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# Analysis of Biogenic Silica Fibers From Rice Farming Operations

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A provisional method has been developed for the quantitation of biogenic silica fibers. Particles were sampled onto track-etched filters prepared by the direct-clearing method for transmission electron microscopy analysis. Fibers were identified by their length/width ratio, X-ray spectrum, and electron diffraction pattern. Biogenic silica fibers were found to contain mainly silicon and no aluminum, and to have an amorphous crystal structure. Approximately 100 samples from rice farming operations were analyzed. Cyclones were used to exclude large, nonrespirable particles when sampling near farming operations. The optimum fiber loading was found to be 100 to 1000 fibers/mm$^2$. To prevent overloading, in most cases the sampled volume was held to 30 L using 25-mm filters, giving a limit of detection of 0.1 fibers/cc. Higher volumes were practical when sampling away from farming operations, giving limits of detection ranging from 0.02 to 0.004 fibers/cc. For a given microscopist, the coefficient of variation of fiber counts was 20 to 30 percent. For fibers with lengths $\geq 5$ $\mu$m, one microscopist counted 1.9 times more fibers than another microscopist. Although the method yielded a useful assessment of emissions from rice farming, further development of the method, including proficiency testing with standard samples and interlaboratory testing, is recommended. SCALES, D.; JOHN, W.; LAWSON, R.; SCHMIDT, J.: ANALYSIS OF BIOGENIC SILICA FIBERS FROM RICE FARMING OPERATIONS. APPL. OCCUP. ENVIRON. HYG. 10(8):685–691; 1995.

The rice plant has a very high silica content: rice straw is approximately 12 percent silica by weight. Rice farming operations produce aerosols containing silica particles of plant origin (biogenic silica). Biogenic silica has a noncrystalline atomic structure termed amorphous. It has been previously shown that biogenic silica particles are produced by the burning of sugar cane and that some of the particles are fibrous and in the respirable size range.[1] Little is known about the health effects of biogenic silica fibers; however, there is concern because of the analogy to asbestos.[2] Additionally, there are reports of increased incidence of lung cancer among workers in the sugar cane industry.[3]

Because California is one of the largest rice-producing states in the United States, the Environmental Health Laboratory (previously named the Air and Industrial Hygiene Laboratory) has been involved in several investigations of biogenic silica fibers from the burning of rice plants. Scales and John[4] reevaluated the air samples taken during an investigation sponsored by the *Sacramento Bee* newspaper of silica fibers emitted during field burning of rice straw and stubble. The presence of biogenic silica fibers was verified by transmission electron microscopy (TEM). Biogenic silica fibers were also found in air filter samples from the burning of rice plants in the wind tunnel facility at the University of California, Davis.[5] Samples of ash from the burning of rice hulls (which are extremely high in silica) in the Wadham energy plant in the Sacramento Valley were also analyzed.[4]

Since there are no established methods for the sampling and analysis of rice smoke particles, the work at the Environmental Health Laboratory employed ad hoc methods, drawing on methods used in the analysis of other types of particles, especially asbestos fibers. This experience pointed up the need for a more systematic development of an analytical method for the analysis of biogenic silica fibers. The opportunity for such a development arose in connection with a study of emissions of biogenic silica fibers from rice farming.[6] This article reports the development of the analytical method used in the study.

## Method Development

The objective was to develop a laboratory method for the analysis of biogenic silica fibers on air sample filters. Parameters to be determined were the number and concentration of silica-containing fibers, the fiber size distribution, and characterization of the silica as amorphous or crystalline. The method was based on TEM, which permits the classification of individual particles as fibers from the morphology and examination of the crystallinity by electron diffraction. In addition, TEM affords elemental analysis by energy-dispersive X-ray spectrometry, which proved very useful in the identification of biogenic silica, as will be shown. It should be noted that bulk analytical techniques are impractical since the fibers are only a minute fraction of all the particles in the sample.

