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An Evaluation of the Adhesive Tape Sampling Method for Estimating Surface Asbestos Concentrations

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This study was designed to compare surface asbestos concentrations measured directly from a contaminated surface via scanning electron microscopy (SEM) with concentrations measured by adhesive tape sampling. The tape samples were analyzed by both SEM and polarized light microscopy (PLM). Two different length criteria were used for fiber counting: 3 μm and 5 μm. Tape samples analyzed by SEM measured 30.6 and 37.9 percent (3 and 5 μm, respectively) of the surface concentrations. PLM-tape samples measured 6.8 and 18.3 percent, respectively. Based on the 3-μm criterion, the mean fiber concentrations of the three sample types (tape-SEM, tape-PLM, and drywall-SEM) were all significantly different ($p = 0.01$). Based on the 5-μm criterion, the mean fiber concentrations of the tape-SEM and tape-PLM were not significantly different from one another ($p \geq 0.10$) but were significantly less than that for the drywall-SEM samples ($p = 0.05$). None of the regression analyses (for either length criterion) produced statistically significant correlations of tape concentrations versus drywall concentrations. Fibers thinner than 0.1 μm were not observed in the SEM, while fibers thinner than 0.75 μm were not observed with PLM. The failure to demonstrate statistically significant correlations was felt to be due to the inability to produce a uniform surface of known concentration. If one could produce a uniform surface concentration, tape sampling might very well demonstrate statistically significant correlations. Overall, tape sampling must be regarded as a qualitative method at this time. If one were successful in validating significant correlations, field use of the method will still pose problems due to the amount of surface (environmental) variability that exists. This, however, becomes a sampling strategy problem as opposed to a limitation of the method itself. RYAN, G.; BUCHAN, R.M.; KEEFE, T.J.; MCCAMMON, C.S.: AN EVALUATION OF THE ADHESIVE TAPE SAMPLING METHOD FOR ESTIMATING SURFACE ASBESTOS CONCENTRATIONS. APPL. OCCUP. ENVIRON. HYG. 12(4):288–292; 1997. © 1997 AIH.

Tape sampling, which has become a common method for evaluating surface asbestos contamination, holds a potential advantage over the traditional wipe sample since the surface particles are removed by the tape while maintaining their original positions in relation to one another. Because the particle distribution is intact, the tape can be analyzed in a manner similar to an asbestos air sample, resulting in an analysis that should be more quantitative relative to the wipe sample. To date, however, information on the validation of the method has not appeared in the literature.

Previously we reported on the results of a 2-year asbestos removal study in which both airborne and surface concentrations of asbestos were compared before and after the removal of asbestos-containing materials.[1] In preparation for that study, this evaluation was conducted to assess the capability of the adhesive tape method for measuring surface concentrations of asbestos.

Experimental Methods and Materials

Two pieces of painted drywall, one in a horizontal orientation and one in a vertical orientation, were placed into a 1.0-m^3 plastic-lined plexiglass chamber. Five milligrams of National Bureau of Standards standard grade 1866 chrysotile was aerosolized in the chamber and allowed to settle for 24 hours before retrieving the drywall. Tape samples from the drywall surface were collected and analyzed by polarized light microscopy (PLM) and scanning electron microscopy (SEM) methods. To estimate the actual surface concentration, pieces of the drywall surface were excised, mounted on SEM stubs, and analyzed by SEM.

A major concern existed as to whether the aerosol deposition onto the drywall would be uniform. If the deposition patterns were not uniform and a large variation of surface asbestos levels resulted, comparison of adjacent tape samples to the excised surface samples would be difficult. A 4 × 6 grid was drawn on the two 20-cm^2 pieces of drywall. This pattern resulted in six columns and four rows for each piece of drywall. A coin toss was used to determine the type of sample to be taken from the first column (tape or drywall surface). Sample types were then alternated by column.

A decision was made to attempt to produce a surface concentration of 15 to 30 fibers per square millimeter (f/mm^2), or one fiber for every five to ten counting fields in PLM. This concentration would minimize the amount of asbestos used, but would ensure that the samples would be in a quantifiable range. Based on a review of previous work of this type,[2,3] 5 mg of chrysotile was aerosoled by a Misto$_2$® aerosol generator.

An initial SEM bulk sample analysis of the asbestos to be used found that the majority of the fibers were larger than 0.3 μm in width and 1.0 μm in length. The first quartile mean width and length were 0.6 and 2.4 μm, respectively. An asbestos fiber of this size would possess a terminal settling velocity of 3.5×10^{-3} cm/s (adjusted for both a dynamic

shape factor and Cunningham's slip correction factor) and would require 7.9 hours to fall 1 m in still air. To ensure that the majority of fibers had settled, the fibers were allowed to settle for 24 hours prior to sampling.

