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Exposure to Amorphous Silica During Rice Farming Operations

CALIFORNIA ENVIRONMENTAL PROTECTION AGENCY

AIR RESOURCES BOARD
Research Division

EXPOSURE TO AMORPHOUS SILICA DURING RICE FARMING OPERATIONS

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DISCLAIMER

The statements and conclusions in this report are those of the contractor and not necessarily those of the California Air Resources Board. The mention of commercial products, their source or their use in connection with material reported herein is not to be construed as either an actual or implied endorsement of such products.

LIST OF INVENTIONS REPORTED AND PUBLISHED

No inventions were produced as a result of the project.

ABSTRACT

A method for collecting and analyzing silica fibers in air has been developed. Environmental release of amorphous silica fibers by rice harvest, rice straw and stubble burning and field preparation after burning has been demonstrated. Fibers were irregularly shaped, often having no parallel sides, and did not appear to be hair-like or needle-like. Aspect ratios seldom exceeded 20:1 and aspect ratios of 5:1 were more common. Airborne levels for employees conducting these operations, which are likely to exceed exposures to the public, has been demonstrated up to 22.9×10^6 fibers per cubic meter of air, using a respirable fraction sampling head (not a PM10 sampling head). Silica fibers were found downwind of field edge and in urban areas in the vicinity of rice straw burning. Results indicate that decreased burning is likely to reduce the levels of respirable amorphous silica fiber emissions from rice farming, but that rice harvest and field preparation are likely to continue to be significant sources of emissions. Further work will be needed to devise sampling and analytical methods which are less sensitive to field conditions and less time consuming and to characterize the potential health effects of respirable amorphous silica fibers and fiber emissions.

SECTION 1.a.I SCOPE AND PURPOSE OF THE PROJECT

Objective #1: Develop laboratory methods for the analysis of silica-containing fibers on air sample filters. Parameters to be determined are the number and concentrations of silica-containing fibers, the fiber size distributions and characterization of the silica as amorphous or crystalline.

Objective #2: Evaluate emissions of amorphous silica fibers from rice farming by performing personal exposure and area air sampling of rice harvest, field burning and field preparation, and by performing ambient air sampling for biogenic silica fibers in municipal areas in the Sacramento Valley on rice straw burn days. Evaluate other airborne hazards associated with rice farming, particularly crystalline silica exposures.

SECTION 1.a.II BACKGROUND ON RICE FARMING IN CALIFORNIA

California is the largest rice producing state in the United States and production is focused in the Sacramento Valley [1]. Seven local counties (Butte, Colusa, Glenn, Sacramento, Sutter, Yolo and Yuba) account for over 90 percent of rice acreage in the state and produced nearly 1.5 million tons of rice in 1988 [2]. There are approximately 1500 commercial rice farmers in California, and the average farm size is approximately 200 acres [1]. Rice production is not as labor intensive as cultivation of row crops; consequently, there are few migrant or seasonal farm workers employed in its production. Farmers frequently perform much of the labor themselves, aided at times by a small number of employees or family members. Activities such as aerial seeding and pesticide application are usually contracted out and usually are not labor intensive. The total amount of time spent on any given activity depends largely on the amount of acreage the farmer has in rice. For example, the smallest farms may complete harvest in one day. The largest farms may harvest continuously for weeks.

The rice growing cycle may begin in the fall or the spring with field preparation. The field is first disced and harrowed to turn the soil, then planed, rolled and fertilized. Employees may spend 10-12 hours engaged in field preparation per day. In the spring, the field is seeded, followed by flooding and irrigation through the late summer. Herbicides, primarily directed against broadleaf grasses, are applied in the late spring and early summer.

In the fall, the rice is harvested and dried for storage. Rice harvest involves the use of a combine harvester, a bank out wagon, which transfers harvested rice from the combine in the fields to trucks waiting at the field edge, and trucks to transport rice to processing or storage. Almost all combine harvesters in use now have an enclosed cab with air conditioning. Enclosed cabs are becoming more common on bank out wagons but still may represent less than half of the equipment now in use. Employees may spend eight to ten hours engaged in harvest per day.

After harvest, rice straw and stubble in the field is burned off. Fields not burned in the fall because of regulatory limitations may be burned in the spring prior to planting. Rice stubble and straw is typically set on fire by

employees on foot carrying torches. From one to three employees will set fire to a field, typically working upwind. The fire may be set by an employee on a tractor that tows a torch. Employees typically spend one to three hours engaged in burning per day.

Burning of rice straw is still popular with farmers for several reasons. Farmers do not want rice straw to remain on the surface of the field after harvest. Firstly, leaving old straw on the surface of the field tends to increase the amount of fungal disease in the next rice crop. Secondly, the straw is tough and is difficult to disc or plow under; it may even damage field preparation equipment. Farmers do not want to collect and remove the straw from the field because of the expense, and because there are no good markets for the straw. Therefore, burning is still the method preferred by farmers to eliminate rice straw after harvest.

SECTION 1.a.III BACKGROUND ON AMORPHOUS SILICA IN PLANTS

Silica (SiO_2, from Latin *silex*, flint) is taken up from the soil and incorporated in the plant's vascular skeleton and tissues [3]. Silica in the plant has a less ordered structure, termed amorphous or biogenic silica [3]. Rice straw contains approximately 12 percent silica by weight [4], characterized as amorphous or biogenic silica. Silica commonly occurs in many grasses and woody plants. [5,6].

Airborne emissions of biogenic silica from plant have been demonstrated when silica containing plant material is burned, and some of the biogenic silica particles liberated as an aerosol during burning have a fibrous form and are in the respirable size range [1,7,8]. Rice and sugar cane are examples of crops that are burned at some stage in the production cycle and yield aerosols containing fibrous biogenic silica. Wind tunnel studies conducted at the University of California, Davis, have demonstrated the release of biogenic silica fibers during the burning of agricultural wastes [9,10,11]. The silica fiber amazons from rice harvesting detected by this study are the first seen from an operation other than burning. However, given the prevalence of biogenic silica in plants, it appears likely that respirable plant aerosols of many types will contain amorphous silica fibers.

SECTION 1.a.IV BACKGROUND ON HEALTH ISSUES AND EXPOSURE STUDIES

Farming is an important industrial sector in the United States. It is especially significant in California, the nation's leading agricultural state [12]. Farming requires a wide range of skills and abilities for specialized tasks such as planting, harvesting, pesticide application and operating machinery. These farming activities entail exposures to a variety of agents, including toxic gases, inorganic and organic dusts and synthetic chemicals that may have adverse consequences for respiratory health [13]. Occupational respiratory disorders that have been described in farmers include mucous membrane irritation [14], asthma [15], hypersensitivity pneumonitis [16], toxic lung injuries such as silo filler's disease [17], organic dust toxicity syndrome [18], allergic rhinitis and bronchitis [19]. Farming is also thought to be a major contributor to environmental dust levels.

Although other silicon based compounds such as quartz and asbestos are known to cause lung disease [20], little is known about the health effects of biogenic silica. Concern over biogenic silica has arisen because it also occurs in fibrous forms similar to asbestos [7,21].

The pathogenicity of asbestos appears to relate in part to fiber size and shape [20]. Because of the similarity in form to asbestos fibers, it is thought that fibrous biogenic silica may have similar disease causing properties. However, the health effects of amorphous silica fibers are not known at this time.

Respiratory hazards of rice cultivation: Potentially hazardous respiratory exposures to soil dust aerosols or ash from burned rice straw and stubble may occur during the pre-planting preparation of the fields. Similarly, dust exposure from soil and plant materials may occur during harvest. In many cases, personal dust exposures from field preparation and harvest are minimized because farmers now typically work with farm machinery with enclosed cabs, providing significant protection from aerosols during field operations [22]. However, historical exposures occurred before these engineering improvements were widely instituted over the past decade and may be associated with chronic respiratory outcomes. Many minor tasks associated with field preparation and harvest, such as removal of dikes and operation of bank out wagons involves use of open cabs. Most burning is done by unprotected employees on foot.

Currently, the major respiratory hazard for the public is believed to occur after harvest when the remaining rice straw and stubble is burned in the fields. Rice generates approximately three tons of straw per acre. Approximately one million tons of straw are burned off annually in the Sacramento Valley area, discharging 5000 tons of particulates into the air and contributing up to four percent of the area's airborne particle mass component of air pollution [23]. In response to public complaints about the effect of rice straw and stubble burning on air quality, in 1981 California initiated the Variable Acreage Allotment Program, a permit system for agricultural burning. The program attempts to reduce the effect on air quality by limiting burning to times of favorable atmospheric conditions.

Public concern about rice straw burning initially focused on reduced visibility and acute respiratory complaints. In addition to these concerns, exposure to rice straw and stubble smoke may have important chronic effects on respiratory health. Rice straw and stubble smoke contains polycyclic aromatic hydrocarbons (PAHs) [24], which are carcinogenic and may lead to lung cancer. There is concern that inhaled biogenic silica fibers may cause restrictive lung disease, cancer and pulmonary conditions similar to those seen with asbestos [7,21]. Although some morphologic similarity between fibrous biogenic silica and asbestos has been demonstrated [25,7,21,8], the behavior of biogenic silica in biologic assay systems has not been thoroughly evaluated, and there have been no assays of the respiratory toxicity of biogenic silica fibers.

Animal studies of biogenic silica: Animal data suggest that fibrous biogenic silica can act as a promoter of skin cancers in cutaneously exposed mice [26].

The proposed mechanism is that the fibers cause irritation and inflammation with consequent increased cellular turnover and activity, leading to promotion of cancer in cells with a previous initiation exposure. No animal data are available to evaluate the respiratory health effects in a controlled laboratory setting.

Exposure levels: Few data are currently available characterizing the exposure of rice farmers to total or respirable agricultural dust or to biogenic silica from rice straw smoke. Data are available related to burning of sugar cane. Investigators from the National Institute for Occupational Safety and Health (NIOSH) have evaluated personal exposures among sugar cane workers in Florida and Hawaii. In the Florida study [25], total particulate matter concentrations sampled from the subjects' personal breathing zone ranged from 0.1 to 5.2 mg/m^3. Total dust exposure was higher among burners (median 2.1 mg/m^3) than among cane cutters (median 0.8 mg/m^3). Inorganic fiber concentrations ranged up to 300,000 $fibers/m^3$; fiber length ranged from 3.5 to 65 μm (mean 12 μm) with an average diameter of 0.6 μm. In comparison, the NIOSH recommended exposure limit for asbestos is 100,000 fibers greater than 5 μm in length per cubic meter [27]. In the study among sugar cane workers on the Hawaiian island of Maui, investigators found inorganic fiber concentrations in the subjects' personal breathing zones between 1,250 and 56,280 $fibers/m^3$ [8]. The majority of the fibers were between 0.5 and 2 μm in diameter and 10-40 μm in length.

Epidemiologic studies: Epidemiologic studies of farmers have generally shown lower than expected mortality for all causes and for lung cancer [28]. The low lung cancer death rate has been attributed to low smoking prevalence among farmers [28]. California occupational mortality data for 1979-81 shows an increased risk for mortality (Proportionate Mortality Ratio =1.65; $p<0.05$) from non-malignant respiratory disease [29]. This finding is unlikely to be explained by smoking because the figures are adjusted for age, sex, race and smoking prevalence.

In spite of the importance of farming and the recognition of work related respiratory disease, epidemiologic data describing pulmonary function and symptoms in farmers and addressing possible risk factors in the workplace are limited. Dosman and coworkers [30] studied 1824 farmers selected from tax roles in Saskatchewan. Smoking history, prevalence of respiratory symptoms and spirometry in this group was compared to 556 non-farmer town dwellers. With the data adjusted for age and smoking, the farmers had a significantly increased prevalence of respiratory symptoms, including morning phlegm production (14.5 percent vs. 9.2 percent), wheezing (27.4 percent vs. 9.9 percent), shortness of breath (33.3 percent vs. 18.2 percent), and chronic bronchitis (11.1 percent vs. 7.7 percent). Forced vital capacity (FVC) and forced expiratory volume in one second (FEV_1), expressed as percentage of expected value adjusted for height, weight and age, were also studied and farmers had significantly reduced FVC (97.7 percent vs. 106.1 percent) and FEV_1 (96.1 percent vs. 102.8 percent) compared to the non-farmer town dwellers.

Heller and coworkers [31] studied a group of 428 farmers and an equal number of non-farm control workers in England and Wales. No significant differences

were found between the groups in the prevalence of chronic bronchitis symptoms. However, farmworkers demonstrated a decreased FEV_1 compared to the control group of workers. Neither of these studies attempted to quantify exposures to respiratory hazards in the work place on the basis of industrial hygiene measurements, limiting the ability to compare with other exposure settings and to evaluate validity of exposure assignment, causality and dose-response relationships.

In addition to the mortality and respiratory health studies cited above, several investigations have produced results justifying concern about human health effects of respiratory amorphous silica exposure. A case series from India [32] described five cases of mesothelioma in young men (age 27-37) from an agricultural community. There were no known asbestos exposures; all five worked for the sugar industry, and four were involved in farming, milling or burning cane. In addition, a case-control study in Louisiana has implicated sugar cane farming as a risk factor for lung cancer [33]. The association persisted after controlling for smoking history. No data exist currently to evaluate whether smokers are at markedly increased risk for lung cancer from biogenic silica, as has been demonstrated for asbestos [34].

A case-control study was conducted of the risk of lung cancer and mesothelioma from environmental and occupational exposures associated with sugarcane production in Florida. A slight, not statistically significant, excess risk of lung cancer was observed among participants who reported working in the sugarcane industry (odds ratio 1.8, 95% confidence interval 0.5-7.5). No increased risk was observed among the studied population, associated with living near sugarcane growing areas. Little difference was observed between cases and controls in years employed in the industry or jobs performed. Only one mesothelioma case and no controls reported working in the sugarcane industry [35].

Further epidemiologic data are being developed by NIOSH investigators studying sugar cane workers in Hawaii. A preliminary study of current and retired workers was conducted by NIOSH investigators in 1988 [36]. A group of 176 retired and current workers was randomly selected from the union roster; medical records were obtained for 168 (96 percent) of the selected workers. Of these, 98 had chest radiography and their radiographic interpretations were reviewed. Of these 98 workers, 26 (27 percent) were noted to have findings suggestive of parenchymal scarring and/or pleural thickening.

Although the 27 percent prevalence of parenchymal scarring and/or pleural thickening is significantly greater than one would expect based on prevalences in low exposure groups in cohort studies of asbestos workers [37], these results must be interpreted with caution. The report is based on review of written radiography reports; chest films were not examined and scored using the standard ILO system for pneumoconiosis [38]. In addition, the films were ordered for clinical purposes and were not obtained systematically on all subjects. Nevertheless, these findings are suggestive and the results of a focused cross-sectional study by NIOSH to evaluate possible pulmonary fibrosis in sugar cane workers is underway. Although data collection has been completed, analysis is still in progress.

A case control study of mesothelioma in Hawaii sugarcane workers was recently completed by NIOSH. The study did not find clear evidence that exposure to biogenic silica fibers presented a risk of malignant mesothelioma [39].

An epidemiologic study of the health of rice farmers is being conducted by the UC Davis Division of Occupational and Environmental Medicine. Results of the study are expected to be released in the summer of 1993.

SECTION 1.a.V BACKGROUND ON EXISTING SAMPLING AND ANALYTICAL PROBLEMS

The Air and Industrial Hygiene Laboratory (AIHL) has been involved in the analysis of airborne particles and bulk samples of ash from several investigations of the rice burning problem. AIHL has reevaluated the samples Taken during an investigation sponsored by the Sacramento Bee of silica fibers emitted during field burning of rice straw and stubble. AIHL has also analyzed ash from the burning of rice hulls in the Wadham energy plant. Currently, AIHL is analyzing air filter samples from the burning of rice plants in the wind tunnel facility of Prof. Bryan Jenkins, U.C. Davis. This work is supported by the California Air Resources Board. AIHL is also currently involved in advising the Toxics Substances Control Division, California Department of Health Services, on methods for sampling and analysis of rice smoke particles in support of ongoing field investigations.

There are no established methods for the sampling and analysis of rice smoke particles. The work thus far has employed ad hoc methods, drawing on sampling and analytical methods used in the analysis of other types of particles. It is clear, however, that the proper assessment of the possible health hazard associated with the rice smoke particles requires validated methods of analysis.

SECTION 1.b STUDY DESIGN, MATERIALS AND METHODS

I. Design

The number and distribution of samples was primarily based on a desire to provide a useful sample pool for development and testing of the analytical method. A secondary goal was to characterize emissions of silica fibers from rice farming operations by measuring exposures to employees, as well as ambient levels in towns located in the rice farming region. The study was not designed to provide sufficient samples to fully characterize exposures or emissions.

Exposure samples were collected on six days of rice harvest, four days of rice stubble burning, four days of field preparation and four days of town sampling. Five to fifteen samples were collected on each day of monitoring. Sampling locations were not chosen randomly. Locations were selected based on the availability of rice producers who volunteered to cooperate. Exposure sampling was distributed between rice harvest, burning of rice straw and stubble, and field preparation and ambient levels in towns. Samples were either "personal," representing the exposure of an employee, or "area," representing the level in a fixed location. The personal exposure samples taken for this study, and some of the upwind samples, were collected with a respirable dust size selective sampling head, not a PM10 sampling head. The respirable size fraction, as defined by the American Conference of Industrial Hygienists is the rough equivalent of a fraction of all particles with an aerodynamic diameter of 5 μm, and is described by a cumulative lognormal function with a median aerodynamic diameter of 3.5 μm (plus or minus 0.3 μm) and with a geometric standard deviation of 1.5 (plus or minus 0.1).

The field blank samples were compared to all other samples. It was assumed that the number of fibers found in analysis of field samples above that found on blanks represented airborne fibers. Samples taken upwind of field operations and ambient samples from towns were compared to personal exposure samples taken from employees performing rice farming operations and samples taken short distance downwind from those operations. It was assumed that fiber levels found in the occupational and downwind samples above those found in upwind and town samples represented airborne fiber levels attributable to the rice farming operation. Rice farming operation samples were split into major categories of harvest, burn, and field preparation, and subcategories by type of farming operation. Exposures were grouped by subcategories and compared.

Ten percent of collected samples were field blanks. Ten percent of collected samples were side by side duplicates. One set of eight side by side replicate samples was collected for harvest and another set of eight was collected for burning of rice straw and stubble. The laboratory was blinded to the identity of the duplicate and replicate samples. Duplicate samples were taken within four centimeters of each other and were run simultaneously. Replicate samples were all taken within twelve centimeters of each other and were run simultaneously.

II. Equipment and materials

A. Personal exposure air monitoring - total dust, respirable dust, crystalline silica, and amorphous silica fibers

i. personal sampling pumps (MSA-G)
ii. 25 mm diameter, 0.4 μm track etched polycarbonate filters with five μm cellulosic diffuser disc in carbon filled cassette with 50 mm cowl (Poretics)
iii. 25 mm diameter, 0.45 μm mixed cellulose ester filters in carbon filled cassette with extended cowl (Environmental Express)
iv. 37 mm diameter, 5 μm low ash Polyvinyl Chloride (PVC) filters (SKC)in polystyrene cassette with cellulose backup pad
v. 10 mm nylon respirable fraction cyclones(MSA)
vi. precision rotameter(Brooks)

B. Area monitoring: respirable amorphous silica fibers

i. 12 volt battery powered pumps and 110 volt generator powered pump
ii. 25 mm diameter, 0.4 μm polycarbonate filters in carbon filled cassette with extended cowl (Nuclepore or Poretics)
iii. 25 mm diameter, 0.45 μm mixed cellulose ester filters in carbon filled cassette with extended cowl (Nuclepore)
iv. respirable fraction cyclones(Gilian)
v. paired rotameters (Dwyer)

C. Carbon monoxide monitoring

i.carbon monoxide diffusion indicator tubes (MSA)

D. Laboratory analysis: respirable amorphous silica fibers

i. transmission electron microscope with electron diffraction and scanning transmission modes (Hitachi H-600/H-601A).
ii. X-ray Fluorescence Spectrometer and Image Analyzer (Kevex Delta Class)
iii. instrumentation for optical microscopy.

E. Laboratory analysis: Total dust, respirable dust/crystalline silica

i. precision electrobalance (Cahn 25)
ii. x-ray diffractometer (Diano XRD 8000)

III. Methods

A. Sample collection

i. Calibration
Personal sampling pumps were calibrated before and after each use with the precision rotameter. The precision rotameter was calibrated before the start of the project.

Area monitoring pump flow rates were set with rotameters built into the pump carrying case. These rotameters were used with the manufacturer's calibration provided on purchase.

ii. Personal exposure samples: respirable amorphous silica fibers
Personal sampling pump flow rates were 1.7 l/min and a respirable dust cyclone was used. Samples were collected using 25 mm diameter filters with 5 μm pore size cellulosic diffuser membrane behind the filter. Filters were 0.4 μm pore size track etch polycarbonate membranes or 0.45 μm mixed cellulose ester membranes. The filter cassette had a 50 mm extended cowl and were made of electrically conductive carbon filled polypropylene cassette. The cyclone filter holder bracket was modified to accommodate the extended cowl cassette.

Baskets containing multiple samplers were used to collected most of the samples. Only one pump was used when collecting a personal exposure sample from an employee on foot.

Personal sample locations were as follows:
If the person was working on foot, the sampling pump was placed on their belt and the filter was placed in their breathing zone. The filter was connected to the pump using flexible Tygon tubing. A person was not asked to wear more than one pump.
If the person was working on a machine, the sampling pump was placed in a basket, the filter was clipped to the basket, and the basket was attached to the machine. Multiple samplers were used. Samples were collected both inside the cab and outside the cab of closed cab harvesters, or tractors. Samples were collected either from inside the cab or outside the cab of closed cab bank out wagons. In all cases samples were located within two meters from the person's breathing zone and the sample location was noted. Sample times were selected to achieve useable loading for the analytical method.

ii. Upwind and downwind area samples: respirable amorphous silica fibers
A portable weather station was located on site to indicate wind speed, direction, air temperature, relative humidity and solar radiation. The upwind sampler was placed so that at no time during the burn was it exposed to the plume. Two samplers were placed at the downwind edge of the field, and the fourth sampler was placed approximately 1.6 km (one mile) downwind. The filters were placed at a height of approximately 2 m. A fifth sampler was placed at the downwind edge of the field on a mast approximately six meters in the air. Flow rates were generally set at the maximum the pumps could pull, which was between 8 and 10 l/min. All samples were collected on 0.4 μm polycarbonate filters in 25 mm diameter carbon filled polyethylene cassettes. Cassettes were either run open without upstream particle size separation or with Gilian cyclone size selectors. Cassettes were electrically grounded. A respirable dust cyclone was used on some upwind samples.

iii. Ambient air sampling in municipal areas: respirable amorphous silica fibers

The collection methodology was the same as described above for upwind and downwind samples.

iv. Personal exposure samples: crystalline silica, respirable dust and total dust
Sampling locations were as described above for amorphous silica fiber personal exposures. If samples were taken for an individual on foot, only a respirable dust/crystalline silica sample was collected. If the person was on a machine both total dust and respirable dust/crystalline silica samples were collected.

Respirable dust and crystalline silica samples were collected at a flow rate of 1.7 l/m using a respirable dust cyclone. Samples were collected using 37 mm diameter, 5 μm pore size, PVC filters in polystyrene cassettes. Total dust samples were collected at a flow rate of 2.0 l/m, and were collected open face using 37 mm diameter, 5 μm pore size, PVC filters in polystyrene cassettes. Sample times were one to three hours.

v. Carbon monoxide personal exposure monitoring
Diffusion indicator tubes were placed in the breathing zone of personnel performing rice straw and stubble burning. They were left in place for the duration of the operation. One tube was placed with amorphous silica fiber monitoring equipment downwind of a burn.

B. Sample Analysis

i. Respirable amorphous silica fibers
The "direct method", described in detail in Appendix A, was used to analyze for amorphous silica fibers on air sample filters. The basic approach to the analysis was the use of transmission electron microscopy (TEM) to identify and count fibers (aspect ratio ≥3:1). A known area of the sample was scanned and silicon-containing fibers are identified by the characteristic fluorescent X-rays. Electron diffraction was used to determine whether the silica had a crystalline structure.

The identification of amorphous silica fibers was based partly on a comparison of the elemental composition of unknown particles with particles of known rice-ash origins. Examination of such reference samples indicated that rice ash was composed mainly of amorphous silica. Similar structures were found on harvest and burn air samples and were categorized by fiber length. Because samples are carbon coated during the analysis the method cannot distinguish between fibers which are entirely amorphous silica and fibers which may contain carbon as well.

Laboratory results were reported as amorphous silica fibers found less than 5 μm in length or greater than or equal to 5 μm in length, and aluminum silicate fibers less than 5 μm in length or greater than or equal to 5 μm in length. Blanks were analyzed to determine the correction to apply for contamination of the media with amorphous silica fibers. Reported results were not corrected for ambient background levels of amorphous silica fibers.

Each result was reported as a statistical confidence interval statement. The statistical methodology is standard for asbestos fiber analysis [40]. For example, a typical result was reported as a calculated airborne concentration of 220,000 fibers/m^3, with a 95 percent confidence range of 96,000 to 440,000 fibers/m^3. The analysis consisted of microscopic examination of randomly selected small portions of each air sample filter membrane. The amount of the filter actually examined in a laboratory analysis typically ranged from 0.01 percent to 0.03 percent of the total filter surface. The width of the confidence range was narrower for samples for which a high volume of air was collected. The width of the confidence range was also narrower if more of the filter was analyzed.

In this study a maximum of ten fields (0.03% of the filter) were analyzed to limit the amount of time needed to analyze each sample. This resulted in some loss of sensitivity, but was necessary because of the large number of samples and limited time in which to complete the analyses.

Two analysts were used. Ten percent of analyzed samples were subject to recount analysis, being analyzed twice each by two different analysts. Reported results were not corrected for variance between the analysts.

A more complete description of the provisional analytical method is given in Appendix A.

ii. Respirable dust, crystalline silica and total dust

Filters were preweighed on a Cahn 25 electrobalance. After use in the field, filters were postweighed on the same balance. The electrobalance was located in a temperature and humidity controlled room. Filters were allowed to remain in the room for a minimum of twenty-four hours before weighing. Sample tare weights were adjusted based on measured changes in field blank filters.

Sample preparation followed NIOSH method 7500. The sample PVC filters were ashed in a muffle furnace, dispersed ultrasonically and filtered on silver membranes. Standards on silver membranes were prepared from NIOSH reference quartz (Q-1) and contained 20, 40, 100, 200 and 500 micrograms per filter. Silver membranes were mounted on an aluminum sample holder with double-stick tape for x-ray analysis.