TEM requires a very thin sample, that is, most of the filter material must be removed before microscopy. Two techniques have been developed for the analysis of asbestos fibers; the so-called direct-clearing method and the indirect transfer method.[7,8] In the direct method, the particles are sampled onto track-etched polycarbonate membranes (Nuclepore or Poretics). These filters have circular pores penetrating straight through the membrane. Most of the particles sampled with these filters lie on the flat top surface. The filter is carbon

coated to encapsulate the particles and then the filter is etched away with solvent. In the indirect transfer method, the particles are sampled onto a filter medium such as cellulose ester membrane. The filter is then dissolved in solvent and the particles are transferred to a polycarbonate filter by liquid filtration. The final preparation is as in the direct method.

Both methods have advantages and disadvantages. The direct method has less chance of altering the particles by breakage, deagglomeration, or agglomeration, and less chance of losing particles or contaminating the sample during transfer. The principal disadvantages of the direct method are the restriction on the filter loading to prevent the overlapping of particles and the tendency of the filters to plug in certain high moisture conditions. This places a limit to the airborne particle concentration that can be detected. Further, this requires field personnel to control the filter loading, a difficult task involving trial and error. The indirect transfer method has the advantage of allowing the use of filters such as cellulose ester membranes that capture particles in depth, permitting high loading before clogging. Since the particles are transferred to another filter before microscopy, the particle loading on the final sample can be adjusted and optimized. Another advantage is the removal of soluble particles that are simply a background in the present application.

In a study of silica fibers from the burning of sugar cane in Florida, Boeniger *et al.*[9] and Yamate *et al.*[10] used a modified version of the Environmental Protection Agency (EPA) provisional method for the TEM analysis of asbestos fibers. This method employs direct clearing for sample preparation. Electron diffraction was used to verify that the silica fibers were amorphous. From the X-ray spectra, it was concluded that most of the fibers were silicates. In another study, Boeniger *et al.*[1] analyzed smoke samples from sugar cane burning in Hawaii. Cellulose ester filters were used. No details of the sample preparation were given; it is assumed than an indirect transfer method was used. It was found that most of the fibers contained only silicon, in contrast to the findings in Florida. It is of interest that personal samples were taken with open-faced filters, while area samples were taken with dichotomous samplers that collect the PM10 fraction, excluding larger particles.

Our choice for the initial development of the analytical method was the direct-clearing method, despite the sampling limitations, because the particles are unaltered by sample preparation. The indirect transfer technique requires verification that the particles are not significantly altered or lost during transfer. However, because of the advantages of the indirect method for sampling, over 50 samples were taken on cellulose ester membrane in parallel with the polycarbonate samples with the intention of validating the indirect method by comparison with the direct method. Unfortunately, we did not have the time and resources to undertake this comparison.

Preliminary trials were conducted to identify problems before establishing procedures for the field sampling. Initially, open-faced filters were used. It was found that samples taken in the vicinity of operations in the rice fields contained large plant structures that obscured the biogenic silica fibers. Most of the large interfering structures were not rice particles; many of them were fungus particles. To exclude large structures which could interfere with the analysis, particle size-selective samplers were used to select the respirable particles, as defined by the American Conference of Governmental Industrial Hygienists.[11] Thoracic samplers might have been better for this purpose because particles deposited in the upper airways within the thoracic region might cause lung cancer. However, at the time of the study no personal thoracic samplers were available. For this first study, it was decided that respirable samplers would be used, but for future studies the use of thoracic samplers should be considered. Subsequently, samples for the analysis of biogenic silica fibers were taken with cyclones restricting the particles to the respirable fraction, eliminating the large, interfering structures. The data obtained are thus restricted to assessment of exposure to the respirable fraction. The cyclones were modified to accept filter cassettes with a 50-mm extended cowl, which was necessary to obtain uniform deposits on the filter. Open-faced filters were used upwind and downwind of the rice fields and in communities at some distance from the fields. Experience was also gained in the permissible particle loading.