After 24 hours the drywall was retrieved, the chamber's air intake was blocked off, and a Nilfisk® vacuum cleaner was used to evacuate the air inside the chamber's plastic liner. After the liner was collapsed, it was removed, double-bagged, and disposed of as a hazardous waste. The inside of the chamber was vacuumed and wet-wiped. Due to the potential hazards of working with asbestos, the work was isolated in a separate airtight room with dedicated exhaust. The investigator wore personal protective equipment, including full-body Tyveck® coveralls and a full-face powered air purifying respirator with high efficiency particulate air cartridges. At the completion of the study, the airtight room was decontaminated; personal air sampling of the investigator and clearance samples of the room indicated that contamination did not occur.

Tape sampling as described by Nichols® was utilized to assess surface asbestos contamination. 3M Scotch 810 Magic Tape® was used to sample the drywall surface. Drywall samples were collected by excising a 10 × 20 mm piece of drywall surface with a scalpel. Samples were then mounted on SEM stubs. Phase contrast microscopy (PCM), PLM, and SEM were all investigated for analyzing the tape samples. Of the light microscopic methods, PCM naturally provided the best resolution. The major limitation of PCM is its inability to differentiate asbestos and nonasbestiform fibers. Since it was anticipated that numerous nonasbestos fiber interferences would exist with field use, an analysis method capable of fiber differentiation was desirable. Since PLM methods were capable of partially differentiating between asbestos and nonasbestos fibers, it was chosen as the light microscopic method. Once mounted and cleared, samples were counted on a Zeiss binocular microscope equipped with phase contrast and polarizers mounted above and below the microscope stage. A 10× eyepiece and 40× achromat phase contrast objective (NA 0.65) was utilized. Although not used in this study due to availability, a combination of PCM and PLM can be utilized to enhance the resolution of PLM as long as the microscope's illumination source is greater than 100 W.

Tape samples were counted by a method that is currently employed by the Occupational Safety and Health Administration's (OSHA's) National Analytical Laboratory, as described by Dixon.[5] Samples were examined by PLM under crossed polars with a quarter wave length quartz compensator (550 nm of retardation). Using this system, a determination of whether a fiber was isotropic or anisotropic was made. Depending on a fiber's orientation to the slow ray of the compensator, fibers greater than 1 μm in diameter demonstrate a different color for each refractive index. Asbestos fibers are all anisotropic and appear blue or yellow depending on their orientation to the compensator.

The counting start and stop rules specified by National Institute for Occupational Safety and Health (NIOSH) Method 7400 were utilized.[6] Fibers with aspect ratios ≥3:1 and lengths ≥3 μm were examined. Since asbestos tape sample results are primarily used for assessing cleanliness, the 3-μm length criterion was chosen to avoid ignoring short fibers. When using light microscopy, the limits of resolution for the human eye (0.1 mm)[7] made it difficult to determine whether a fiber shorter than 3 μm was indeed a fiber. The dimensions of all fibers counted were recorded, allowing the use of different length criteria (i.e., 5 μm) during statistical analysis.

When morphology and characteristics under polarized light indicated that a fiber was an isotropic mineral fiber, it was eliminated from the count; otherwise, the fiber was presumed to be asbestos and counted (all anisotrophic fibers). This procedure allowed for the elimination of most interfering fibers while not eliminating fibers that, while suspicious, could not be proven to be asbestos. It has been estimated that the above protocol will eliminate 90 to 95 percent of interference from the presence of nonasbestos fibers.[8]

Tape and drywall samples were also analyzed by SEM. Sections of tape and drywall (8 × 19 mm) were mounted on aluminum stubs. The edges of the samples were coated with colloidal paint to assure conductivity and reduce excess charging in the SEM. Samples were sputter coated with approximately 20 nm of a gold–palladium alloy. A Philips 505 scanning electron microscope equipped with a secondary electron detector and Kevex Super 8000 microanalysis system was used for SEM fiber analysis. A spot size of 20 nm, accelerating voltage of 30 KeV, working distance of 12 mm, tilt angle of 15°, and magnification of 2500× were used.

