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Asbestos can be screened quickly in the field by a two-part test. In this test magnesium is liberated from chrysotile, and a blue-colored complex is formed; or iron is liberated from amosite or crocidolite, and a red-colored complex is formed. Before the acid wash steps were added to the procedure, the presence or absence of asbestos was correctly indicated in 126 out of 174 samples. Forty-eight samples gave false positive reactions, but since there were no false negative reactions, the test was considered practical. After adding the acid wash steps, the chemical test correctly indicated the presence or absence of asbestos in all 41 samples tested. There were no false positive or negative reactions. The results were verified by transmission electron microscopy, X-ray diffraction, or dispersion staining.

Quick screening test for asbestos

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introduction

The three most common types of asbestos are chrysotile ($3\ MgO \cdot 2\ SiO_2 \cdot 2\ H_2O$), amosite ($1.5\ MgO \cdot 5.5\ FeO \cdot 8\ SiO_2 \cdot H_2O$), and crocidolite ($Na_2O \cdot 3\ FeO \cdot Fe_2O_3 \cdot 8\ SiO_2 \cdot H_2O$). Chrysotile accounts for about 90% of all asbestos processed. Amosite is used primarily for heat and pipe insulation, and crocidolite is used to make acid-resistant cement pipe and electric battery cases. Almost 3000 asbestos products are manufactured today; the construction industry uses about two-thirds of all asbestos for soundproofing and fire-proofing.[1]

Because of the dangers to humans from asbestos and the need to be aware of its presence in any environment, the National Institute for Occupational Safety and Health (NIOSH) developed a quick and simple method for on-the-spot testing for asbestos in samples of bulk material. In this test, magnesium or iron is released from asbestos and reacts with color-forming reagents to indicate the possible presence of asbestos.[2] The chemical test can be used directly in the field; and, if the presence of asbestos is indicated by the chemical test, samples should be submitted to the laboratory for more exhaustive confirmation by the use of dispersion staining, transmission electron microscopy (TEM), or equivalent methods.[3-4] X-ray diffraction analysis, for example, has been shown to be feasible for the detection of asbestos in bulk samples and filter samples.[5-8]

apparatus

Teflon dish or white plastic plate
Plastic or glass rod
Plastic squeeze bottle for quick wash-down
Dropping plastic pipet
15 mL disposable plastic beaker
25 mm size 0.8 μm mixed cellulose ester filter
25 mm size polyvinyl chloride filter
25 mm Swinnex filter holder and gasket
10 mL disposable plastic syringe

reagents (reagent grade)

Phosphoric Acid, concentrated
10 N sodium hydroxide –
Dissolve 40 g of NaOH in 100 mL of water
Mg Reagent –
Dissolve 1 mg of p-nitrophenylazo-α-naphthol in 100 mL of 2 N NaOH. Age at least a day. This reagent has a shelf life of 3 months.
Hydrofluoric acid –
Dilute 20 mL of HF with 20 mL of water. Add 0.6 mL of concentrated HCl.
Fe Reagent –
Dissolve 2 g of 1,10-phenanthroline in 50 mL of ethanol. This reagent is stable over 3 months.
Glycerin, reagent grade
Acetic acid, concentrated glacial
Sulfuric acid, concentrated
Double de-ionized water

procedure

magnesium test

1. In those instances where the presence of acoustical plaster or other calcium-bearing materials is suspected, positive reactions will be obtained with the magnesium test. To eliminate the calcium interference, the following procedure is used. Place a portion of the bulk sample, about the size of a large pea, in a plastic beaker. Add a few drops of

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Mg Reagent directly to the sample. If blue color appears, wash the sample with glycerin before the test. Any sample containing plaster is also washed with glycerin.

glycerin wash

a. Place a small portion of sample, about the size of a large pea, in a plastic beaker.
b. Add 5 drops of glycerin and mix well with a plastic rod or spatula.
c. Rinse the plastic rod with a small amount of water into the beaker.
d. Filter the sample through the filtration assembly consisting of a syringe attached to the Swinnex filter holder loaded with a mixed cellulose ester filter and a gasket.
e. Filter with a minimum of 5 washings or about 50 mL of water.
f. Transfer the filter to a Teflon dish for the magnesium test.

