background levels measured in any test component.

A potential hazard calculation may be attempted based on the above measurements. The Nuclear Regulatory Commission uses a standard based on Maximum Permissible Concentration (MPC), (defined as 1 MPC = 2^{-10} μCi/cc) and a standard human breathing rate of 1.2^{6} cc/hr. For a person to inhale 1 MPC in 1 hr, he would inhale 2.4^{-4} μCi in that hour. The NRC requires action if a person is exposed to an environment where he would receive an exposure of 40 MPC-hr in any one-quarter year. The NRC requires the submittal of a report if a person is exposed to an environment of 520 MPC-hr in any one-quarter year. From the above, it may be seen that if a person inhales 40 times 2.4^{-4} or 9.6^{-3} μCi in any quarter-year, action is required.

The results of the wipe tests can be used to compare with this requirement. The wipe tests covered only ~20 percent of the total surface area. Furthermore, a 24 percent collection efficiency should be assigned to the measured values (Personal communication with R. Schweiss of the 3M Corp. Static Control Center). Assuming an average contamination reading of 100 counts/min (cpm), the total amount of escaped Po-210 material which might be inhaled is found to be 9.5^{-4} μCi, from:

$$100 \text{ cpm} \times (1/0.24 \text{ eff}) \times (1 \text{ μCi}/2.2^{6} \text{ cpm}) \times (1/0.20 \text{ area})$$

Thus if all the material which escaped from the strips after testing became airborne and were inhaled by a single person, his exposure as a result of the testing would be about an order of magnitude less than that requiring NRC action. This calculation is not conservative in the sense that it reflects the results of testing in one tube, and an operator might be exposed to several tubes. It is however very conservative in the sense that it is highly unlikely that all the material escaping from the strips would first become airborne, then inhaled entirely by a single operator. Furthermore, contamination readings of this level were taken after a number of tests with an individual tube. In fact tubes were used until they exhibited contamination, i.e., they were effectively failure tested. This level of escaped material was not normally measured after a single test.

DISCUSSION OF RESULTS

As postulated by Berlad et al. (ref. 1), the adhesion observed in the PCCE tests is undoubtably electrostatic in origin, and not mechanical, since: (1) ADP levels differed greatly in similarly cleaned tubes whose only known difference was the inclusion or exclusion of the source strips; (2) the scanning electron microscope pictures revealed only edge-to-edge contact; and (3) mechanical adhesion due to oil deposited on the wall could not account for the observed multilayered adhesion. However, in contradiction to the postulate in (ref. 1) regarding "saturated" walls, there was no apparent limit to the number of layers of adhered lycopodium which could develop in the absence of alpha sources.

The observations from the baseline experiments are consistent with existing literature studies of coal dust travelling through iron pipes and corn flour rubbing against brass plates (ref. 8). In fact, Loeb states "The real problems occur, where, while one side of the charge generating system may be metal and grounded the other part of the system is a plastic, a fabric, or a nonconductor of some sort. This can lead to ..undesirable adhesion.. (and).. accumulation of dusts by electrostatic action."

The following variables were seen to aggravate adhesion: mixing time, mixing vigor, and the number of lycopodium particles. This observation may be generalized to state that adhesion is aggravated with the number, frequency, and force of particle-particle and particle-wall collisions.

These results confirm that the adhesion and agglomeration levels are governed by a rate process. Berlad et al. (ref. 4) envisioned a simple kinetic scheme involving agglomeration and deagglomeration processes. In the absence of a mechanism to neutralize the charged particles, clusters of polymeric particles will form. However when the source strips provided this mechanism at a sufficient magnitude and rate, the particles remained monomeric. The photographs of adhered lycopodium displayed monomeric particles, so the strips were a sufficient source to overcome the agglomeration due to particle collisions.

It should be noted that the concentration of particles in the tubes was selected to be excessive by 5 to 10 times that expected to be used in the PCCE, thus representing a severe test of the technique. For the eventual application to the PCCE, the number and frequency of collisions can and should be minimized. This may be accomplished by positioning the lycopodium particles prior to mixing as remotely from each other as possible, and by reducing the time during which the speaker is energized to a minimum.

There are several other implications of these results for the PCCE. The exact PCCE mixing operation in reduced gravity is undetermined currently, but might require the speaker to cycle on and off to achieve a uniform cloud. During the off cycles the source strips will continue to ionize air and tapper should continue to operate and minimize wall adhesion and agglomeration. Also, if the PCCE is flown on the Shuttle, the last access to the experimental hardware is usually 60 to 120 days prior to flight. The polonium-210 has a half-life of 138 days. The experiment then must be constructed initially with an amount of Po-210 which will retain sufficient strength at the time of the actual experiment. The experiments described above suggest a source strength of 0.08 μCi/2.54 cm/strip is satisfactory if the mixing time is maintained at 60 sec or less. The strips can be bought commerically with a strength of 0.2 μCi/2.54 cm/strip, while maintaining the epoxy overlays for safety. The calculated shelf life (i.e., the time between last access and the conduct of the experiment) is then on the order of 200 to 250 days. While in storage, the strips will be producing helium inside the flame tube. Assuming a 25 cm length flame tube, about 36 μCi of Po-210 would be used during the experiment. If the storage time is picked as 138 days (for convenience in this calculation), then 72 μCi are needed initially to be loaded in the tube. During this 138 day waiting period, it is calculated that about 15^{-8} g of helium will be produced, equivalent to a partial pressure of 6.04^{-7} atm and should have no effect on the observed combustion results (see Appendix 2 for calculation).

The reason the source material escapes the strips is postulated as being caused by the lycopodium particles scrubbing and eroding the epoxy overlay during mixing, analogous to "sand blasting." This process is such that not only the epoxy may be eroded, but individual microspheres also may be dislodged or fractured. The microsphere construction is designed such that if it remains integral, polonium-210 retained in its fissures will settle rapidly in normal gravity. Also if ingested, the Po-210 leachibility from the microsphere is so low as to allow the microsphere to pass through a person's system without significant effect. However, if the microsphere integrity is compromised by the mixing process, a greater hazard exists. It was undetermined in the reported tests whether the microspheres were merely dislodged or if they fractured.

One possible method of eliminating the scrubbing process is to recess the aluminum strip in the flame tube and cover it with a <25 μm mesh screen. This mesh size would prevent lycopodium particles from reaching the epoxy but would still allow the air inside the tube to ionize. This design emulates the commerical product's "sealed source" construction.

While the potential health hazard should not be minimized, it should be noted that several tests were conducted when no contamination was detected. The guidelines of "safe" lifetimes for use of the weak strips should be followed. Furthermore, when these strips were failure-tested, the amount of Po-210 which escaped the weak strips was small, and below NRC guidelines. Lastly, it should be noted that the strips when mounted inside a flame tube with its endcaps in place are effectively "sealed sources," regardless of the use of a wire mesh. Official approval of this classification requires submittal to the NRC of the design and operating plans for the experiment.

The problem of lycopodium lodging under the aluminum strips was potentially solved by fabricating thin aluminum strips with curved backs and knife edges. The curvature matched the tube curvature. A thin double-sided tape was then applied to the strip back. A special fixture was designed for pressing the strips into place in a 30 in. tube. Pressure was applied near the strip edges, with care being taken to avoid any pressure or contact with the Polonium-210 elements. Figure 13 displays a full length flame tube with the strips mounted inside. No tests, however, were conducted to verify this solution.

POTENTIAL PROBLEMS AND CONCERNS

To ensure safety, the integrity of the strips should be verified when a combustion event occurs. The epoxy overlays are expected to survive temperatures up to 90 °C. It is known that the flame passage is sufficiently rapid that the outer wall temperature change due to combustion is not noticable to the touch. The Lexan tube and aluminum strips have a large heat capacity and should prevent the inner wall temperature from rising significantly as well. However tests with bare aluminum strips and polonium-free microspheres should be performed to determine the range of inner wall temperatures which can be expected to occur in the actual experiment.

There are two additional concerns regarding use of this technique in reduced gravity. First any airborne Po-210 will not settle as readily in reduced gravity. Thus, caution should be ensured that the experimental hardware is well-contained and not exposed to humans in reduced gravity. Secondly, it is the author's common experience that experiments in reduced gravity do not behave as predicted. Also the removal of adhered particles by tapping may

not work as well in reduced gravity. Therefore, the effectiveness of the technique should be tested in short duration reduced gravity tests prior to its planned use in a space-based experiment.

CONCLUSIONS AND RECOMMENDATIONS

1. As postulated by Berlad and Joshi, the nature of adhesion is clearly electrostatic. The ADP levels are aggravated by increased mixing time, vigor, and the number of particles.

2. Increases in the electrical conductivity of the air and the tube wall were insufficient to reduce ADP levels to an acceptable value.

3. The observed adhesion can be reduced to acceptable ADP levels by use of an alpha emitter of strength 0.08 μCi/2.54 cm/strip position in the flame tube to ensure complete and effective wall exposure to the strips. This strength is not necessarily a limiting value, but was instead the lowest strength strip which was tested. This statement may not be true for richer mixtures, longer mixing times, or increased acoustic power.

4. Alpha emitters were capable of removing previously adhered particles, as well as preventing adhesion occurrences. However, if the particles are adhered prior to exposure to the source, it may take several minutes of exposure to induce their removal. This time frame is at least two orders of magnitude less than the time frame required to reduce adhesion by coating the walls with conductive material.

5. No contamination was measured when 0.1 μCi/2.54 cm/strip sources were used in cumulative testing up to 300 sec of mixing. Serious contamination was observed however with strips of strength 0.8 to 2.4 μCi/2.54 cm/strip, due to their different manufacturing processes.

6. The levels of contamination with the failure-tested weak strips were below the cited NRC requirements.

7. The shelf life of the PCCE employing commerically-available strips is on the order of 200 to 250 days. During this time period, an insignificant amount of helium will be produced by the level of Po-210 required inside the flame tube assembly.

8. Further tests should be performed in order to verify: (a) the strip integrity during a combustion event; (b) the technique's performance in reduced gravity; and (c) the fabrication procedure to prevent particles from reaching the strip surface and being lodged under the strip.

