Bell Laboratories

subject: ACGIH Asbestos Symposium

date: April 26, 1979

from: Vicki R. Cohen

On April 9, 1979, I attended an ACGIH Symposium on techniques for evaluating airborne asbestos dust. After some opening remarks by David M. Trayer, Chairman ACGIH, the objectives of the program were discussed by James S. Ferguson of NIOSH, the program chairman. The program aims were to discuss the following:

(1) Identification of asbestos; finding a practical, cost-effective technique.

(2) The NIOSH method, an evaluation not identification technique.

(3) Methods of analysis now in use.

(4) Asbestos in schools

(5) Insulators in industry, hair dryers, etc.

Speakers scheduled to speak in the morning included Walter C. McCrone, McCrone Research Inst., Dr. Voelker A. Mohnen and Mr. Roger Cheng S.U.N.Y., Dr. Eric Steel, N.B.S., Willard Dixon, OSHA, Ian Stewart, McCrone, and Steven Bayer and Dick Coupell, NIOSH.

Walter McCrone spoke about optical techniques. He claimed that the NIOSH procedure is good, but must be refined so as to discriminate between asbestos and other fibers. He discussed dispersion staining in detail.

Ian Stewart stressed the importance of crystallographic identification for distinguishing asbestos from nonasbestos material. He claimed that the SEM is not able to detect fibers less than 300Å in width.

Eric Steele of the National Bureau of Standards discussed the use of the analytical electron microscope. With this technique resolution and contrast as well as identification of serpentine from amphibole varieties may be accomplished. Small fiber morphology may be seen using selected area x-ray diffraction (SAED) as well as chemical composition using the electron diffraction pattern (EDS). Although SEM can see and identify most fibers, the polymorphs of the same serpentine mineral (ie., antiguerite, chrysotile), can not be distinguished. SEM is also quite costly, and a standardized technique and practical time problems must be resolved.

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Dick Coupell from NIOSH has developed a spot test for identifying asbestos bulk. The test is actually a screen for the presence of magnesium. To date, there have been no false negatives from 150 samples tested. A copy of this relatively simple screening test is attached.

Willard Dixon, OSHA Analytical Lab, spoke about quick tests which could be done in the field to readily identify whether asbestos is present. Some examples he gave were that fiberglass melts and makes a crinkling noise. Fiberglass is also easier to grind with mortar and pestle than asbestos.

Voelker Mohnen's main topic of discussion was the importance of describing the relationship between the mass of asbestos dust to the fibers present. He stressed that an adequate fiber size distribution relating particle size to mass is of great importance when determining health effects from exposure to airborne fibers. An EPA round robin experiment was conducted in which seven well known laboratories were given the task of analyzing the same air filter sample. Each of the laboratories came up with different results. Mohnen stressed that some means of standardizing asbestos fiber determinations was necessary. If these highly reputable labs showed such disagreement, what kinds of results can we expect from other labs lacking in their expertise.

A panel discussion with the various speakers was held in the afternoon, chaired by Benjamin Levadie, State of Vermont. He began the discussion by asking for an honest definition of asbestos. He also asked the audience to consider which forms of asbestos were most hazardous, serpentine or amphibole. Although amphibole is known to be very hazardous, this variety comprises only 5% of the asbestos which is used. The serpentine variety whose health effects are not entirely understood comprises 95% of the asbestos which is used. Questions then came from the floor concerning the rational for the current aspect ratio used for fiber counting. A suggestion was that all physiologically active fibers ought to be regulated.

Each panel member was then asked to comment on which analytical technique is best and how it should be standardized.

McCrone commented that the problems are not with the methods, but with the analysts. He claimed that an optical crystallographic method was the only way to determine the crystallographic mineralogic species.

Mohnen didn't recommend any technique and wished to leave the standardization of methods to Willard Dixon.

Dixon made the recommendation that standard asbestos fibers for calibration purposes be made available through NBS. He suggested field by field instruction and analysis during counting procedures to standardize the NIOSH method and to employ dispersion staining techniques to verify the crystallographic structure.

Ian Stewart spoke about standardizing techniques for crystallographic identification using the electron microscope.

Steven Sayer, who runs the NIOSH course in phase contast microscopy claimed that he has been training individuals to count asbestos in a uniform manner since 1971. Whether or not this method is best, he has standardized it to the best of his ability.

Jim Ferguson's comment was that one technique can not be used for all purposes. The technique to be used should be dependent upon the type of sample to be analyzed.

No standardized or best method for the identification and evaluation of airborne asbestos was decided upon at this symposium. All the methods discussed had various drawbacks including time, cost, accuracy, and reproducibility.