Samples, standards and blank silver filters were x-rayed with a Diano XRD 8000 diffractometer, with a Cu tube operated at 50 kV and 15 Ma. The x-ray reference was Permaquartz (novaculite). Standards and samples were step-scanned from 26.30 to 27.10 degrees 2-theta for quartz and 37.70 to 38.60 degrees for silver, at a scan rate of 0.01 degrees per step and counting rate of ten seconds per step. Following background correction on either side of the peaks, the area under the peak was integrated to obtain net counts for quartz or silver. Nets counts were adjusted for absorbance of x-rays by the sample according to the NIOSH method 7500. Concentration of quartz was calculated from the mass of quartz in each sample (from standards calibration curve) and the volume

of sampled air. A few samples were scanned for cristobalite and tridymite. Neither were detected. A few samples were also scanned from two to 40 degrees 2-theta for qualitative analysis. Feldspars and layer silicates were the major mineral constituents other than quartz.

iii. Carbon monoxide diffusion tubes

The carbon monoxide diffusion tubes were direct reading. The length of the color stain on the tube was read and compared to a table of reference values based on length of exposure.

SECTION 1.c RESULTS

I. Number and types of samples collected and analyzed

A total of 262 samples of all types were collected. Because of time and budget constraints only 136 samples were analyzed. The distribution was as follows:

- o Two hundred and thirty samples were collected for airborne amorphous silica fibers, of which 169 were collected on polycarbonate filters for analysis by the direct method, and 61 were collected on mixed cellulose ester filters for analysis by the indirect method. One hundred and four of 169 polycarbonate filters were analyzed; none of the mixed cellulose ester filters were analyzed.

- o Twenty-six samples for total dust, respirable dust and crystalline quartz, were collected on PVC filters and all were analyzed.

- o Six samples for carbon monoxide collected on direct reading diffusion tubes and all were analyzed.

II. Silica fiber analytical results

A. General comments

Fibers which were composed solely of silicon and oxygen were always found to be amorphous and appeared similar to fibers found in reference material prepared from ashed rice straw. Fibers which contained metals, such as aluminum or magnesium, were seen only in field samples, not in the reference material, and were assumed to be soil particles, such as silicates. Therefore, the discussion that follows focuses on results for amorphous silica fibers which did not contain metals.

The analytical method is still considered provisional and not all sources of variability have been determined or quantified. No correction factor was applied for contamination of the sampling media as no fibers were found in ten fields examined on any of the blanks. Results were not corrected for ambient background levels of amorphous silica fibers. Reported results were not corrected for variability between analysts. Samples times were typically in the range of 15 minutes, so individual results may reflect temporal variability in the process. All results are reported as measured airborne concentrations. Occupational exposures have not been converted to eight hour time weighted averages, nor have ambient results been converted to 24-hour averages. The personal exposure samples, and some of the upwind samples, taken for this study were collected with a respirable dust size selective sampling head (not a PM10 sampling head). Other samples were collected as total dust, without a size selective sampling head.

Fibers were irregularly shaped, often having no parallel sides, and did not appear to be hair-like or needle-like. Aspect ratios seldom exceeded 20:1 and aspect ratios in the range of 5:1 were more common. A

discussion of the nature of the particles is included in section 1.d.II of this report.

Many of the personal exposure samples were limited to approximately 30 liters of sample volume to prevent overloading. This gave a limit of detection in the range of 0.1 X 10^6 fibers/m^3. Limits of detection were proportionately lower for samples with higher sample volumes.

B. Summary of results

The distribution and summary of the 104 amorphous silica fiber analysis samples are presented in table 1 and figures 1 and 12. Details of analyses, grouped by sample type, are presented in figures 2-11 for total fibers and figures 13-22 for fibers ≥5 μm in length. Figures 1-22 are located at the end of this section.

C. Analysis of blanks

There were no amorphous silica fibers detected in ten fields on any of the five analyzed blanks.

D. Upwind air samples (ambient background samples)

Results for the eleven upwind samples indicated a very low ambient background concentration of amorphous silica fibers upwind of rice farming operations. There were no fibers detected on three samples. There was one fiber each on eight of the samples. The geometric mean for the eight filters was 1.4 X 10^4 fibers/m^3. Of these eight samples, six were relatively low samples volumes and two had relatively high samples volumes. For each of the two high volume samples the calculated concentration was 4 X 10^3 fibers/m^3. Results of upwind samples are summarized in figures 2 and 13.

Looking only at fibers greater than 5 μm in length, no fibers were detected on ten of eleven upwind samples. One fiber was detected on one of the upwind samples. The calculated concentration for the sample with one fiber detected was 2.4 X 10^4 fibers/m^3.

E. Rice harvest

Amorphous silica fibers were detected on all analyzed rice harvest samples. Samples from the exterior of harvesters were the highest. The geometric mean of airborne levels for harvest operations was 4.16 X 10^6 fibers/m^3 for the harvester exterior, 0.896 X 10^6 fibers/m^3 for harvester interior (inside the enclosed cab), and 0.815 X 10^6 fibers/m^3 for bank out wagon operations. Results for total fibers are summarized in figures 3-5.

Looking only at fibers greater than 5 μm in length, Amorphous silica fibers were detected on all analyzed rice harvest samples except one harvester cab interior sample. Samples from the exterior of harvesters were the highest. The geometric mean of airborne levels for harvest operations was 1.04 X 10^6 fibers/m^3 for the harvester exterior, 0.201 X 10^6 fibers/m^3 for bank out wagon operations and 0.120 X 10^6 fibers/m^3 for harvester interior (inside the enclosed cab). Results are summarized in figures 14-16.

Table 1. Summary of Amorphous Silica Fiber Sampling.

ACTIVITY Location	No. of Samples Analyzed	No. of Samples w/fibers	Total Fibers/m^3 x 10^6		Fibers >5μm/m^3 x 10^6	
			Geo. Mean[3]	Range	Geo. Mean[3]	Range
RICE HARVEST						
Harvester (exterior)[1]	10	10	4.16	0.85-9.10	1.04	0.14-2.70
Harvester (interior)[1]	4	4	0.90	0.19-1.70	0.12	n.d.-0.23
Bank Out Wagon[1]	3	3	0.81	0.27-1.96	0.20	0.09-0.42
BURNING						
By Foot[1]	18	6	0.22	n.d.-0.89	0.21	n.d.-0.53
Tractor[1]	11	11	2.29	0.17-7.40	1.74	n.d.-4.90
Down Wind	6	3	0.01	n.d.-0.02	0.01	n.d.-0.01
Town	14	7	0.01	n.d.-0.03	0.01	n.d.-0.01
FIELD PREPARATION AFTER BURNING						
Tractor/Bulldozer[1]	7	6	8.44	n.d.-23.0	4.06	n.d.-9.90
Tractor (interior)[1]	2	2	2.35	1.15-4.80	0.50	0.13-1.90
ALL ACTIVITIES						
Up Wind[2]	11	8	0.01	n.d.-0.02	0.02	n.d.-0.02

1 Samples collected using respirable (<5 μm aerodynamic diameter) not PM10 particle sizing device.
2 Samples collected using respirable (<5 μm aerodynamic diameter) not PM10 particle sizing device.
3 Calculated from non-zero values only
n.d. None detected

F. Rice burning

Rice straw burning samples fell into four categories: personal samples from employees on foot burning rice straw in the field; personal samples from an employee on a tractor using a tow behind device to light the straw burning rice straw in the field; samples taken downwind from fields being burned; and samples taken within towns in the rice growing region on days when there were field burns.

The geometric mean of airborne levels for samples taken from employees on foot was 0.217×10^6 fibers/m^3, with no fibers found in twelve of eighteen analyses. The geometric mean of airborne levels for samples taken from an employee using a tractor to burn was 2.28×10^6 fibers/m^3. The geometric mean of airborne levels for samples taken downwind of burns was 1.17×10^4 fibers/m^3, with no fibers detected in three of six analyses. The geometric mean of airborne levels for samples taken in towns was 7.71×10^3 fibers/m^3, with no fibers detected in seven of fourteen analyses. Results for total fibers are summarized in figures 6-9.

For fibers greater than 5 μm in length result, the geometric mean of airborne levels for samples taken from employees on foot was 0.206×10^6 fibers/m^3, with no fibers found in fourteen of eighteen analyses. The geometric mean of airborne levels for samples taken from an employee using a tractor to burn was 1.74×10^6 fibers/m^3. The geometric mean of airborne levels for samples taken downwind of burns was 1.40×10^4 fibers/m^3, with no fibers detected in four of six analyses. The geometric mean of airborne levels for samples taken in towns was 6.89×10^3 fibers/m^3, with no fibers detected in ten of fourteen analyses. Results are summarized in figures 17-20.

The high number of samples with zero fibers detected for burning by foot and downwind samples was consistent with visual observations made in the field. Employees performing burning of rice straw on foot take care to stay out of smoke plumes. The downwind sampling was very difficult and was characterized by erratic and weak burns during the burn events sampled for this project.

G. Field preparation (after burning)

Fibers were found on all eight samples taken of field preparation activities after a rice straw and stubble burn. The time between burning and field preparation varied from one hour to two months for the field preparation operations sampled. Airborne levels detected from field preparation activities were the highest of all activities, on average.

The geometric mean of airborne levels for field preparation samples was 8.43×10^6 fibers/m^3. Two samples taken inside closed cab tractors during field preparation were analyzed, with an average result of 2.35×10^6 fibers/m^3. Results for total fibers are summarized in figures 10-11.

For fibers greater than 5 μm in length, the geometric mean of airborne levels for field preparation samples was 4.06 X 10^6 fibers/m^3. Two samples taken inside closed cab tractors during field preparation were analyzed, with an average result of 0.501 X 10^6 fibers/m^3. Results are summarized in figures 21-22.

An anomalous non-asbestos crystalline fiber form containing silicon, oxygen, aluminum and magnesium was observed in some of the field preparation samples. The fibers may have been soil silicates but were interesting as they were not seen in samples from harvest or burning.

H. Repeat analyses of the same sample (fiber recounts)

There were seven samples which were analyzed twice each by both microscopists. For the first microscopist the coefficient of variation for the analyses ranged from 1% to 63% and averaged 25% (16% to 47% and an average of 29% for fiber greater than 5 μm in length). For the second microscopist the coefficient of variation ranged from 0 to 84% and averaged 22% (0 to 94% and an average of 30% for fibers greater than 5 μm in length). A comparison of the results of one microscopist with the second microscopist showed a systematic variation, with one microscopist reading higher than the other. For the seven recount analyses the average counts varied by a factor of 0.90 to 14.86, averaging 3.66 with a standard deviation of 4.97 (0.67 to 2.94, averaging 1.86 with a standard deviation of 0.85 for fibers greater than 5 μm in length). This difference between readers was significant at the 95% confidence level using ANOVA and Wilcoxon Matched Pairs tests. Results indicated a higher level of variability when comparing total counts, indicating that the smallest fibers contributed the most to the variability between the analysts.

I. Field duplicate and replicate samples, airborne amorphous silica fibers

Forty two of the samples collected for airborne amorphous silica fibers were duplicates and replicates. Of these, 20 were analyzed. Six pairs of duplicate analyses had coefficients of variation ranging from 11% to 47.1%, averaging 24% for analysis of total fibers. Results for replicate analyses were reported only for fibers greater than 5 μm in length.

For fibers greater than 5 μm in length there was less variability. Seven pairs of duplicate analyses had coefficients of variation ranging from 4.3% to 32.6%, averaging 18%. Analysis of the three replicates analyzed by the same microscopist showed a coefficient of variation of 26%. The other three replicate samples could not be used for meaningful comparisons. One replicate analysis was lost due to lab error. One analysis was by the second microscopist, and one was started by one microscopist and finished by the second.

These results indicate that the smallest fibers contribute the most to overall variability in the analyses.

III. Total dust, respirable dust/crystalline quartz analysis results

A summary of total dust and respirable dust/crystalline quartz analyses is shown in tables 2 and 3.

Dust exposure levels were highest for field preparation after a burn, followed by harvesting and rice straw and stubble burning. Some exposure samples for field preparation and rice harvest exceeded the Cal/OSHA personal exposure limit for nuisance particulate of 10 mg/m^3. One sample from field preparation exceeded the Cal/OSHA personal exposure limit for respirable dust of 5 mg/m^3. However, these samples were taken on the exterior of equipment and represent worst case exposures. The harvesters and most of the field preparation equipment were closed cab so actual exposures to personnel in the cab could be considerably lower.

The ratio of respirable dust to total dust was an average of roughly 10-15 percent, indicating that most of the total dust exposure was very coarse, with particle size of 10 μm or greater. The exception to this was the single sample set taken downwind from a burn, where the ratio of respirable to total dust was roughly 85 percent. This is consistent with reports that particulate from burns generally has a particle size less than 1 μm [25].

Table 2. Summary of Total Dust Sampling.

ACTIVITY Location	Number of Samples	Total Dust mg/m^3 Geo. Mean	 Range
RICE HARVEST			
Harvester	5	9.80	4.02-22.07
BURNING			
Tractor	1	1.83*	
Down Wind	1	1.39*	
FIELD PREPARATION			
Tractor	3	26.2	11.05-72.1

* only one observation

Table 3. Summary of Respirable Dust and Crystalline Quartz Sampling.

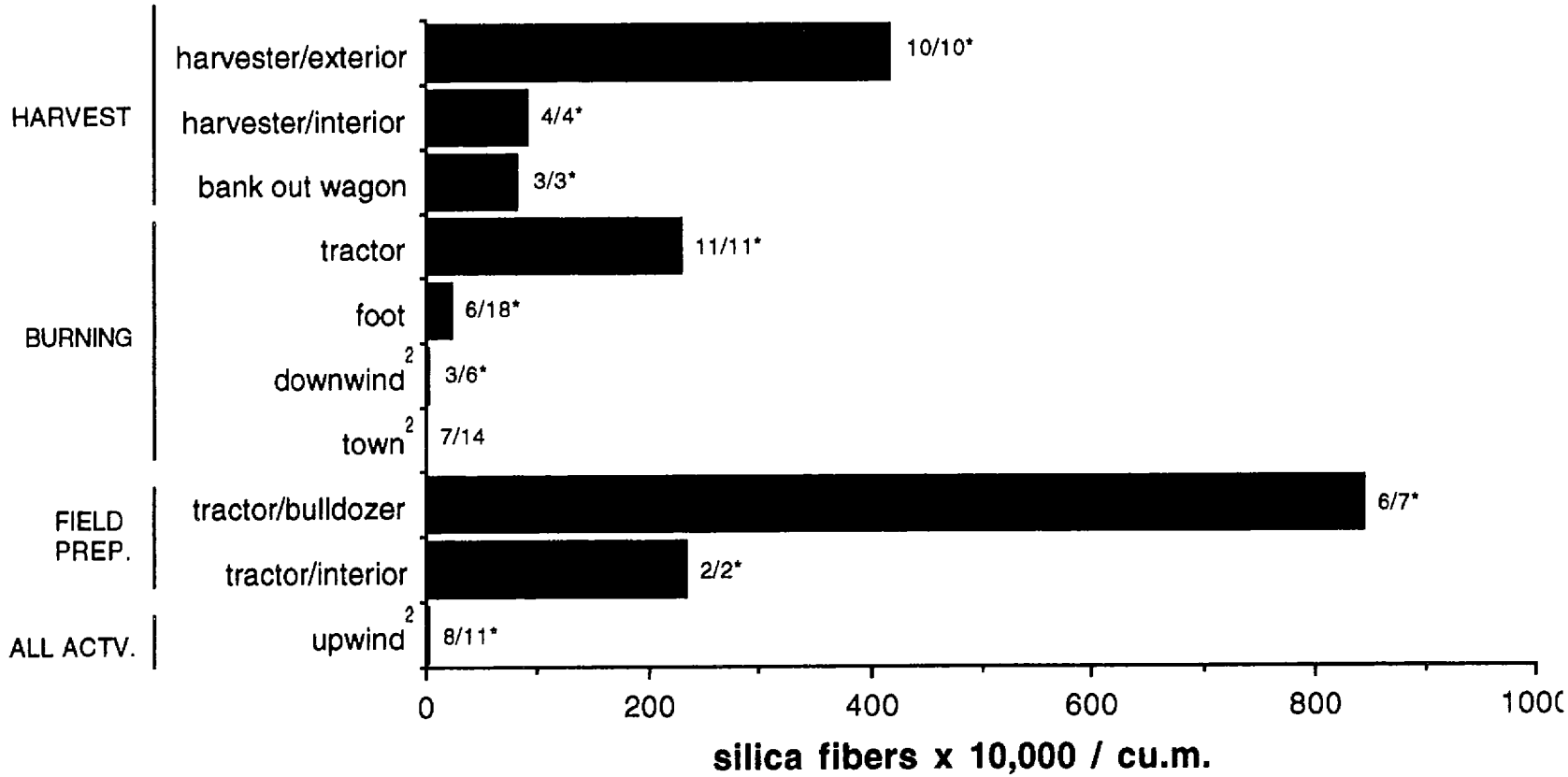
ACTIVITY Location	Number of Samples	Respirable Dust mg/m^3		Respirable Quartz mg/m^3	
		Geo. Mean	Range	Geo. Mean	Range
RICE HARVEST					
Harvester	5	1.04	0.52-2.16	0.03	0.02-0.04
BURNING					
By Foot	2	0.20	0.14-0.31	0.03	0.02-0.03
Tractor	1	0.30*		0.07*	
Down Wind	1	1.19*		0.02*	
FIELD PREPARATION					
Tractor	3	3.17	1.77-5.24	0.08	0.08-0.09

* only one observation

IV. Carbon monoxide results

All carbon monoxide results were below the limit of detection, which ranged from 25 to 50 ppm.

FIGURE 1. GEOMETRIC MEAN[1] OF TOTAL AMORPHOUS SILICA FIBER CONCENTRATION BY RICE GROWING ACTIVITY



1 Geometric mean of non-zero results only
2 Downwind, town, and upwind are the only area samples, all others are personal samples
* Samples with fibers detected / number of samples analyzed