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APPENDIX 1

TEST PROCEDURE AND RESULTS FOR BASELINE TESTS

A number of tests were conducted with different variables to determine their effects of lycopodium particle adhesion on gold-coated Lexan tubes. The variables investigated were:

1. Effect of grounding (busbar).
2. Antistatic coating - ANTISTAT-79.
3. Effect of tube material (copper versus gold-coated Lexan).
4. Mixing time.
5. Amount of lycopodium.
6. Tube orientation - the direction in and frequency of particle impacts against the walls.
7. Type of mixing.

Before discussing the results of these variables, a brief description of preparation of the experiment, the manner of performing the experiment, and qualitative observations are presented.

PROCEDURE

The general test procedure used in the tests was:

1. Cleaning of tube with ethanol and air drying.
2. Transport to dry box.
3. Bottom end cap attachment and ground connection.
4. Loading of lycopodium on bottom end cap.
5. Top end cap connection.
6. Mixing of particles.
7. Observation of particle adhesion characteristics.
8. Tapping of tube to dislodge "loosely held" particles.
9. Observation of particle adhesion characteristics.
10. Removal of tube from dry box.
11. Quantitative particle counts.
12. Cleaning of tube with dry air for next test (go to 2).

This general procedure is now expanded:

1. Cleaning

   The tubes were cleaned initially by spraying the inside walls with 190° ethyl aclohol. The alcohol dried in air for roughly 1 min. (Earlier tests with various cleaning agents revealed ethyl alcohol as the best cleaning agent amongst R113, alconox soap, ultrasonic scrubbing w/distilled water, and ethyl alcohol.)

2. Transport to Dry Box

   The tubes were handled primarily at the ends to minimize static charge buildup during transport.

3. Bottom End Cap Attachment and Ground Connection

   Aluminum foil was used for end cap material for most of the tests. It was held snuggly to the tube with rubber bands. Copper end plugs, which snuggly fit inside the ends of the tube, started to be used on the 37th test. When aluminum foil was used to seal the tube, grounding was provided by means of silver paint strips and copper tape overlapping the gold-coated inside surface of the tube. Alligator clips were then attached to the copper tape "ears" and connected to the dry box outlet which, in turn, was connected to the building ground. When the copper end plugs were used, the grounding was slightly more direct (no alligator clips) leading straight to the box outlet and from there to the building ground. The measured resistance between the end cap and the building ground was 0.5 Ω.

4. Loading of Lycopodium on bottom End Cap

   Lycopodium was placed on the bottom end cap (tube in vertical position.) In early tests, the amount of lycopodium was not measured, but was around 1 g (i.e., several times stoichiometric amount.) In this way, it proved to be "worst case" testing (to be discussed later).

5. Top End Cap Connection

   Same as discussed in paragraph 3. When 2 grounds were used, the second ground was to the top connection.

6. Mixing

   Manual Method: The tube was held vertically by the end caps and vigorously shaken up and down and then quickly turned upside down and shaken again. Early tests were not timed but were on the order of 10 sec in each position. In a few tests, ground connection was lost during mixing (noted on attached sheets).

7. Observation of Particle Adhesion Characteristics

   While still in the dry box, the tube was examined for patches of particularly heavy particle adhesion.

8. Tapping of Tube to Dislodge Loosely Held Particles

   With the ground still connected, the tube was placed on the floor of the dry box and tapped three times with tongs.

9. Observation of Particle Adhesion Characteristics

   The tube was again observed to see if particles of lycopodium had been dislodged.

10. Removal of Tube from Dry Box

    At this point, the ground was disconnected. The copper end caps, when used, were removed. The tube was taken out of the dry box and a more thorough qualitative observation was made.

11. Quantitative Particle Counts

    If little general adhesion was observed, then the tube was placed under the microscope and particle counts were taken at representative locations along the length of the tube. Both axial and radial profiles were taken.

12. Cleaning of Tube with Dry Air

    The lycopodium particles adhering to the tube were removed by blowing compressed air across the tube walls. This removed all particles.

    Subsequent tests with the tubes proceeded as above, beginning with paragraph 2, i.e., the ethyl alcohol cleaning was not repeated.

MODIFICATIONS TO ABOVE PROCEDURE

1. Some tests were done out of the dry box to test the effect of using room air and lycopodium exposed to room air.

2. One test was done with gold-coated lycopodium. assuming a 100 Å thick layer of gold on the lycopodium, 100-mg test was compared with a 66-mg uncoated lycopodium tests. The weights were selected to have an equal (approx.) number of particles.

3. Some tests were done with the tube held horizontally. This produced more frequent collisons with the tube walls.

4. With the copper tubes, grounding was directly to the tube wall.

DISCUSSION OF RESULTS

The following is a brief description of the findings from this set of tests.

1. Effect of Grounding

   Grounding did not make much difference in particle adhesion. Inconsistency in initial results, however, did occur. This was due to inconsistency in experimental technique. (These tests were the first done). Busbar did not improve the particle adhesion.

2. Antistatic Liquid Coating (ANTISTAT-79)

   Initially it appeared that ANTISTAT-79 wiped on the outside surface of the tube greatly lowered the particle adhesion. However, further testing showed that the early success was due to the inability to shake the tubes. The early tests were with 10" tubes which were difficult to handle in the dry box. Shaking with them was less vigorous, thus less static charge was built up. Later tests with the 10 in. tubes in a horizontal position showed antistat to make little difference.

3. Effect of Tube Material

   Adhesion was similar (excessive) in the copper tubes and the gold-coated Lexan.

4. Mixing Time

   The clear trend was that longer mixing produced more adhesion. The reason for this trend is believed to be electrostatic. Longer mixing produces more particle collisions, and likely more charged particles and therefore more adhesion.

5. Amount of Lycopodium

   The amount of powder originally placed in the tube had a clear influence on how much stuck to the walls; the more powder placed inside the tube, the more powder stuck to the walls. The denser clouds created more static charge because of the more frequent particle to particle interaction.

6. Tube Orientation

   The adhesion was strongly related to the way the tube was held and shaken. If the tube was held horizontally and shaken up and down, the adhesion was much heavier than if the tube was held vertically and shaken up and down. The number and frequency of particle-wall and particle-particle collisions increased greatly when the tube was horizontal as opposed to vertical.

7. Type of Mixing

   Mixing via acoustics produced a cloud only in the lower portions of the 10 in. tube. In those portions of the tube where vigorous mixing was achieved acoustically, heavy adhesion was observed. Because mixing was confined to a small region, a large number of particle collisions occurred producing more static charge.

General Notes

1. Particle counts with a microscope were not taken when it was obvious that the particle density was excessively high. (Greater than 50 particles/mm^2).

2. Most of the time the particles were unevenly distributed throughout the tube. There were some patches heavily coated and there were spots almost clear of particles.

TABLE A-1

BASELINE TESTS

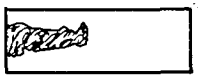
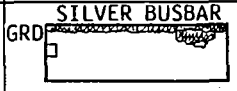
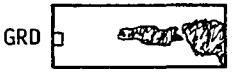
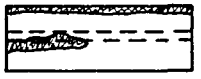
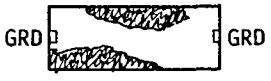
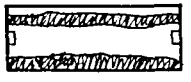
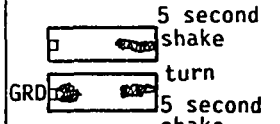
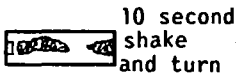
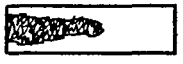
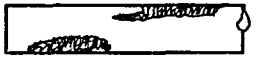
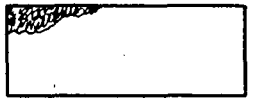
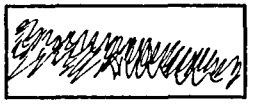
Test number, date and tester	Tube I.D. type/length material	LYCO mixing method	Grounding method	Antistat	Testing for ?	Tube appearance and particle count (Number 16 mm^2)	Remarks, results
1 1/31/86 HR	A trans. temp. 6" straight	Shake turn shake	Copper tape 1 connection at top	None	Effect of copper tape		Heavy patch of LYCO on portions of tube
3 1/31/86 HR	A trans. temp. 6" straight	Shake turn shake	Copper tape 1 connection at top	Yes	Effect of liquid anti-station outside of tube		Patches of LYCO remain, but less heavy patch located opposite end of ground connection
6 2/3/86 HR, RK	B trans. temp. 6" straight	Shake turn shake	Copper tape 2 conections, top/bottom	Yes	Effect of 2 grounds connections		Several patches, opposite from trend of previous tests
14 2/3/86 HR, RK	A trans. temp. 6" straight	Shake turn shake	Copper tape 1 connection direct to bldg ground	Yes	Effect of 3 day room LYCO storage (exposed to humidity)		Heavy patch of LYCO-very poor results
15 2/4/86 HR	A trans. temp. 6" straight	Shake turn shake	Copper tape 2 connects to ground	Yes	Effect of extra anti-stat, very careful handling and NO ground!!		Heavy patch of LYCO, NO GROUND! Too many particles to warrant counting
21 2/4/86 HR	A trans. temp. 6" straight	Roll, slow turn	Copper tape 2 ground connects	Yes	Effect of LYCO mixing method (rolling)		Rolled LYCO onto surface then let it slide down, back and forth. Heavy streaks on surface when I let LYCO roll down. Repeated test on other side with same result. Rolling is source of streaking.
22 2/4/86 HR	B trans. temp. 6" straight	Shake (no turn)	Copper tape 2 ground connects	Yes 1/31 and 2/3	Effect of LYCO mixing method		Heavy patch near busbar. Probable source due to initial shaking.
24 2/5/86 HR	A	Shake turn shake	Copper tape	Yes	Effect of poor handling-hand around outside of tube		Heavy blotches, especially near hand-print
27 2/5/86 HR	B trans. temp. 6" straight	Hori- zontal shake	Copper tape 2 ground connects	Yes	Effect of horizontal shake		Super heavy coating on bottom. Then tube rotated 180° bottom to top. Shaking again left super heavy coating on new bottom heavy coating on top remained.