Initially an attempt was made to count and size fibers directly from the CRT screen in full-frame scan mode. Due to the amount of signal noise, resolution was not sufficient. Instead, the "TV-rate" scan mode was used, and each counting field was photographed using a video print system. Fibers greater than 3 μm with aspect ratios greater than 3:1 were counted from the video print. Due to cost considerations, counting was limited to 20 fields as specified in the old NIOSH P&CAM 239 rules.[9]

Quality assurance procedures (field blanks, laboratory blanks, and replicate analyses) were utilized to assure the accuracy and precision of the analysis. Field and laboratory blanks revealed no significant contamination. Ten percent of the samples were randomly chosen for a blind recount. At the 5 percent significance level there were no differences found between the original counts and recounts. To determine whether the results were consistent with those of a certified laboratory, the PLM recount samples were submitted to a primary reference laboratory for NIOSH's analytical proficiency test program. Results indicated that two of the six samples were out of control limits ($p = 0.05$). One sample contained one counting field that represented 31.4 percent of the total count, or 8.0 standard deviations from the average count per field. The second sample had three fields that together represented 40.5 percent of the total count, or 4.5 standard deviations from the average count per field. Based on the observed frequencies and coefficients of variation among the counting fields, it was concluded that the observed bias was probably caused by nonuniform deposition of fibers and was not due to quality control problems. When the fields in question were eliminated from the counts, no significant differences were observed ($p = 0.05$). A random 10 percent blind recount of the SEM video prints was also conducted. Again, there were no statistical differences at the 5 percent level of significance.

The data collected were analyzed by two-way analysis of

TABLE 1. Tape Sample Validation Descriptive Statistics

Sample Type	Mean (f/mm²)	Standard Deviation	Geometric Mean (f/mm²)	Coefficient of Variation (%)
Drywall-SEM				
3 μm	541.3	281.8	485.1	52.1
5 μm	143.4	111.0	109.6	77.4
Tape-SEM				
3 μm	165.7	108.4	131.4	65.4
5 μm	54.3	54.9	32.5	101.1
Tape-PLM				
3 μm	36.6	14.8	32.4	40.4
5 μm	26.2	13.8	22.0	52.7

Based on 24 samples for each sample type.

TABLE 2. Tukey's Pairwise Comparison of Tape Sample Concentration Means: Surface Asbestos Concentration by Sample Type

Sample Type	No. of Samples	ln Mean Concentration (f/mm²)	Tukey's HSD*
≥3 μm			
Drywall-SEM	24	6.184	A
Tape-SEM	24	4.878	B
Tape-PLM	24	3.478	C
≥5 μm			
Drywall-SEM	24	4.697	D
Tape-SEM	24	3.481	E
Tape-PLM	24	3.089	E

*Fiber count means with the same letter are not significantly different at a significance level of $p \geq 0.1$. Note: The means from the 3-μm criterion were not compared with the means from the 5-μm criterion.

variance (ANOVA), with the main effects being sample type and orientation of drywall (vertical or horizontal). The asbestos concentrations were log-transformed to satisfy the assumption of normality in the ANOVA procedures.[10] Tukey's honestly significant difference (HSD) method was used to contrast the mean asbestos concentrations with the sample types and drywall orientation. Linear regression analysis was used to explore the potential for predicting the surface concentration of asbestos on the basis of results received from tape sampling.[10] The data were also explored by dichotomizing the concentrations into high and low categories, thereby treating the data as qualitative indicators of drywall concentrations rather than quantitative indicators. The log means for the data were used as cut points for the categorization. A 2 × 2 table was produced, and the data were evaluated using chi square and odds ratio statistics. Fisher's exact test was utilized whenever an expected cell value was less than five.[11]

Results and Discussion

Data in the form of 24 triplets were collected (drywall-SEM, tape-SEM, and tape-PLM). Table 1 summarizes descriptive statistics for these triplets. Using a 3.0-μm length criterion, a mean concentration of 541.3 f/mm² was observed on the drywall, with the concentrations ranging from 157.9 to 1552.6 f/mm² (coefficient of variation, 52.1%). A 5.0-μm length criterion resulted in a mean concentration of 143.4 f/mm², with concentrations ranging from 6.6 to 578.9 f/mm² (coefficient of variation, 77.4%). The increase in variation with the 5.0-μm criterion was felt to be due to the lower concentration range observed rather than to the length criterion. Based on what is known about the variation associated with fiber counting, this would be expected.[6] Despite efforts to control the variability in the concentration of asbestos on the drywall surface, considerable variability was obviously produced. Producing a known uniform concentration of dust has historically plagued aerosol researchers. The variation produced here was probably the result of the aerosolization process and the electrostatic forces within the plastic chamber. Future research of this kind might attempt to minimize the electrostatic forces through the use of conductive materials and high humidity conditions. The aerosolization process, however, may still prove to be problematic.