### Analysis of Samples from Field Emissions Study

The companion field emissions study is reported elsewhere; however, a very brief summary of the sampling for biogenic silica fibers is given here since the samples were used to evaluate the capability of the analytical method.[6] In all, 86 air samples taken during rice farming operations were analyzed. Sampling was conducted during rice harvesting, inside the enclosed cabin and on the outside of the harvester which separates the rice (with hull) from the mature, dry rice plants left in the field, and on the bank out wagon which transfers the rice (with hull) from the harvester to a truck at the edge of the field. Additional samples were taken upwind of the rice field. Air samples were taken during open field burning of the rice straw, which is air-dried, unharvested plant material including stems, leaves, and panicles (branchlike structures to which the rice hulls are attached before harvesting). Samples were also taken during the field preparation after burning inside and outside the tractors. The fields had been burned from 1 day to 2 months previously. Ash residue from previous burning was visible in most but not all cases.

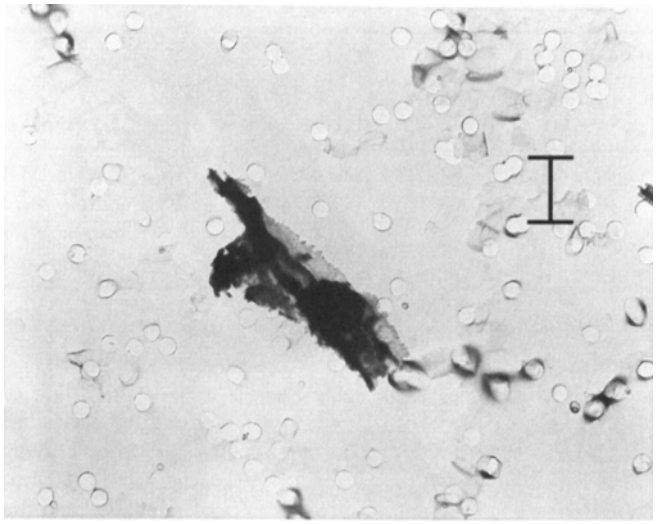
All of the samples were taken on 25-mm diameter, 0.4-$\mu$m pore size track-etched polycarbonate filters backed by 5-$\mu$m cellulose diffuser disks. Ten percent of the collected samples were duplicates taken with side-by-side samplers operating simultaneously. One set of eight side-by-side replicate samples was collected during harvest and another set of eight was collected during field burning. Field blanks were also taken. The laboratory was blinded to the identity of the samples.

Two experienced microscopists participated in the sample analysis. To evaluate the precision of the results, seven samples chosen at random were analyzed twice by both analysts.

### Method Details

#### *Sample Preparation*

The direct-clearing method was used, closely similar to that of the EPA provisional method for asbestos fibers.[10] 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.



FIGURES 1 to 4. Photomicrographs of amorphous silica fibers from rice farming operations. Fibers consist mainly of silicon and oxygen and are noncrystalline. Scale bar indicates one micron.

### *Reference Samples*

Our identification of biogenic silica fibers found on harvest and burn samples was based partly on a comparison of the elemental composition of unknown particles with that of particles of known rice ash origins. Rice char was obtained from Prof. Bryan Jenkins, University of California, Davis. The sample was collected from the ash bin after rice straw was burned in a large-scale wind tunnel. Samples of unburned rice straw were also obtained.

Ten milligrams of the rice char ash was pulverized by mortar and pestle. One hundred milligrams of 0.1 $\mu$m filtered water and 1 ml of 0.1 percent 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-$\mu$m track-etched polycarbonate filter was used to collect the particles from the water. The filter sample was then prepared by the direct-clearing method and examined by TEM and energy-dispersive X-ray analysis. The X-ray spectra were simple: most of the particle spectra contained only silicon and oxygen peaks. A small number of particles also contained traces of magnesium and/or potassium, as well as chlorine.

### *TEM Analysis*

A Hitachi H-600 scanning transmission electron microscope (STEM) was used. Although a STEM was not essential for this application, it had some advantages over a TEM, as will be seen. For scanning, the instrument was operated at 75 kV in TEM mode at a magnification of 35,000×.

Ten grid openings were selected at random and scanned to locate fibers. A fiber is defined to be a structure having an aspect ratio of 3:1 or greater by convention. The fibers observed on rice harvest and burn samples did not generally have the parallel sides or the appearance of asbestos or mineral fibers (Figures 1–4). After all, the rice fibers are fragments of either the plant or the charred residue. None of the fibers are identical. Some are perforated, while others have spikes (papillae) or other irregular characteristics. Some are branched like twigs.