FIGURE 2. TOTAL AMORPHOUS SILICA FIBER CONCENTRATION AND 95% CONFIDENCE RANGE UPWIND OF RICE BURNING

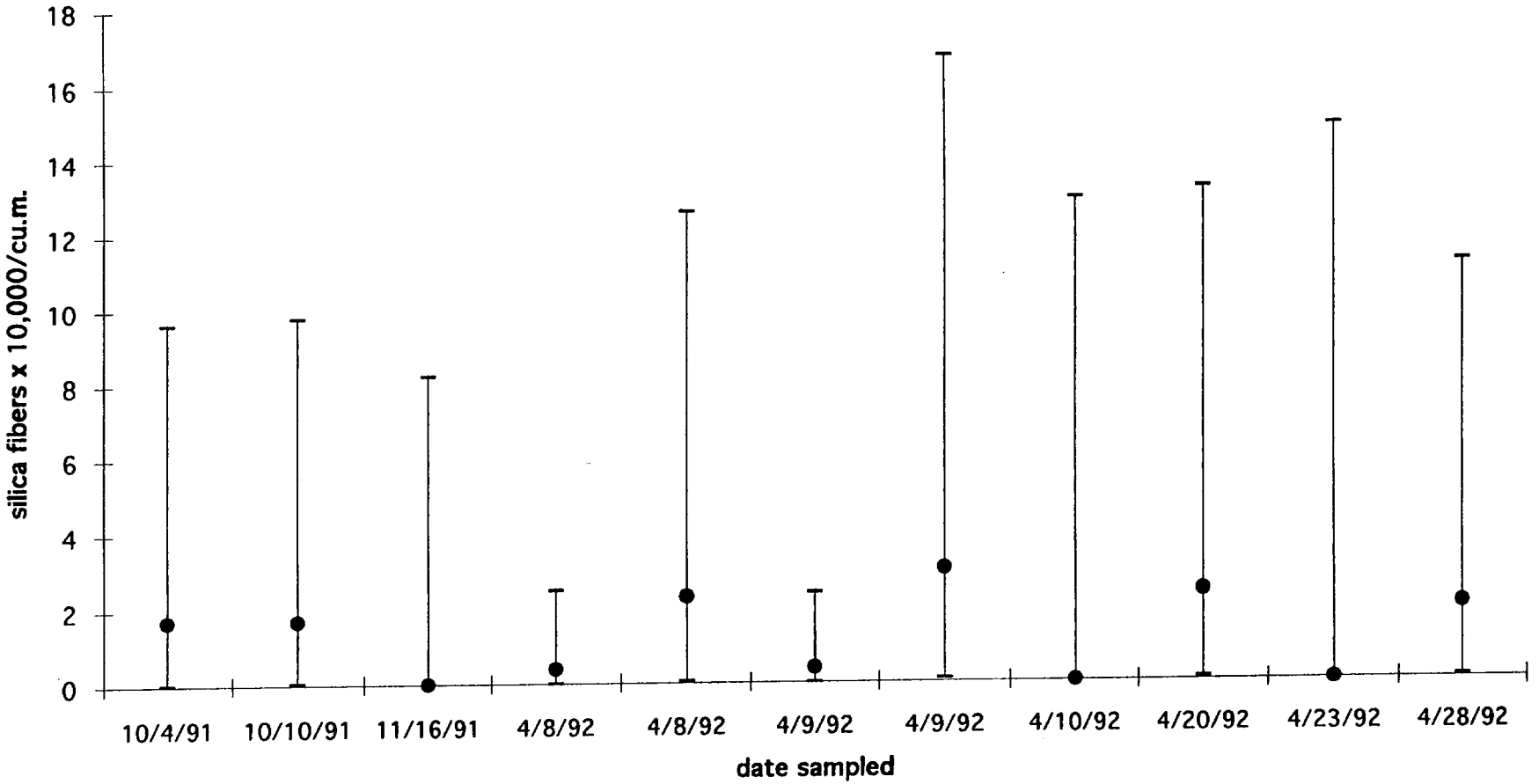


FIGURE 3. TOTAL AMORPHOUS SILICA FIBER CONCENTRATION AND 95% CONFIDENCE RANGE FOR RICE HARVESTER

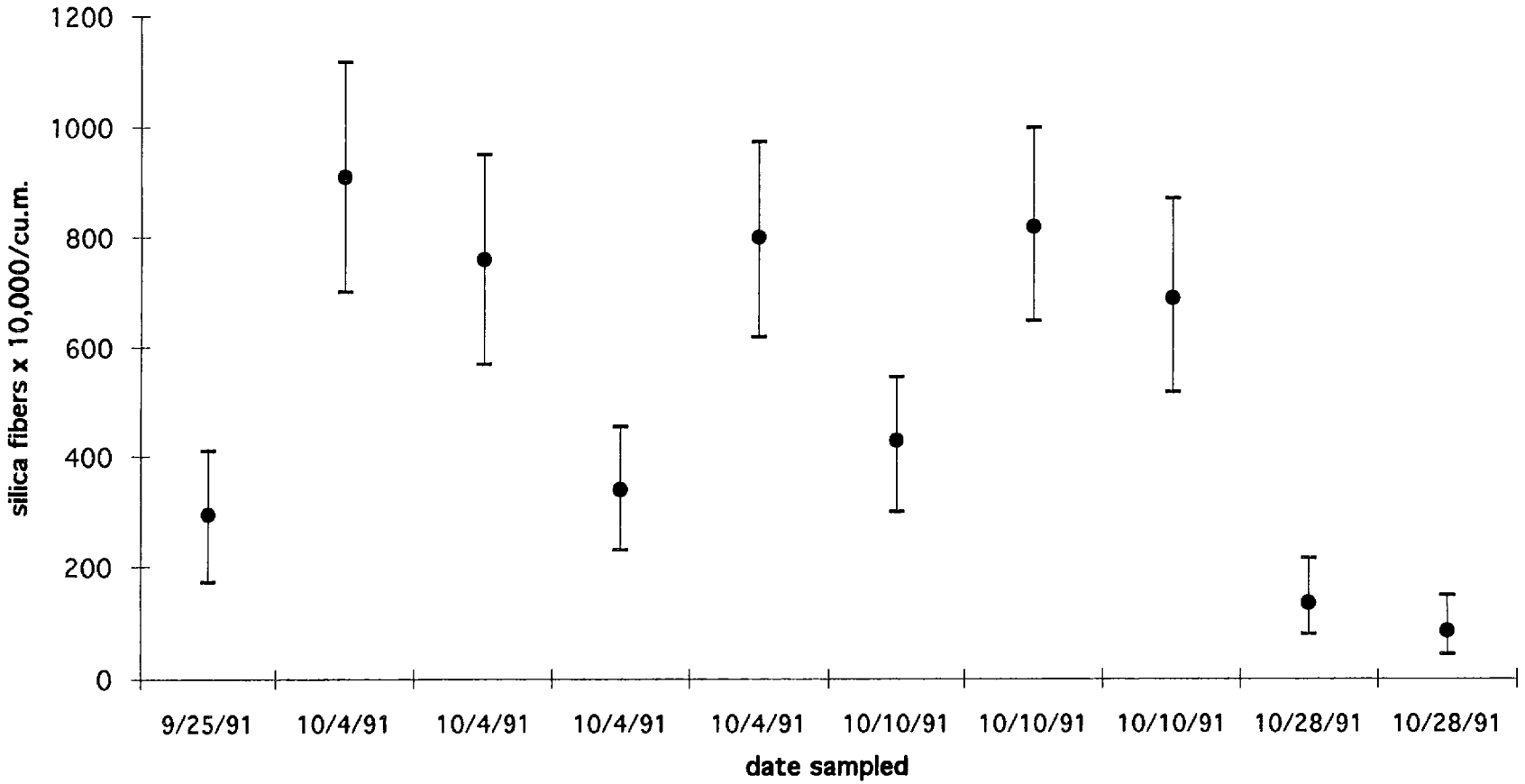


FIGURE 4. TOTAL AMORPHOUS SILICA FIBER CONCENTRATION AND 95% CONFIDENCE RANGE FOR RICE HARVESTER INTERIOR

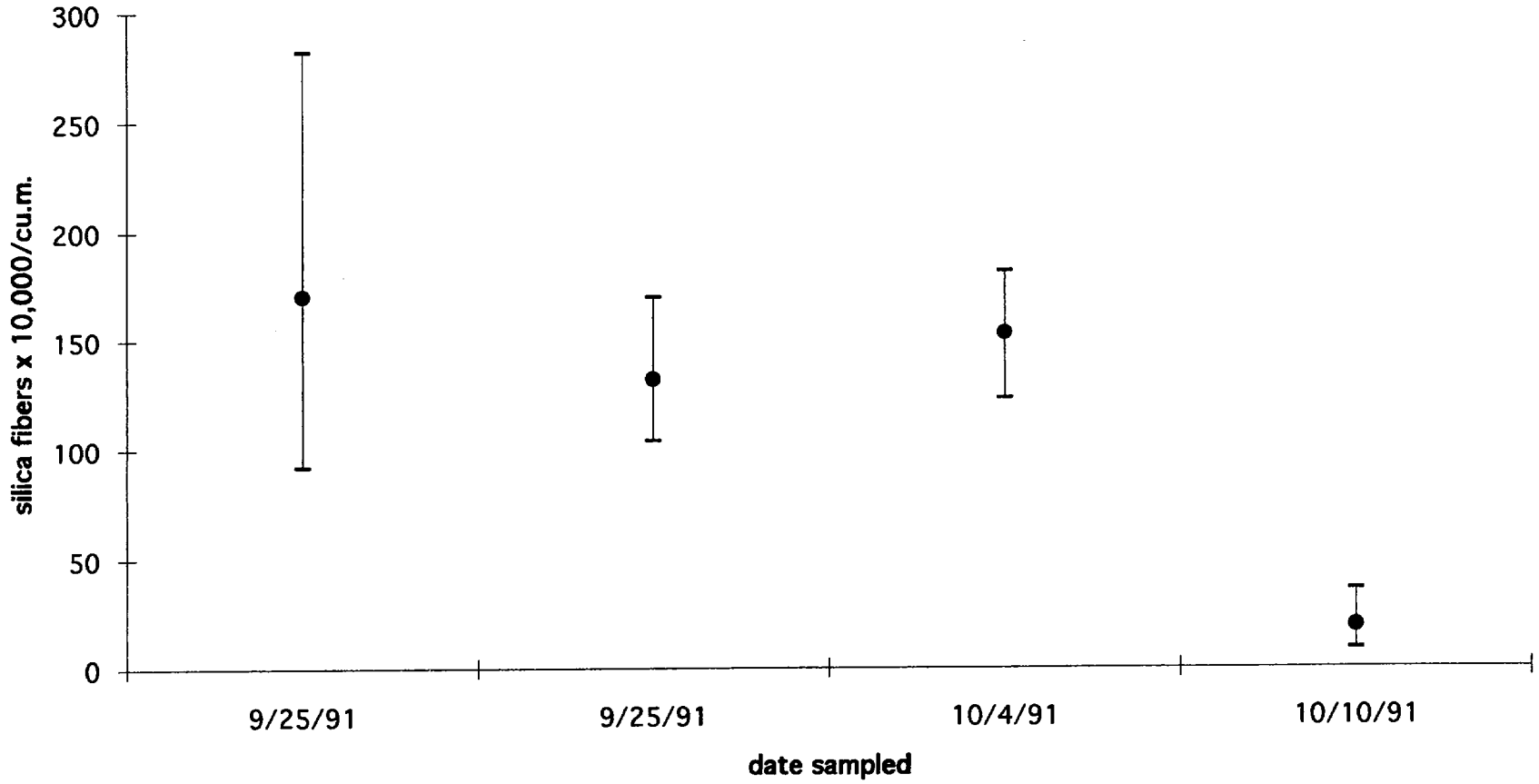


FIGURE 5. TOTAL AMORPHOUS SILICA FIBER CONCENTRATION AND 95% CONFIDENCE RANGE FOR BANK OUT WAGON DURING RICE HARVESTING

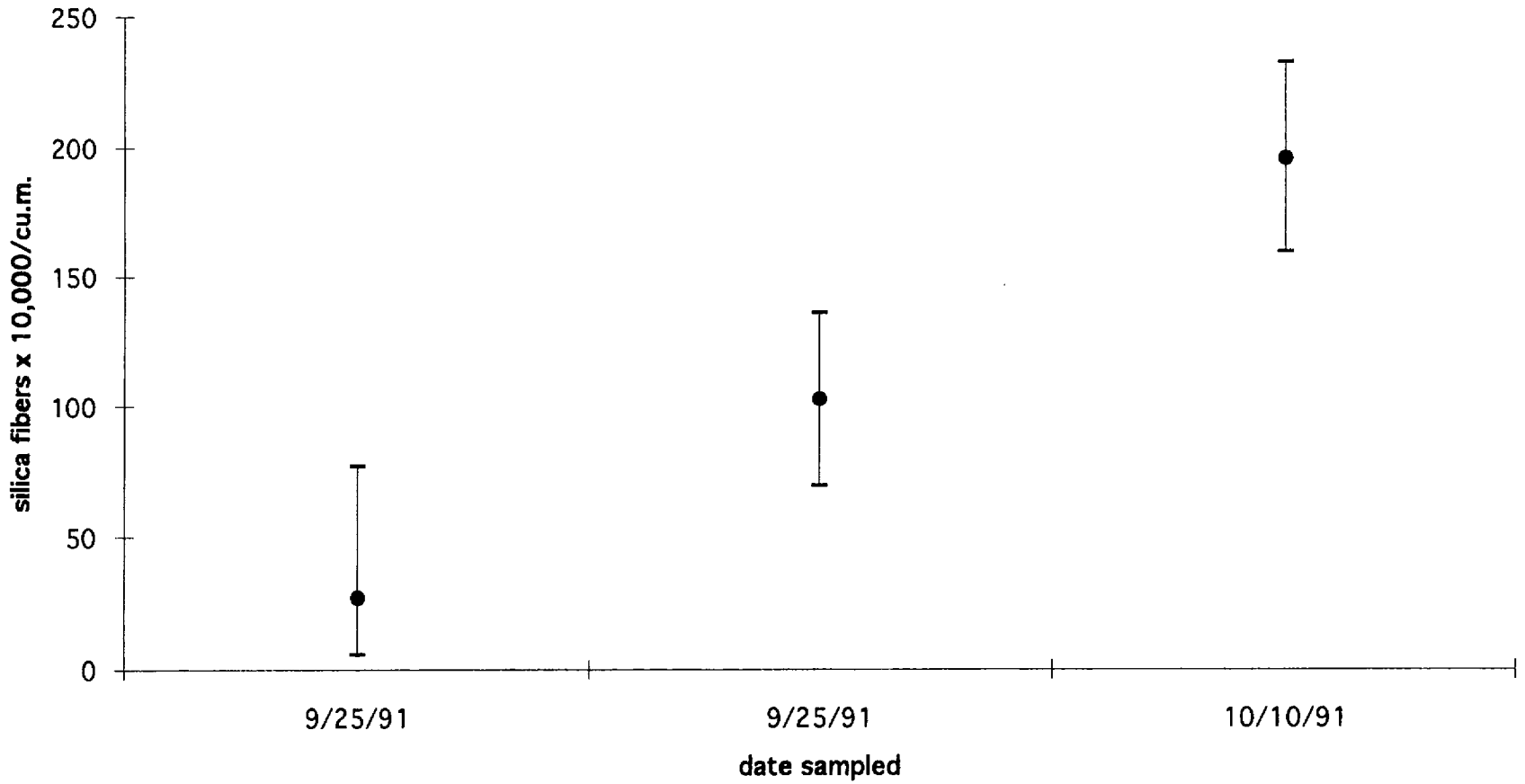


FIGURE 6. TOTAL AMORPHOUS SILICA FIBER CONCENTRATION AND 95% CONFIDENCE RANGE FOR RICE STUBBLE BURNING BY FOOT

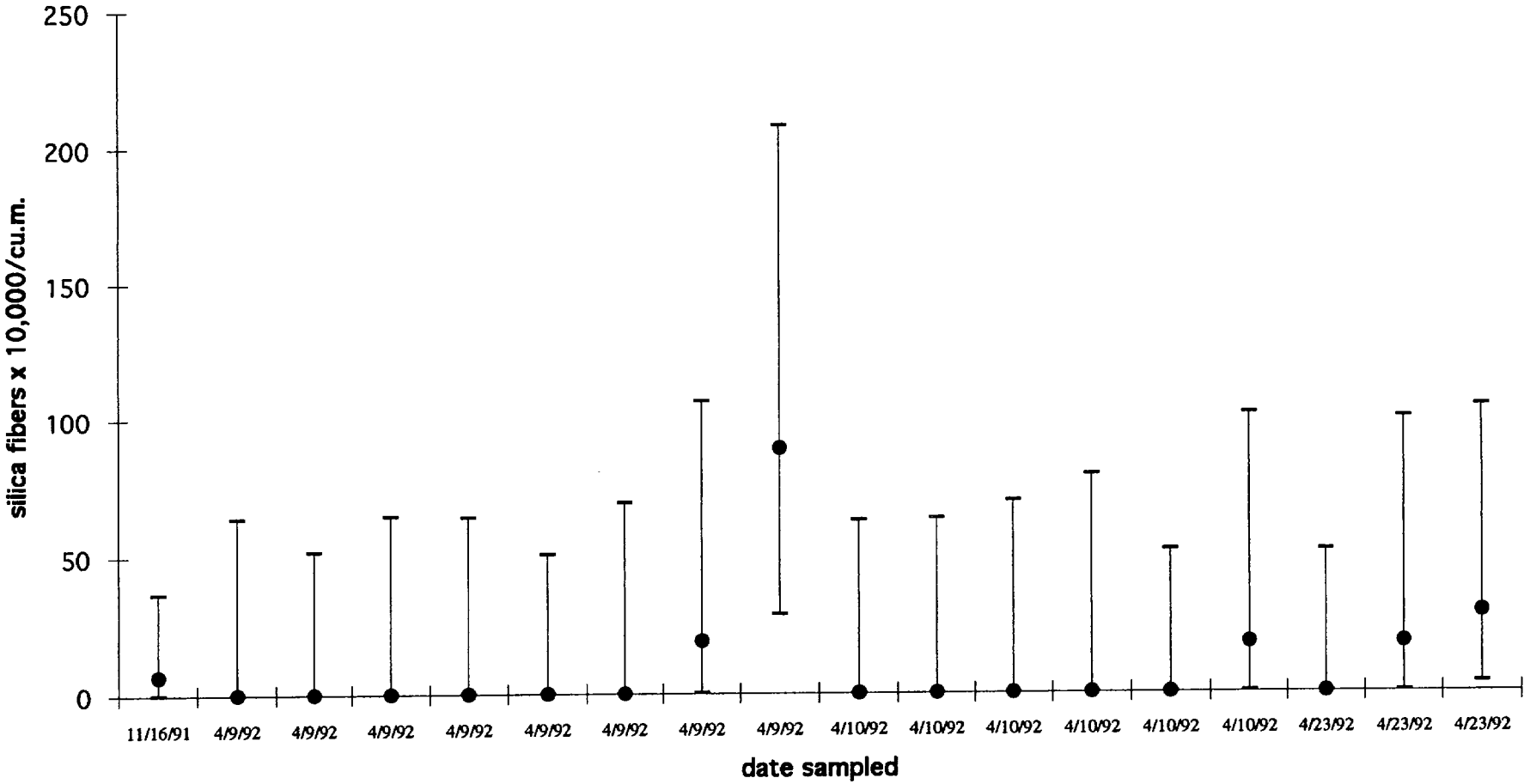


FIGURE 7. TOTAL AMORPHOUS SILICA FIBER CONCENTRATION AND 95% CONFIDENCE RANGE FOR RICE STUBBLE BURNING BY TRACTOR

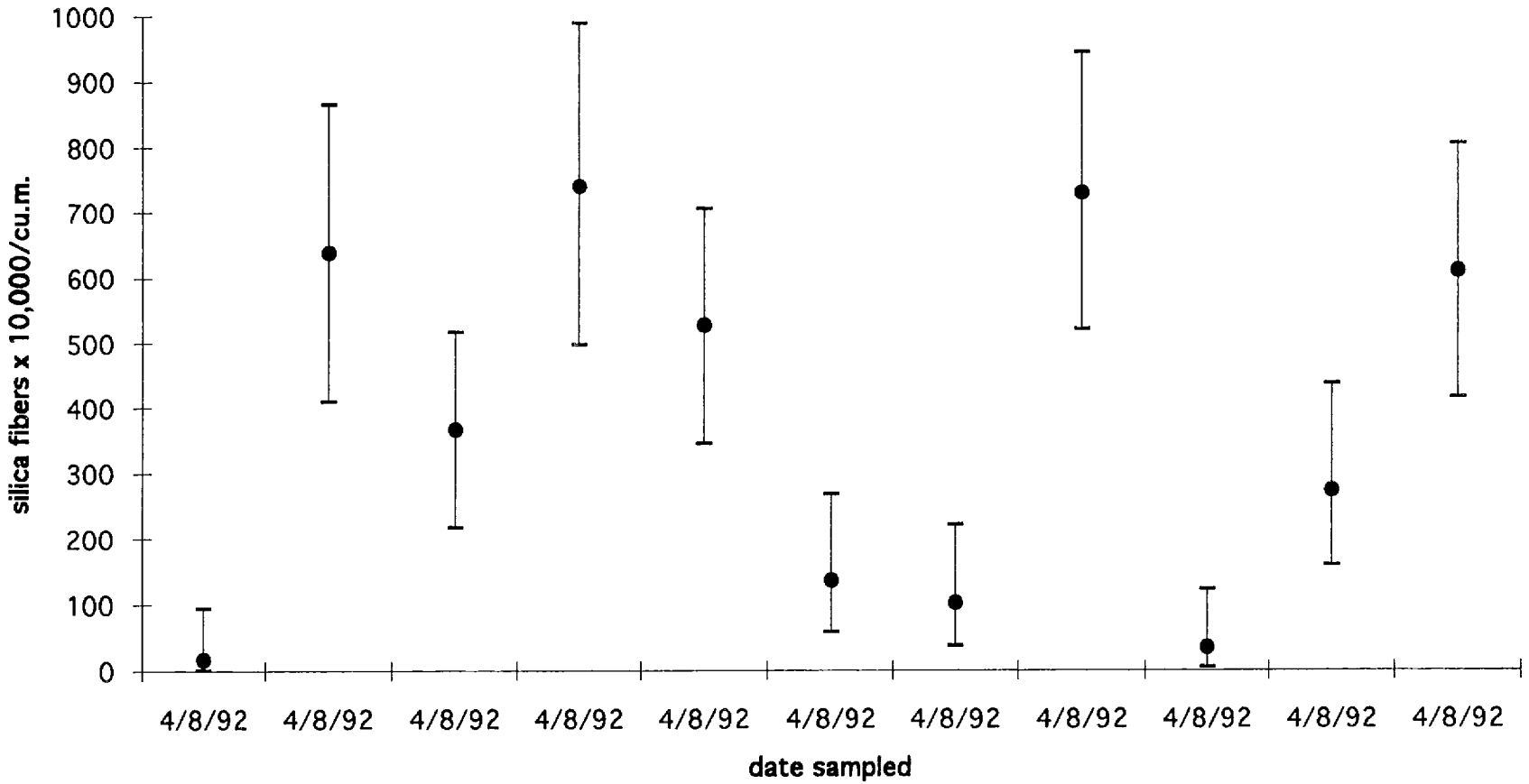


FIGURE 8. TOTAL AMORPHOUS SILICA FIBER CONCENTRATION AND 95% CONFIDENCE RANGE DOWNWIND OF RICE STUBBLE BURNING

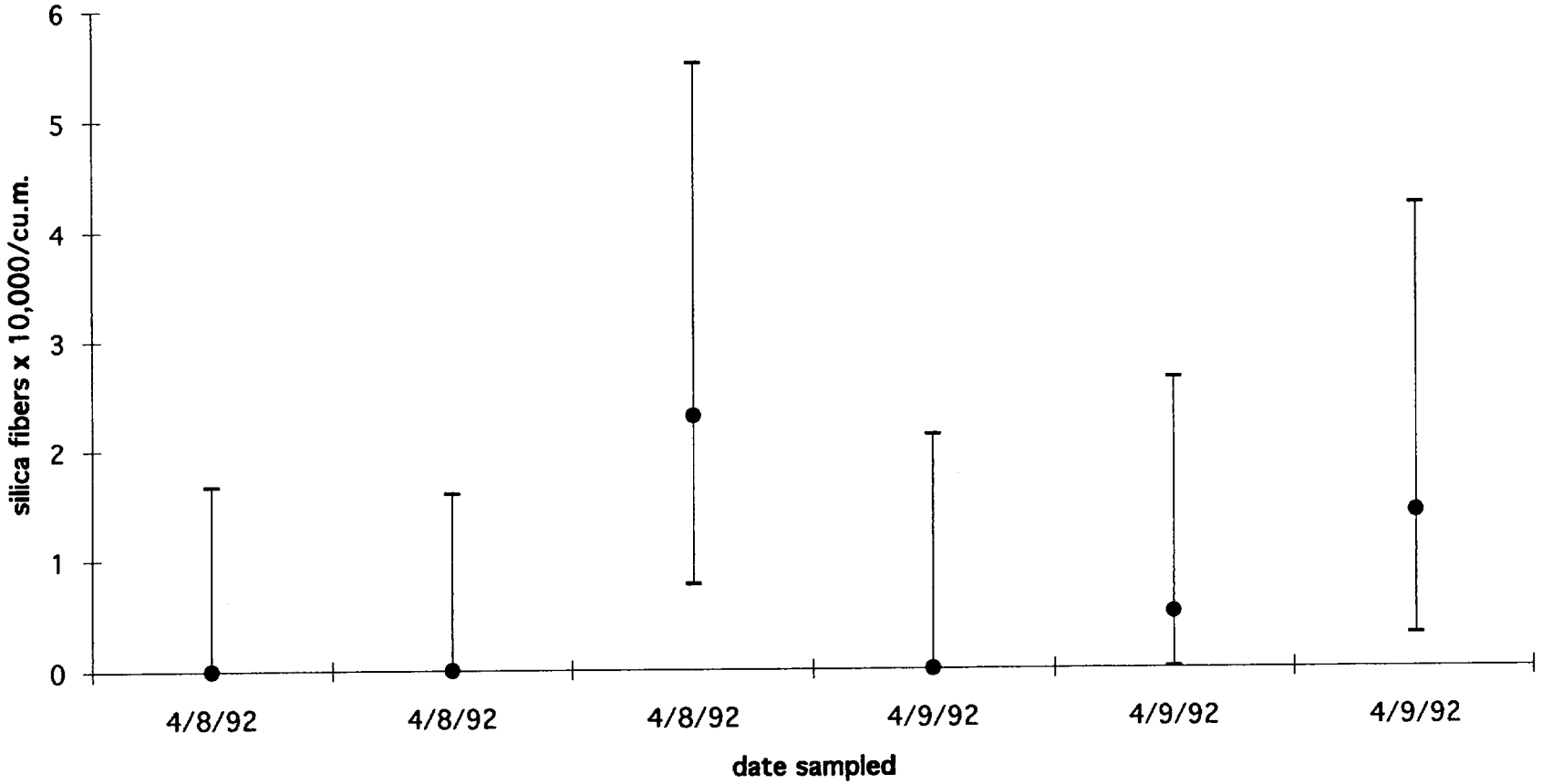


FIGURE 9. TOTAL AMORPHOUS SILICA FIBER CONCENTRATION AND 95% CONFIDENCE RANGE IN NEARBY COMMUNITIES ON DAYS WITH RICE STUBBLE BURNING

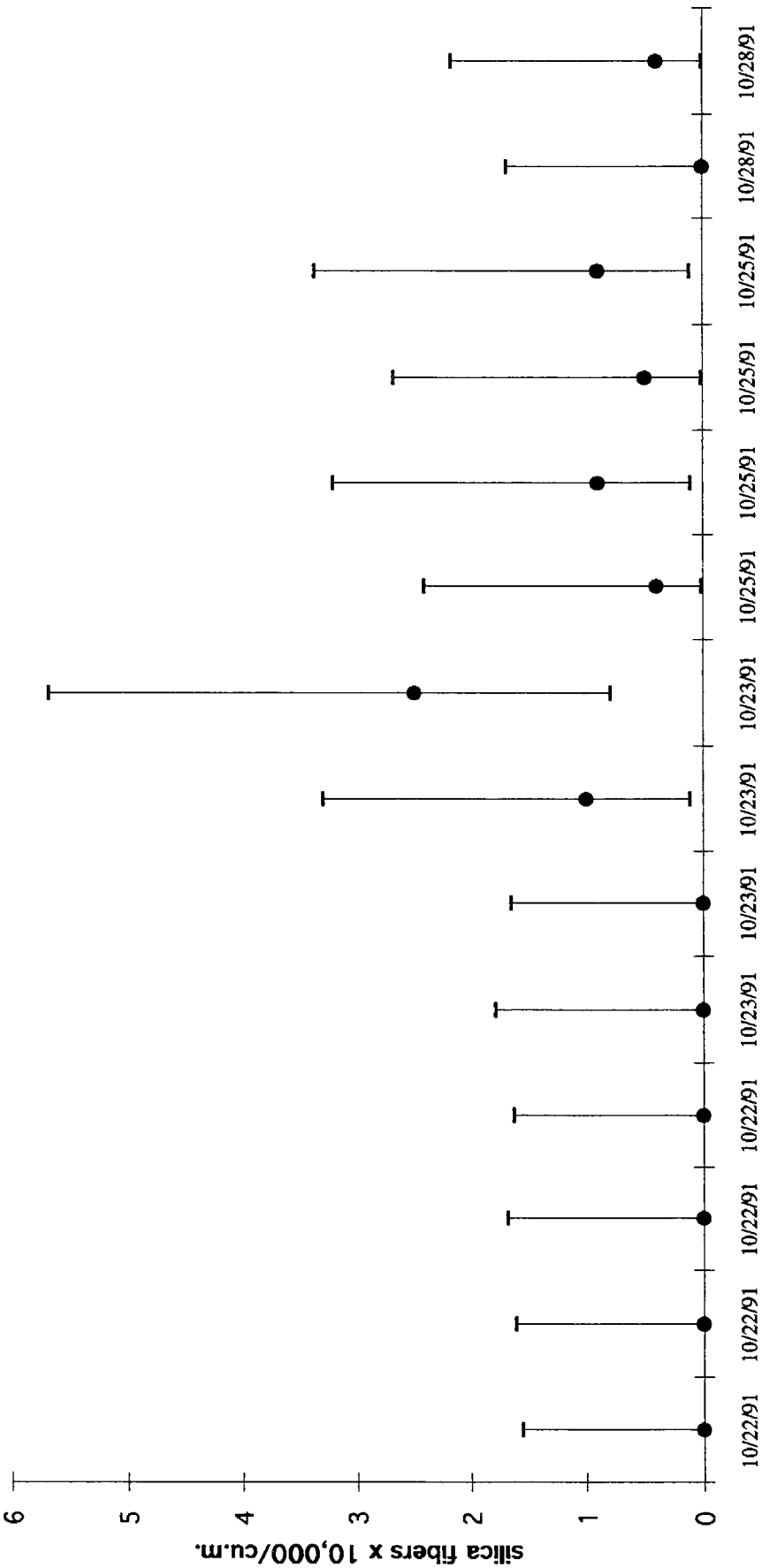


FIGURE 10. TOTAL AMORPHOUS SILICA FIBER CONCENTRATION AND 95% CONFIDENCE RANGE FOR TRACTOR/BULLDOZER DURING FIELD PREPARATION OF A BURNED RICE FIELD

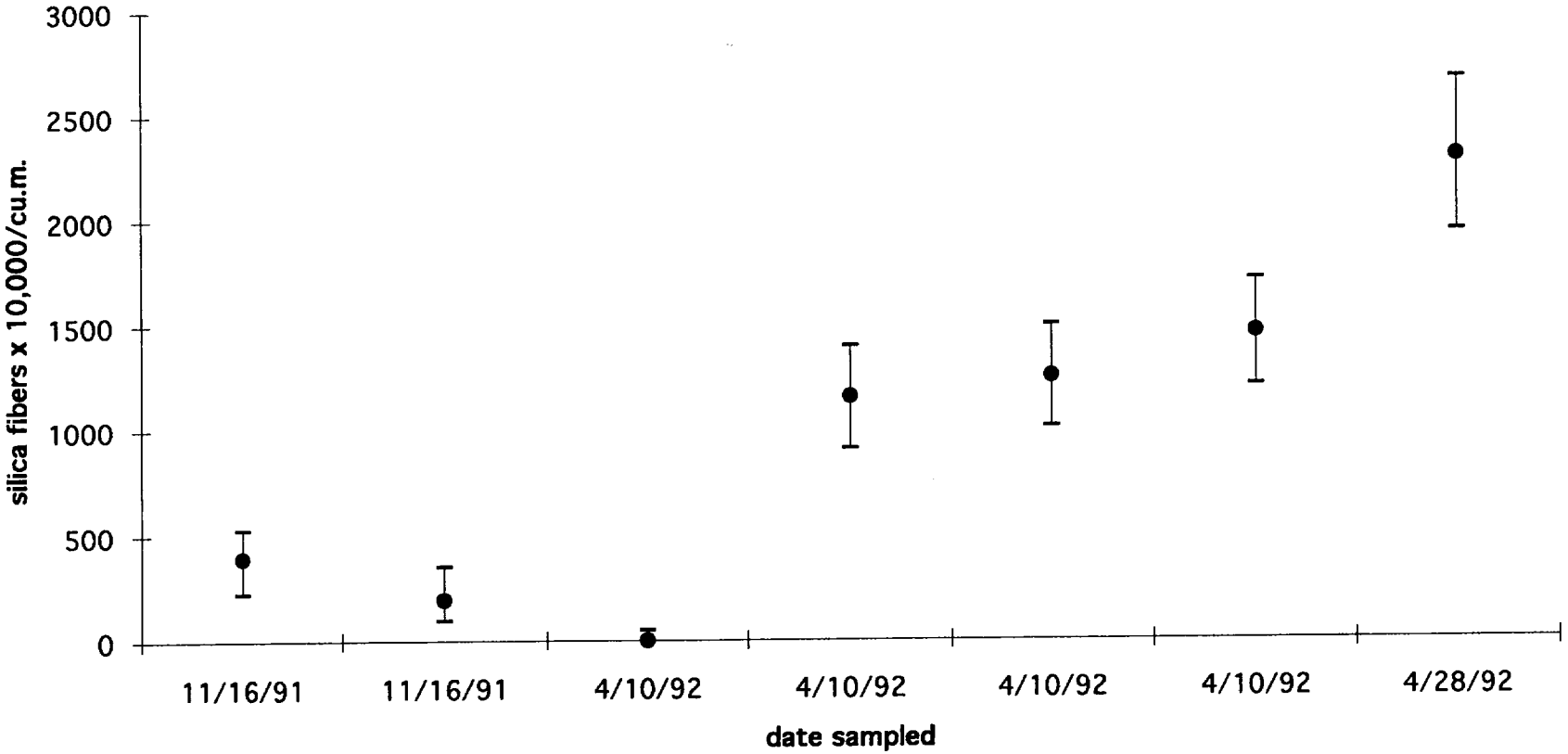


FIGURE 11. TOTAL AMORPHOUS SILICA FIBER CONCENTRATION AND 95% CONFIDENCE RANGE IN TRACTOR INTERIOR DURING FIELD PREPARATION OF A BURNED RICE FIELD

