

ETHYL CORPORATION

INTER-OFFICE

To Donald E. Johnson ADDRESS

FROM Robert F. Cope, Jr. ADDRESS

SUBJECT Screening Method for Presence of Asbestos DATE Nov. 6, 1979
in Insulation Material

Two NIOSH employees, Walter S. Kim and Richard E. Kupel, developed a quick and inexpensive test to detect the presence of asbestos in bulk samples of insulation material. A copy was obtained and is attached for possible use in our industrial hygiene program. It will be published soon in the AIHA Journal.

It was developed to test for the presence of magnesium and iron in asbestos of such materials as spray on insulation and ceiling tiles. The reagents applied in the test will change color in the presence of magnesium or iron which are in asbestos and this will indicate a fairly strong possibility that the material contains asbestos.

NIOSH has conducted tests with this method and the following are the results; from a total of 70 field samples tested, 52 (74%) were positive and 18 samples gave a false/positive value. The test was 100 percent accurate in 22 samples of materials which did not contain asbestos (reagents did not change color). No false negatives occurred in 200 sample tests.

Babc
Robert F. Cope, Jr.
Industrial Hygienist

RFC:sbb
Attach.

cc: J.A. Jacoby
W.F. Jordan
P.E. Kennedy
T. Harbourt
T.W. Maples
M.M. Monroe
A. Mulholland
B.P. Pancamo
J.T. Rucker
P.J. Sanders
R.L. Smith
G.L. Ter Haar
L. Simpson
File:Oper.(BR-Asbestos)

ETH 002376

K² Test for Screening Asbestos

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 K² test is simple and can be readily used in the field to screen for the presence or absence of asbestos.

II. PRINCIPLE

The K² 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 K² 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 K² test, plaster (CaSO₄) 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 K² test as to whether asbestos was present or absent. Only 18 samples or 26% gave false positive reactions. The K² test was 100% accurate in identifying 22 samples that did not contain asbestos. The K² test results were verified using the transmission electron microscope.

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 μ m 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 - Dissolve 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

- 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.

Magnesium Test

If plaster is known to be absent, take a portion of sample about the size of a large pea and begin with Step 2.

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

1. Caution: Asbestos Dust. A scriptographic booklet by Channing L. Bete Co., Inc., Greenfield, Mass. Prepared in cooperation with U.S. DHEW, PHS, CDC, National Institute for Occupational Safety and Health, Cincinnati, Ohio (1973).
2. Fritz Feigl: Spot Tests - In Inorganic Analysis. Translated by Ralph E. Oesper. 5th Edition. Elsevier Publishing Company, Amsterdam. (1958) p. 225-227.
3. Walter S. Kim, James W. Carter, II, and Richard E. Kupel: Quick Screening Test for Asbestos (K^2 Test). U.S. DHEW, PHS, CDC, NIOSH, Cincinnati, Ohio. To be published in AIHAJ.

Walter S. Kim

Richard E. Kupel — 573 - 684 - 4219

U.S. Department of Health, Education, and Welfare
Public Health Service
Center for Disease Control
National Institute for Occupational Safety and Health
Division of Physical Sciences and Engineering
Measurement Support Branch
Cincinnati, Ohio 45226

April 1979