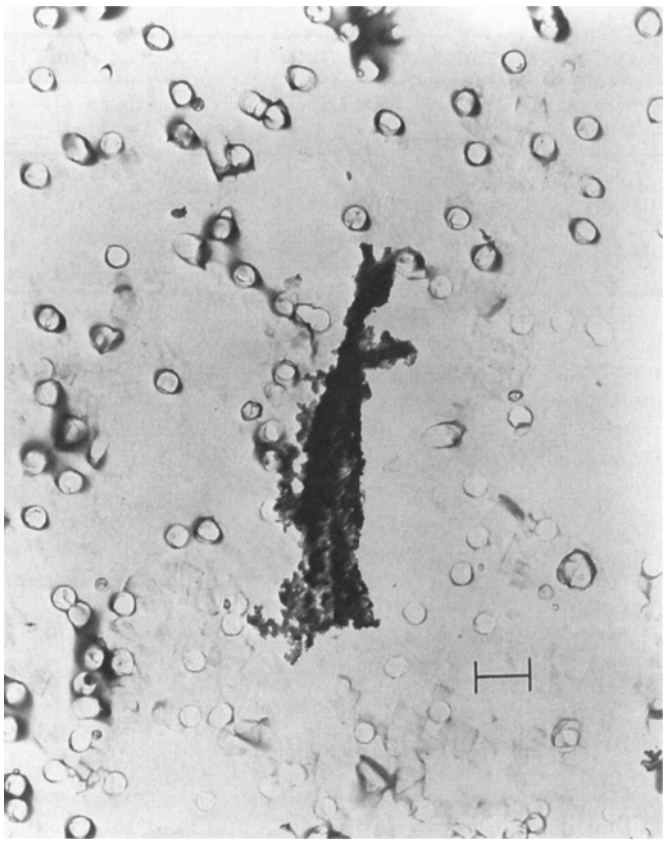


FIGURE 2.

When examined in three dimensions (stereoscopic image pairs obtained by changing the parallax angle), the structures are seen not to be flat and are thus not adequately described in two dimensions. Nevertheless, the term fiber still describes the essential features of the rice particles with respect to inhalation hazard. The aerodynamic diameter of the fiber and the site of lung deposition are determined mainly by the small dimension, the width. The long dimension, the length, will resist clearance from the lung, leading to longer residence times. Since the rice fibers penetrated the cyclone sampler, they are in the respirable fraction. However, their actual aerodynamic diameters would be difficult to calculate, and the length and width parameters are only rough descriptors.

The length of the fiber was taken to be the largest dimension. Since most of the fibers did not have a fixed width, we arbitrarily chose to measure the width at the midpoint of the structure's length using the two-dimensional image. Length to width aspect ratios seldom exceeded 20:1 and ratios closer to 5:1 were more common.

### *Fiber Composition and Crystallinity*

Once a fiber was selected, the TEM was switched to STEM/scanning electron microscope scanning mode. In this mode the surface features of the fiber could be observed. This was helpful in positioning the electron beam spot; for example, small, agglomerated particles adhering to the fiber could be avoided. A fluorescence X-ray spectrum was then acquired for 20 seconds using a Quantex (thin-window) X-ray detector and a