Table 2 contrasts the mean asbestos concentrations by sample type. Based on the 3.0-μm length criterion, the mean fiber concentrations of the sample types were all significantly different ($p = 0.01$). Based on the 5.0-μm length criterion, the mean fiber concentrations of the tape samples (SEM and PLM) were not significantly different from one another ($p \geq 0.1$). The tape samples were, however, significantly different from the drywall samples with respect to the mean asbestos concentrations ($p = 0.01$). There was no clear trend with respect to orientation.

As noted above, linear regression analysis was used to explore the value that tape sampling had for predicting the surface concentration of asbestos-contaminated drywall surface. None of the regression analyses produced statistically significant regression lines (p values of 0.652 to 0.115). The amount of variation explained by the regression lines (i.e., R-sq values) ranged from 0.0 to 6.9 percent. As in the case of the quality control samples, examination of the data revealed that the drywall data set contained what appeared to be several data points that were outliers, the greatest of which was a sample with a concentration of 1552.6 f/mm², or 3.58 standard deviations from the mean. The corresponding concentrations ≥3.0 μm for the tape samples for this triplet were 131.6 and 59.2 f/mm² for SEM and PLM, respectively. This observation, in conjunction with the amount of variability observed in the drywall counts, indicated that the disparity was probably due to surface variability and not necessarily to variability from the sample methods. Figure 1 represents a typical plot of the tape fiber concentrations versus the drywall fiber concentrations. After the data triplet referred to above was eliminated from the analysis (data point in lower right corner of plot), the regression equation was found to be statistically significant [ln drywall-SEM concentration = 3.65 + 0.382(lntape-PLM concentration); $p = 0.045$; R-sq = 17.8%].

When the tape data were further explored by dichotomizing the concentrations into high and low categories (i.e., treating them as qualitative indicators of drywall concentrations rather than quantitative indicators), none of the tape sets proved to be statistically significant indicators. Table 3 summarizes the tape sample predictions of drywall asbestos fiber concentrations. The tape samples predicted averages from 6.8 to 37.9 percent of the concentrations measured directly from the drywall surface. This estimate of the sampling percent recovery (percent