2. Add a drop of H_3PO_4 and mix well by grinding the sample with a plastic rod.
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. Samples giving a positive test should be sent to a laboratory for confirmation. If the magnesium test is negative, the sample must be tested for iron.

The color changes from violet to blue if magnesium is present. The blue color thus indicates that chrysotile may be present in the sample. Anthophyllite (7 $MgO \cdot 8\ SiO_2 \cdot H_2O$) and tremolite ($2CaO \cdot 5\ MgO \cdot 8\ SiO_2 \cdot H_2O$) also give the blue color with this magnesium test. No color change with the use of the Mg Reagent indicates the absence of magnesium, and thus the sample may not contain these types of asbestos. Although the presence of 0.1 mg of pure chrysotile gives a faintly positive reaction, 0.2 mg of pure chrysotile gives a definitely positive reaction.

iron test

1. If no blue color develops from the magnesium test, the samples are tested for the presence of iron released from amosite and crocidolite. Place a small portion of the sample, the size of a large pea, on a Teflon dish. Add a few drops of Fe Reagent directly to the sample. If a red color appears, wash the sample with the acetic – sulfuric acid mixture (equal volumes) before the test. Samples containing fiberglass or mineral wool are also washed with the acid mixture.

acid wash

a. Place a small portion of the sample, about the size of a large pea, in a plastic beaker.
b. Add 5 drops of concentrated acetic acid.
c. Add 5 drops of concentrated sulfuric acid.
d. Mix well and ultrasonicate if available for 5 minutes.
e. Filter the sample through a *polyvinyl chloride* filter with a minimum of 5 washings or about 50 mL of water.
f. Transfer the filter to a Teflon dish and proceed with the iron test.

2. Add a frop of HF solution and mix well.
3. Add 5 drops of Fe Reagent and observe the development of red color. As little as 20 μg of ferrous iron produces the dark red color and is considered the lowest limit of detection.
4. The red color indicates that amosite or crocidolite may be present.

negative tests

Even if both parts of the test are negative, additional portions of the sample should be tested to confirm these results. If both parts of the test give negative responses, there is a low probability that any asbestos is present. If on further testing the presence of magnesium or iron, or both, is indicated, laboratory analyses are needed to confirm the presence of asbestos, identify the type, and, if required, quantitate the percentage.

TABLE I
Results of Chemical Tests Compared to Results of Verification Tests

Source	Test	Acid Wash	True Pos.	True Neg.	False Pos.	False Neg.	Verification[b]
Various Field	Mg	no	23	17	10	(2)[a]	TEM
Various Field	Fe	no	5(+2)[a]	5	8	0	TEM
Acoustical Tile	Mg/Fe	no	25	49	30	0	XRD/DS
Field	Mg/Fe	yes	5	8	0	0	TEM
LRC/ NASA	Mg/Fe	yes	8	20	0	0	DS/TEM

[a]Counted as "true positive." Although the magnesium test was negative, these samples contained amosite and gave positive reactions to the iron test.
[b]TEM — transmission electron microscopy
XRD — X-ray diffraction
DS - microscopical dispersion staining

results and discussion

Before the acid wash steps were added to the iron test procedure, the chemical tests were applied to 70 field samples of insulating and building materials collected during many different surveys. To verify the presence or absence of asbestos, these samples were analyzed by TEM. The results of the spot tests confirmed by TEM are presented in Table I.

The number of samples that gave positive reactions to the test and that were confirmed by TEM are listed under "true positive." Samples with negative responses to the test and that TEM confirmed as not containing asbestos are listed under "true negative". The number of samples falling under "false negative" indicates the unreliability of the spot tests.