TABLE A-1 (concluded)

Test number, date and tester	Tube I.D. type/length material	LYCO mixing method	Grounding method	Antistat	Testing for ?	Tube appearance and particle count (Number 16 mm^2)	Remarks, results
32 2/6/86 HR, RK	A trans. temp. 6" straight	(Sec pix) 2 to 5 second shakes	Copper tape 1 ground	Yes	Effect of bottom versus top orientation		The longer time we shake, the more LYCO sticks. Heavy patches of LYCO, grow and grow in formerly clean areas.
33 2/6/86 HR, RK	A trans. temp. 6" straight	10 second shake	Copper tape 1 ground	Yes	Effect of shaking time		
38 2/7/86 HR,RK	K Copper tube 1-1/2" diam 6" length	Horizontal	Ground direct to copper	No	Effect of highly conducting surface (unclean)		Heavy patches
39 RK	K Copper tube 1-1/2" diam 6" length	Horizontal	Direct to copper	No	Effect of highly conductive surface (clean)		Heavy patches NOTE: The inside surface was sand-blasted to have a clean copper surface. Still heavy particle adhesion.
40 RK	L Copper tube 2" diameter 3" length	Horizontal and vertical	Direct to copper	No	Same as above		Heavy patches
41 2/19/86 RK,HR	B trans. temp. 6" straight	Shake (5 g) turn shake (5 g)	Copper tape direct to ground	Yes 2/3/86	Effect of mixing time and amount (77 mg)		Count ~100 p/gm^2 before tapping. Some agglomeration, but not severe
47 2/20/86 HR	0 trans. temp. 6" straight	Shake turn shake second	No ground connect	No	152 mg, then add >500 mg		-Very heavy LYCO pathes on much of tube-amount of LYCO has strong effect -Most of Mylar diaphragm clean - heavy patch on a corner end

APPENDIX 2

PRODUCTION OF HELIUM IN A FLAME TUBE

Final activity in each tube, I_o

$$\begin{aligned} &4 \text{ strips} * 30"/\text{strip} * 0.25 \text{ mCi/in.} && = 30 \text{ mCi/tube} \\ +\ &12 \text{ strips/end} * @ \text{ ends} \times 1" * 0.25 \text{ mCi/in.} && = \underline{6} \\ & && \quad 36 \text{ mCi/tube} \end{aligned}$$

138 days earlier, need 72 mCi/tube, i.e. 2x

Time constant of decay:

$$0.5 = \frac{I}{I_o} = e^{-\tau t} \text{ where } t = 138 \text{ days (from literature)}$$

$$\tau = 5.023 \times 10^{-3} \text{ days}^{-1}$$

Activity per tube as FN of time

$$I = I_o e^{-\tau t} = (2)(36 \text{ mCi}) \exp(-5.023 \times 10^{-3} t)$$

$$= 2(36 \text{ mCi}) \left(\frac{3.168 \times 10^{12} \ \alpha/\text{day}}{\text{mCi}} \right) \exp(-5.023 \times 10^{-3} t)$$

Each α disintegration provides 1 He atom; $\therefore$ the rate of generation of the helium is:

$$\dot{N}_{He} = I = (2)\ (35 \text{ mCi}) \left(\frac{3.168 \times 10^{12} \ \alpha/\text{day}}{\text{mCi}} \right) \exp(-5.023 \times 10^{-3} t)$$

$$= 2 * 1.14048 \times 10^{14} \ \frac{\text{He atoms}}{\text{day}} \exp(-5.023 \times 10^{-3} t)$$

Now, $MW_{He} = 4$ g/mol and there are 6.023×10^{23} atoms/mole

$\therefore$ so $\dot{N}_{He}$ in terms of grams

$$\dot{N}_{He} = 2 * \left(1.14048 \times 10^{14} \text{ at/day} \right) \left(\frac{4 \text{ g/mole}}{6.023 \times 10^{23} \text{ atms/mole}} \right) \exp(-5.023 \times 10^{-3} t)$$

or

$$\dot{N}_{He} = (2)(7.5742 \times 10^{-10} \text{ g/day}) \exp(-5.023 \times 10^{-3} t)$$

Assume 138 days in storage

$$N_{He} = \int_0^{138} \dot{N}_{He}\, dt$$

$$= \int_0^{138} 7.5742\times10^{-10} * 2\, \exp(-5.023\times10^{-3}\, t)dt$$

$$= 2 * \frac{7.5742\times10^{-10}}{-5.023\times10^{-3}} \exp(-5.023\times10^{-3}\, t) \Bigg|_0^{138}$$

$$= 2 * \frac{7.5742\times10^{-10}}{-5.023\times10^{-3}} (0.5 - 1)$$

$$N_{He} = 15.08\times10^{-8} \text{ grams He}$$

Therefore there are 15.08×10^{-8} g He in a tube after 138 days. This may be compared with air:

$$1.5 \text{ liter air} * 129 \text{ g/liter} = 1.93 \text{ g air}$$

In terms of partial pressure:

$$P_{He} = \rho RT$$

$$= \frac{(2)7.54\times10^{-8} \text{ g}}{1.5 \text{ liter}} \quad R\ (293\ K)$$

where $R = \frac{Ru}{MW} = \frac{0.082055 \text{ lit-atm/mole/K}}{4 \text{ g/mole}} = 0.0205$

so $P_{He} = 6.04\times10^{-7}$ atm

or $\frac{V_{He}}{V_{TOT}} = 6.04\times10^{-7}$

This amount of additional diluent in the tube is well within the PCCE requirement and is less than the uncertainty in the concentration of diluent used in typical combustion experiments. It therefore is considered negligible in its effect.

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TABLE I. - PARTICLE CLOUD COMBUSTION EXPERIMENT INITIAL TEST PARAMETERS

Tube length: 75±0.2 cm
Tube diameter: 5±0.2 cm

Ignition section diameter and length: 6.0 cm, 12 cm (±0.2 cm)

REACTANTS: Lycopodium-air, in following quantities (note: equivalence ratio = 1 corresponds to 230 mg)

Equivalence ratio: 0.72, 0.78, 0.84, 0.92, 1.0, 1.1, 1.2, 1.3

All values to be within ±5 percent

PRESSURE: 1 atm (±5 percent)

TEMPERATURE 294 ±6 K

G-Level and Recording Frequency: <0.0005 g at 1 Hz

TABLE II. - ADHESION ACCOMPANYING ACOUSTIC MIXING

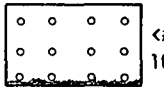
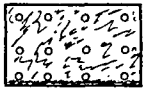
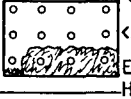
Test number and date and tester	Tube I.D. and description	Amount of LYCO used	Mixing method	Grounding method	Testing for ?	Tube appearance and particle counts (after tapping)	Remarks - results
2 9/3/86	(FF) clear straight	250 mg	Acoustic 60 sec power: 13 W 200 Hz 200 Hz	None	To get a baseline measure of adhesion on clear tube w/o sources	BLACK RED BLUE GREEN  <#> > 100/mm^2	Tapped 3x after test using tongs. The tube was initially cleaned by spraying with ethanol. Relative humidity: 20 percent
3 9/3/86	(FF) clear straight	250 mg	Acoustic 60 sec mix 60 sec tap (together)	None	To check the effect of continuous tapping	<#> > 100/mm^2 	Continuous tapping with impact device during the mix. Impact device settings: 1-7, 1-7. The tapping solenoid was pointed at black dots on the tube, and placed as low as it would go - slightly below center of the tube - this will be the case on all subsequent tests R.H. = 20 percent
4 9/8/88	(BB) gold straight	250 mg	Acoustic 60 sec mix 60 sec tap (together)	None	To check the effect of gold	 <#> ≈ 100/mm^2 HEAVY LAYER >500/mm^2	A large patch formed near the bottom of the tube R.H. = 20 percent
6 9/8/88	Gold	250 mg	Acoustic 60 sec mix 60 sec tap		To check a tube with no sources and no ground	B1 B R G  <#> > 100/mm^2 <#> > 100/mm^2 EXTREMELY HEAVY COVER	Extremely heavy patch covering almost the whole bottom half or the tube. Rest of the tube is moderately covered.
60 4/30/86 RK, HR, NJ	(J) 6"	238 mg	Acoustic 4" woofer mixed for 5 min. orientation	None	To observe adhesion after acoustically mixing for long time (5 min)	Heavy adhesion. The particles seemed to have charged up and the powder stuck to the walls like glazing clumping at end of test	The mixing was stopped after 1 min and adhesion observed little adhesion was notieced and it was very uniform. Then the mixing was resumed for the next 4 min. Initially the mixing was started at low power ≈10 W. Also some hunting for optimum frequency was done The best mixing occurred at 465 and 238 Hz. The power was increased to 55 W.
61 4/30/86 RK, HR	(R) 6"		Acoustic speaker at bottom (238 Hz) same as in No. 60 source(d) at bottom	No ground	More observation of acoustic mix for long time - 3 min	Heavy adhesion around the center part of the tube where the powder was swirling. Only little adhesion at ends 	During the mixing a strange swirling motion was observ. the powder stuck to places where the swirling was greatest.

TABLE III. - POLONIUM-210 SOURCE STRIP DESCRIPTIONS

Model number	Source strength	Construction
PM 24	40 μCi/15 cm	no epoxy
PM 20	12 μCi/15 cm	1 coat epoxy
7B8L	1.5 μCi/15 cm	2 coat epoxy
204	5 μCi per spot	?

Notes: μCi = milliCuries
Model 204 has a single spot sized ~1 cm diameter.