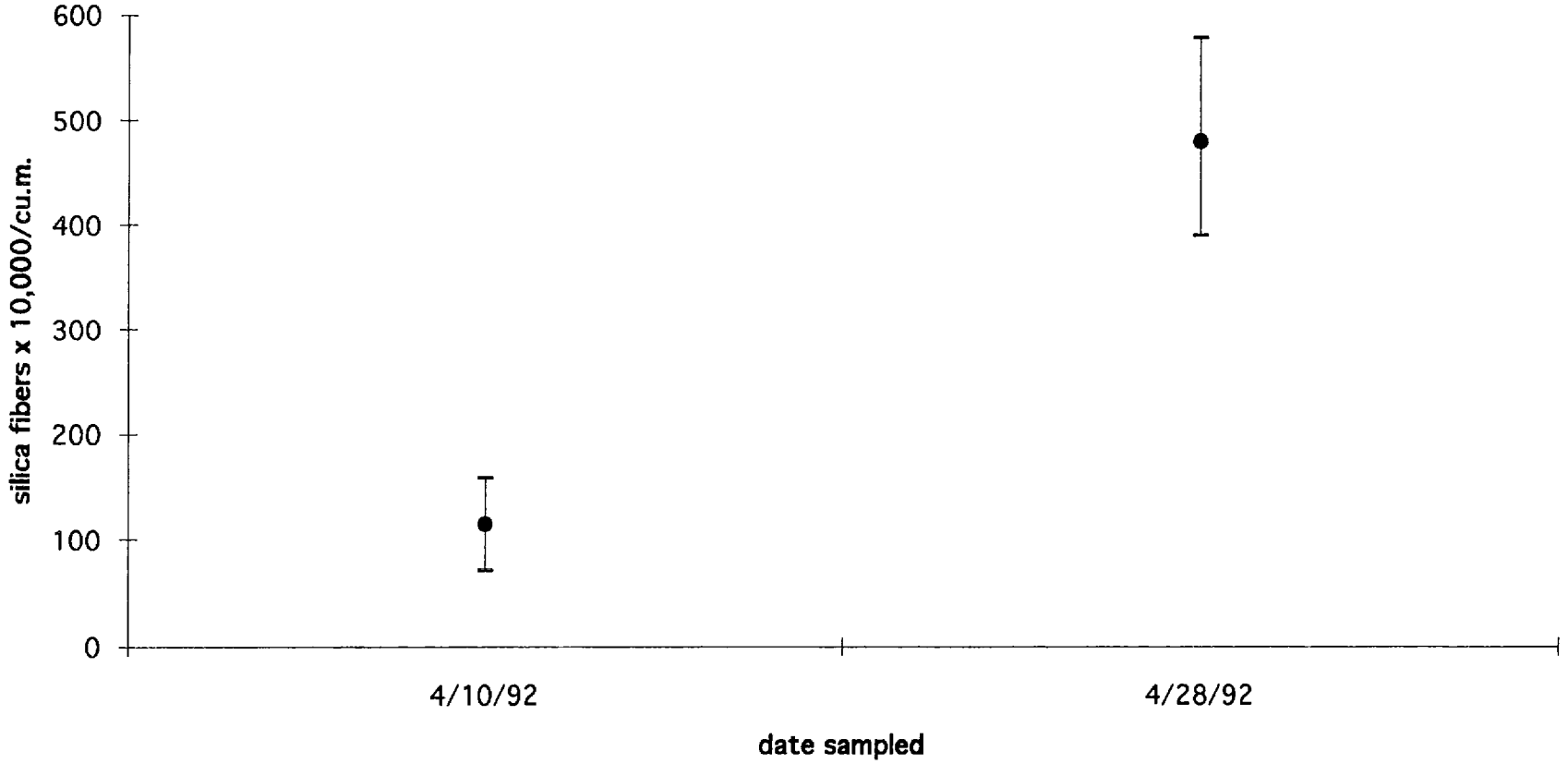
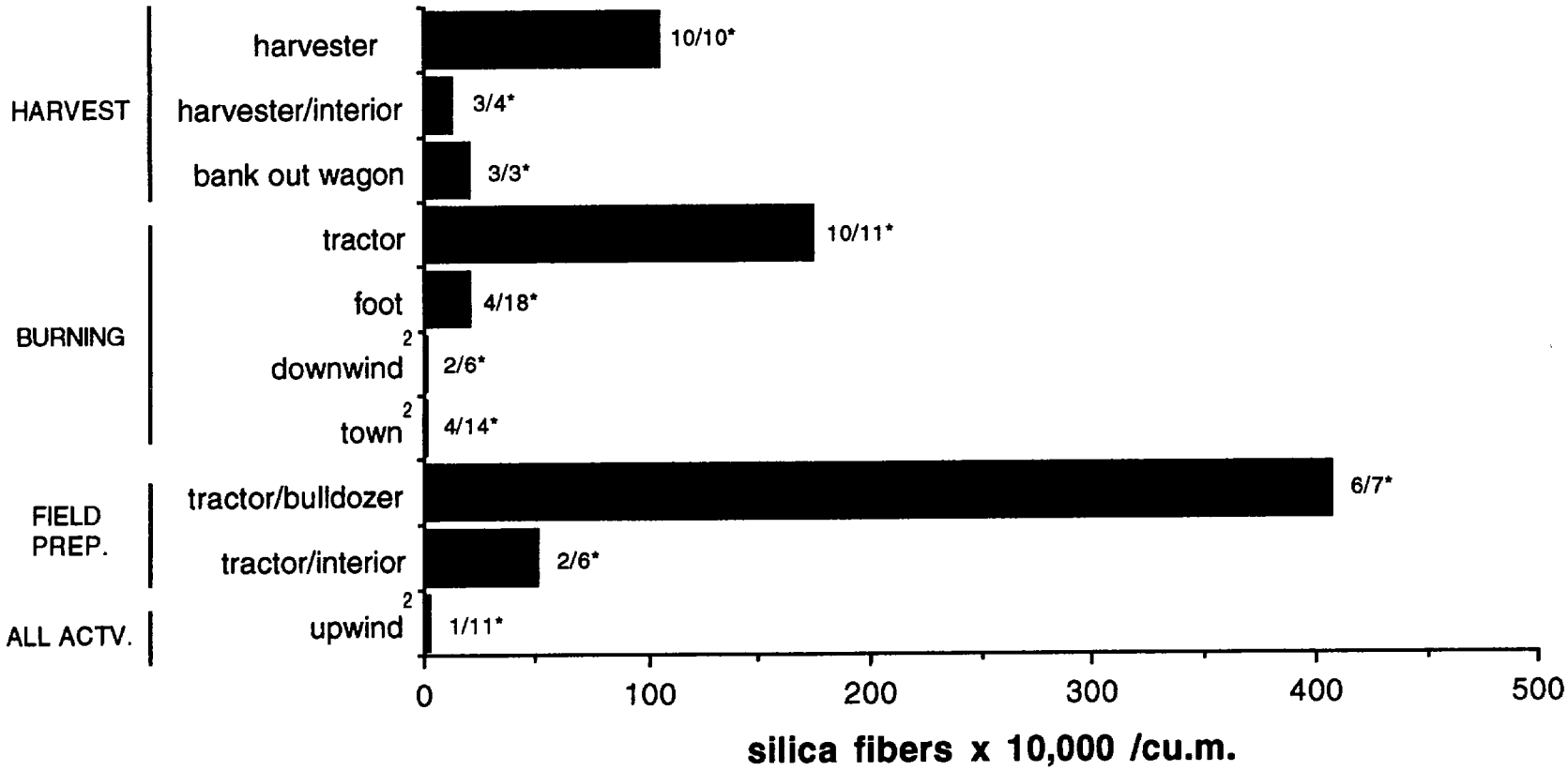


FIGURE 12. GEOMETRIC MEAN[1] OF AMORPHOUS SILICA FIBER (length ≥ 5 μm) CONCENTRATION BY RICE GROWING ACTIVITY



1 Geometric mean of non-zero results only
2 Downwind, town, and upwind are the only area samples, all others are personal samples
* Samples with fibers detected / number of samples analyzed

FIGURE 13. AMORPHOUS SILICA FIBER CONCENTRATION (length ≥ 5 μm) AND 95% CONFIDENCE RANGE UPWIND OF RICE BURNING

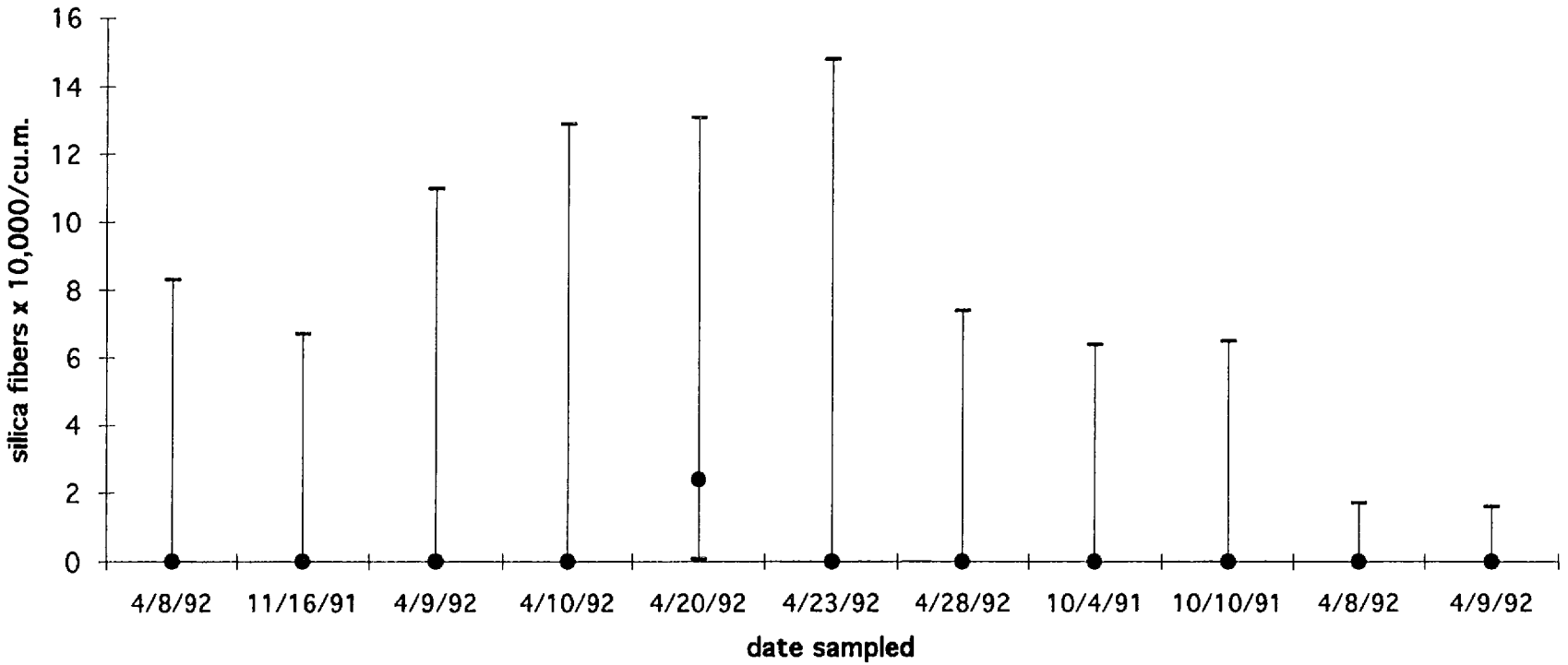


FIGURE 14. AMORPHOUS SILICA FIBER CONCENTRATION (length ≥ 5 μm) AND 95% CONFIDENCE RANGE FOR RICE HARVESTER

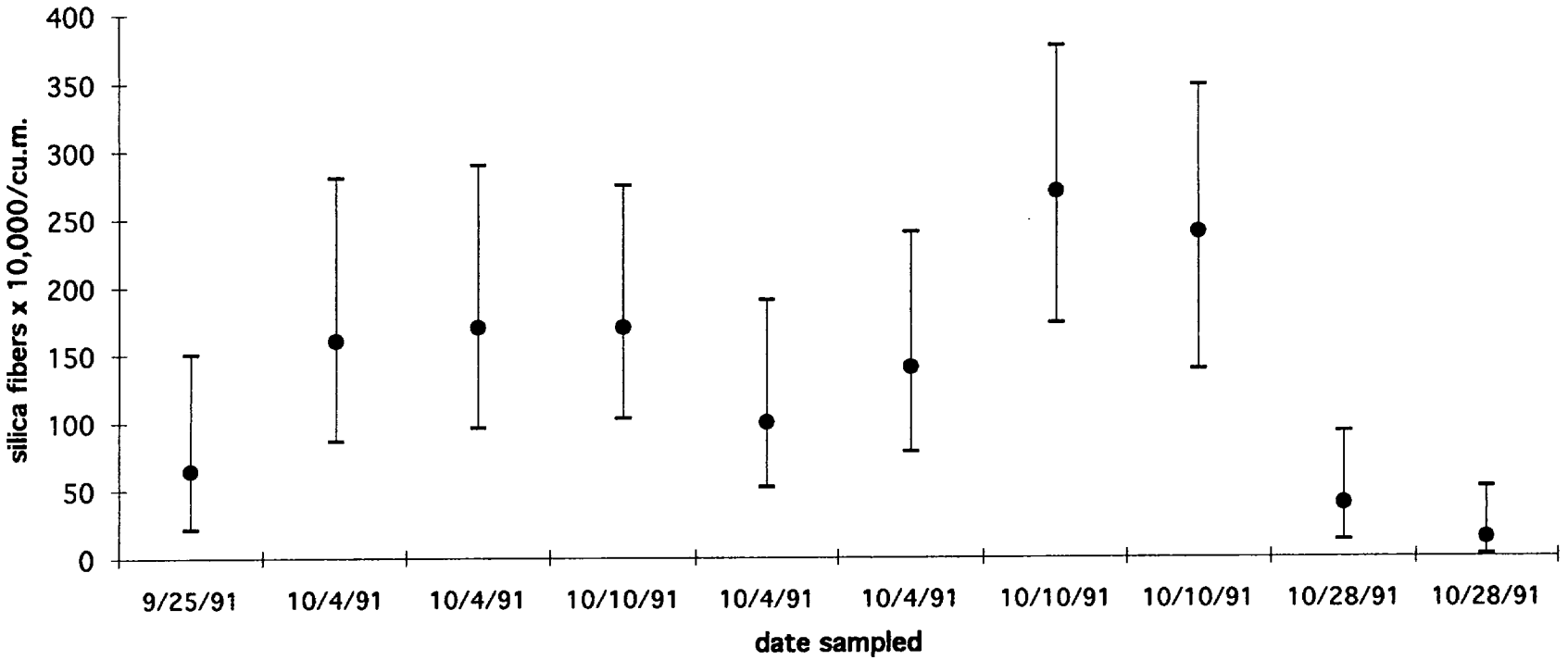


FIGURE 15. AMORPHOUS SILICA FIBER CONCENTRATION (length ≥ 5 μm) AND 95% CONFIDENCE RANGE FOR RICE HARVESTER INTERIOR

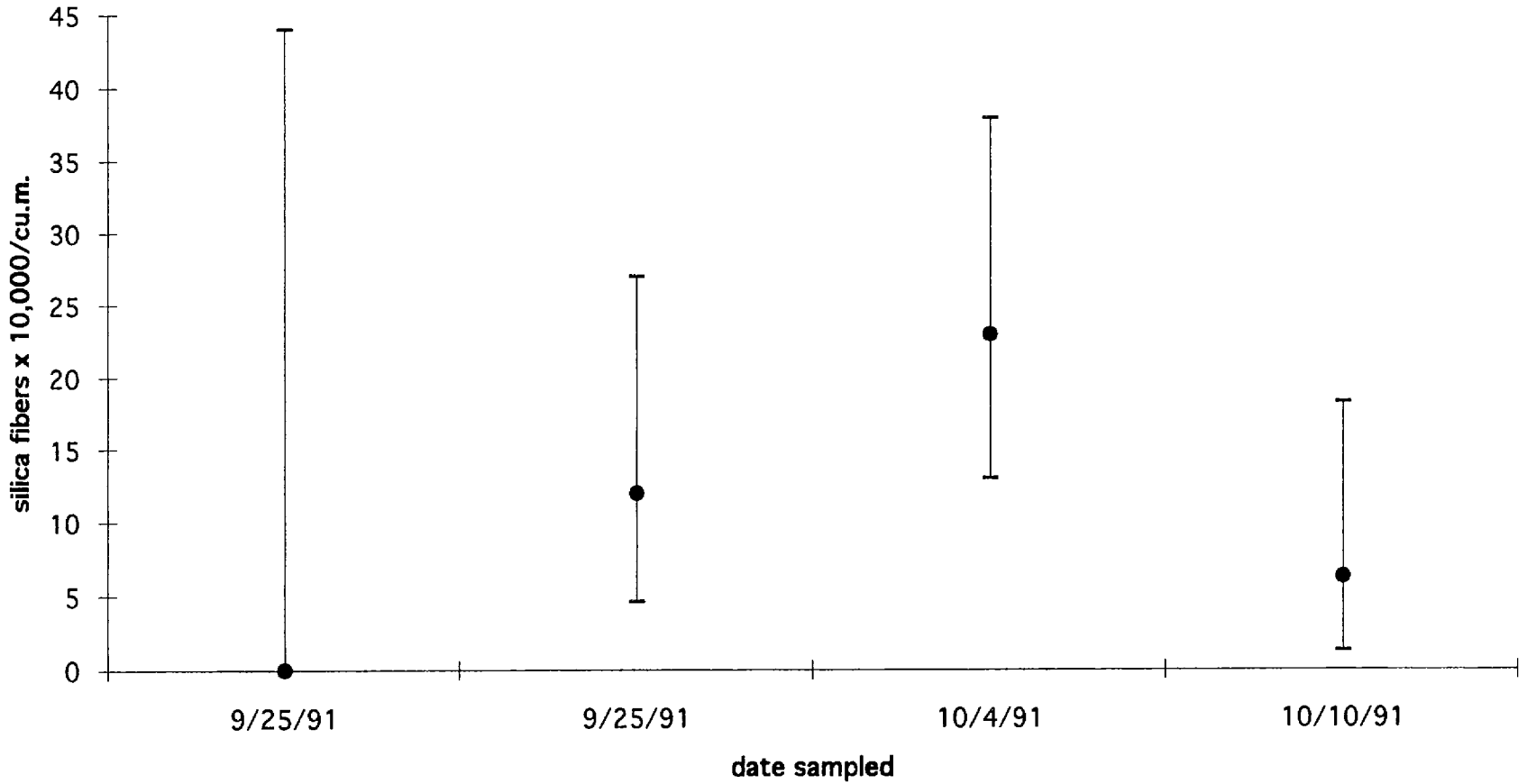


FIGURE 16. AMORPHOUS SILICA FIBER CONCENTRATION (length ≥ 5 μm) AND 95% CONFIDENCE RANGE FOR BANK OUT WAGON DURING RICE HARVESTING

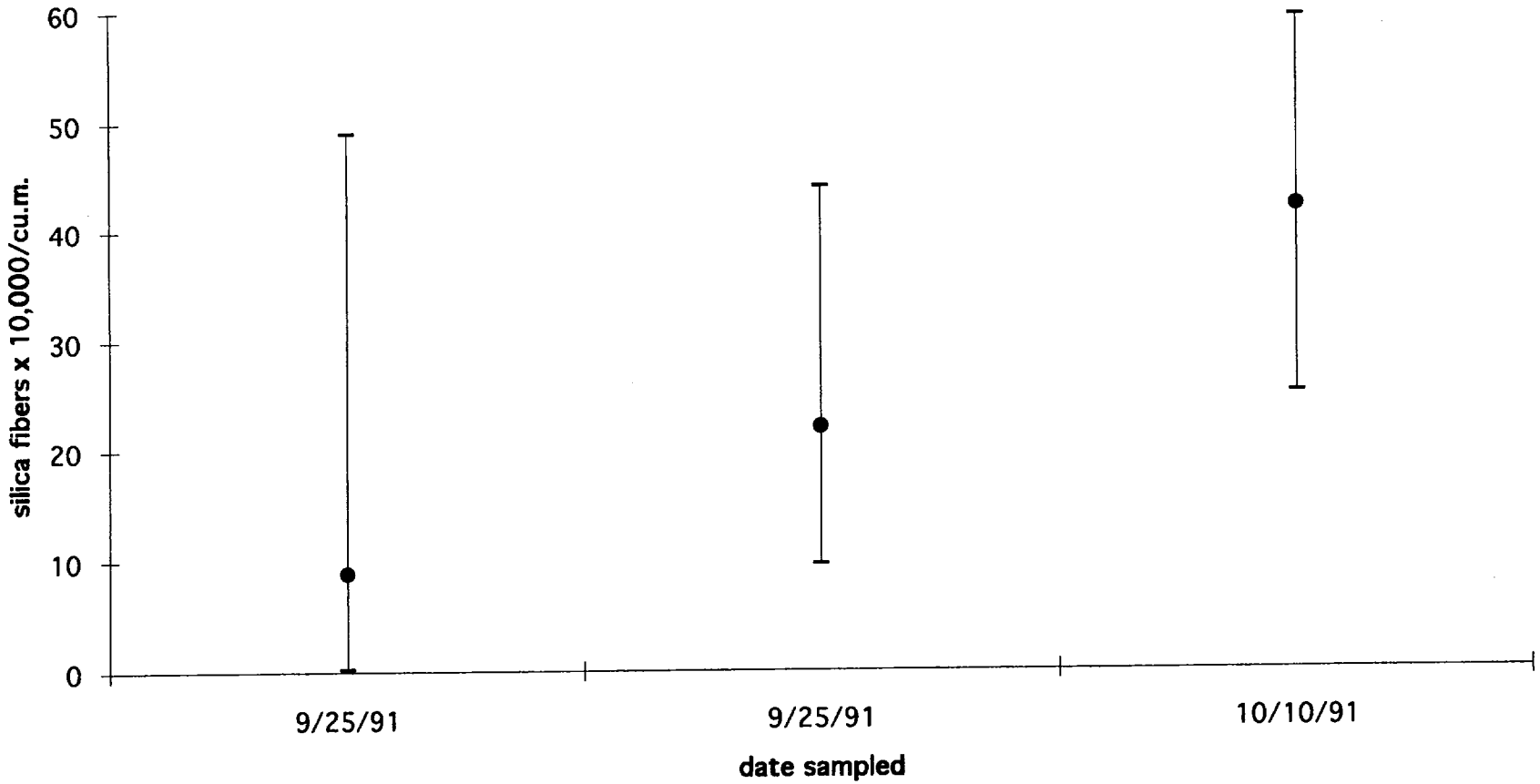


FIGURE 17. AMORPHOUS SILICA FIBER CONCENTRATION (length ≥ 5 μm) AND 95% CONFIDENCE RANGE FOR RICE STUBBLE BURNING BY FOOT

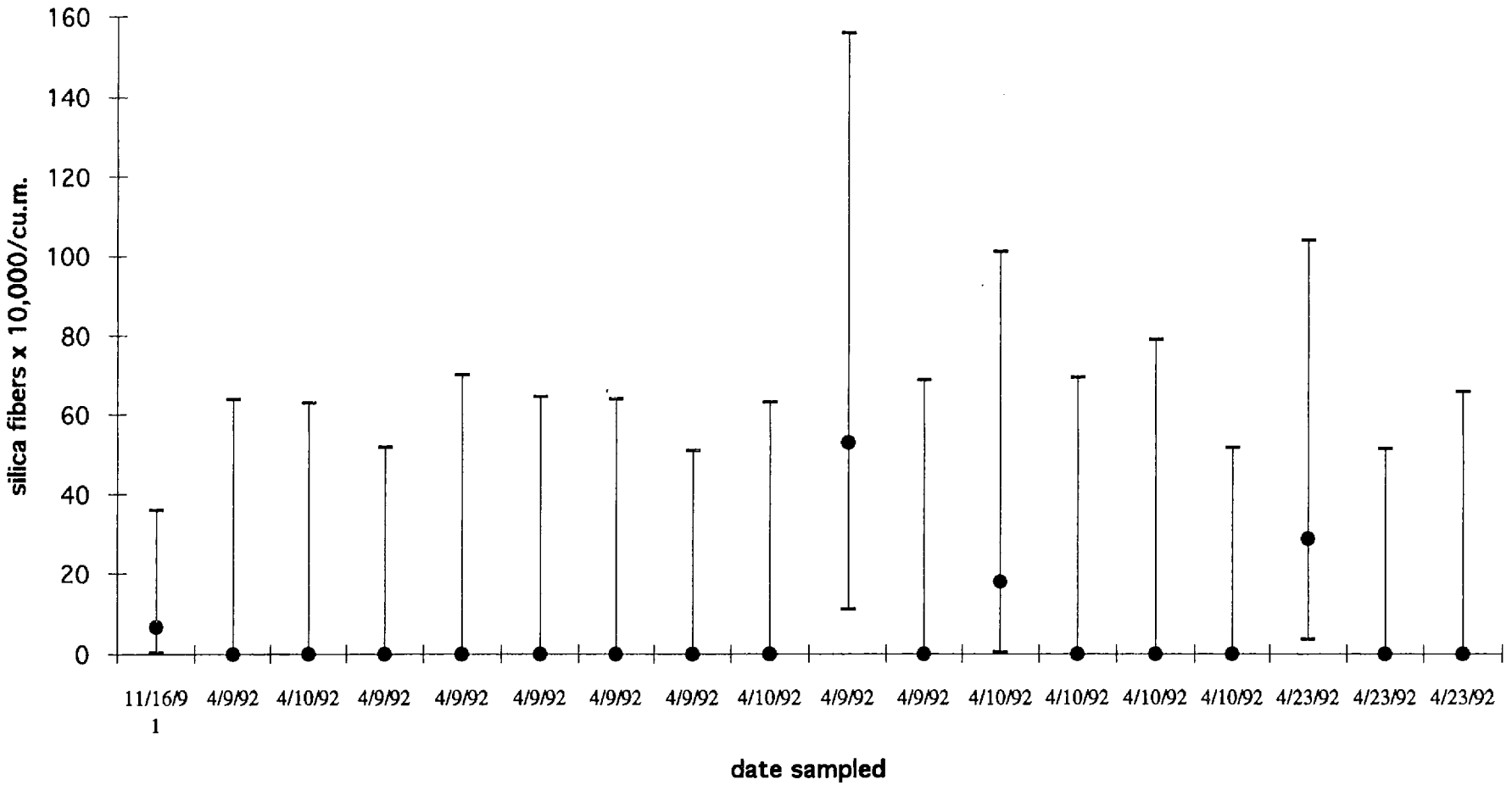


FIGURE 18. AMORPHOUS SILICA FIBER CONCENTRATION (length ≥ 5 μm) AND 95% CONFIDENCE RANGE IN RICE STUBBLE BURNING BY TRACTOR