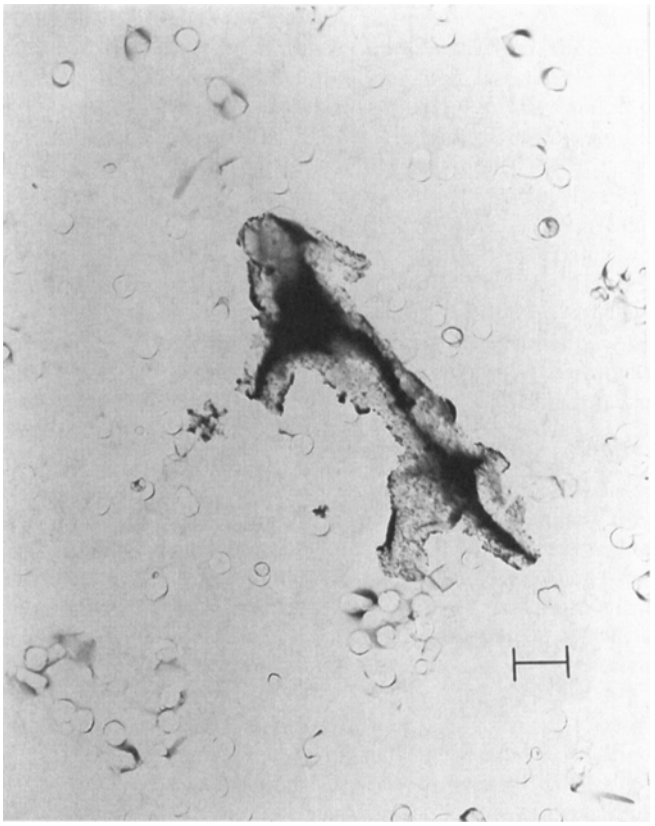


FIGURE 3.

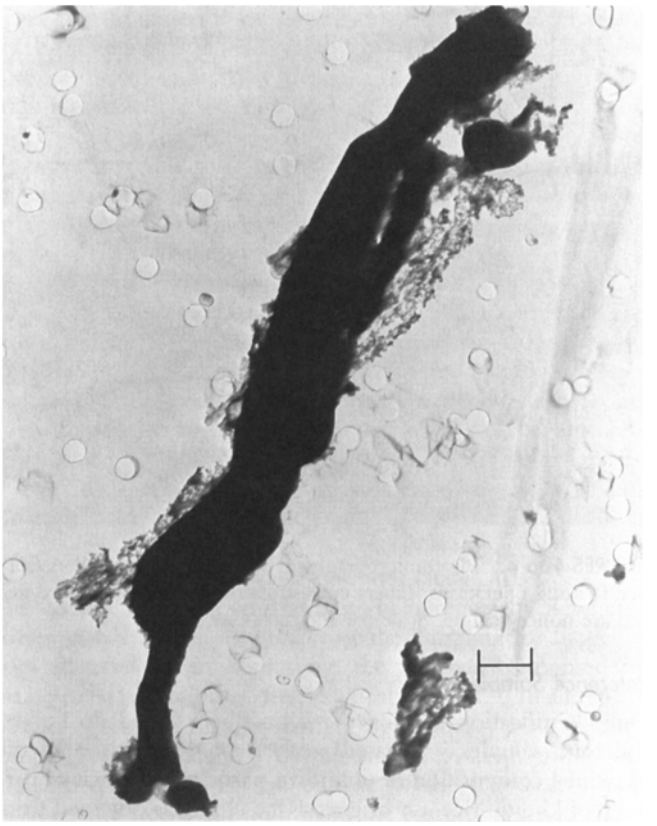


FIGURE 4.

Kevex Delta Class Analyzer. Only fibers with dominant silicon peaks were entered into the data record. The spectra were normalized to the highest peak and the relative peak heights of the identified elements were recorded.

All of the silica fibers were observed to fall into one of two classes:

1. silica fibers: fibers showing a dominant silicon X-ray peak and no aluminum peak; and
2. aluminum–silica fibers: fibers showing an aluminum and a silicon X-ray peak.

Because the samples were carbon coated, the method could not distinguish between fibers that contain silicon only and those that contained carbon in addition to silicon.

Each fiber was also examined by electron diffraction to determine its crystallinity. A microdiffraction technique was used, rather than selected area electron diffraction, because microdiffraction minimizes the background from other particles. The STEM affords a finely focused beam for this purpose and the Hitachi H-600 can be switched from scanning mode to microdiffraction mode under computer control. The diffraction pattern was observed visually to determine whether it showed an array of bright spots, indicating a crystalline structure, or no bright reflections, indicating an amorphous structure. Some particles were thick and had to be examined along the edges where the electron beam could penetrate.