TABLE IV. - QUALITATIVE EFFECTS OF SOURCES IN TUBES

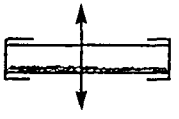
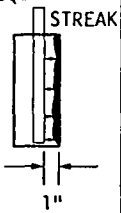
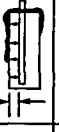
Test number and date and tester	Tube I.D. and description	Mixing method	Grounding method	Testing for ?	Tube appearance and particle counts (after tapping)		Remarks - results	
62 5/22/86 HR, RK	A 6"	Horizontal position - mix up and down 30 sec 	No ground	To observe effect of "nuclear bar" newly arrived α-sources		A. Dropped from 1/2" (instead of tapping) - streak on one side. B. Placed in the source bar (picture) kept it in for 1 min drop 3x- some powder fell off	C Allowed to sit in dry box for 6 min drop 3x-almost nothing fell off D. Placed the source bar in the tube for 6 min-drop 3x-the strip almost fully fell off	Amt. LYCO ≈250 mg Conclusion: the α-source had a definite effect on adhesion
63 5/22/86 HR, RK	I 6" expanded	Horizontal position - mixed up and down (20 sec) turned 180° mixed another 20 sec	No ground	To observe effect of "nuclear bar" newly arrived α-sources	A. Let sit for 5 min - dropped 3x no powder fell off B. Placed in the 6" source for 1 min-drop 3x-much powder fell off- a distinct outline where the source was acting was visible C. Pointed the source to opposite side of the tube for 2 min some powder fell off but nothing drastic		D. Allowed to sit for 11 min in the drybox (no source) dropped 3x - just little fell off E. Pointed the source 6" at the same patch as in step C for 2 min-drop 3x-almost cleaned the wall completely 	Amt. LYCO ≈ 2000 mg (wanted heavy adhesion) very promising results-a clear line of demarcation is visible where the source was pointing
64 5/28/86 RK, HR	B 6"	Horizontal up and down 30 sec	None	Effect of ionizing air with α-source	A. Took the existing tube and dropped it 3x-no powder fell off the tube sat in the dry box all week B. Exposed to source (6") for 1 min dropped it 3x-only little powder fell off the source was 1.5" away from the wall		C. Allowed to sit in dry box for 10 min-drop 3x none fell off D. Exposed to source for 5 min-drop 3x none fell off	HUMIDITY = 15% (DRYBOX)  Since the source only has an active range of 1" it is not going to work at 1.5".
65 5/29/86 RK, HR	B 6"	Horizontal up and down 30 sec	None	Effect of α-source on small amt of LYCO (125 mg)	Initially (after shaking) the tube was lightly and evenly coated everywhere A. Tap (drop) 3x-little fell off B. Placed the source in the tube for 30 sec-drop 3x-almost perfectly clean 			Amt of LYCO = 125 mg This experiment shows that small amounts of lycopodium can be discharded faster and more completely than large amounts

TABLE IV. - (cont.)

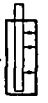
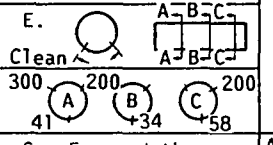
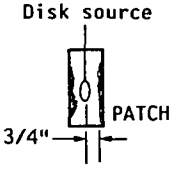
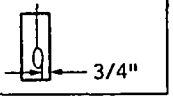
Test number and date and tester	Tube I.D. and description	Mixing method	Grounding method	Testing for ?	Tube appearance and particle counts (after tapping)	Remarks - results	
66 5/29/86 RK, HR	B 6"	Horizontal up and down 30 sec	None	Same as 65 but with more powder (250 mg)	A. Quite a bit fell off during the initial 3 drops B. Allowed to sit 6 min-drop 3x-some came off but not much	C. Exposed to source for 1.5 min-the whole patch fell off 	Amt of LYCO = 250 mg
67 5/29/86 RK, HR	B	Horizontal up and down 30 sec	None	Same as 66 but more vigorous shaking	A. Drop 3x-patch remains B. Exposed to source for 5 min drop 3x-most of the patch fell off, some remains C. Allowed to sit for 1-1/2 min no source drop 3x only little fell off patch remains	D. Exposed to source for 7 min. patch gone after 1 drop sparse uniform layer still remains E. 	Amt of LYCO = 250 mg. The α-source got the particle count down to acceptable range-but it took minutes to do it
68 5/28/86 RK, HR	A 6"	Tube horizontal shaking up and down 30 sec	None	The effect of disk α-source has on adhesion-the disk source is approx. 3x stronger, per area, than the 6" bar	A. Initial shaking produced long and heavy patch. Drop 3x some fell off patch remains B. Exposed to disk source for 5 min drop 3x patch thinned out but the length stayed about the same	C. Exposed the tube to disk source for 6 min-drop 3x the whole patch fell of	Amt of LYCO = 250 mg 
69 5/8/86 RK, HR	A 6"	Same as above	None	To obtain a baseline for data in test 68 using no sources	A. Drop 3x long patch remained B. Allowed to sit for 5 min drop 3x long patch remained	C. Allowed to sit (in drybox) for another 6 min-drop 3x patch still remained	Amt of LYCO = 250 mg This comparison between test 68 and 69 showed that without the α-source the powder remained on the walls
70 5/29/86 RK, HR	B 6"	Tube horizontal mixing and rolling the tube. Rolled the tube 7"	None	To see the effect the disk source has on heavy adhesion	A. The rolling produced even and very heavy coating of LYCO which didn't come off even with drops B. Exposed to disk source for 5 min drop 3x a patch fell off in a circular shape just where the source was	C. Left sitting in in drybox overnight with 6" source in it-drop 3x-the the whole side of LYCO fell off, only where the source was pointing	Amt of LYCO = 1000 mg This test proved without a doubt that the source has a definite effect on dislodging particles from the wall. 

TABLE IV. - (concl.)

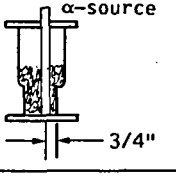
Test number and date and tester	Tube I.D. and description	Mixing method	Grounding method	Testing for ?	Tube appearance and particle counts (after tapping)	Remarks - results	
71 5/30/86 HR, RK	A 6"	Tube held horizontal mixed by shaking up and down	None	To obtain one more comparison betweem the use of a α-source and no source	A. The shaking produced a long streak which persisted even after 3 drops (done on 5/24/86) B. Left overnight (no source) drop 3x-no movement, almost all the powder remained	C. Placed 6" source into the tube pointing at the streak for 5 min-drop 3x-all powder (the whole streak) fell off	Amt of LYCO = 250 mg
72 6/3/86 HR, RK	M 6" with flanges	Acoustic 2 min of mixing cloud only half way up the tube (speaker on bottom)	None	The effect of α-sources on adhesion generated by acoustic mixing	A. Heavy adhesion on bottom half tap on top with tongs no effect on adhesion B. Expose to source for 1-1/2 min (unbolt one flange) tap with tongs some powder fell off	C. Expose to source for 4 min tap 3x w/tongs-more powder fell off but much much remains D.	Amt of LYCO = 243 mg 
73 6/3/86 RK, HR	M 6"	Acoustic 1 min speaker on top ~185 Hz	None	Same as above	A. Heavy adhesion on lower half; tapping dislodged nothing B. Exposed to 6" source for 2 min tap 3x cleaned the side where source was facing (close to wall) distinct boundary	C. Exposed to source for 2 min (other side) the source was for from the wall ≈1.5"-tap 3x some fell off but not much	Amt of LYCO = 246 mg During mixing the cloud momentarily filled the whole tube but most mixing in bottom half this test proves that distance of the source to the wall is very important

TABLE V. - EFFECTS ON ADHESION OF SOURCES OF VARYING STRENGTH

Test number and date	Tube I.D. and description	Amount of LYCO used	Mixing method	Testing for?	Tube appearance	Remarks - Results
7 RS 9/16/86 at 3M	(BB) gold	250 mg	Acoustic 60 sec mix 60 sec tap	Effect of 4 PM24 sources. Tap at 1.7 Hz 1.7 width	Blue Black Red Green GRD GRD <#> = 7.75/mm^2 <#>/11 photo = 3.8/mm^2	Grounding was used - with copper tape. Both ends of the tube were grounded to the building ground. Fabricated in plastic bag filled with N_2 to try to control humidity. 3M personel mounted sources w/350 1 G/A epoxy and heat treat. Test for emission afterwards: Powder hot!... Some microspheres escaped top diaphragm's little hot! Further tests with PM24 were terminated.
8 RS 9/16/86 at 3M	(EE) gold	250 mg	Same as above	Effect of 4 7B8L sources in a gold tube	<#> = 8.5/mm^2 <#>/10 photo = 4.4/mm^2	Fabricated in room air since the plastic bag was too restrictive. All subsequent tests will be performed in room air. No emission from test: powder clean! the 7B8L sources are better able to withstand the mixing process. No agglomeration.
9 RS 9/17/86 at 3M	(FF) clear	250 mg	Same as above	Effect of 4 7B8L sources in a clear tube.	<#> = 4/mm^2 Some single layer adhesion	Some scratches on tube Safety check: CLEAN!! No loose microspheres detected Compared to test 8 RS, ahdesion on clear tube only slightly worse than on gold tube.
10 RS 9/17/86 at 3M	(DD) gold with clear island where gold was wiped off prior to test. (Reason unknown)	250 mg	Same as above	Effect of 2 PM20 sources with less than 100 percent coverage	<#> = 4.5/mm^2 <#>/10 photo = 1/mm^2 No gold patch	Safety check: top gasket hot! 5000 counts/min. Leakage of powder behind source strips, due to curvature of the tube. No adhesion visible even on the clear strip. Further tests with PM20 sources were terminated for safety reasons. All further tests will be with 7B8L sources.
11 RS 9/17/86 at 3M	(EE) gold	250 mg	Acoustic 60 sec mix 60 sec tap	Tube upside down to be compared with test 8 RS. This is to see if the tube orientation has an effect	Blue Green Black Red Top Bot <#> = 2.15/mm^2 <#>/11 photo = 0.5/mm^2 Heavy layer	Safety: CLEAN! Heavy layer near the bottom diaphragm (top in test 8 RS). This indicates that there is nothing peculiar with the tube surface, but that adhesion is heavier near the bottom diaphragm where the mixing is most vigorous. The results, in general, were excellent (except for the patch). worst count <> = 20/mm^2, next worst <> = 1.5/mm^2
12 RS 9/17/86 at 3M	(FF) gold	250 mg	Same as above	Tube upside down to be compared with test 9 RS	Top Bot	Safety: CLEAN! no radiation at all. Same results as in test 11 RS. About the same as test 18 RS.
13 RS 9/17/86 at 3M	(GG) clear expanded	250 mg	Same as above	Effect of tube w/o alpha strips and w/o tapping intended as a baseline measure	Heavy layer everywhere <#> >30/mm^2	The bottom diaphragm (28) tore. Must use large hole diaphragms on both sides of the tube from now on. (It was very fortunate that the diaphragm tore during a test without any alpha strips-points out extra safety concern)

TABLE V. - (concl.)