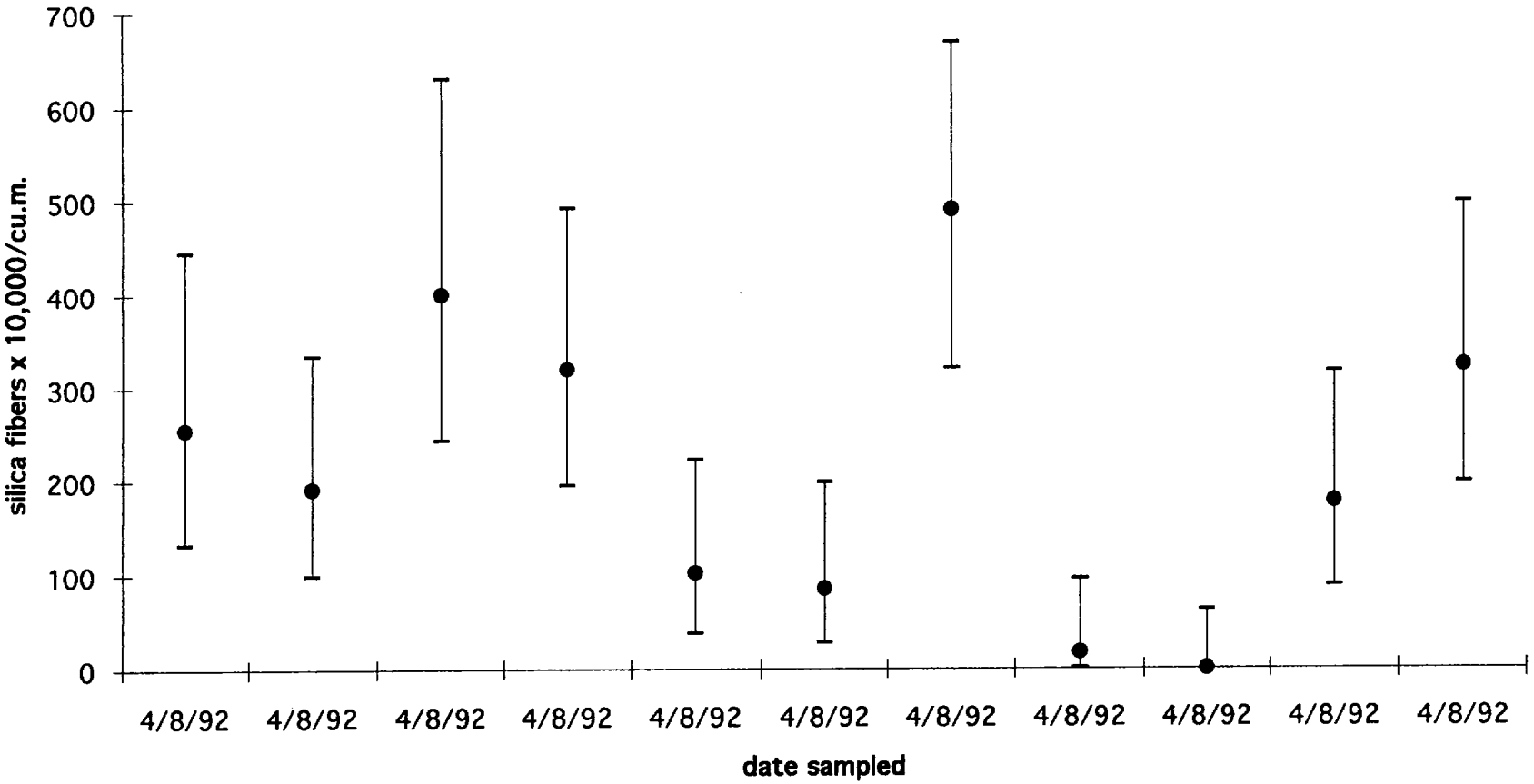


FIGURE 19. AMORPHOUS SILICA FIBER CONCENTRATION (length ≥ 5 μm) AND 95% CONFIDENCE RANGE DOWNWIND OF RICE STUBBLE BURNING

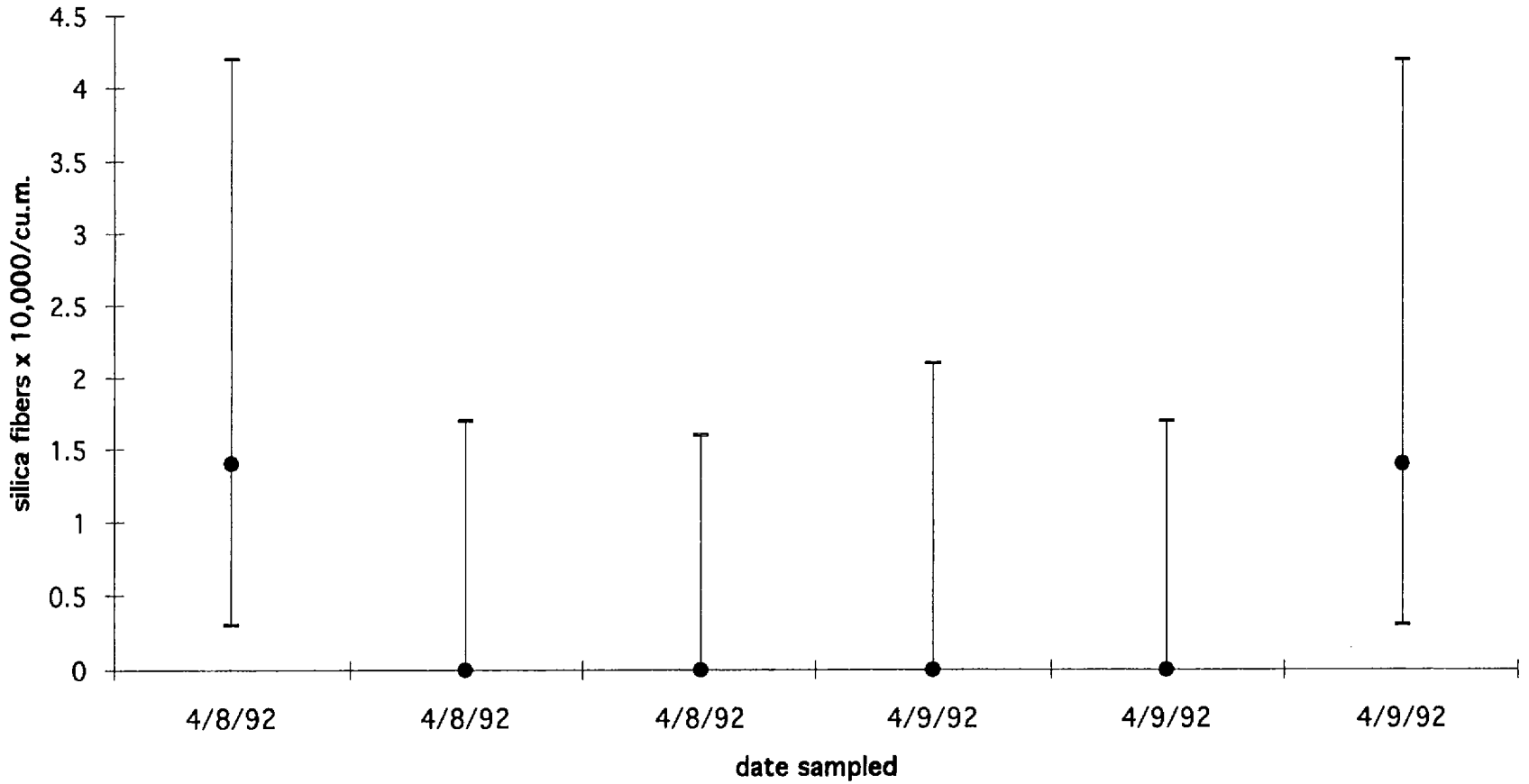


FIGURE 20. AMORPHOUS SILICA FIBER CONCENTRATION (length ≥ 5 μm) AND 95% CONFIDENCE RANGE IN NEARBY COMMUNITIES ON DAYS WITH RICE STUBBLE BURNING

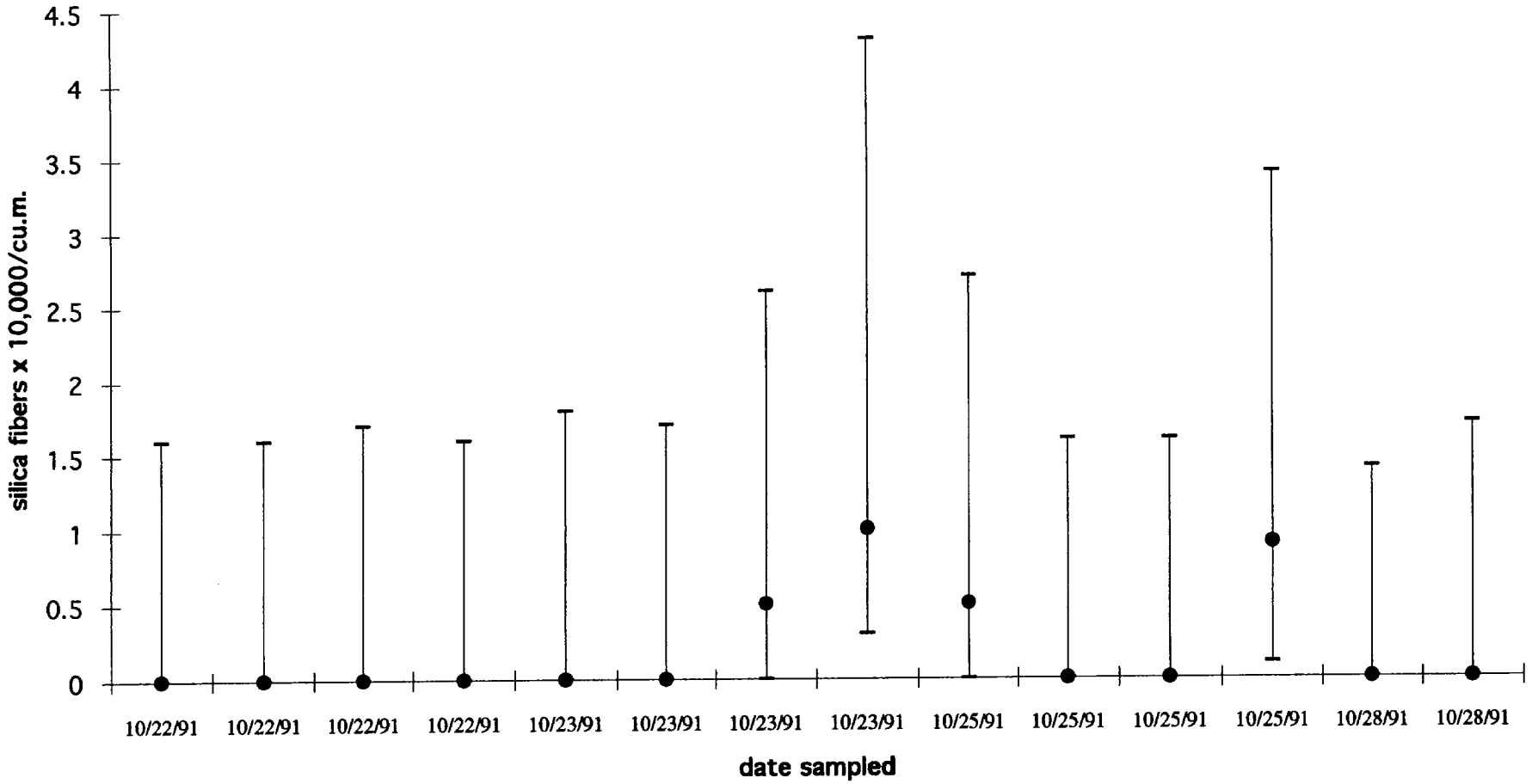


FIGURE 21. AMORPHOUS SILICA FIBER CONCENTRATION (length ≥ 5 μm) AND 95% CONFIDENCE RANGE FOR TRACTOR/BULLDOZER DURING FIELD PREPARATION OF A BURNED RICE FIELD

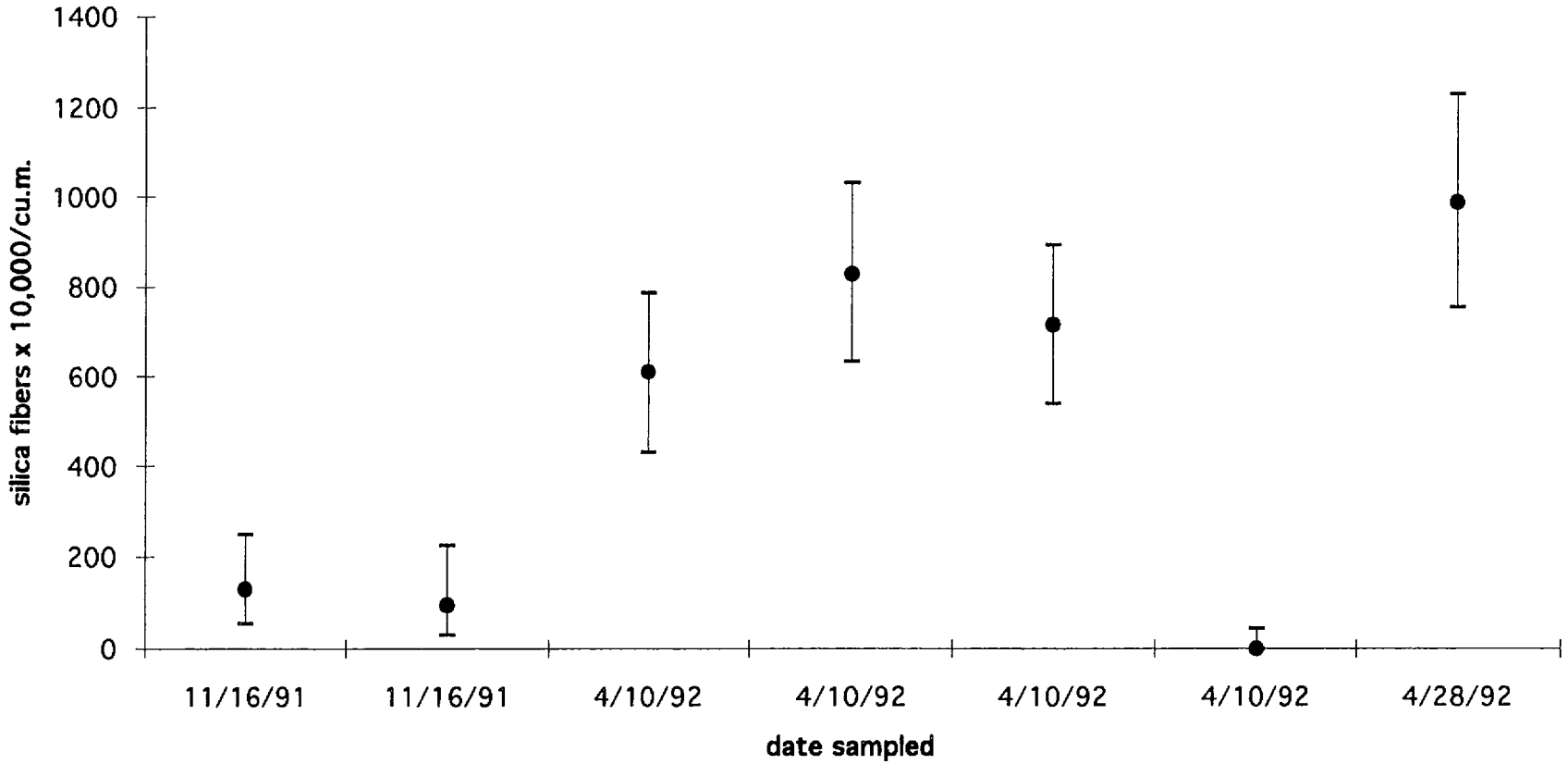
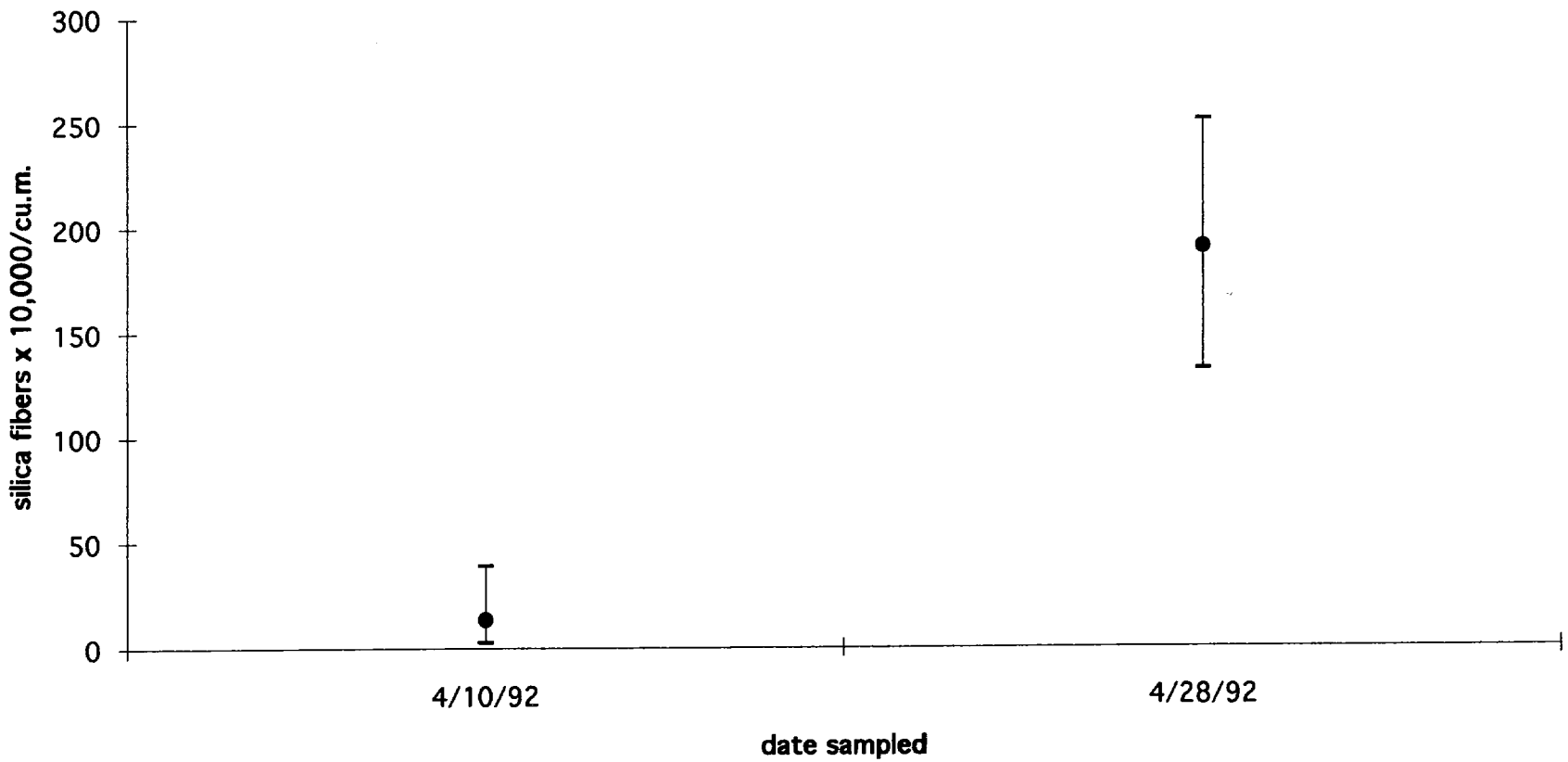


FIGURE 22. AMORPHOUS SILICA FIBER CONCENTRATION (length ≥ 5 μm) AND 95% CONFIDENCE RANGE IN TRACTOR INTERIOR DURING FIELD PREPARATION OF A BURNED RICE FIELD



SECTION 1.d. DISCUSSION

A method has been developed and demonstrated for the sampling and analysis of silica fibers. Many of the variables that affect the silica sampling and analytical method have been demonstrated. The method has some important limitations, but it provides a standard against which future method development may be compared. Some of the most significant emissions and occupational exposures from rice farming have been measured.

Fibers which were composed solely of silicon and oxygen were always found to be amorphous and appeared similar to fibers found in reference material prepared from ashed rice straw. Fibers which contained metals, such as aluminum or magnesium, were seen only in field samples, not in the reference material, and were assumed to be soil particles, such as silicates. Therefore, the discussion that follows focuses on results for amorphous silica fibers which did not contain metals.

As discussed in section 1.d.III.D. below the personal exposure samples taken for this study, and some of the upwind samples, were collected with a respirable dust size selective sampling head, not a PM10 sampling head. Other samples were collected without use of a size selective sampling head. Many of the personal exposure samples were limited to approximately 30 liters of sample volume to prevent overloading. This gave a limit of detection in the range of 0.1×10^6 fibers/m^3. Limits of detection were proportionately lower for samples with higher sample volumes.

The focus of the measurements was airborne amorphous silica fibers. The highest airborne fiber levels documented in this study were 22.9×10^6 fibers/m^3. The highest airborne fiber levels previously documented was 0.3×10^6 fibers/m^3 seen in sugar cane workers [25]. Fiber emissions were detected from rice harvest and field preparation, as well as from burning of rice straw. The results suggest that more work is needed before an estimate can be made of the relative contribution of rice straw burning to total emissions of silica fibers. A limited study was also made of airborne total dust, respirable dust, crystalline quartz and carbon monoxide. A detailed discussion of the results of this project follows.

I. Theoretical approach to the sampling and analysis of amorphous silica fibers

We focused our efforts on development of the direct method of analysis. The method was derived in part from established microscopy methods for sizing and counting asbestos fibers. Because it involved little manipulation of the sample it was unlikely to produce artifacts or to alter the appearance of the particles.

Air samples were collected to provide a pool of samples to use in development of the analytical method. The samples also were used to aid in characterization of exposures and emissions from rice farming operations. A limited number of samples were collected and analyzed. We focused on

occupational exposures for two reasons: occupational exposure samples reliably provided useable sample loading, which was critical for method development; and occupational exposure samples were useful in identifying activities which produce high emissions, as employee exposures typically were orders of magnitude higher than environmental samples.

II. The nature and shape of amorphous silica fibers of rice farming origin

The fibers that were recognized as being of rice farming origin contained only amorphous silica and no other metallic elements. We noted that fibers containing only silicon and oxygen were always amorphous. Because the method obscures the presence of carbon in the sample, it is possible that the fibers contained carbon as well. The fibers differed in significant ways from mineral fibers or most man-made fibers. None were identical. Some fibers were perforated, while others had spikes or other irregular characteristics. They did not appear to be hair-like or needle-like. Fibers examined by stereoscopic techniques often had tortuous or complex three dimensional shapes. Mineral fibers are easily described by parameters of length and shape. The amorphous silica fibers seen in this study were not so easily characterized, as dimensions of width and thickness often varied greatly within individual particles. Aspect ratios seldom exceeded 20:1 and aspect ratios in the range of 5:1 were more common. As the width of individual fibers was often irregular, determination of the aspect ratio was difficult. In some cases fibers showed distinctive patterns and shapes seen in samples of rice plant material.

Photographs of typical amorphous silica fibers seen in this study are included as Appendix D.

III. Development of the silica fiber sampling and analytical method

Seven methodological issues were identified as important for the development of a validated method for the sampling and analysis of rice smoke particles. Achievements towards the resolution of these issues and remaining problems will be reviewed.

A. Choice of direct or indirect method of filter clearing

A direct method was developed and used in this study to analyze approximately 100 samples. The method is presented in Appendix A. The direct method was chosen because it is based on methodology previously developed for the analysis of asbestos fibers. The direct method minimizes the possibility of losing or altering the fibers during sample preparation, which requires dissolving the filter in order to provide a thin sample for transmission electron microscopy.

The principal limitation to the method is the inability to analyze samples which are overloaded. Fiber counts could not be obtained for nine of 104 attempted analyses because of overloading. Analysis was not attempted on several other samples when preliminary examination by SEM indicated overloading. This imposes a requirement for care in field sampling and for training of the field sampling personnel. Even with

care, in some instances field conditions make it difficult to avoid the problem.

Some samples also suffered from a problem of plugging when located in the plume immediately downwind of the fire. Under microscopic inspection, the filter pores were seen to be open, suggesting that the plugging was a result of moisture condensing on the filter, which evaporated prior to inspection. Laboratory experiments confirmed that the polycarbonate filters are subject to plugging in high humidity environments and that the water is held very tightly in the pores. The cellulose ester filters did not plug, nor were there problems of plugging with the polycarbonate filters on the upwind and far-downwind samplers.

The indirect method involves the dissolution of the filter and redeposition of the particles on a second filter for analysis. This allows adjustment of the particle loading on the second filter by taking an aliquot of the solution. Also, the first filter does not have to be a Nuclepore (track etch polycarbonate) filter, permitting more sampling without plugging the pores of the filter. Validation of the indirect method requires verification that particles are not lost or altered during sample preparation. A preliminary investigation of the indirect method was made by placing glass beads on a cellulose ester membrane filter, dissolving the filter and filtering onto a Nuclepore (track etch polycarbonate) filter which was then processed for analysis as in the direct method. The loading of glass beads on the cellulose ester and Nuclepore filters was compared. The result was promising. However, some problems were encountered from residue of the cellulose ester filter depositing on the Nuclepore filter. No investigation was made of the possibility that rice smoke particles would be altered in the sample preparation; this would involve considerable effort.