Another pattern emerged: the silica fibers were always amorphous and the aluminum–silica fibers were always crystalline. The crystalline, aluminum–silica fibers were believed to be of geological mineral origin (i.e., soil particles). These fibers were simply airborne particles of soil that happened to fit the 3:1 length/width ratio. Furthermore, aluminum was never observed in the reference samples. Conversely, the amorphous, silica fibers were believed to originate from the rice plant. These observations formed the basis for the identification of the silica fibers.

By analogy with asbestos fibers, it is anticipated that silica fibers $\geq 5$ $\mu$m in length may be more hazardous to health. Therefore, such fibers were tabulated separately. The division of the fibers into two classes by length was also experimentally significant in that the variance of the counts between two microscopists was less for fibers $\geq 5$ $\mu$m in length.

To check against contamination, both laboratory and field blanks were carried through the analysis. No silica fibers were ever detected on the blanks. Interestingly, some asbestos fibers were seen on the blanks. The asbestos fibers appeared to be in the polycarbonate filter material. This was confirmed through communication with the filter manufacturer.

### *Fiber Counts and Calculations*

Four numbers were determined for each sample:

1. number of silica fibers $<5$ $\mu$m in length;
2. number of silica fibers $\geq 5$ $\mu$m in length;
3. number of aluminum–silica fibers $<5$ $\mu$m in length; and
4. number of aluminum–silica fibers $\geq 5$ $\mu$m in length.

TABLE 1. Fiber Concentrations Obtained in Field Emissions Study

| Activity | No. of Samples | Range, Silica Fibers | | Range, Aluminum–Silica Fibers | |
|---|---|---|---|---|---|
| | | Total (f/cc) | ≥5 μm (f/cc) | Total (f/cc) | ≥5 μm (f/cc) |
| Rice harvest | 17 | 0.19–9.10 | 0.00–2.70 | 0.00–0.81 | 0.00–0.23 |
| Field burning | 49 | 0.00–7.40 | 0.00–4.90 | 0.00–0.36 | 0.00–0.36 |
| Field preparation | 9 | 0.00–22.95 | 0.00–9.90 | 0.00–2.79 | 0.00–0.89 |
| Upwind | 11 | 0.00–0.03 | 0.00–0.02 | 0.00–0.12 | 0.00–0.06 |

The average area of a grid square opening was determined by light microscopy and computer-aided image analysis for each vial, containing 100 grids, of 200-mesh copper TEM grids. The fiber density (fibers/square millimeter) on the filter surface was calculated. From the record of the volume of air sampled in the field the airborne fiber concentration (fibers/cubic centimeter of air) was calculated. Ten percent of the samples were recounted by the original microscopist and these were then recounted twice by a second microscopist.

## Results from Field Emissions Study

A total of 86 samples from the companion field emissions study were analyzed. Concentrations ranged from none detected to 9.90 fibers/cc (f/cc) for fibers >5 μm in length (Table 1). The median fiber length was 2.8 μm, with a range from 0.5 to 20 μm. (Fibers <0.5 μm were not counted.) Ninety percent of the fibers were <9 μm in length. The median fiber width was 0.9 μm, with a range from 0.2 to 7 μm. Ninety percent of the fibers were <2.5 μm in width.

Seven samples were counted twice by each microscopist. Microscopist A consistently obtained higher fiber counts than did microscopist B. For total fibers, the ratio of the results A/B was 3.7 ± 5.0 (Table 2), while for fibers ≥5 μm the ratio was 1.9 ± 0.9 (Table 3). The difference in fiber counts obtained by the two microscopists was significant at the 95 percent confidence level.

Six sets of duplicate samples from side-by-side samplers were analyzed by microscopist A. For total fibers, the average coefficient of variation was 24 percent (Table 4). For fibers ≥5 μm, the average coefficient of variation was 18 percent (Table 5).

From one of the two sets of eight replicate samples, taken with side-by-side samplers, five samples were analyzed. Some of the samples were analyzed by microscopist A, some by microscopist B, and one by both. The coefficient of variation of the fiber concentration was 43 percent (Table 6).