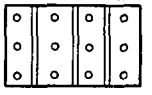
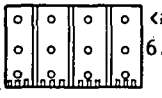
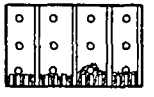
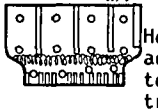
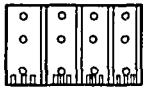
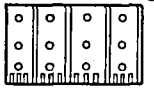
Test number and date	Tube I.D. and description	Amount of LYCO used	Mixing method	Testing for?	Tube appearance	Remarks - Results
14 RS 9/17/86 at 3M	EE gold	250 mg	Acoustic 180 sec mix 180 sec tap	Effect of longer mixing time. The tube is mounted upside down. To be compared to test 11 RS	<#> >1.5/mm^2 	Safety: CLEAN! Some powder leaked outside the tube. Adhesion did not seem any worse than for 60 sec mix. No microspheres escaped after a total of 5 min of mixing with this tube and these same sources.
15 RS 9/18/86 at 3M	FF clear	250 mg	Acoustic 60 sec tap 60 sec mix	Attempt to eliminate adhesion near the bottom of the tube by adding short strips near the bottom	Blue Green Black Red Top  Bot <#> = 6.9/mm^2	12 short strips have been glued on the bottom of the tube in addition to the 4 long (6") strips. The short strips are 0.75 in. long and contain two active areas. The short strips were quite effective in reducing static adhesion near the bottom.
16 RS 9/18/86 at 3M	EE gold	250 mg	Acoustic 300 sec mix 300 sec tap	More severe test Longer mixing time and large amount of lycopodium.	<#> = 18/mm^2  Worst = 43/mm^2	On top of short sources, a small pile of LYCO settled Some agglomeration near the bottom of the tube Small islands of LYCO all throughout the tube including on lowest sources. Safety: Some 7B8L microspheres shook loose! This tube has been used several times before, therefore 5 min < "safe" lifetime <10 min.
17 RS 9/18/86 at 3M	GG clear expanded	250 mg	Acoustic 60 sec mix no tap	To observe adhesion with no tapping	<#> = 59/mm^2  Heavy adhesion to the transition	Source strips have been newly installed; due to tube curvature, sources could not be mounted in the transition section. This may affect strength of alpha coverage. Tapped the tube after 5 min with tongs, much powder fell off. Average <#> after taps was <#> = 10.5/mm^2
18 RS 9/18/86 at 3M	FF clear straight	250 mg	90 sec mix and tap then 60 sec tap only then 60 sec mix and tap then 5 sec tap only	Attempt to simulate near flight configuration	<#> >1.8/mm^2 	Safety: CLEAN! no leaching of microspheres. Extremely low particle counts. Post-mix tapping was very effective.
19 RS 9/19/86 at 3M	FF clear straight	250 mg	90 sec mix and tap, 10 sec tap, 90 sec mix and tap, 3 sec tap	Near flight configuration with 500 mg of LYCO.	<#> = 5.6/mm^2 	Safety: CLEAN! Test was initially aborted after 10 sec because of diaphragm leakage. Diaph 30 was replaced by diaph 24 the test was then resumed. After 8-1/2 min of mixing, no microspheres escaped from the source strip.

TABLE VI. - EFFECTS ON ADHESION OF VARYING HUMIDITY

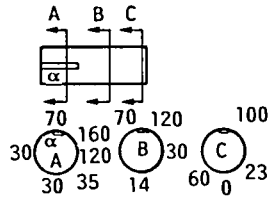
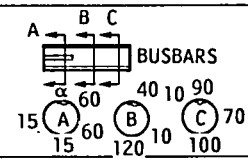
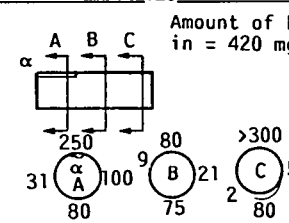
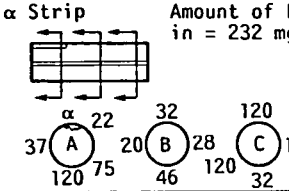
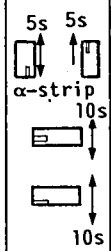
Test number date and tester	Tube I.D. and description	LYCO mixing method	Amount of LYCO used	Mixing method	Grounding method	Testing for ?	Tube appearance and particle counts (after tapping)	Remarks - Results
52 4/16/86 HR, RK	J 6" trans. temp. 2.5" nuclear bar mounted inside the tube	Shake turn shake. Tube held vert-tically shaken 5 sec in each orient-ation			Copper end plugs (both ends grounded)	Effect of decreasing static by ionizing the with alpha particle emitting strip placed inside the tube. NOTE: only small portion of tube covered by source		Very light and uniform coating of particles. No big patches but small clumps (≈20 particle) are common NOTE: The tube was acciden-tially dropped inside the dry box. After the drop a streak of LYCO could be seen. Humidity inside the box = 10 percent
53 4/16/86 HR, RK	G 6" trans. temp. 2.5" nuclear bar	Same as above			Same as above	Same as above using differ-ent tube.		No noticeable patches. Fair before tapping, much better after tapping. Slightly better (cleaner) around the α-source. Tapped the tube again after 30 min-more LYCO fell off.
54 4/16/86 HR, RK	HDR 1/4/86 2.5" nuclear bar	Same as above Tube held hori-zontal shaken for 10 sec			Both ends capped with alum-inum foil ground using alligator clips α-source on bottom	Effect of ionizing the air to decrease static, while generating static by mixing powder in a horizon-tal held tube		Patches near ends of the tube. Tapped tube 3x and LYCO is still sticking. Thin LYCO patch Tapped tube again after 30 min the bot-tom patch fell of (around the α-strip) but top patch remained LYCO patch α-strip Humidity ≈ 0% as will be
55 4/18/86 HR, RK	G 6" trans. temp. 2.5" nuclear bar	Same as test 54			Same as above	Same as test 54 (worst case)		Two long patches along the length of the tube Smaller patch on top Initially before tapping Wide patch on bottom side Tube was turned vertically and trapped 3x most of the par-ticles fell off after tapping
58 4/23/86 RK, HR	J				No ground	Mixed in all directions to get extremely heavy coat of LYCO. Lots of static charging	Too many particles for counting. Before tapping extremely heavy coating everywhere in large blothes After tapping some LYCO fell off but lot of it is still sticking-large blotches very heavey near the α-strip. Tapped again after 20 min (in the drybox) some more fell off mostly near the α-source	Amount of LYCO in = 859 mg In general the α-strip appeared to relieve sticking but it was very time dependent

TABLE VI. - (concl.)

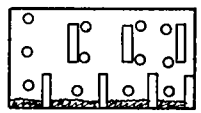
Test number date and tester	Tube I.D. and description	LYCO mixing method	Amount of LYCO used	Mixing method	Grounding method	Testing for ?	Tube appearance and particle counts (after tapping)	Remarks - Results
1 8/31/86	AA with sources		250 mg	Acous- tic 60 sec mix		To check the effect of α-source	 $\langle\#\rangle = 10/mm^2$	Tapped 3x after the test
5 RS 9/8/86	Gold		250 mg	Acous- tic 60 sec mix 60 sec tap (to- gether)		To check the effect of α-sources and tapping	$\langle\#\rangle = 5.8/mm^2$  $\langle\#\rangle$/11 photo $= 2.3/mm^2$	Some agglomeration observed on the bottom 1/8 of the tube. However no other heavy patches were observed R.H. = 20 percent Sources placed in the tube.

TABLE VII. - WIPE TEST NASA TUBES

Tube		Source	Loc number	Wall test	Source test, cm	Results
BB	AA	PM-24	2	0 c/m		
	AB				3 238	
	AC			0 c/m		
	AD				602	
						Wall test-4 smears taken in a spiral the length of the tube between sources
	BA			0		
	BB				18 650	
	BC			6		
DD	AA	PM-20	2	0		
	AB				64	
	AC			0		
	AC				18	
	BA			60		
	BB				0	
	BC			4		
EE	AA	7B8L	4	2		
	AB				2	
	AC			0		Source test-3 smears taken in a spiral on the sources.
	AD				4	
	BA			8		
	BB				10	
	BC			26		
FF	AA	7B8L	4	0		
	AB				0	
	AC			2		
	AD				12	
	BA			0		
	BB				26	
	BC			64 [a]93		
GG	AA	7B8L	4	0		
	AB				28	
	AC			0		
	AD				60	
	BA			4		
	BB				0	
	BC			40 [a]162		

[a]On strip itself.

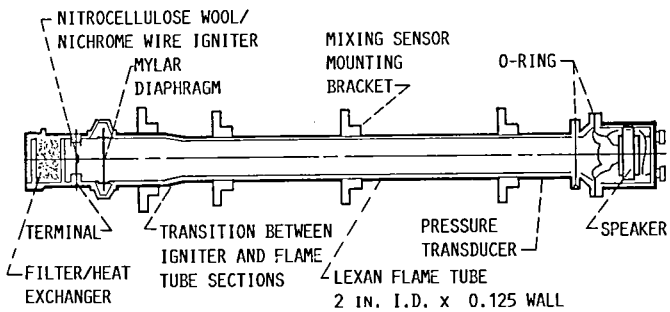
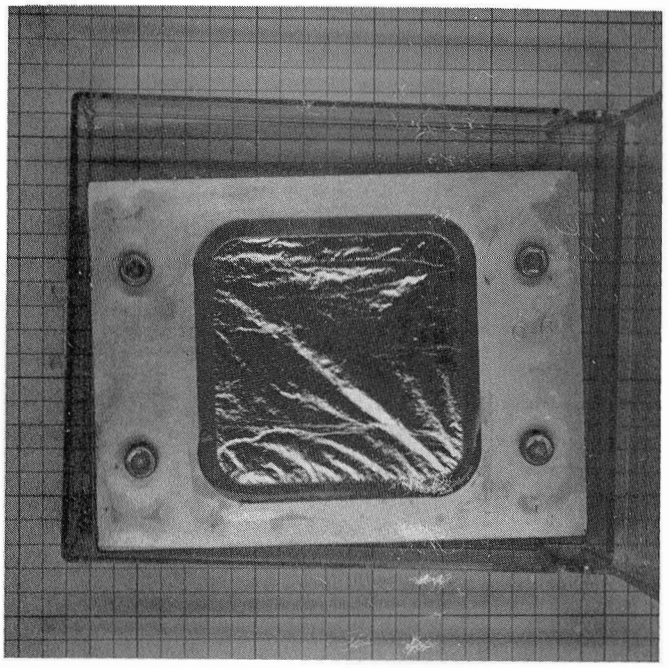
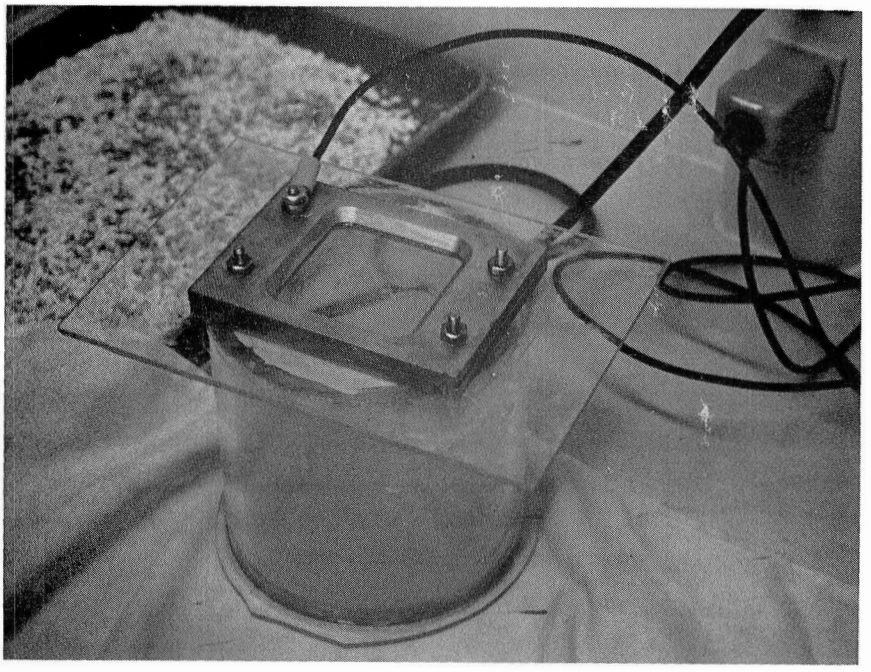