As shown in Table I, two samples gave false negative results for the magnesium (chrysotile) test. These samples contained amosite with 5% to 15% chrysotile. Both samples gave positive reactions to the iron (amosite and crocidolite) test. Thus, even though the magnesium test was negative, these samples were correctly identified as containing iron (∴ possible presence of amosite or crocidolite — subsequently confirmed by TEM). With the use of the Mg Reagent, the blue color was bleached when equal amounts of chrysotile and amosite were tested. With three times higher concentration of amosite, the blue color of the chrysotile was completely bleached. The presence of chrysotile does not, however, affect the iron test for amosite or crocidolite. Table I lists the overall results from various types of samples. The presence or absence of asbestos ("true positive" and "true negative") was correctly identified by the test in 52 of 70 samples. Eighteen samples gave false positive reactions. The test was 100% accurate in having no false negative reactions.

Of the 10 "false positives" for magnesium, 5 were talc samples, 4 contained mineral wool, and 1 was a ceiling tile. The eight "false positives" for the iron test were the samples that contained mineral wool. These samples were tested before adding the Acid Wash steps to the procedure, and thus the number of false positives would be considerably less if the samples were re-tested by the complete procedure that includes the acid wash steps.

The chemical test without the acid wash was also applied to 104 acoustical plaster and ceiling tile samples from New York City and Indiana schools. These samples were analyzed by X-ray diffraction or dispersion staining before submission for the chemical test. The chemical test results, verified by X-ray diffraction and dispersion staining, are also tabulated in Table I.

From these 104 ceiling tiles, the test without the acid wash steps correctly indicated the presence or absence of asbestos in 74 samples. If these 74 samples, 25 were "true positives" and 49 were "true negatives." Thirty samples gave false positive reactions. The false positive reactions occurred because of other magnesium and iron compounds present in the samples. Since glycerine treatment removes soluble magnesium, iron, and plaster, the insoluble compounds interfere with the test by giving false positive reactions. The test was 100% correct in identifying samples that contained more than 1% asbestos, which is the EPA lower limit for asbestos in bulk material.

After adding the acid wash steps to the iron test, 13 field samples were tested by the complete procedure. The results of the chemical test were confirmed by TEM, and are presented in Table I. The presence or absence of asbestos was correctly indicated without any false results.

The complete chemical test was applied in an asbestos survey conducted at the Lewis Research Center of the National Aeronautics and Space Administration (NASA). Various samples were collected from building materials, such as ceiling tile, ceiling and pipe insulation, concrete, and plaster. The results of the chemical test were compared against the results of dispersion staining, and presented in Table I. Of the 28 samples, 5 were also analyzed by transmission electron microscope for confirmation of both results. No false positive or negative result was obtained.

summary and recommendations

For the 174 samples tested without the acid wash steps, the test correctly indicated that 126 contained or did not contain asbestos. Forty-eight samples that did not contain asbestos were indicated as containing asbestos, and no samples containing asbestos were indicated as not containing asbestos.

With the addition of the acid wash steps to the procedure, the false positive results were eliminated for the building material samples. For a total of 41 samples, the chemical test correctly indicated that all 41 samples contained or did not contain asbestos. No false positives were obtained, and non-asbestos samples were indicated as not containing asbestos.

The magnesium and iron chemical tests can screen samples of bulk materials quickly and simply for the possible presence or definite absence of asbestos. Thus industrial hygienists can use this field method for determining the presence or absence of asbestos material quickly before conducting an environmental survey, particularly for screening asbestos in the materials used for soundproofing and fireproofing in schools, office buildings, etc.

This test is recommended for screening use in determining compliance with the EPA lower limit of 1% asbestos in bulk material and in any situations where a quick screening test is needed to determine the presence of asbestos in bulk material to preclude unnecessary expensive laboratory testing for asbestos.

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