Overall, the recount data show that each microscopist counted fibers ≥5 μm in length with a coefficient of variation in the range of 20 to 30 percent, but one microscopist counted about twice as many fibers as the other. The coefficient of variation is comparable to or slightly larger than that for the counting of asbestos fibers. The coefficient of variation might be reduced by counting more than ten grid openings; however, this would increase the already long analysis time of about 1 day per sample. As with asbestos fibers, the variance between microscopists would undoubtedly be reduced by proficiency tests by which the microscopist calibrates his/her discrimination of fibers. The much larger variance for total fibers reflects the increasing difficulty of discriminating small fibers.

The optimum fiber loading was in the range of 100 to 1000 fibers/mm$^2$. Even with care exercised in the field, 9 of 104 samples were overloaded. To avoid overloading, the total sampled volume of air through the 25-mm filters was held to 30 L for most samples collected from farming operations, resulting in a limit of detection of 0.1 f/cc. The sampled volume could be increased for most samples collected some distance from farming operations, resulting in limits of detection ranging from 0.02 to 0.004 f/cc.

Despite the current limitations of the method, its use made possible a successful study of emissions from rice field operations. The highest airborne concentration of biogenic silica fibers reported thus far was found. An unexpected result was higher silica fiber concentrations during harvesting than during field burning.

TABLE 2. Rice Sample Recounts

| Sample | $B_1$ | $B_2$ | $\sigma_B/B_{av}$ | $A_1$ | $A_2$ | $\sigma_A/A_{av}$ | $A_{av}/B_{ab}$ |
|---|---|---|---|---|---|---|---|
| RH002 | 3 | 12 | 0.85 | 15 | 10 | 0.28 | 1.67 |
| RH007 | 20 | 25 | 0.16 | 66 | 40 | 0.35 | 2.36 |
| RH1004 | 6 | 8 | 0.20 | 105 | 103 | 0.01 | 14.9 |
| RB1045 | 24 | 32 | 0.20 | 43 | 38 | 0.09 | 1.45 |
| RB1051 | 10 | 10 | 0.00 | 13 | 5 | 0.63 | 0.90 |
| RB1063T | 20 | 16 | 0.16 | 46 | 34 | 0.28 | 2.22 |
| RB1066T | 17 | 17 | 0.00 | 32 | 36 | 0.08 | 2.00 |
| Averages | | | 0.22 | | | 0.24 | 3.64 |

Same sample counted twice by each analyst. Counts are total number of amorphous silica fibers detected.
$B_1$ = microscopist B, count 1; $A_1$ = microscopist A, count 1; $\sigma$ = standard deviation; av = average.

TABLE 3. Rice Sample Recounts

| Sample | $B_1$ | $B_2$ | $\sigma_B/B_{av}$ | $A_1$ | $A_2$ | $\sigma_A/A_{av}$ | $A_{av}/B_{av}$ |
|---|---|---|---|---|---|---|---|
| RH002 | 1 | 2 | 0.47 | 3 | 5 | 0.35 | 2.67 |
| RH007 | 5 | 8 | 0.32 | 6 | 7 | 0.11 | 1.00 |
| RH1004 | 6 | 8 | 0.20 | 16 | 13 | 0.15 | 2.07 |
| RB1045 | 8 | 14 | 0.38 | 15 | 17 | 0.09 | 1.45 |
| RB1051 | 5 | 4 | 0.16 | 5 | 1 | 0.94 | 0.67 |
| RB1063T | 7 | 9 | 0.18 | 31 | 16 | 0.45 | 2.94 |
| RB1066T | 11 | 7 | 0.31 | 20 | 20 | 0.00 | 2.22 |
| Averages | | | 0.29 | | | 0.30 | 1.86 ± 0.85 |

Same sample counted twice by each analyst. Counts are number of amorphous silica fibers ≥5 μm in length.
$B_1$ = microscopist B, count 1; $A_1$ = microscopist A, count 1; $\sigma$ = standard deviation; av = average.

## Summary and Conclusions

A provisional method has been developed for the quantitation of biogenic silica fibers. Particles were sampled onto track-etched filters that were prepared by the direct-clearing method for TEM analysis. Fibers are defined by a length/width ratio ≥3:1 by convention. The identification as a biogenic silica fiber was based on obtaining an X-ray spectrum with a dominant silicon peak and no aluminum peak coupled with an amorphous crystal structure as revealed by the lack of bright spots in the electron diffraction pattern. On each sample, ten grid openings chosen at random were scanned for fibers.