FIGURE 1. - SCHEMATIC OF FLAME TUBE ASSEMBLY.



(a) TOP SURFACE SPECIMEN HOLDER CONTAINING A 1-MICRON GOLD FOIL.



(b) TOP SURFACE MOUNTED ON ACRYLIC TUBE (4 IN. DIAM, 6 IN. HIGH). WIRES ARE TO BUILDING GROUND.

FIGURE 2. - BERLAD AND JOSHI's TEST SETUP.

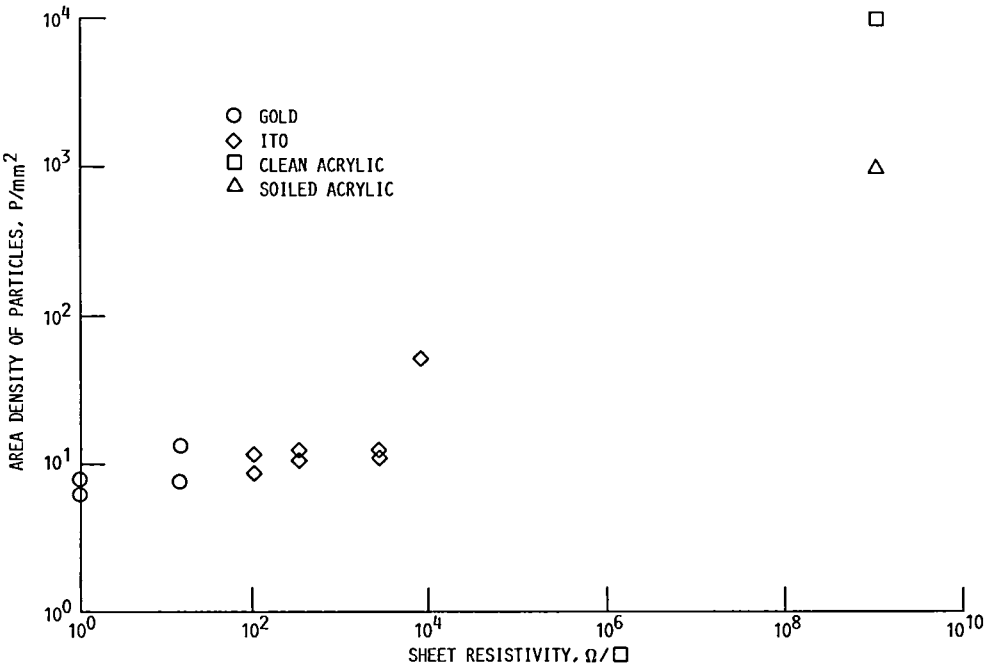
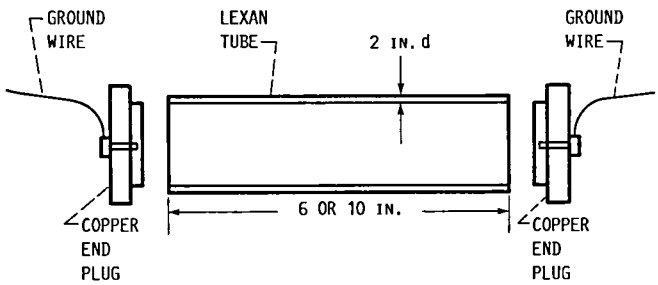
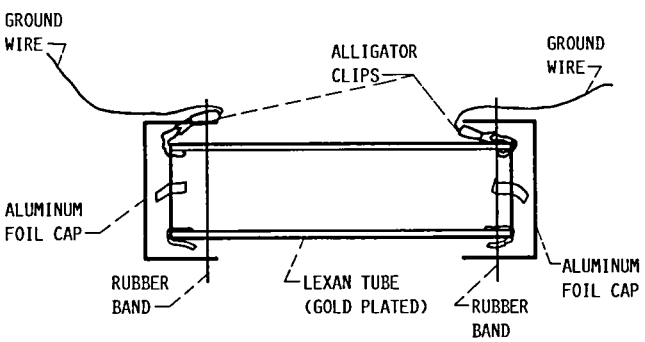


FIGURE 3. - OBSERVED ADP VALUES VERSUS SHEET RESISTIVITY FOR GOLD, ITO, AND ACRYLIC SAMPLES.



(a) COPPER END PLUG SYSTEM, FRICTION FIT.



(b) ALUMINUM FOIL END CAP.

FIGURE 4. - SCHEMATIC OF TEST SECTION. TWO ENDCAP ARRANGEMENTS WERE USED. COPPER TAPE WAS USED TO PROVIDE A DIRECT CONNECTION FROM TUBE WALL TO BUILDING GROUND.

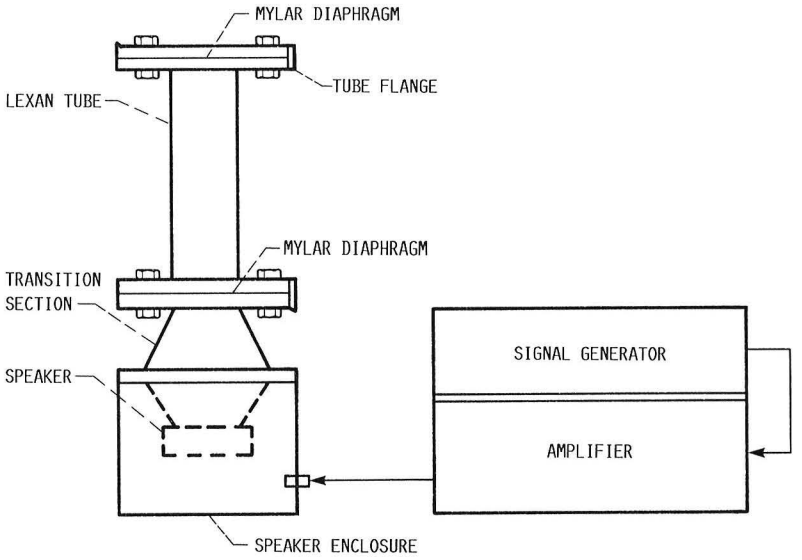


FIGURE 5. - SCHEMATIC OF ACOUSTIC TEST APPARATUS.

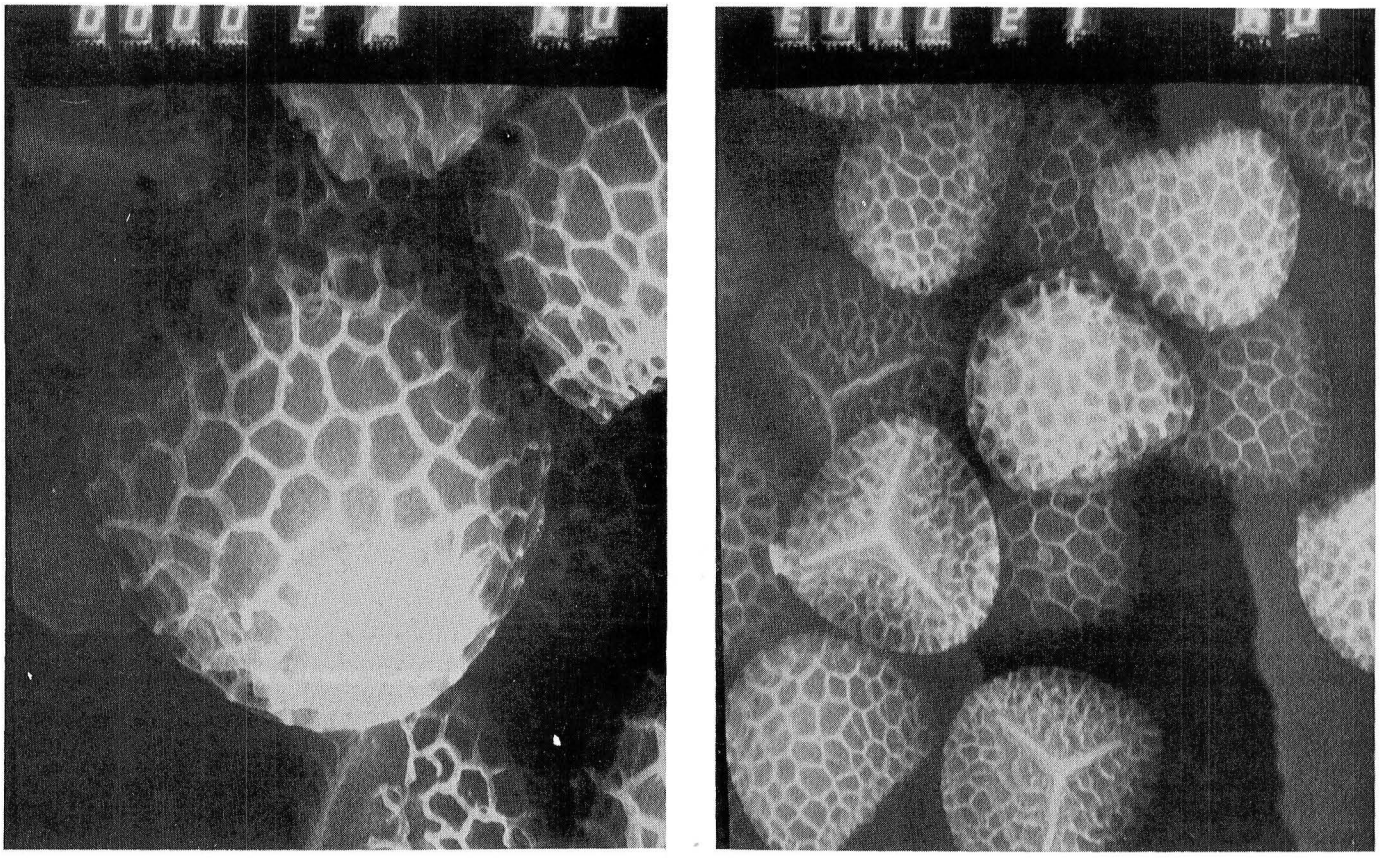
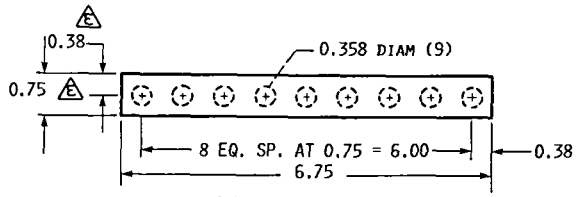
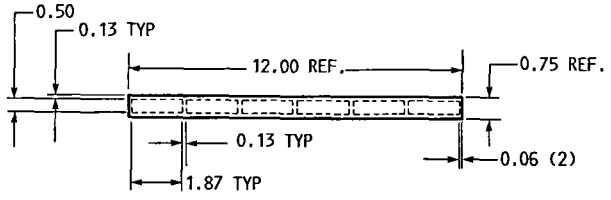