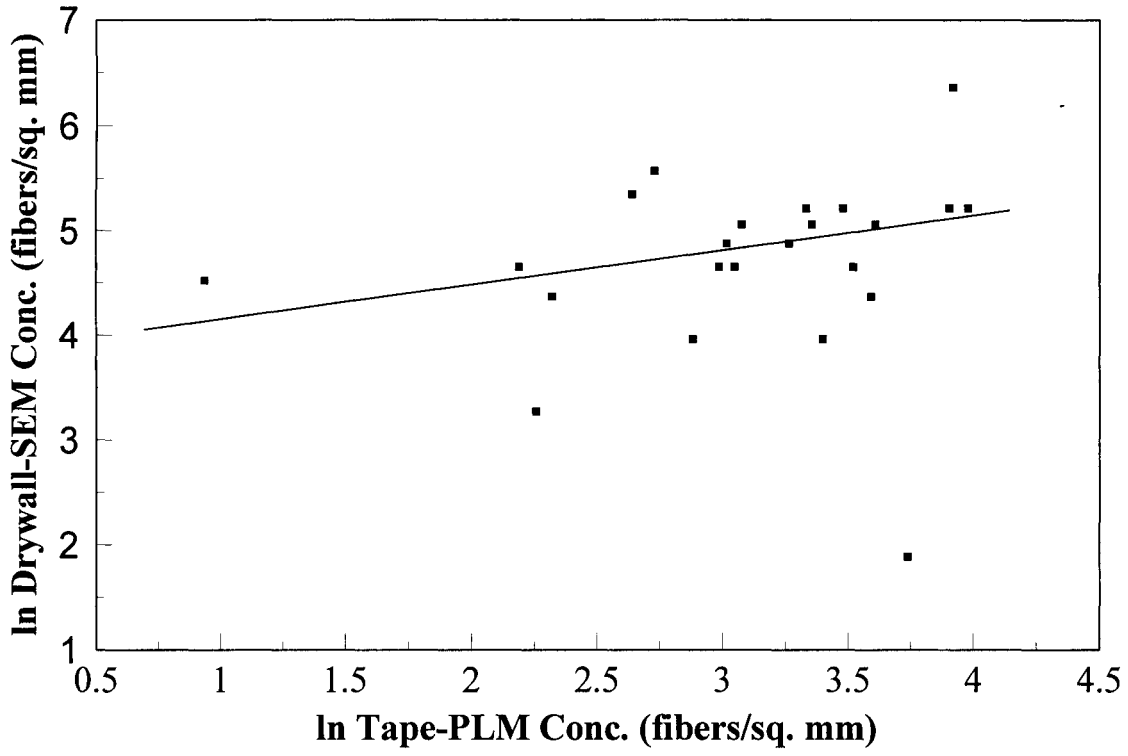

FIGURE 1. Correlation between concentrations of asbestos on tape samples (PLM) and drywall samples (SEM). ln drywall-SEM concentration = 4.11 + 0.191(ln tape-PLM concentration); R^2 = 2.3 percent; p = 0.475; n = 24.

removed) is consistent with previously reported work. Lichtenwalner[12] reported a 55 percent removal rate for repeat wipe samples of metals, while Chavalitnitikul and Levin[13] reported an 85 percent removal rate of lead oxide on a smooth Formica surface and 37 percent on a rough surface, plywood.

Table 4 summarizes descriptive statistics for the asbestos fiber sizes observed. No fibers thinner than 0.1 μm were observed in the SEM, while no fibers thinner than 0.75 μm were observed with PLM. Middleton[7] reported the practical limit of resolution for SEM when counting asbestos fibers to be approximately 0.1 μm. For light microscopy, NIOSH has reported that 0.3 μm is the lower limit of resolution for PCM.[6] Based on these reports, our observations that 0.1-μm fibers were the thinnest fibers that could be observed for SEM and 0.75-μm fibers for PLM appear to be reasonable estimates of the limits of resolution for asbestos counting on tape samples.

It was observed during PLM analysis that fibers within a counting field were contained within the same depth of field plane; however, fibers in different counting fields were frequently observed in different planes (depths). These observations indicate that surface topography and the pressure used during sampling resulted in the particulate becoming embedded at various depths within the tape mastic. This observation raises a concern for the use of SEM. The electron beam in a scanning electron microscope is not capable of penetrating

TABLE 3. Tape Predictions Versus Drywall Asbestos Surface Concentrations

Sample Type	Sampling Percent Predicted*	Pearson's Correlation (Product Moment)	Coefficient of Variation (%)
Tape-SEM			
3 μm	30.6	0.10	65.4
5 μm	37.9	0.19	101.1
Tape-PLM			
3 μm	6.8	0.33	40.4
5 μm	18.3	0.15	52.7

*Represents the amount recovered from surface and the amount detected by the analytical method.

TABLE 4. Tape Sample Fiber Size Descriptive Statistics

	Width (μm)	Length (μm)	Aspect Ratio
Drywall-SEM			
Geometric mean	0.79	3.53	4.51
Geometric standard deviation	1.75	1.72	1.49
Range	0.1–5.9	1.0–27.7	3.0–41.0
Tape-SEM			
Geometric mean	0.51	3.18	6.20
Geometric standard deviation	2.52	2.36	2.11
Range	0.1–5.9	0.6–35.3	3.0–29.3
Tape-PLM			
Geometric mean	1.12	6.96	6.64
Geometric standard deviation	1.66	2.38	2.11
Range	0.75–2.5	3.0–200	3.0–150

more than approximately 10 μm into a substrate such as the adhesive. According to the 3M Corporation, the average depth of adhesive on their 810 Scotch Brand Tape is 1.0 mils or 27.6 μm.[14] Thus, SEM probably was only detecting the particulate embedded in the top 50 percent of the adhesive.

Conclusions

Not unexpectedly, the concentrations observed on the three tape sample types were significantly different from one another. These differences would not detract from a viable sampling and analytical method as long as significant correlations existed, which would allow accurate predictions. While this evaluation did not have success in demonstrating such correlations, this failure is believed to be due to surface variability produced by the aerosol generation, and not necessarily to the method itself. When outliers were eliminated from the data sets, statistically significant trends began to emerge.

If one could produce a uniform surface concentration, tape sampling might very well demonstrate statistically significant correlations with coefficients of variation similar to those found when fiber counting air samples by PCM (i.e., 20 to 50%). The magnitude of the variation would depend on the number of fibers observed. Overall, tape sampling must be regarded as a qualitative method at this time. If one were successful in validating significant correlations, field use of the method would still pose problems due to the amount of surface (environmental) variability that exists. This, however, becomes a sampling strategy problem as opposed to a limitation of the method itself.

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