Several of the attendees I spoke with, however, mentioned that they would be taking the McCrone course in dispersion staining.

In summary, the symposium was informative both in terms of the discussions of the various techniques available for asbestos identification as well as the problems associated with those techniques. However, those problems were not resolved.

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Att.
K² Test

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K^2 Test for Screening Asbestos (Evan Campbell NIOSH)

I. SCOPE AND APPLICATION

This colorimetric test is applicable to the detection of magnesium (II) and iron (II) from asbestos in bulk samples. These samples include sprayed on asbestos as well as ceiling tiles. The K^2 test is simple and can be readily used in the field to screen for the presence or absence of asbestos.

II. PRINCIPLE

The K^2 test is based upon the formation of color complexes with Mg^{+2} and Fe^{+2} released from asbestos. The Mg^{+2} from chrysotile is complexed with p-nitrobenzeneazo-α-naphthol. The Fe^{+2} from crocidolite and amosite is complexed with 1, 10-phenanthroline. A positive test is indicated by the formation of colored complex for Mg^{+2} and/or Fe^{+2}, and it indicates possible presence of asbestos.

III. INTERFERENCES

The K^2 test is a colorimetric spot test for Mg^{+2} and Fe^{+2}, and is not specific for asbestos.

The bulk sample may contain Fe compounds other than asbestos. Without treating the sample as in the Procedure, the color forming reagent is added directly to the sample. If red color forms, Fe is present as an interference and these samples can be washed with water prior to the test to eliminate the interference. In the K^2 test, plaster ($CaSO_4$) interference is removed from the sample prior to the Mg test.

IV. SENSITIVITY, PRECISION, AND ACCURACY

With the glycerine treatment the detection limit is about 2 mg of pure chrysotile per test.

From the total of 70 various field samples tested, 52 samples or 74% were correctly identified by the K^2 test as to whether asbestos was present or absent. Only 18 samples or 26% gave false positive reactions. The K^2 test was 100% accurate in identifying 22 samples that did not contain asbestos. The K^2 test results were verified using the transmission electron microscope. ~3%

V. APPARATUS

1. Teflon dish
2. Microspatula or glass rod
3. Dropping plastic pipet
4. 15 mL disposable plastic beaker
5. 25 mm size 0.8 μm membrane filter
6. 25 mm Swinnex filter holder and gasket
7. 10 mL disposable plastic syringe

VI. REAGENTS (Reagent Grade)

1. Phosphoric acid, concentrated.

2. 10 N sodium hydroxide - Dissovle 40 g of NaOH in 100 mL of water.

3. Mg Reagent - Dissolve 1 mg of p-nitrobenzeneazo-α-naphthol in 100 mL of 2 N NaOH. Age at least a day. This reagent is stable over a month.

4. Hydrofluoric acid - Dilute 20 mL of HF with 20 mL of water. Add 0.6 mL of concentrated HCl.

5. Fe Reagent - Dissolve 2 g of 1,10-phenanthroline in 50 mL of ethanol. This reagent is stable over a month.

6. Glycerine, reagent grade.

7. Double de-ionized water.

VII. PROCEDURE

A: Magnesium Test

1.

a. Place a small portion of sample in a plastic beaker.

b. Add 5 drops of glycerine and mix well with a spatula.

c. Rinse spatula with a small amount of water into the beaker.

d. Filter the sample through the filtration assembly consisting of syringe attached to the Swinnex filter holder loaded with a membrane filter and a gasket.

e. Filter with minimum of 5 washings or about 50 mL of water.

f. Transfer filter to a Teflon dish.

2. Add a drop of H_3PO_4 and mix well by grinding the sample with a spatula.

3. Add 2 drops of 10 N NaOH and mix well.

4. Add 5 drops of Mg Reagent and stir briefly. Note any color change.

5. Add 5 more drops of Mg Reagent and observe the final color.

6. A blue color indicates that chrysotile may be present.

7. No color change with Mg Reagent indicates the absence of chrysotile. Even though amosite contains magnesium, this test does not produce the blue color. The iron test would produce a positive reaction indicating the presence of asbestos.

8. Check for Mg interference as in Section III if positive result is obtained.

B. Iron Test

1. Place a small portion of sample on a Teflon dish.

2. Add a drop of HF solution and mix well.

3. Add 5 drops of Fe Reagent and observe the development of red color.

4. The red color indicates that amosite or crocidolite may be present.

5. Check for iron interference as in Section III.

C. Notes

If both tests give negative responses there is a low probability that any asbestos is present. Once the presence of magnesium and/or iron is indicated, further analysis if needed is done in the laboratory to confirm, identify the type, and quantitate the percentage of asbestos.

VIII. REFERENCE

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