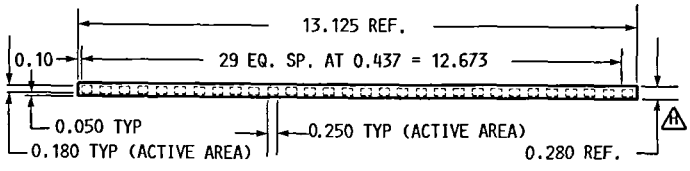
FIGURE 6. - SCANNING ELECTRON MICROSCOPE PICTURES OF ADHERED LYCOPODIUM. MAGNIFICATION: 2000.



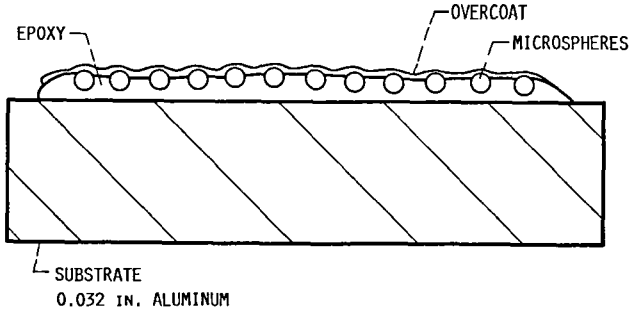
(a) PM 24 STRIP.



(b) PM 20 STRIP.

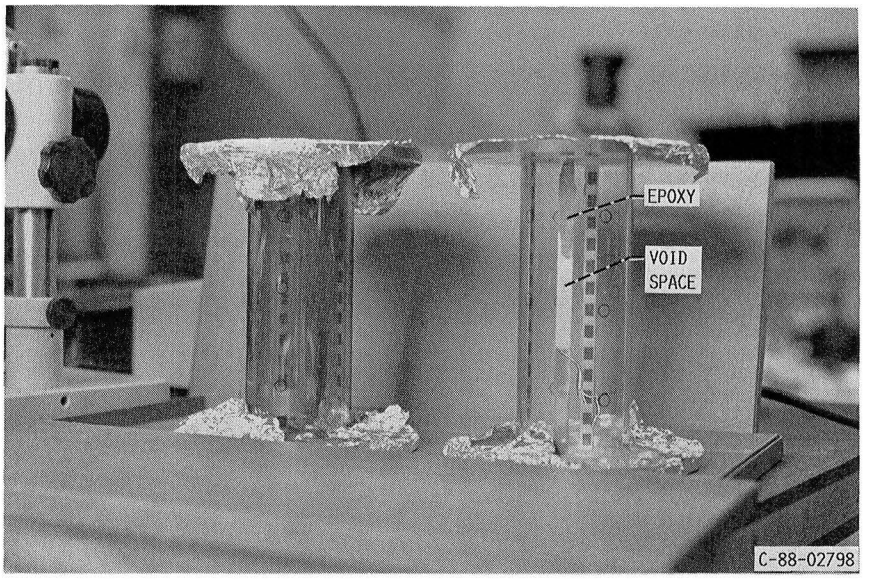


(c) 7B8L STRIP.



(d) TYPICAL CONSTRUCTION TECHNIQUE FOR (b) AND (c).

FIGURE 7. - POLONIUM-210 SOURCE STRIPS.



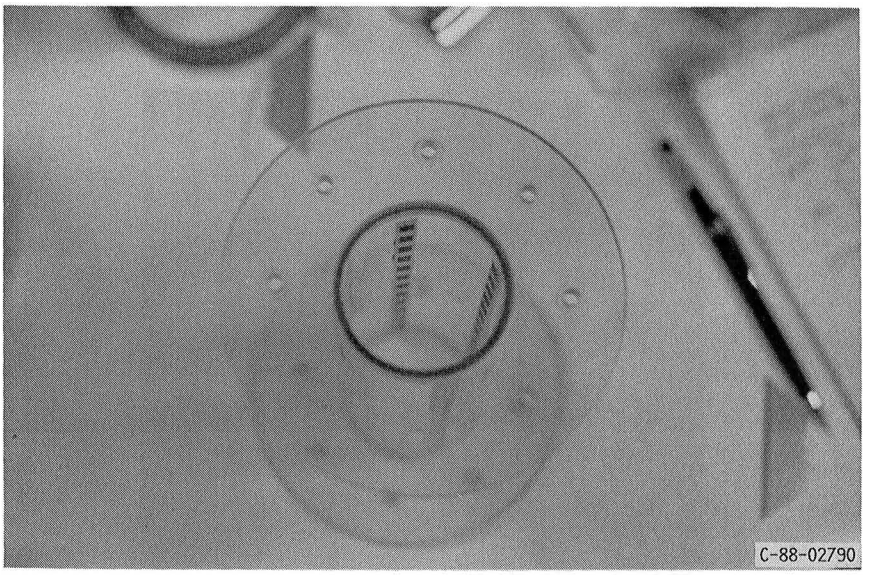


FIGURE 8. - POLONIUM-210 7B8L STRIPS MOUNTED INSIDE TUBE. NOTE VOIDS IN EPOXY HOLDING STRIPS TO TUBE WALL.

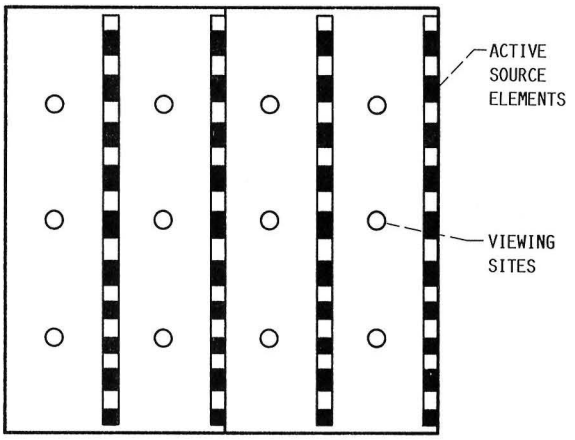
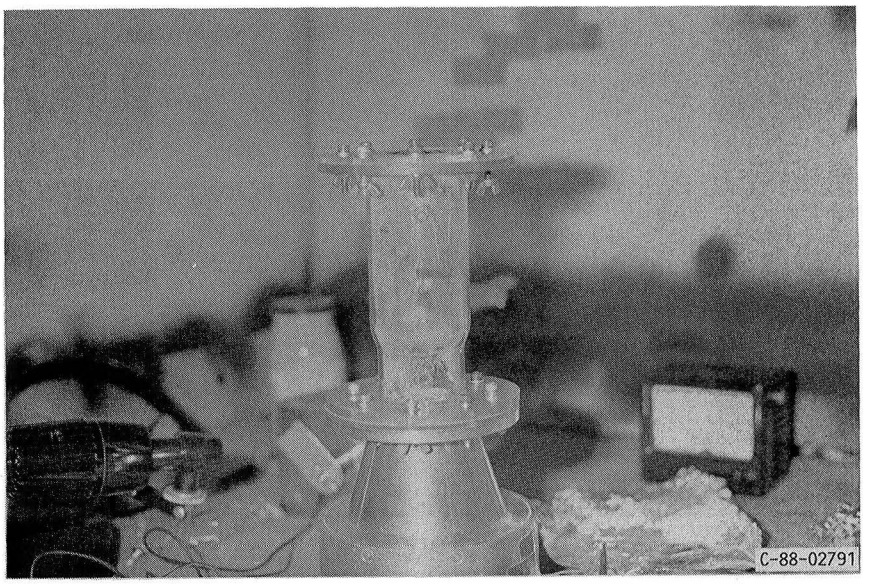
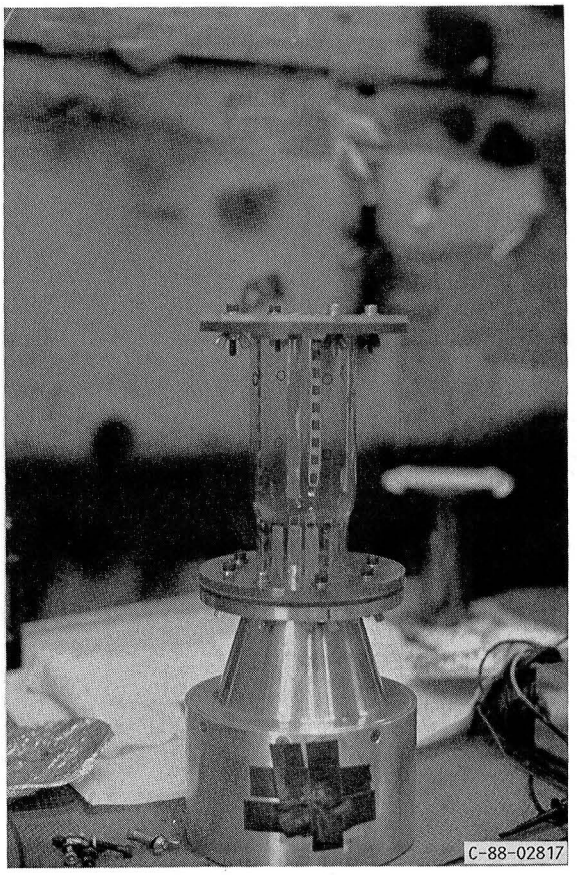


FIGURE 9. - "UNFOLDED" VIEW OF TUBE WITH MEASURING STATIONS CIRCLED. EACH CIRCLE IS ABOUT 12 sq. mm.