Another advantage of the indirect method is the possibility of removing some interfering background particles. Soluble material can be eliminated. Biological material could be eliminated by low temperature ashing, however it would have to be verified that ashing did not alter rice smoke fibers. Background particles were especially prominent in wind tunnel samples collected in previous projects to derive an emission factor for silica fibers from rice straw and stubble burning. The background appeared to consist of potassium chloride particles. These were seen in lower amounts in some of the field samples. Also, some of the harvest samples had considerable amounts of fungi present. In fact, harvest samples required refrigeration immediately after sampling in order to prevent further contamination by ubiquitous genera of fungi.

B. Selection of filter medium

The direct method restricts the choice of filter medium to Nuclepore filters. In the indirect method, the final filter is again a Nuclepore filter. However, the first filter can be, for example, a cellulose ester filter which tolerates higher loading without plugging than a Nuclepore filter. Again, further work is necessary to validate the indirect method.

C. Determination of the optimum particle loading

Our experience from the present project was that the range of optimum particle loading for microscopy is 100-1000 fibers/mm^2 of filter surface. Fewer requires the scanning of more grid squares and more operator time. If the maximum number of grid openings scanned is limited to ten and the number of fibers is still less than that required to maintain the sensitivity, then the statistical precision will be reduced. Conversely, if the loading exceeds the above range, particles begin to overlap and it will not be possible to obtain an accurate count.

A more practical problem was overloading of samples with soil and fungi particles which were not of interest and which obscured fibers or made the samples too thick for TEM analysis. In most cases personal exposure sampling time had to be limited to 15 minutes to prevent overloading of the filters with soil particles and fungi. As a result, the limit of detection was in the range of 0.1 X 10^6 fibers/m^3 for these samples. Samples from upwind or from within closed cabs could be run for several hours without overloading with debris, and gave better limits of detection. Overloading with fibers was never a practical problem.

D. Determination of the type of particle size selection necessary in the air sampling

Preliminary samples contained some large structures which obscured the particles of interest. Subsequent personal exposure and field edge samples were taken with a respirable dust fraction cyclone preceding the filter cassette to exclude large, non-respirable particles. This eliminated the problem of the large structures.

The personal exposure samples, and some of the upwind samples, were collected with a respirable dust size selective sampling head, not a PM10 sampling head. A PM10 head was not used because it is too large to use for personal exposure monitoring of mobile employees. The study design called for collection of personal exposure samples for a variety of tasks, including employees on foot. The respirable fraction cyclone used was 10mm long, and is can be attached to an employee's collar. The respirable size fraction, as defined by the American Conference of Industrial Hygienists is the rough equivalent of a fraction of all particles with an aerodynamic diameter of 5 μm, and is described by a cumulative lognormal function with a median aerodynamic diameter of 3.5 μm (plus or minus 0.3 μm) and with a geometric standard deviation of 1.5 (plus or minus 0.1).

On the basis of this study, the use of a cyclone or other device to exclude large particles from the filter is recommended. The impact of the use of a cyclone with fiber count methods was not fully explored, but the cyclone was not expected to lead to significant breakdown or alteration of the fibers. It may have lead to some loss of fibers with larger aerodynamic diameters which would have been collected using a PM10 head. Fibers appeared to be evenly and randomly distributed on the filter, but this was not investigated in detail. An analysis of fibers on one heavily loaded sample did indicate that their distribution was

random. Visual inspection of the samples showed evenly distributed material, without the appearance of rings or streaks in the deposition pattern.

E. Investigation of the use of electron diffraction to determine the crystallinity of the silica

It has proven to be practical to use electron diffraction to determine the crystallinity of the silica particles. This was done in the microdiffraction mode, which minimized the interference from nearby particles. In the case of a particle too thick to allow the electron beam to penetrate, it was possible to obtain diffraction spots from thinner areas near the edge of the particle. It was noted that particles containing both a silicon and an aluminum X-ray peak were invariably crystalline. These were undoubtedly mineral particles from soil which were expected to be crystalline. This lent credence to the diffraction method.

Scanning electron microscopy (SEM) views showed the presence of surface particulates on some fibers. The provisional method does not distinguish fiber diffraction from diffraction arising from such surface contaminants.

F. Application of X-ray diffraction to determine the crystallinity of the silica particles

This was not pursued during the present project. We noted that the X-ray diffraction method is essentially a bulk method rather than a single particle method. Thus, since fibers composed a relatively small proportion of the total number of particles, X-ray diffraction could not be used to analyze the crystallinity of the silica fibers.

G. Testing of the method

During the project, duplicates and replicates were obtained and analyzed. Duplicates consisted of two samples obtained from side-by-side samplers operating simultaneously. The duplicates were mixed into the other sample batches and numbered without identifying them as duplicates. The microscopist analyzed the samples at different times without knowing they were duplicates. Replicates were similar to duplicates except that more than two samplers were used. Samples were also recounted by each of the two operators. The data on duplicates, replicates and recounts are included in Appendix C.

A limited analysis suggests that fibers less than 5 μm in length contribute the most to overall variability in analyses. Experience from asbestos fiber counting shows that reliability of fiber count methods drops severely when applied to the smallest particles at the limits of resolution of the method.

The coefficient of variation for recounts by the same analyst is also within the range of fiber count analytical methods. The variability between analysts was higher than acceptable for total fibers, but in an acceptable range and is comparable to that found on other fiber count methods for fibers greater than 5 μm in length[41].The variability could

be reduced with the preparation of standard slides and a systematic reanalysis of the standard slides and comparison of results between analysts, as is done in the Proficiency Analytical Testing program for asbestos fiber count analysis [41].

The coefficients of variation for duplicate samples, which averaged 24% (18% for fiber greater than 5 μm in length), is comparable to other field industrial hygiene sampling and analytical methods, and is relatively low, as it reflects both the variability of the analytical method as well as small amounts of variability between side by side measurements.

IV. Interpretation of results of silica fiber analyses

Fibers which were composed solely of silicon and oxygen were always found to be amorphous and appeared similar to fibers found in reference material prepared from ashed rice straw. Fibers which contained metals, such as aluminum or magnesium, were seen only in field samples, not in the reference material, and were assumed to be soil particles, such as silicates. Therefore, the discussion that follows focuses on results for amorphous silica fibers which did not contain metals. No analysis was made of results for fibers which contained metals.

The reported value of analyses should be interpreted with caution. The analytical method is still provisional. Variability between analysts was noted, but results were not adjusted for this factor. Results have not been converted to eight hour time weighted averages or 24-hour averages. Sample times were typically in the range of 15 minutes, so individual results may reflect temporal variability in the process. Insufficient samples were collected to analyze temporal variability.

Each result is reported with a statistical confidence interval. For example, a typical result is reported as a calculated airborne concentration of 220,000 fibers/m^3, with a 95 percent confidence range of 96,000 to 440,000 fibers/m^3. The statistical methodology is standard for asbestos fiber analysis [40]. The analysis consists of microscopic examination of randomly selected small portions of each air sample filter membrane. The amount of the filter actually examined in a laboratory analysis typically ranged from 0.01 percent to 0.03 percent of the total filter surface. The width of the confidence range is a factor of the percent of the filter analyzed and the total volume that had been pulled through the sample. The confidence range is narrower for samples for which a high volume of air was collected. The ability to influence the confidence range by analyzing more of the filters is limited by the time required for the analysis. In this study a maximum of ten field were analyzed.

In many of the analyses only a small number of fibers were detected, making interpretation of results difficult. For example, in samples from employees on foot who were burning fields, no fibers were found on twelve of eighteen samples. However, the results were more meaningful when viewed as a data set and sample distribution, as shown in figure 9.

Caution is also required because of the small number of samples in each exposure type data set. The number of samples per exposure type ranged from a high of eighteen for personal exposure to employees on foot burning rice stubble, to a low of two from inside the cab of a tractor during field preparation.

Detailed analyses were made only of results for fibers which contained amorphous silica and which did not contain other metallic elements such as aluminum. The aluminum silicate fibers are thought to be of soil origin and are not specific to rice farming. The method does not distinguish between particles which are composed exclusively of amorphous silica and particles which contain amorphous silica and carbon or other traces of plant material. Samples are coated with carbon in the TEM analysis preparation process. Therefore the presence of carbon in the samples would be obscured by the method.

V. Measurements of airborne amorphous silica fibers

This study confirmed the generation of airborne amorphous silica fibers associated with all types of rice farming operations. Fibers were also found downwind of farming operations and in communities in the rice farming region. Results of upwind and community samples showed a very low background level of airborne silica fibers, probably less than 9,000 fibers/m^3. Samples taken in rice fields during harvest, burning of straw and stubble, field preparation after burning, and downwind of those operations all showed elevated airborne silica fiber levels. The highest levels observed were samples from field preparation, which were up to 22.9 X 10^6 fibers/m^3. These results exceeded the highest levels of airborne silica fibers recorded in previous studies, which was 300,000 fibers/m^3 seen in sugar cane workers [25].

A. Samples from towns in the rice farming region

Fourteen airborne samples taken in towns in the rice farming region on days when there were burns indicated an geometric mean concentration probably less than 9,000 fibers/m^3, and less than 5,000 fibers/m^3 for fibers greater than 5 μm in length. The concentration in town samples could not be calculated with great accuracy because of the very light loading of the samples, despite relatively high samples volumes, and the limited number of samples. It is not possible to state that these fibers were the result of rice burning operations. Sampling indicated that fibers are released by harvest, as well as field preparation. Fibers may be released by vegetation, fires or other farming activities.

B. Samples upwind of rice farming operations

Eleven samples taken upwind of rice farming operations yielded results similar to the town samples. The samples indicated that the geometric mean upwind concentration was probably less than 14,000 fibers/m^3 in air. For fibers greater than 5 μm in length, there was one fiber found on one sample, giving a calculated concentration of 24,000 fibers/m^3, with no fibers found on ten of the samples. More precise conclusions are not possible because of the small number of samples and the relatively wide confidence range of the results. These results reflect

the ambient background level of amorphous fibers found in rural farm areas.

C. Samples from rice harvest operations

Samples from rice harvest operations had sufficient loading to be interpreted with some confidence. The samples were divided into three subsets: harvester exterior, harvester interior (within the harvester cab) and bank out wagon. Average results were well above upwind values and represented measurements of emissions from harvest operations. It is not clear if the particles detected contain only amorphous silica or amorphous silica within plant tissue, as the analytical method cannot distinguish between these two types of particles.

Samples from the harvester exterior had a geometric mean of 4.159×10^6 fibers/m^3, and 1.04×10^6 fibers/m^3 for fibers greater than 5 μm in length . Eight hour time weighted average levels would be higher, as work days typically included eight to ten hours of harvesting. This was the second highest average level detected for all rice farming operations. This shows that harvest activities may be a major contributor to total silica fiber emissions from rice farming. This is the first time that silica fiber measurements have been made on harvest of a crop that had not been burned first. Sugar cane harvest has been investigated, but sugar cane is typically burned to remove leaves before it is harvested, therefore, samples from sugar cane harvest can be interpreted as representing exposure hazards of burned plant matter. Further investigation will be needed to show if the emissions measured are unique to rice harvest, or if they are found in other harvest operations and other dusty operations involving plant matter. As amorphous silica has been reported from a wide variety of plants, it is likely that silica fiber emissions will be found in a variety of operations involving many different types of crops and vegetation.

Samples taken from the interior of harvesters and from the bank out wagon interior showed that enclosed cabs provided a measure of protection from exposures outside the cab. On average, fiber concentrations inside the cab were approximately twenty percent of the fiber concentration outside the cab.

D. Burning of rice straw and stubble in the field

The samples confirmed the release of fibers from burn operations and accurately reflect employee exposure. However, we do not think that the fiber levels detected by this monitoring are an accurate reflection of silica fiber emissions from rice straw and stubble burning operations because we observed that employees on foot usually were able to stay clear of the smoke. The employees had very little actual exposure to the smoke. Employee exposure levels for burning rice straw in the field were low, with a geometric mean of 0.217×10^6 fibers/m^3, and 0.206×10^6 fibers/m^3 for fibers greater than 5 μm in length. This number is not precise because of the small number of samples and the small number of fibers found on the samples, however, it is clear that there was very little employee exposure to silica fibers when they stayed clear of the

smoke plume. Eight hour time weighted averages would be lower as employees typically spent one to three hours burning in a day.

It was also clear from field operations that very little smoke from the burns reached downwind monitoring locations. Early efforts to monitor for airborne fibers in concentrated downwind plumes at the immediate field edge were complicated when the polycarbonate filters used for direct analysis plugged, shutting off the sampling pumps. This problem was attributed to high levels of moisture in the plume. Subsequent samples were taken a greater distance from the field edge. It was clear that little of the visible plume reached downwind sampling locations.

The highest field burn airborne levels, a geometric mean of 2.29 X 10^6 fibers/m^3, and 1.74 X 10^6 fibers/m^3 for fibers greater than 5 μm in length, were detected on one day of monitoring when a farmer used a tractor towing a trailer which set the rice straw on fire. Field observations showed that the tractor raised dust and sometimes raised ash by driving over areas that had burned. The tractor driver's path also frequently passed through areas that were a little smokey. It was not possible to tell from the analysis whether the fibers where from dust and ash raised by the tractor or from smoke.

E. Field Preparation

Two field preparation activities were included in this study. Samples taken on 11/16/91 were for removal of levees from a burned field using a bulldozer. All other field preparation samples were for discing or ripping in fields which had been burned. Fiber levels from field preparation activities were higher than from harvest or burning. The geometric mean of fiber levels was 8.44 X 10^6 fibers/m^3, with a peak level of 22.9 X 10^6 fibers/m^3 observed on one sample. For fibers greater than 5 μm in length, the geometric mean of fiber levels was 4.06 X 10^6 fibers/m^3, with a peak level of 9.9 X 10^6 fibers/m^3. Eight hour time weighted averages would be higher as employees typically spent 10-12 hours daily in field preparation. The presence of elevated fiber levels seen did not depend on having fresh ash in the field. Results were obtained from one field which had fresh ash, one field which was idle for about two months since it had been burned and which had been rained on, and one field which had already been disced once since it had been burned. The origin of amorphous silica fibers may be attributed to rice plant ash which was on the soil surface or which had become incorporated into the soil, or to particulate released from decomposing plant matter which has become incorporated into the soil.

Two samples taken inside the cabs of tractors being used for field preparation gave average results of 2.34 X 10^6 fibers/m^3, and 0.501 X 10^6 fibers/m^3 for fibers greater than 5 μm in length. These results confirmed observations from rice harvest that enclosed cabs provide a significant but incomplete protection from exposures outside the cab. We observed that one driver preferred to operate with the cab windows open in the morning when the weather was cool.

VI. Total dust, respirable dust and crystalline silica

Total dust and respirable dust levels varied by type of rice farming operation. Levels were highest for field preparation, followed by rice harvest and rice straw burning. We have only analyzed these results in a limited way because of the small number of samples collected. Typically one total dust and one respirable dust sample was collected on each day or sampling; a total of 22 exposure samples and 4 blanks were collected.

Field preparation activities generated the highest levels of total airborne dust and the highest levels of fine dust in the respirable dust size fraction. Two out of three total dust samples exceeded Cal/OSHA regulatory limits for total dust of 10 mg/m^3 and one of three dust samples exceeded the Cal/OSHA regulatory limits for respirable dust of 5 mg/m^3. Because these were all samples from the exterior of closed cab equipment, exposures to employees would have been lower. The ratio of total dust to respirable dust levels indicated that most of the dust was not respirable.

Dust levels from harvest operations were high, but lower than field preparation. Total dust levels exceeded Cal/OSHA regulatory limits on two out of three samples. Respirable dust levels were all below regulatory limits. Because these were all samples from the exterior of closed cab equipment, exposures to employees would have been lower. The ratio of total dust to respirable dust levels indicated that most of the dust was not respirable.

The dust levels on samples collected from rice burning operations were lower than from harvest and field preparation and were well below regulatory limits. There were too few samples to draw other meaningful relationships. The single pair of total dust and respirable dust samples taken downwind of a rice burn suggested that most of the particulate in the smoke was in the respirable range. However, more samples would be needed to confirm this.

An airborne crystalline silica concentration was calculated for all respirable dust samples. The samples were all positive for quartz and negative for tridymite and cristobalite. None of the samples exceeded the 0.1 mg/m^3 Cal/OSHA limit for respirable quartz exposure. Average levels were 0.09 mg/m^3 for plowing and for burning by tractor. The relatively low percentage of quartz observed in the samples is consistent with the clay soil type typical to rice farming. Clay soils have a very low quartz content. Note that these samples were from the exterior of the vehicles. Tractors used for plowing were all closed cab and employee exposures would be lower. Average levels for burning by foot, downwind of a burn and from the harvester exterior were 0.02-0.03 mg/m^3. The lower levels of quartz exposure reflect the lower levels of respirable dust exposure seen in these operations. The percent quartz seen from the single burning by tractor sample was the highest seen for all operations. Because there was only one sample the meaning of this result was unclear.

VII. Carbon monoxide

Carbon monoxide levels were measured as a general indicator of exposure to smoke and combustion by products in the air during rice burn activities. All but one were personal exposure samples taken in the breathing zone of

employees setting fire to rice straw in the fields. The results confirmed that the employees had very little exposure to the smoke plume. All results were below the detection limit, ranging from <25 ppm CO to <50 ppm CO for the duration of exposure which ranged from one to three hours, and 8 hour time weighted average exposure would be even lower. Carbon monoxide exposures seen in wildland firefighters average about 14.4 ppm [42] and would probably be higher than in employees burning rice straw and stubble. These exposures may be compared to exposures in structural firefighters, which have been measured in excess of 1500 ppm [43].

VIII. Implications of results and findings

The direct method of analysis developed in this project can serve as a standard against which other methods may be tested. Because of the limited manipulation of the sample in the direct method, the sample is unlikely to suffer from fiber loss or alteration. Limitations of the direct method, such as overloading with extraneous debris, suggest that efforts should be made to develop the indirect method of analysis.

These results indicate that a complete inventory of silica fiber emissions must include emissions from harvest and field preparation. Some work has been done to characterize fiber emissions from burning, but we are not aware of any work to characterize fiber emissions from harvest or field preparation. The ability of burning to spread particulate widely is obvious. It is not clear how widely particulate from other operations would disperse.

Results of this study indicate that many occupations may involve exposure to silica fibers, as we have documented emissions from harvest and field preparation activities, as well as from burning of agricultural waste. Farming and processing operations involving any of the materials reported to contain biogenic silica could result in exposure. Exposures seen from occupational samples were much higher than area samples taken in towns. Samples taken in towns in this study showed a relatively low level of amorphous silica fibers, and suggest that the potential hazard of occupational exposures may be more significant.

Peak airborne fiber levels documented in this study were 22.9 X 10^6 fibers/m^3, and, for fibers greater than 5 μm in length, 9.9 X 10^6 fibers/m^3, far in excess of airborne fiber silica fiber levels documented in previous studies of amorphous silica exposure. Until more is known about the health effects of amorphous silica fibers the most conservative approach is to consider them as hazardous as asbestos fibers. The current Cal/OSHA occupational exposure limit for asbestos is 0.200 X 10^6 fibers/m^3 in air. This suggests that an investigation of the health effects of silica fibers will be an important follow-up measure. A more complete investigation of occupational and public exposure may also be desirable.

SECTION 2: SUMMARY AND CONCLUSIONS

Recently, attention has focused on exposure to respirable amorphous silica contained in aerosols generated from burning of rice straw and stubble and other agricultural wastes. There are no established methods for the sampling and analysis of silica fibers. The work thus far has employed ad hoc methods, drawing on sampling and analytical methods used in the analysis of other types of particles. It is clear, however, that the proper assessment of the possible health hazard associated with the rice smoke particles requires validated methods of analysis.

We have developed a laboratory method which we call the "direct method" for the analysis of silica fibers on filters from air samples. In the direct method a section of the filter is analyzed by transmission electron microscopy. Nuclepore (track etch polycarbonate) filters with 0.4 μm pore size are the medium of choice. Fibers are identified by morphology and by their characteristic X-ray spectrum. Electron diffraction is used to determine crystallinity. This method is based on established methods of asbestos fiber analysis. Other fibers may be visualized by this method, but identification of a fiber by this method is only possible if the fiber has a unique combination of morphology, x-ray spectrum and crystallinity. As the direct method involves little manipulation of the sample, it is unlikely to produce artifacts or to alter the appearance of the particles.

The method produced useful results, and it is the only tested method available for amorphous silica fibers. The major limitation was the tendency of soil, fungi and other particles not of interest to obscure the fibers. To overcome this, most samples were collected with a size selective sampling head and very low sample volumes, often 30 liters or less, were used. The existence of respirable amorphous silica fibers in ambient air was confirmed, and environmental release of the fibers by rice harvest, rice straw and stubble burning and field preparation after burning was demonstrated. Fibers were irregularly shaped, often having no parallel sides, and did not appear to be hair-like or needle-like. Aspect ratios seldom exceeded 20:1 and aspect ratios in the range of 5:1 were more common. Personal exposure air samples were collected from employees performing rice farming operations, and area air samples were collected upwind and downwind of those operations and from communities in the rice farming area. The small number of samples taken and the nature of the samples did not permit full characterization of employee or public exposure to airborne silica fibers, nor did it permit calculations of emissions.

Fibers which were composed solely of silicon and oxygen were always found to be amorphous and appeared similar to fibers found in reference material prepared from ashed rice straw. Fibers which contained metals, such as aluminum or magnesium, were seen only in field samples, not in the reference material, and were assumed to be soil particles, such as silicates. This project focused on the particles which did not contain metal ions. No analysis was made of the results for fibers which contained metal ions.

A small number of individual samples were reanalyzed twice each by two microscopists. These data show that the coefficient of variation of the

measured concentrations was of the order of 25 percent for a given microscopist. But there was a systematic bias factor between the two microscopists, with one microscopist finding about 3.66 (1.9 for fibers greater than 5 μm in length) times as many fibers as the second microscopist. This result was not unexpected. In the similar case of the analysis of asbestos fibers, microscopists learn to adjust their discrimination of fibers on the basis of repeated intercomparison tests. Rice fibers are more irregular than asbestos fibers, seldom displaying simple parallel-side morphology. With more experience, the bias between microscopists could no doubt be reduced. At this time, the method should be considered provisional, pending more complete validation.

A limited number of duplicates and replicates, samples collected side-by-side, were submitted for analysis so that the microscopists were blinded to their identity. The coefficient of variation for these analyses averaged 24% (18% for fibers greater than 5 μm in length).

The method was used to analyze approximately 100 field samples. About half of the samples were taken during field burning of rice straw and stubble. The remainder included samples from rice harvesting, plowing of rice fields after burning, locations at the field edge, and in communities in rice farming regions. The personal exposure samples taken for this study, and some of the upwind samples, were collected with a respirable dust size selective sampling head, not a PM10 sampling head. The respirable size fraction, as defined by the American Conference of Industrial Hygienists is the rough equivalent of a fraction of all particles with an aerodynamic diameter of 5 μm, and is described by a cumulative lognormal function with a median aerodynamic diameter of 3.5 μm (plus or minus 0.3 μm) and with a geometric standard deviation of 1.5 (plus or minus 0.1). The results summarized in this study focus only on the fibers which contained amorphous silica and no other metallic elements, but the method does not distinguish between particles which are composed exclusively of amorphous silica and particles which contain amorphous silica and carbon or other traces of plant material.

The number and distribution of samples was primarily based on a desire to provide a useful sample pool for development and testing of the analytical method. A secondary goal was to characterize emissions from rice farming operations by measuring exposures to employees, as well as ambient levels in towns located in the rice farming region. Sample locations were not random. Locations were selected based on the availability of rice producers who volunteered to cooperate. Because of the limited number of samples that could be collected within the scope of the project, emphasis was placed on occupational exposure samples. Occupational exposure samples reliably provided useable sample loading, which was critical for method development. Also occupational exposure samples gave some indications of fiber levels that occur in plumes emitted by rice farming operations.

Results of upwind and community samples show a very low background level of airborne silica fibers, probably less than 9,000 fibers/m^3, or 5,000 fibers/m^3 for fibers greater than 5 μm in length. Samples taken in rice fields during harvest, burning of straw and stubble, field preparation after burning and downwind of those operations all show elevated airborne silica fiber levels.

The highest levels observed were samples from field preparation in fields which had been burned. The geometric mean of field preparation samples was 8.43×10^6 fibers/m^3, and 4.06×10^6 fibers/m^3 for fibers greater than 5 μm in length. These results exceed the highest level of airborne silica fibers recorded in previous studies which was 0.300×10^6 fibers/m^3 seen in sugar cane workers [25]. Results of air sampling are summarized below in Table 4, which is a duplicate of Table 1 found in Section 1.c.

Results must be interpreted with caution because of the limited number of samples and because of the uncertain accuracy of many of the results where the airborne concentration was very low. The number of samples per exposure type ranged from a high of eighteen for personal exposure to employees on foot burning rice stubble, to a low of two from inside the cab of a tractor during field preparation. In some of the analyses only a small number of fibers were detected, making interpretation of results difficult. For example, in town samples taken in the rice farming region of burn days no fibers were found on ten of fourteen samples.

We have shown that silica fibers are emitted by rice harvest and field preparation as well by burning. Some work has previously been done to quantify fiber emissions from rice straw and stubble burning, but there have been no estimates or measurements of fiber emissions from harvest or field preparation prior to the work reported here. Although the silica content of the rice plant is unusually high silica has been detected in many other crops, plants and trees. Farming and processing operations and natural processes such as weather and wildland fires involving these crops or plants could all lead to respirable amorphous silica fiber emissions. There have been no estimates of biogenic silica fiber emissions from other sources in California that we are aware of.

Several recommendations are made for additional work. Respirable amorphous silica fibers should be tested in vitro and in vivo to better characterize their health effects. Methods which simplify, speed up and reduce the cost of silica fiber sampling and analysis should be explored, and more work should be done to validate and characterize the reliability of the method developed by this project. Respirable amorphous silica fiber emissions estimates should be made for rice harvest and field preparation and for other farming and natural sources of silica fibers.

Table 4. Summary of Amorphous Silica Fiber Sampling.