The method was used to analyze approximately 100 samples from rice field operations. For sampling in the vicinity of field operations, it was found advantageous to precede the filters with cyclones to exclude large, nonrespirable structures that obscured fibers on the filter. The optimum fiber loading on the filter was found to be 100 to 1000 fibers/mm$^2$. To prevent overloading, the sampled volume was held to 30 L through the 25-mm filters, giving a limit of detection of 0.1 f/cc.

Sample recounts by a given microscopist gave fiber counts with a coefficient of variation in the range 20 to 30 percent. However, one microscopist consistently obtained higher fiber counts than another microscopist; for total fibers the ratio of counts was 3.7 and for fibers ≥5 μm the ratio was 1.9. The difference between microscopists could be reduced by proficiency type testing such as that used for asbestos fibers.

The method produced useful results in the rice emissions study. The present method represents a further development of the TEM method for biogenic silica fibers, the only general method currently available for biogenic silica fibers. The method must be regarded as provisional because of the lack of

TABLE 4. Rice Sample Duplicates

| Sample | Total Fibers (f/cc) | Average (f/cc) | Standard Deviation | Coefficient of Variation (%) |
|---|---|---|---|---|
| RB1084P | 12.5 | 13.55 | 1.48 | 11.0 |
| RB1086P | 14.6 | | | |
| RH1001 | 9.1 | 8.35 | 1.06 | 12.7 |
| RH1002 | 7.6 | | | |
| RB1050T | 6.3 | 6.85 | 0.78 | 11.4 |
| RB1056T | 7.4 | | | |
| RB1060T | 1.34 | 1.18 | 0.23 | 19.2 |
| RB1061T | 1.02 | | | |
| RB1064T | 0.34 | 0.26 | 0.12 | 47.1 |
| RB1065T | 0.17 | | | |
| RH1003 | 4.3 | 6.25 | 2.76 | 44.1 |
| RH1010 | 8.2 | | | |
| Average | | | | 24.3 |

Side-by-side sampling, submitted blind for analysis. Counts of all amorphous silica fibers.

TABLE 5. Rice Sample Duplicates

| Sample | Fibers ≥5 μm (f/cc) | Average (f/cc) | Standard Deviation | Coefficient of Variation |
|---|---|---|---|---|
| RB1084P | 8.3 | 7.75 | 0.78 | 10.0 |
| RB1086P | 7.2 | | | |
| RH1001 | 1.6 | 1.65 | 0.07 | 4.24 |
| RH1002 | 1.7 | | | |
| RB1050T | 2.5 | 3.25 | 1.06 | 32.6 |
| RB1056T | 4.0 | | | |
| RB1060T | 1.0 | 0.93 | 0.11 | 11.4 |
| RB1061T | 0.85 | | | |
| RB1064T | 0.17 | (At limit of detection) | | |
| RB1065T | 0.00 | | | |
| RH1003 | 1.7 | 2.20 | 0.71 | 32.1 |
| RH1010 | 2.7 | | | |
| Average | | | | 18.1 |

Side-by-side sampling, submitted blind for analysis. Counts of amorphous silica fibers ≥5 μm in length.

TABLE 6. Rice Sample Replicates; Five Side-by-Side Samples, Submitted Blind for Analysis

| Sample Number | Fibers/cc | Analyst |
|---|---|---|
| RB1052T | 1.9 | B, A |
| RB1058T | 3.2 | A |
| RB1063T | 4.9 | A |
| RB1066T | 1.8 | B |
| RB1069T | 3.2 | A |
| Average ± Std. Dev. | 3.0 ± 1.3 | |
| Coeff. of Variation (%) | 43 | |

Counts of amorphous silica fibers ≥5 μm in length.

interlaboratory testing and use by other workers. A basis for further development has been provided, both for refinement of the present method and for the possible development of a method based on the indirect clearing of filters. The latter would require comparison with the direct method that minimizes alteration of the fibers.

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## Disclaimer

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.

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