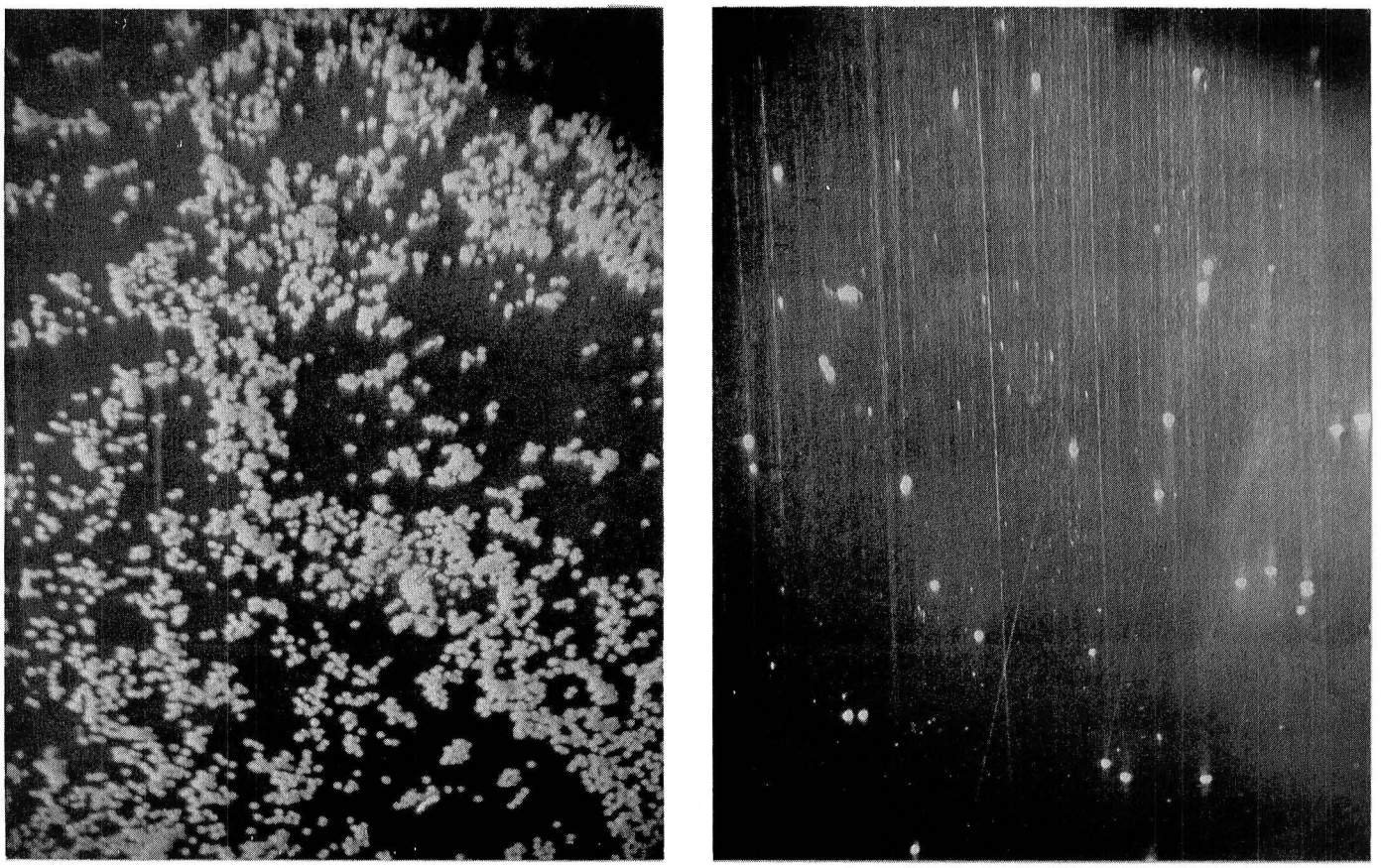


(a) NO SOURCE STRIPS.



(b) SOURCE STRIPS IN ALL BUT TRANSITION SECTION OF TUBE.

FIGURE 10. - EFFECT OF POLONIUM-210 ON ADHESION.



(a) RADIOACTIVE SOURCES NOT IN USE.

(b) POLONIUM-210 SOURCES IN USE.

FIGURE 11. - WALL PARTICLE ADHESION FOR A CLEAR LEXAN (UNGROUNDED) TUBE. MIXING: 250 MG LYCOPODIUM AT 13 W AND 200 Hz FOR 60 SEC.

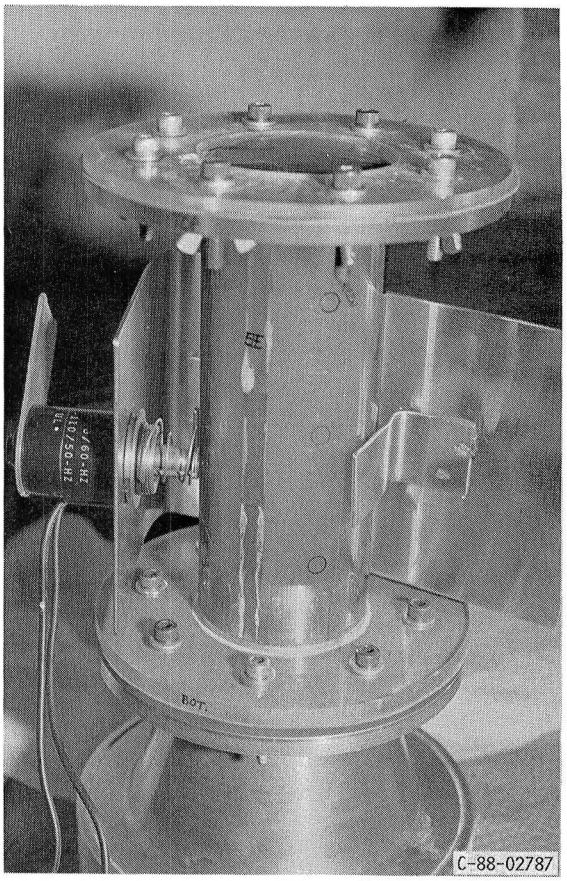


FIGURE 12. - LYCOPODIUM LODGED BEHIND SOURCE STRIP.
SEE ALSO VIEW OF TAPPER.

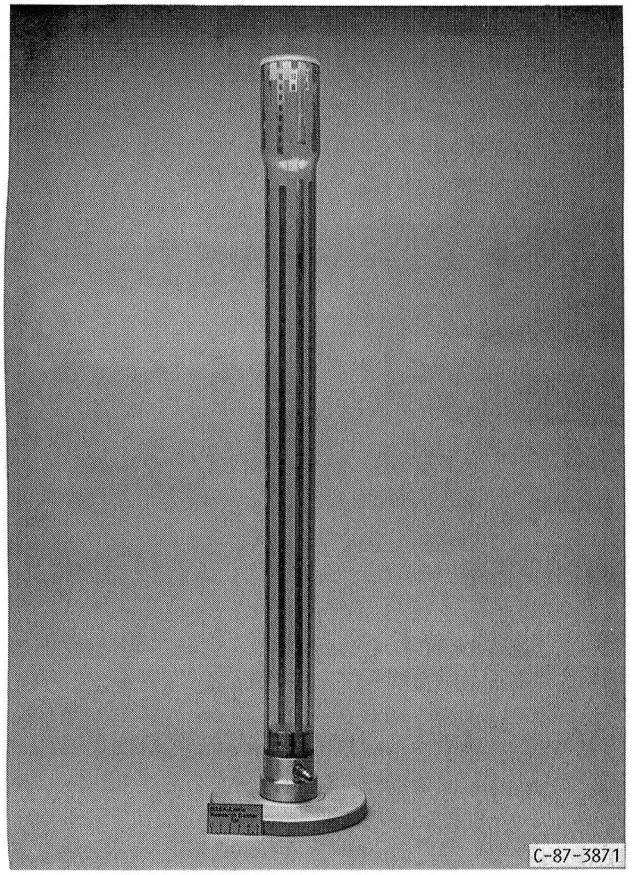


FIGURE 13. - PCCE TUBE WITH MOUNTED SOURCE STRIPS.

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15. Supplementary Notes

16. Abstract

The study of combustible particle clouds inside flame tubes is of fundamental scientific interest as well as a practical concern. Only the suspended concentration is important to the combustion process, so that assurances must be provided that a minimum of particles are adhered to the tube wall. This paper demonstrates experimentally the ability to minimize adhesion and agglomeration of acoustically-mixed lycopodium particles within a 5 cm diameter lexan flame tube. The area density of particles (ADP) adhered to the wall of bare lexan tubes was measured at greater than 100 particles/mm^2. The nature of adhesion was found to be clearly electrostatic, with the ADP level aggravated by increased mixing time, vigor, and the concentration of particles. Increases in the conductivity of the air and the tube wall did not affect ADP levels substantially. However, the observed adhesion was reduced to less than 10 p/mm^2 when the air was ionized by use of an alpha emitter mounted on the inner walls of the flame tube.

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