ACTIVITY Location	No. of Samples Analyzed	No. of Samples w/fibers	Total Fibers/m^3 x 10^6		Fibers >5μm/m^3 x 10^6	
			Geo. Mean[3]	Range	Geo. Mean[3]	Range
RICE HARVEST						
Harvester (exterior)[1]	10	10	4.16	0.85-9.10	1.04	0.14-2.70
Harvester (interior)[1]	4	4	0.90	0.19-1.70	0.12	n.d.-0.23
Bank Out Wagon[1]	3	3	0.81	0.27-1.96	0.20	0.09-0.42
BURNING						
By Foot[1]	18	6	0.22	n.d.-0.89	0.21	n.d.-0.53
Tractor[1]	11	11	2.29	0.17-7.40	1.74	n.d.-4.90
Down Wind	6	3	0.01	n.d.-0.02	0.01	n.d.-0.01
Town	14	7	0.01	n.d.-0.03	0.01	n.d.-0.01
FIELD PREPARATION AFTER BURNING						
Tractor/Bulldozer[1]	7	6	8.44	n.d.-23.0	4.06	n.d.-9.90
Tractor (interior)[1]	2	2	2.35	1.15-4.80	0.50	0.13-1.90
ALL ACTIVITIES						
Up Wind[2]	11	8	0.01	n.d.-0.02	0.02	n.d.-0.02

1 Samples collected using respirable (<5 μm aerodynamic diameter) not PM10 particle sizing device.
2 Samples collected using respirable (<5 μm aerodynamic diameter) not PM10 particle sizing device.
3 Calculated from non-zero values only
n.d. None detected
Note: This table is a duplicate of Table 1 which appears in Section 1.c.

SECTION 3: RECOMMENDATIONS

Additional work is needed in the following areas:

a. Characterization of biological effects of amorphous silica fibers
b. Sampling and analytical method development
c. Emissions characterization
d. Exposure evaluation

a. CHARACTERIZATION OF BIOLOGICAL EFFECTS OF AMORPHOUS SILICA FIBERS

Work should be done to more fully characterize the toxicology of amorphous silica fibers. Cell culture studies or animal exposure studies could be particularly helpful. Detailed study of clearance mechanism and efficiency and the effect of respirable amorphous silica fibers in study animals in vivo would also be very helpful. Development of a method to purify gram qualities of silica fibers will be very useful in these studies.

b. SAMPLING AND ANALYTICAL METHOD DEVELOPMENT

I. Interlaboratory comparison

The next logical step in the validation of the direct method is to carry out interlaboratory comparisons. This would test the practicality of the method for general usage. It would also provide more definitive data on the accuracy of the method.

II. Fiber count standardization

A procedure for standardizing counts should be developed. The procedure should be similar to that used for standardizing asbestos fiber counts, known as Proficiency Analytical Testing (PAT). Fiber analysis results tend to vary widely between qualified microscopists. A count standardization procedure ensures comparability of results between analysts. In the PAT program for asbestos standard slides are prepared and analyzed by a panel of microscopists. Their results are used as a standard against which the results of other analysts may be compared.

III. Development and validation of the "indirect" method

An "indirect" method of analysis should be developed and compared to the "direct" method developed in this project. The indirect method involves the inclusion of a step in which the sample is washed off the original filter and then redeposited onto a Nuclepore (track etch polycarbonate) filter for analysis. Nuclepore filters or mixed cellulose ester filters could be used as the first filter.

Use of the indirect method would permit higher loading of samples from the field, which would result in an improved limit of detection over the direct method. During the transfer of the particles to Nuclepore filters for microscopy, the loading can be adjusted to an optimum level. Also, soluble background material can be removed. This would be a major advantage for the analysis of wind tunnel samples, which contain high loadings of potassium chloride particles. A further possibility is the use of low temperature ashing, which would remove background organic particles. Ashing could be performed with an oxygen plasma temperature of less than 100 degrees Celsius, and would not lead to conversion of amorphous silica to a crystalline form.

The indirect method could not be used without testing to see whether the particles are altered, i.e., broken up or agglomerated during sample preparation. or whether particles are lost in the sample preparation. Nuclepore filters are not ideally suited as a first filter, as heavy sample loads tend to fall off the filter surface. Mixed cellulose ester filters may trap fibers in the filter matrix, making the low temperature ashing an essential step.

During the present project, some parallel samples were taken with cellulose ester filters, but were not analyzed due to time and budget constraints. These could be analyzed by the indirect method for comparison to the results obtained with the direct method. Use of these samples would save considerable effort.

IV. The Phase Contrast Microscopy (PCM) method of analysis

Phase contrast microscopy (PCM) analysis of samples should be attempted and results compared to those from the "direct" method. The PCM method, currently in use for analysis of airborne asbestos, offers the possibility of inexpensive and rapid analysis of samples. The PCM method, however, is not able to distinguish the chemical nature of particles. In this method samples are collected on cellulose ester filters. Samples are processed by placing a small slice of the filter on a microscope slide and adding a few drops of solvent to solubilize the filter. Particles collected on the filter are then sized and counted using phase contrast microscopy.

c. EMISSIONS CHARACTERIZATION

With modeling and additional sampling it may be possible to develop emissions factors (ie. amount of airborne substance emitted per acre burned)for harvest and field preparation activities. Further work should be done to quantify emissions of amorphous silica fibers from burning of rice straw and other agricultural and crop processing operations. Development of indirect methods of analysis will aid wind tunnel studies which have been hampered so far by potassium chloride contamination which obscures the samples.

d. EXPOSURE EVALUATION

As amorphous silica fiber exposures does not appear to depend on the presence of burned plant material further work should be done to identify the fiber emissions from other farm operations, grain handling and other processing operations. Wildland fires and other crop and vegetation burning should also

be evaluated as sources of respirable amorphous silica emissions. Area and personal exposure evaluation have proven to be efficient means to identify sources of respirable amorphous silica fiber emissions.

SECTION 4: GLOSSARY OF TERMS, ABBREVIATIONS AND SYMBOLS

AIHL: Air and Industrial Hygiene Laboratory
amorphous: without a definite structure; non-crystalline
biogenic silica: silica absorbed from the soil incorporated in the structure of the plant
Cal/OSHA California Department of Industrial Relations - Division of Occupational Safety and Health
cyclone a dust-collecting device which has the ability to separate respirable from non-respirable size particles
cowl an extension of the inlet of the air sampling cassette
direct method of analysis the method of used in this project for analysis of biogenic silica air samples. This a transmission electron microscopy analysis of samples collected on track etch polycarbonate filters. The method is described in detail in appendix A to this report.
FEV_1 forced expiratory volume in one second
fiber a particle which is at least three times as long as it is wide
fibers/m^3 fibers per cubic meter of air
FVC forced vital capacity
indirect method of analysis this is a method of analysis for biogenic silica fiber air samples. In this method the sample is washed of the collection filter and redeposited on a second filter before analysis by transmission electron microscopy
km kilometer
kV killivolt
l/min liters per minute
mA milliampere
mg/m^3 milligram per meter cubed
mm millimeter
NIOSH National Institute for Occupational Safety and Health
Nuclepore filters track etch polycarbonate filters
PAHs polycyclic aromatic hydrocarbons
PCM phase contrast microscopy
PMR proportionate mortality ratio
ppm parts per million
PVC polyvinyl chloride
respirable able to penetrate deep into the lungs upon inhalation; describes particles with a median aerodynamic diameter of 3.5 μm and geometric standard deviation of 1.5 μm
rotameter: a flow meter, consisting of a precision-bored, tapered, transparent tube with a solid float inside
SEM scanning electron microscopy
TEM transmission electron microscopy
μm micrometer
XRD x-ray diffraction

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APPENDIX A:

Summary of a Provisional Analytical Method For the Determination of Biogenic Silica Fibers Emitted During Harvesting and Open Field Burning of the Rice Plant

4 August 1992

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Summary

An analytical method has been developed and tested using transmission electron microscopy to identify and count biogenic silica fibers on air-filter samples taken during routine agricultural processing of the rice plant in the Sacramento Valley.

(1) Air samples were taken during typical rice harvest operations. These operations involve the use of three large agricultural machines: a sophisticated combine for separating the rice (with hull) from the mature, dry rice plants, a bank out tractor for both emptying the combine and transporting the rice (with hulls) to the third vehicle, a truck at the edge of the field.

(2) Air samples were taken during regulated open burnings of rice straw. Rice straw is air-dried, unharvested plant material and includes mainly stems, leaves and panicles (branch-like structures to which the rice hulls were attached before harvesting).

(3) Air samples were taken during field preparation in fields which contained ash residue from previous burns.

The AIHL method is adapted from standard analytical techniques used for identifying asbestos fibers in ambient air samples:

Methodology for the Measurement of Airborne Asbestos by Electron Microscopy, G. Yamate, S. Agarwal, and R. Gibbons, E.P.A. Contract No. 68-02-3266 (1984).

Revised Draft Interim Superfund Method for the Determination of Asbestos in Ambient Air, E. Chatfield, D.W. Berman, E.P.A. Contract No. 68-01-7290, Task 117 (1989).

The scheme used to develop our method is outlined below.

A. Preparation of plastic sampling filters for TEM.

B. Preparation and determination of the elemental composition of TEM rice-ash reference samples.

C. Visual selection of fibers observed by TEM on a known area of filter surface.

D. Determination of fiber composition by identification of X-ray fluorescence peaks.

E. Examination of fiber by electron diffraction to distinguish amorphous and crystalline structures.

F. Classification of fibers based on morphology and a comparison of fluorescence spectra with spectra of the reference samples.

G. Count of the number of fibers that resemble the reference samples in composition and morphology.

H. Calculation of biogenic silica fiber concentration in air (fibers per cc air).

I. Sample recounting to check reproducibility of results.

METHOD DETAILS

A. Sample Preparation

TEM sample preparation of plastic filters was done by the method described in Yamate, et al., 1984. We concentrated our efforts on the **Direct Transfer Technique** and we used 0.4-micron Nuclepore or Poretics (polycarbonate) filters throughout the study.

The advantage of the Direct Transfer preparation scheme is that the filter is not subjected to severe chemical or physical treatments, minimizing particle alteration and loss. The resulting electron images are easy to interpret since preparation artifacts are minor.

The disadvantage of the Direct scheme is that filters can easily become overloaded with particulates. This occurs, for example, when the sampling unit is placed directly into the smoke plume. To use the Direct scheme, one is forced to reduce the volume of air pumped through the filter. This results in poor counting statistics and rather high detection limits. Nevertheless, we were able to achieve high fiber densities with low air volumes under favorable conditions.

An **Indirect Transfer Technique** is suggested by Yamate, et al. and by Chatfield and Berman for use with heavily loaded Millipore (cellulose ester) filters. This method is susceptible to contamination and requires careful study of its reliability. We have already taken over 50 samples on Millipore filters in parallel with many of the Nuclepore filters examined by the Direct technique. They are being stored until we complete analysis of all Nuclepore samples.

B. Reference Samples

Our identification of biogenic silica fibers found on harvest and burn samples is based partly on a comparison of the elemental composition of unknown particles with particles of known rice-ash origins. Rice char was obtained from Dr. Bryan Jenkins, Associate Professor of Agricultural Engineering at University of California in Davis. The sample was collected from the ash bin after rice straw was burned in a large scale wind tunnel.

Ten milligrams of the rice char ash was pulverized by mortar and pestle. 100 ml of 0.1-micron filtered water and 1 ml of 0.1% DT surfactant were added to the ash particles. The solution was sonicated for 15 minutes and 5 ml was placed into a filter funnel apparatus. A 47 mm, 0.4-micron Nuclepore filter was used to collect the particles from the water.

The **Direct Transfer Technique** was used to prepare the filter after it was air dried in a laminar flow hood. The loaded filter was carbon coated. Five pieces of the coated filter were placed carbon side up on 200-mesh copper TEM grids. The grids were then placed in a Jaffe Solvent Washer with chloroform for 72 hours.

The girds were examined by TEM operating at 75 kV. X-ray fluorescence spectra of particles and fibers were acquired with a Quantex (thin-window) X-ray detector and a Kevex Delta Class Analyzer. All spectra were normalized to the highest peak and relative peak heights for all identified elements were recorded.

Elemental Composition of Rice Ash Reference Samples

The composition of rice-char ash particles was simple: Silicon and oxygen. A small number of particles also contained traces of magnesium and/or potassium.

C. TEM analysis of Samples

Ten grid openings are selected at random and scanned visually at a magnification of 35,000X on a Hitachi H-600 Scanning Transmission Electron Microscope.

We define a **fiber** to be a structure having an aspect ratio of 3:1 or greater.

The kind of fibers observed on harvest and burn samples do not have parallel sides, like asbestos or mineral fibers. They are frequently "twig-like" or display other irregular features.

Most of the fibers observed do not have a fixed width. We arbitrarily chose to measure the width at the midpoint of the structure's length.

D. Fiber Composition & Structure

Once a fiber is selected, the analytical TEM was switched to STEM/SEM scanning mode. In this mode, the surface features of the fiber can be observed.

Then an X-ray fluorescence spectrum is acquired for 20 seconds. Only fibers with dominant **silicon** peaks are recorded on data sheets.

Two classes of **silicon fibers** emerged from this data:

Silica Fibers: those that demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

Aluminum-Silica Fibers: those that demonstrate both an aluminum and a silicon X-ray fluorescence peak.

Each fiber is also investigated by electron diffraction using a microdiffraction technique to determine amorphous (no bright reflections) or crystalline (bright spots observed in electron diffraction pattern) structure. Thick particles were examined along their edges, where the electron beam was more likely to penetrate the sample.

Another pattern emerged from our data: only the Aluminum-Silica fibers were crystalline.

E. Classification of Fibers

The crystalline, Aluminum-Silica Fibers are believed to be of geological mineral origin (soil). These fibers are simply airborne particles of soil that happen to fit our definition of a fiber (3:1 or greater).

Aluminum is **never** observed in the reference samples.

The Silica Fibers are believed to originate from the rice plant.

> See Williams, R.J.P., "General Introduction to Silicon Biochemistry," Silicon Biochemistry, John Wiley & Sons (United Kingdom), 1986, page 1.

Our method can distinguish between SiO_2 and Mg, Al, & SiO_2 (believed to be plant soil), but cannot distinguish between burned versus unburned plant material.

F. Fiber Counts & Calculations

Four numbers are determined for each sample:

(1) Number of Silica Fibers less than 5 microns in length.

(2) Number of Silica Fibers equal to or greater than 5 microns in length.

(3) Number of Al-Silica Fibers less than 5 microns in length.

(4) Number of Al-Silica Fibers equal to or greater than 5 microns in length.

The area of grid square openings is determined by light microscopy and image analysis for each vial (100) of 200-mesh TEM grids.

This allows us to calculate a fiber concentration (per cc air) from our measurements of fiber density (per square mm filter surface). These calculations are identical to similar calculations of asbestos fiber densities from the standard asbestos methods mentioned earlier.

G. Sample Recounts

Selected samples (11% of the counted samples) are recounted by the original observer and then these are recounted twice by a second observer.

APPENDIX B: AIHL ANALYTICAL REPORTS

AIHL Lab. Number: 10463

AIHL Analytical Report

Microscopy Unit

Name of Submitter: *Bryan M. Jenkins, Ph.D.* *Samples Received: 12/17/91*
Submitting Agency: *University of California*
Agricultural Engineering Dept.
Davis, CA 95616 *Total Samples Analyzed: 15*

Sampling Location: *UCD Rice straw field burn: Community Samples* *Date Reported* ___/___/___

Sample Description: **Ambient Air Filter Samples**

Analysis Requested: **Fibrous Silica (per cc Air)**

Method: **AIHL Provisional TEM Method for Analysis of Fibrous Silica***

Instruments: **Hitachi H-600/H-6010A Scanning Transmission Electron Microscope**
Kevex Delta Class Analyzer
JEOL JSM-35C Scanning Electron Microscope

Notes

* - At this time there is no officially adopted analytical method for determining silica fiber concentrations emitted from the burning of biomass fuels. The AIHL provisional method is based on standard techniques used for other sample types.

Details of the **AIHL Provisional TEM Method for Analysis of Fibrous Silica** are available upon request.

Signatures of Analysts *Signature of Supervising Analyst*

> Donald J. Seale 4.28.92 > [signature] 4/28/92

> John C. Schmidt 4-28-92

AIHL Lab. Number: 10463

Analytical Summary

Field Number	Silica Fibers per cc Air <5 microns	Silica Fibers per cc Air >=5 microns	Al-Silica Fibers per cc Air <5 microns	Al-Silica Fibers per cc Air >=5 microns
UCRRB104	0.00	0.00	0.00	0.00
UCRRB105	0.00	0.00	0.004	0.00
UCRRB106	0.00	0.00	0.005	0.005
UCRRB107	0.00	0.00	0.00	0.00
UCRRB108	0.00	0.00	0.00	0.00
UCRRB109	0.00	0.00	0.009	0.00
UCRRB110	0.005	0.005	0.005	0.014
UCRRB111	0.015	0.010	0.010	0.034
UCRRB112	Blank			
UCRRB113	0.00	0.005	0.005	0.005
UCRRB114	0.004	0.00	0.004	0.00
UCRRB115	0.009	0.00	0.00	0.00
UCRRB116	0.00	0.009	0.00	0.028
UCRRB117	0.004	0.00	0.004	0.008
UCRRB118	0.00	0.00	0.005	0.00

*Formula for calculating the **Minimum Detection Limit (MDL)** in fibers per cc air:*

MDL = 1 fiber x Effective Filter Area/[1000 x Grid Area Scanned x Sample Volume],

where Sample Volume is given in liters

*Formula for calculating **Fiber Concentration (C)** in fibers per cc air:*

C = (Number of fibers) x (MDL)

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB104: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	2918 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.004 fibers/cc
Grid Openings Scanned	3	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.031	Operator	D. Scales & J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Silica*		Silica Fibers/cc	95% Confidence Range
length	number		
<5um	0	0.00	0.00 to 0.016 fibers/cc
>=5um	0	0.00	0.00 to 0.016 fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range
length	number		
<5um	0	0.00	0.00 to 0.016 fibers/cc
>=5um	0	0.00	0.00 to 0.016 fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB105: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	2822 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.004 fibers/cc
Grid Openings Scanned	3	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.031	Operator	D. Scales & J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	0	**0.00**	0.00	to 0.016	fibers/cc
>=5um	0	**0.00**	0.00	to 0.016	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	1	**0.004**	0.00	to 0.024	fibers/cc
>=5um	0	**0.00**	0.00	to 0.016	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB106: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate. 0.4um	Sample Volume	2696 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.005 fibers/cc
Grid Openings Scanned	03	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.031	Operator	D. Scales

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	0	**0.00**	0.00	to 0.017	fibers/cc
>=5um	0	**0.00**	0.00	to 0.017	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica†		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	1	**0.005**	0.00	to 0.025	fibers/cc
>=5um	1	**0.005**	0.00	to 0.025	fibers/cc

† - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB107: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	2788 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.004 fibers/cc
Grid Openings Scanned	3	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.031	Operator	D. Scales

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica* length	number	Silica Fibers/cc	95% Confidence Range		
<5um	0	**0.00**	0.00	to 0.016	fibers/cc
>=5um	0	**0.00**	0.00	to 0.016	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+ length	number	Al-Silica Fibers/cc	95% Confidence Range		
<5um	0	**0.00**	0.00	to 0.016	fibers/cc
>=5um	0	**0.00**	0.00	to 0.016	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB108: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	2525 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.005 fibers/cc
Grid Openings Scanned	03	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.031	Operator	D. Scales & J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	0	0.00	0.00	to 0.018	fibers/cc
>=5um	0	0.00	0.00	to 0.018	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	0	0.00	0.00	to 0.018	fibers/cc
>=5um	0	0.00	0.00	to 0.018	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB109: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	2747 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.004 fibers/cc
Grid Openings Scanned	3	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.031	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Silica*		Silica Fibers/cc	95% Confidence Range
length	number		
<5um	0	**0.00**	0.00 to 0.017 fibers/cc
>=5um	0	**0.00**	0.00 to 0.017 fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Al-Silica†		Al-Silica Fibers/cc	95% Confidence Range
length	number		
<5um	2	**0.009**	0.001 to 0.032 fibers/cc
>=5um	0	**0.00**	0.00 to 0.017 fibers/cc

† - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB110: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	2684 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.005 fibers/cc
Grid Openings Scanned	3	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.031	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)		
Silica*		Silica Fibers/cc	95% Confidence Range	
length	number			
<5um	1	**0.005**	0.00 to 0.026	fibers/cc
>=5um	1	**0.005**	0.00 to 0.026	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)		
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range	
length	number			
<5um	1	**0.005**	0.00 to 0.026	fibers/cc
>=5um	3	**0.014**	0.003 to 0.040	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB111: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	2515 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.005 fibers/cc
Grid Openings Scanned	3	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.031	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Silica*		Silica Fibers/cc	95% Confidence Range
length	number		
<5um	3	**0.015**	0.003 to 0.043 fibers/cc
>=5um	2	**0.010**	0.001 to 0.035 fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range
length	number		
<5um	2	**0.010**	0.001 to 0.035 fibers/cc
>=5um	7	**0.034**	0.014 to 0.071 fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB112: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	0 liters
Diameter (mm)	25		Blank
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	******* fibers/cc
Grid Openings Scanned	3	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.031	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Silica*		Silica Fibers/cc	95% Confidence Range
length	number		
<5um	0	****	**********
>=5um	0	****	**********

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range
length	number		
<5um	0	****	**********
>=5um	0	****	**********

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB113: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	2550 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.005 fibers/cc
Grid Openings Scanned	3	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.031	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)		
Silica*		Silica Fibers/cc	95% Confidence Range	
length	number			
<5um	0	0.00	0.00 to 0.018	fibers/cc
>=5um	1	0.005	0.00 to 0.027	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)		
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range	
length	number			
<5um	1	0.005	0.00 to 0.027	fibers/cc
>=5um	1	0.005	0.00 to 0.027	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB114: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	2508 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.004 fibers/cc
Grid Openings Scanned	3	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.035	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	1	**0.004**	0.00	to 0.024	fibers/cc
>=5um	0	**0.00**	0.00	to 0.016	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	1	**0.004**	0.00	to 0.024	fibers/cc
>=5um	0	**0.00**	0.00	to 0.016	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB115: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	2438 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.004 fibers/cc
Grid Openings Scanned	4	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.035	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Silica*		Silica Fibers/cc	95% Confidence Range
length	number		
<5um	2	**0.009**	0.001 to 0.032 fibers/cc
>=5um	0	**0.00**	0.00 to 0.016 fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range
length	number		
<5um	0	**0.00**	0.00 to 0.016 fibers/cc
>=5um	0	**0.00**	0.00 to 0.016 fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB116: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	2326 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.005 fibers/cc
Grid Openings Scanned	4	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.035	Operator	J. Schmidt & D. Scales

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Silica*		Silica Fibers/cc	95% Confidence Range
length	number		
<5um	0	**0.00**	0.00 to 0.017 fibers/cc
>=5um	2	**0.009**	0.001 to 0.034 fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range
length	number		
<5um	0	**0.00**	0.00 to 0.017 fibers/cc
>=5um	6	**0.028**	0.010 to 0.061 fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB117: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	3743 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.004 fibers/cc
Grid Openings Scanned	3	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.026	Operator	D. Scales

IDENTIFIED FIBERS — **CALCULATED FIBER CONCENTRATION (PER CC AIR)**

Silica* length	number	Silica Fibers/cc	95% Confidence Range
<5um	1	**0.004**	0.00 to 0.022 fibers/cc
>=5um	0	**0.00**	0.00 to 0.014 fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS — **CALCULATED FIBER CONCENTRATION (PER CC AIR)**

Al-Silica† length	number	Al-Silica Fibers/cc	95% Confidence Range
<5um	1	**0.004**	0.00 to 0.022 fibers/cc
>=5um	2	**0.008**	0.001 to 0.028 fibers/cc

† - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10463
Field Number: UCRRB118: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	3183 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.005 fibers/cc
Grid Openings Scanned	3	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.026	Operator	D. Scales

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Silica*		Silica Fibers/cc	95% Confidence Range
length	number		
<5um	**0**	**0.00**	0.00 to 0.017 fibers/cc
>=5um	**0**	**0.00**	0.00 to 0.017 fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Al-Silica†		Al-Silica Fibers/cc	95% Confidence Range
length	number		
<5um	**1**	**0.005**	0.00 to 0.025 fibers/cc
>=5um	**0**	**0.00**	0.00 to 0.017 fibers/cc

† - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

State of California-Health and Welfare Agency

Department of Health Services

AIHL/ARB
LABORATORY SAMPLE ANALYSIS REQUEST

10463

DIVISION SUBMITTING SAMPLE(S): [ ]TSD [ ]CD [x]MLD [ ]OTHER _____

Date Submitted 08 19 91 (Mo Day Yr)

e of Submitter James E. McCormack

Phone No. 8-493-2289

Project Engineer Approval James E McCormack

CARB STUDY OR CONTROL NUMBER C-90-041

No. of Samples Submitted 51 ~~21-01~~

Date Lab Results Desired: 10 01 91 (Mo Day Yr)

Date Returned _____ (Mo. Day Yr.)

SAMPLE INFORMATION:
Name of Establishment: Rice straw field burn

PURPOSE: [ ] Emission/Compliance Testing
[x] ~~Special~~ Study [X] Method Development
[ ] Other: E205X

Address:

Lab No. For Lab Use	SAMPLE NO.	Type of sample Air	Type of sample Mat	Volume	FIELD SAMPLE DESCRIPTION	Analysis Required
10463	UCRRB80	X				SEE NOTE BELOW
	UCRRB81	X				"
	UCRRB82	X				" Silica
	UCRRB83	X				~~Asbestos~~
	UCRRB84	X			Cat 3Q	"
	UCRRB85	X				fiber conc
	UCRRB86	X				"
	UCRRB87	X				"
	UCRRB88	X				Cat 3Q
	UCRRB89	X				"
	UCRRB90	X				" DS.
	UCRRB91	X				"
	UCRRB92	X				"
	UCRRB93	X				"
	UCRRB94	X				"
	UCRRB95	X				"
	UCRRB96	X				"
	UCRRB97	X				"
	UCRRB98	X				"
	UCRRB99	X				"
	UCRRB100	X				"
	UCRRB101	X				"
	UCRRB102	X				"
	UCRRB103	X				"
	UCRRB104	X				"
	UCRRB105	X				"
	UCRRB106	X				"
	UCRRB107	X				"

COMMENTS: COUNT FIBERS ON TEN GRID OPENINGS ONLY. NO EXCEPTIONS !!!!!!!!
1. FOR ALL SAMPLES COUNT SILICA AND SILICATE FIBERS. ATTACH COUNT SHEETS
2. CALCULATE FIBER CONCENTRATION (FIBER/CU. METER)
3. NOTE IF KCL PARTICLES ARE PRESENT AND IF THE SAMPLE IS OVERLOADED WITH THEM. USE COUNT SHEET AS PREVIOUSLY USED IN PAST UCD SAMPLES.

LAB-N-607 (Rev 11/90)

Chain of Custody Form:

UC Davis/Rice Research Board/Air Resources Board

Sampling Program for Biogenic Silica Fiber Emission

Originating Form from UC Davis (Prof. B. Jenkins, 916-752-1422)

Sample ID	Date Sampled	Filter #	Cyclone	Flow (LPM)	Elapsed Hours	Total Flow (L)	Location	Collected By	Transferred To	Date Transferred
UCRRB80	10/4/91		yes	2	2.60	312.0	Harvesting	SQT/RBW		10-23-91
UCRRB81	10/4/91		no	2	2.60	312.0	Harvesting	SQT/RBW		
UCRRB82	10/4/91		yes	8	2.60	1248.0	Harvesting	SQT/RBW		
UCRRB83	10/4/91		no	8	2.60	1248.0	Harvesting	SQT/RBW		
UCRRB84	10/4/91	1	no	4	2.60	624.0	Harvesting	SQT/RBW		
UCRRB85	10/4/91	2	no	4	2.60	624.0	Harvesting	SQT/RBW		
UCRRB86	10/10/91		yes	2	2.44	292.8	Harvesting	SQT/RBW		
UCRRB87	10/10/91		no	2	2.44	292.8	Harvesting	SQT/RBW		
UCRRB88	10/10/91		yes	8	2.44	1171.2	Harvesting	SQT/RBW		
UCRRB89	10/10/91		no	8	2.44	1171.2	Harvesting	SQT/RBW		
UCRRB90	10/10/91	4	no	4	2.44	585.6	Harvesting	SQT/RBW		
UCRRB91	10/10/91	3	no	4	2.44	585.6	Harvesting	SQT/RBW		
UCRRB92	10/11/91		yes	2	3.00	360.0	Harvesting	SQT/RBW		
UCRRB93	10/11/91		no	2	3.00	360.0	Harvesting	SQT/RBW		
UCRRB94	10/11/91		yes	8	3.00	1440.0	Harvesting	SQT/RBW		
UCRRB95	10/11/91		no	8	3.00	1440.0	Harvesting	SQT/RBW		
UCRRB96	10/11/91	5	no	4	3.00	720.0	Harvesting	SQT/RBW		
UCRRB97	10/11/91	6	no	4	3.00	720.0	Harvesting	SQT/RBW		
UCRRB98	10/17/91		yes	2	2.61	313.2	Harvesting	SQT/RBW		
UCRRB99	10/17/91		no	2	2.61	313.2	Harvesting	SQT/RBW		
UCRRB100	10/17/91		yes	8	2.61	1252.8	Harvesting	SQT/RBW		
UCRRB101	10/17/91		no	8	2.61	1252.8	Harvesting	SQT/RBW		
UCRRB102	10/17/91	7	no	4	2.61	626.4	Harvesting	SQT/RBW		
UCRRB103	10/17/91	8	no	4	2.61	626.4	Harvesting	SQT/RBW		

10/24/91

Chain of Custody Form:

UC Davis/Rice Research Board/Air Resources Board

Sampling Program for Biogenic Silica Fiber Emission

Originating Form from UC Davis (Prof. B. Jenkins, 916-752-1422)

Sample ID	Date Sampled	Filter #	Cyclone	Flow (LPM)	Elapsed Hours	Total Flow (L)	Location	Collected By	Transferred To	Date Transferred
UCRRB104	10/22/91	none	no	8.4	5.79	2,918	E. Niclaus	BMJ/SQT		10-25-91
UCRRB105	10/22/91	none	no	8.4	5.60	2,822	Olivehurst	BMJ/SQT		
UCRRB106	10/22/91	none	no	8.4	5.35	2,696	Yuba City, Ext.	BMJ/SQT		
UCRRB107	10/22/91	none	no	8.8	5.28	2,788	Yuba City, JFW	BMJ/SQT		

10-24-91

Sample Submittal/Chain of Custody Form:

UC Davis/Rice Research Board/Air Resources Board

Sampling Program for Biogenic Silica Fiber Emission

Originating Form from UC Davis (Prof. B. Jenkins, 916-752-1422)

Sample ID	Date Sampled	Filter #	Cyclone	Flow (LPM)	Elapsed Hours	Total Flow (L)	Location	Collected By	Transferred To	Date Transferred
UCRRB108	10/23/91	none	no	8.25	5.1	2,525	E. Niclaus	SQT/CvM	John/Scalar	12/17/91
UCRRB109	10/23/91	none	no	9.25	4.95	2,747	Yuba City, Ext.	SQT/CvM		
UCRRB110	10/23/91	none	no	9	4.97	2,684	Colusa	SQT/CvM		
UCRRB111	10/23/91	none	no	8.4	4.99	2,515	Robbins	SQT/CvM		
UCRRB112	10/23/91	none	no	0	0	0	Blank	SQT/CvM		
UCRRB113	10/25/91	none	no	7.9	5.38	2,550	E.Niclaus	SQT/CvM		
UCRRB114	10/25/91	none	no	7.8	5.36	2,508	Olivehurst	SQT/CvM		
UCRRB115	10/25/91	none	no	7.8	5.21	2,438	Yuba City, Ext.	SQT/CvM		
UCRRB116	10/25/91	none	no	7.3	5.31	2,326	Yuba City, JFW	SQT/CvM		

Sample Submittal/Chain of Custody Form:
UC Davis/Rice Research Board/Air Resources Board
Sampling Program for Biogenic Silica Fiber Emission
Originating Form from UC Davis (Prof. B. Jenkins, 916-752-1422)

Sample ID	Date Sampled	Filter #	Cyclone	Flow (LPM)	Elapsed Hours	Total Flow (L)	Location	Collected By	Transferred To	Date Transferred
UCRRB117	10/28/91	none	no	8.75	7.13	3,743	Davis	BMJ/SQT	John/Scala	12/12/91
UCRRB118	10/28/91	none	no	8.9	5.96	3,183	Woodland	BMJ/SQT		
UCRRB119	10/28/91	none	no	8	3	1,440	Harvesting	BMJ/SQT		
UCRRB120	10/28/91	none	yes	2	3	360	Harvesting	BMJ/SQT		
UCRRB121	10/28/91	10	no	4	3	720	Harvesting	BMJ/SQT		

AIHL Analytical Report

Microscopy Unit

Name of Submitter: *David Goldsmith*
Submitting Agency: *Division of Occupational and Environmental Medicine University of California Davis, California 95616*
Sampling Location: *Rice Harvest Samples Hofert Brothers Farm Sutter County*

Samples Received: *9/25/91*

Total Samples Analyzed: *5*

Date Reported 12 / 9 / 91

Sample Description: **Ambient Air Filter Samples**

Analysis Requested: **Fibrous Silica (per cc Air)**

Method: **AIHL Provisional TEM Method for Analysis of Fibrous Silica***

Instruments: **Hitachi H-600/H-6010A Scanning Transmission Electron Microscope**
Kevex Delta Class Analyzer
JEOL JSM-35C Scanning Electron Microscope

Notes

* - At this time there is no officially adopted analytical method for determining silica fiber concentrations emitted from the burning of biomass fuels. The AIHL provisional method is based on standard techniques used for other sample types.

Details of the **AIHL Provisional TEM Method for Analysis of Fibrous Silica** are available upon request.

Signatures of Analysts

> Donald J. Scales 12-5-91

> John C. Schmidt 12-6-91

Signature of Supervising Analyst

> [signature] 12/6/91

Analytical Summary

Field Number	Silica Fibers per cc Air <5 microns	Silica Fibers per cc Air >=5 microns	Al-Silica Fibers per cc Air <5 microns	Al-Silica Fibers per cc Air >=5 microns
RH-1	No filter in cassette.			
RH-2	0.18	0.088	0.088	0.088
RH-3	0.81	0.22	0.056	0.084
RH-4	2.3	0.64	0.51	0.13
RH-5	Filter was overloaded. No analysis attempted.			
RH-6	1.7	0.0	0.48	0.12
RH-7	1.2	0.12	0.14	0.083

*Formula for calculating the **Minimum Detection Limit (MDL)** in fibers per cc air:*

MDL = 1 fiber x Effective Filter Area/[1000 x Grid Area Scanned x Sample Volume],

where Sample Volume is given in liters

*Formula for calculating **Fiber Concentration (C)** in fibers per cc air:*

C = (Number of fibers) x (MDL)

Sample Analysis

Project I.D.: UCD-001
Field Number: RH-2: **FIBER DATA**

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	48.1 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.088 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.09	Operator	D. Scales

IDENTIFIED FIBERS — **CALCULATED FIBER CONCENTRATION (PER CC AIR)**

Silica* length	number	Silica Fibers/cc	95% Confidence Range
<5um	2	**0.18**	0.021 to 0.64 fibers/cc
>=5um	1	**0.088**	0.002 to 0.49 fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS — **CALCULATED FIBER CONCENTRATION (PER CC AIR)**

Al-Silica+ length	number	Al-Silica Fibers/cc	95% Confidence Range
<5um	1	**0.088**	0.002 to 0.49 fibers/cc
>=5um	1	**0.088**	0.002 to 0.49 fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.
A = Amorphous fiber, C = Crystalline fiber

Sample Analysis

Project I.D.: UCD-001
Field Number: RH-3: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	152 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.028 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.09	Operator	D. Scales

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	29	**0.81**	0.51	to 1.1	fibers/cc
>=5um	8	**0.22**	0.096	to 0.44	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	2	**0.056**	0.007	to 0.20	fibers/cc
>=5um	3	**0.084**	0.017	to 0.24	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

Project I.D.: UCD-001
Field Number: RH-4:

FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	33.3 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.13 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.09	Operator	D. Scales

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	18	**2.3**	1.4	to 3.6	fibers/cc
>=5um	5	**0.64**	0.21	to 1.5	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	4	**0.51**	0.14	to 1.3	fibers/cc
>=5um	1	**0.13**	0.003	to 0.71	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

Project I.D.: UCD-001
Field Number: RH—5: **FIBER DATA**

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	240 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	XXXX fibers/cc
Grid Openings Scanned	0	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0	Operator	D. Scales

IDENTIFIED FIBERS — **CALCULATED FIBER CONCENTRATION (PER CC AIR)**

Silica*

length	number	Silica Fibers/cc	95% Confidence Range
<5um	**Filter was overloaded.**		
>=5um	**No analysis was attempted.**		

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS — **CALCULATED FIBER CONCENTRATION (PER CC AIR)**

Al-Silica+

length	number	Al-Silica Fibers/cc	95% Confidence Range
<5um			
>=5um			

+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

Project I.D.: UCD-001
Field Number: RH-6: **FIBER DATA**

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	35.2 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.12 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.09	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	14	**1.7**	0.92	to 2.8	fibers/cc
>=5um	**0**	**0.0**	0.00	to 0.44	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	**4**	**0.48**	0.13	to 1.2	fibers/cc
>=5um	1	**0.12**	0.003	to 0.67	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

Project I.D.: UCD-001
Field Number: RH-7: **FIBER DATA**

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	204 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.021 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.09	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	60	1.2	0.93	to 1.6	fibers/cc
>=5um	6	0.12	0.046	to 0.27	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	7	0.14	0.058	to 0.23	fibers/cc
>=5um	4	0.08	0.02	to 0.21	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample #		Time On	Time Off	Elapsed Time	flow On	Flow Off	Avg Flow	Volume
1		11:07 AM	11:17 AM	0:10	2	2	2.00	20.00
2		11:25 AM	11:51 AM	0:26	2	1.7	1.85	48.10
3		11:53 AM	1:15 PM	1:22	2	1.7	1.85	151.70
4		12:19 PM	12:37 PM	0:18	2	1.7	1.85	33.30
5		12:19 PM	2:29 PM	2:10	2	1.7	1.85	240.50
6		12:19 PM	12:38 PM	0:19	2	1.7	1.85	35.15
7		12:39 PM	2:29 PM	1:50	2	1.7	1.85	203.50
1001		9:52 AM	10:13 AM	0:21	1.7	1.53	1.62	33.92
1002		9:52 AM	10:13 AM	0:21	1.7	1.53	1.62	33.92
1003								
1004		9:52 AM	12:48 PM	2:56	1.7	1.61	1.66	291.28
1005		1:32 PM	3:56 PM	2:24	1.7	1.7	1.70	244.80
1006		10:14 AM	10:43 AM	0:29	1.53	1.53	1.53	44.37
1007		10:44 AM	11:11 AM	0:27	1.53	1.53	1.53	41.31

AIHL Analytical Report

Microscopy Unit

Name of Submitter: *Robert Lawson*
Submitting Agency: *Agricultural Health and Safety Center*
University of California
Davis, California 95616-8757
Sampling Location: *Rice Harvest Samples*
Rominger Farms
Highway 45

Samples Received: 10/04/91

Total Samples Analyzed: 6

Date Reported 01/17/92

Sample Description: **Ambient Air Filter Samples**

Analysis Requested: **Fibrous Silica (per cc Air)**

Method: **AIHL Provisional TEM Method for Analysis of Fibrous Silica***

Instruments: **Hitachi H-600/H-6010A Scanning Transmission Electron Microscope**
Kevex Delta Class Analyzer
JEOL JSM-35C Scanning Electron Microscope

Notes

* - At this time there is no officially adopted analytical method for determining silica fiber concentrations emitted from the burning of biomass fuels. The AIHL provisional method is based on standard techniques used for other sample types.

Details of the **AIHL Provisional TEM Method for Analysis of Fibrous Silica** are available upon request.

Signatures of Analysts

> Donald J. Scales 1-15-92

> John C. Schmidt 1-15-92

Signature of Supervising Analyst

> 1/16/92

Analytical Summary

Field Number	Silica Fibers per cc Air <5 microns	Silica Fibers per cc Air >=5 microns	Al-Silica Fibers per cc Air <5 microns	Al-Silica Fibers per cc Air >=5 microns
RH-1001	7.5	1.6	0.75	0.00
RH-1002	5.9	1.7	0.12	0.00
RH-1004	1.3	0.23	0.058	0.00
RH-1005	0.017	0.00	0.035	0.035
RH-1006	2.4	1.0	0.19	0.00
RH-1007	6.6	1.4	0.31	0.00

*Formula for calculating the **Minimum Detection Limit (MDL)** in fibers per cc air:*

MDL = 1 fiber x Effective Filter Area/[1000 x Grid Area Scanned x Sample Volume],

where Sample Volume is given in liters

*Formula for calculating **Fiber Concentration (C)** in fibers per cc air:*

C = (Number of fibers) x (MDL)

Sample Analysis

Project I.D.: UCD-002
Field Number: RH-1001: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	33.9 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.12 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.09	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	60	7.5	5.6	to 9.4	fibers/cc
>=5um	13	1.6	0.86	to 2.8	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	6	0.75	0.28	to 1.6	fibers/cc
>=5um	0	0.00	0.00	to 0.46	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.
A = Amorphous fiber, C = Crystalline fiber

Sample Analysis

Project I.D.: UCD-002
Field Number: RH-1002: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	33.9 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.12 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.09	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	47	5.9	4.2	to 7.5	fibers/cc
>=5um	14	1.7	0.96	to 2.9	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	1	0.12	0.003	to 0.70	fibers/cc
>=5um	0	0.00	0.00	to 0.46	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

Project I.D.: UCD-002
Field Number: RH-1004: **FIBER DATA**

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	291 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.015 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.09	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	89	1.3	1.0	to 1.6	fibers/cc
>=5um	16	0.23	0.13	to 0.38	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	4	0.058	0.016	to 0.15	fibers/cc
>=5um	0	0.00	0.00	to 0.054	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

Project I.D.: UCD-002
Field Number: RH-1005: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	245 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.017 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.09	Operator	J. Schmidt

IDENTIFIED FIBERS — **CALCULATED FIBER CONCENTRATION (PER CC AIR)**

Silica* length	number	Silica Fibers/cc	95% Confidence Range
<5um	1	**0.017**	0.00 to 0.096 fibers/cc
>=5um	0	**0.00**	0.00 to 0.064 fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS — **CALCULATED FIBER CONCENTRATION (PER CC AIR)**

Al-Silica+ length	number	Al-Silica Fibers/cc	95% Confidence Range
<5um	2	**0.035**	0.004 to 0.12 fibers/cc
>=5um	2	**0.035**	0.004 to 0.12 fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

Project I.D.: UCD-002
Field Number: RH-1006: **FIBER DATA**

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	44.4 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.095 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.09	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	25	**2.4**	1.4	to 3.3	fibers/cc
>=5um	11	**1.0**	0.52	to 1.9	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	**2**	**0.19**	0.023	to 0.69	fibers/cc
>=5um	**0**	**0.00**	0.00	to 0.35	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

Project I.D.: UCD-002
Field Number: RH-1007: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	41.3 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.10 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.09	Operator	J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	64	**6.6**	4.9	to 8.2	fibers/cc
>=5um	14	**1.4**	0.78	to 2.4	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	3	**0.31**	0.063	to 0.90	fibers/cc
>=5um	0	**0.00**	0.00	to 0.38	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Lab. No. 10529

Date Received 10/4/91

California State Department of Health
Air and Industrial Hygiene Laboratory
2151 Berkeley Way, Berkeley, CA 94704

LABORATORY SERVICE REQUEST

Plant or Place UCD-002 Rice Harvest Samples No. of Samples 6

Address Romingen Farms, Highway 45 Fund Code 205 N

Submitting Agency UCD Div. of Occ. & Env. Med Phone No.

Date Submitted 10/4/91

Send Analytical Report to Robert Lawson, UC Davis CA 95611

Field No.	Date Coll.	Type of Sample (Air, Material)	Volume	Field Information	Analysis Requested
RH-1001		Air	33.9		Silica
1002		"	33.9		Fiber count
1004		"	291		by TEM
1005		"	245		cat 35
1006		"	44.4		
1007		↓	41.3		

Description of Problems:

Sample #		Time On	Time Off	Elapsed Time	flow On	Flow Off	Avg Flow	Volume
1		11:07 AM	11:17 AM	0:10	2	2	2.00	20.00
2		11:25 AM	11:51 AM	0:26	2	1.7	1.85	48.10
3		11:53 AM	1:15 PM	1:22	2	1.7	1.85	151.70
4		12:19 PM	12:37 PM	0:18	2	1.7	1.85	33.30
5		12:19 PM	2:29 PM	2:10	2	1.7	1.85	240.50
6		12:19 PM	12:38 PM	0:19	2	1.7	1.85	35.15
7		12:39 PM	2:29 PM	1:50	2	1.7	1.85	203.50
1001		9:52 AM	10:13 AM	0:21	1.7	1.53	1.62	33.92
1002		9:52 AM	10:13 AM	0:21	1.7	1.53	1.62	33.92
1003								
1004		9:52 AM	12:48 PM	2:56	1.7	1.61	1.66	291.28
1005		1:32 PM	3:56 PM	2:24	1.7	1.7	1.70	244.80
1006		10:14 AM	10:43 AM	0:29	1.53	1.53	1.53	44.37
1007		10:44 AM	11:11 AM	0:27	1.53	1.53	1.53	41.31

AIHL Lab. Number: 10530

AIHL Analytical Report

Microscopy Unit

Name of Submitter: *Robert Lawson* *Samples Received: 11/16/91*
Submitting Agency: *Agricultural Health and Safety Center*
University of California
Davis, California 95616-8757 *Total Samples Analyzed: 4*

Sampling Location: *Rice Field Burn*

Date Reported 3/26/92

Sample Description: **Ambient Air Filter Samples**

Analysis Requested: **Fibrous Silica (per cc Air)**

Method: **AIHL Provisional TEM Method for Analysis of Fibrous Silica***

Instruments: **Hitachi H-600/H-6010A Scanning Transmission Electron Microscope**
Kevex Delta Class Analyzer
JEOL JSM-35C Scanning Electron Microscope

Notes

* - At this time there is no officially adopted analytical method for determining silica fiber concentrations emitted from the burning of biomass fuels. The AIHL provisional method is based on standard techniques used for other sample types.

Details of the **AIHL Provisional TEM Method for Analysis of Fibrous Silica** are available upon request.

Signatures of Analysts

> Donald J. Scales 3-18-92

> John C. Dahlmidt 3-18-92

Signature of Supervising Analyst

> [signature] John 3/19/92

AIHL Lab. Number: 10530

Analytical Summary

Field Number	Silica Fibers per cc Air <5 microns	Silica Fibers per cc Air >=5 microns	Al-Silica Fibers per cc Air <5 microns	Al-Silica Fibers per cc Air >=5 microns
RB1041	0.00	0.066	0.20	0.00
RB1042	Filter was overloaded. No analysis was attempted.			
RB1043	Filter was overloaded. No analysis was attempted.			
RB1044	Filter was overloaded. No analysis was attempted.			
RB1045	2.6	1.3	0.0	0.0
RB1048	Filter was plugged. No analysis was attempted.			
RB1049	Filter was plugged. No analysis was attempted.			
RB1051	0.96	0.96	0.0	0.0
RB1054	0.0	0.0	0.0	0.0
RB1046	Filter was contaminated in laboratory accident.			

*Formula for calculating the **Minimum Detection Limit (MDL)** in fibers per cc air:*

MDL = 1 fiber x Effective Filter Area/[1000 x Grid Area Scanned x Sample Volume],

where Sample Volume is given in liters

*Formula for calculating **Fiber Concentration (C)** in fibers per cc air:*

C = (Number of fibers) x (MDL)

Sample Analysis

AIHL Lab. Number: 10530
Field Number: RB1041: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	64.6 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.066 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.090	Operator	D. Scales

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*		Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	0	**0.00**	0.00	to 0.24	fibers/cc
>=5um	1 [A]	**0.066**	0.002	to 0.36	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.
A = Amorphous fiber, C = Crystalline fiber

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	3 [3C]	**0.20**	0.04	to 0.57	fibers/cc
>=5um	0	**0.00**	0.00	to 0.24	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.
A = Amorphous fiber, C = Crystalline fiber

Sample Analysis

AIHL Lab. Number: 10530
Field Number: RB1045: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	22.1 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.16 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.11	Operator	D. Scales

IDENTIFIED FIBERS			CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*			Silica Fibers/cc	95% Confidence Range		
length	number					
<5um	16	[5A, 11C]	2.6	1.5	to 4.2	fibers/cc
>=5um	8	[4A, 4C]	1.3	0.55	to 2.5	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.
A = Amorphous fiber, C = Crystalline fiber

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	0	0.00	0.00	to 0.59	fibers/cc
>=5um	0	0.00	0.00	to 0.59	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10530
Field Number: RB1051: FIBER DATA

Filter Data		Air Pump Data	
Type	Polycarbonate, 0.4um	Sample Volume	22.1 liters
Diameter (mm)	25		
Effective Area (mm^2)	380		

TEM Analytical Parameters

Magnification	35,000X	Minimum Detection Limit	0.19 fibers/cc
Grid Openings Scanned	10	Instrument	Hitachi H-600/H6010A
		Accelerating Voltage	75 kV
Grid Area Scanned (mm^2)	0.090	Operator	D. Scales

IDENTIFIED FIBERS			CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Silica*			Silica Fibers/cc	95% Confidence Range		
length	number					
<5um	5	[5A]	**0.96**	0.31	to 2.24	fibers/cc
>=5um	5	[4A, 1C]	**0.96**	0.31	to 2.24	fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.
A = Amorphous fiber, C = Crystalline fiber

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)			
Al-Silica+		Al-Silica Fibers/cc	95% Confidence Range		
length	number				
<5um	0	**0.00**	0.00	to 0.71	fibers/cc
>=5um	0	**0.00**	0.00	to 0.71	fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

Sample Analysis

AIHL Lab. Number: 10530
Field Number: RB1054: FIBER DATA

Filter Data

Type Polycarbonate, 0.4um
Diameter (mm) 25
Effective Area (mm^2) 380

Air Pump Data

Sample Volume 195 liters

TEM Analytical Parameters

Magnification 35,000X
Grid Openings Scanned 10
Grid Area Scanned (mm^2) 0.11

Minimum Detection Limit 0.018 fibers/cc
Instrument Hitachi H-600/H6010A
Accelerating Voltage 75 kV
Operator J. Schmidt

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Silica* length	number	Silica Fibers/cc	95% Confidence Range
<5um	0	0.00	0.00 to 0.067 fibers/cc
>=5um	0	0.00	0.00 to 0.067 fibers/cc

* - **Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate a dominant silicon X-ray fluorescence peak with no aluminum peak.

IDENTIFIED FIBERS		CALCULATED FIBER CONCENTRATION (PER CC AIR)	
Al-Silica+ length	number	Al-Silica Fibers/cc	95% Confidence Range
<5um	0	0.00	0.00 to 0.067 fibers/cc
>=5um	0	0.00	0.00 to 0.067 fibers/cc

\+ - **Al-Silica fibers** are structures that have an aspect ratio of 3:1 or greater and demonstrate both an aluminum and silicon X-ray fluorescence peak.

LN 10530
UCD-003

samplevols.ric

Sample Volume (liters)

Sample #								
A59351		1:00 PM	2:55 PM	1:55	1.7	1.61	1.66	190.33
A59352		1:00 PM	2:55 PM	1:55	1.7	1.55	1.63	186.88
A59353		3:16 PM	4:45 PM	1:29	1.7	1.61	1.66	147.30
A59355		12:52 PM	2:50 PM	1:58	1.7	1.61	1.66	195.29
RB 1041 OK		12:59 PM	1:38 PM	0:39	1.7	1.61	1.66	64.55
RB1042	overloaded	12:59 PM	2:29 PM	1:30	1.7	1.53	1.62	145.35
RB1043	overloaded	1:39 PM	3:02 PM	1:23	1.7	1.53	1.62	134.05
RB1044 small	overloaded (smoker)	2:30 PM	3:02 PM	0:32	1.7	1.7	1.70	54.40
RB1045 small	bulldozer	3:16 PM	3:29 PM	0:13	1.7	1.7	1.70	22.10
RB1048 OK		1:00 PM	2:55 PM	1:55	1.7	0	0.00	0.00 * See note
RB1049		1:00 PM	2:55 PM	1:55	2	0	0.00	0.00 * See note
RB1051 OK	bulldozer	3:30 PM	3:43 PM	0:13	1.7	1.7	1.70	22.10
RB1054		12:52 PM	2:50 PM	1:58	1.7	1.61	1.66	195.29
RB1~~055~~46	~~overloaded bulldozer~~	3:44 PM	4:45 PM	1:01	1.7	1.61	1.66	100.96

I Date Collected 11/16/91
By R. Lawson
Rice Burning

II Filter Type
RB 10... = Polycarbonate, 0.4μ filter, 25mm diameter
A593... = MCE filter, 0.45μ filter, 25 mm diameter

III Note RB1045, RB1049 plugged. Actual sample volume not known

IV Priority for SEM Loading review
RB1043, RB1042, RB1051, ~~RB1055~~ RB1046

Submitted By
R. Lawson
UC Davis
916-752-5180