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PLAINTIFF'S EXHIBIT

DOW-1025

ANALYTICAL METHOD

September 30, 1968 Method No. TC-AM-68-15

DETERMINATION OF ACID-SOLUBLE IRON IN ASBESTOS

1. Scope

This method is suitable for the determination of 0.2% or more of acid-soluble iron in asbestos.

2. Principle

Iron is solubilized by digestion of the asbestos with hot hydrochloric acid. The iron is reduced with stannous chloride and then measured by titration with standard potassium dichromate.

3. Safety Precautions

Strong mineral acids are used in this procedure. Care should be exercised to prevent spilling of the acids on the skin or clothing. If this occurs, wash the affected areas immediately with large quantities of water. The eyes should be protected with safety glasses or chemical goggles.

4. Reagents

(a) Hydrochloric acid, ACS reagent.
(b) Stannous chloride solution. Weigh 6 grams of the dihydrate ($SnCl_2 \cdot 2H_2O$) into a 250 ml beaker and add approximately 60 ml concentrated hydrochloric acid. Heat gently for solution, and then dilute to approximately 100 ml with water. Store in an amber bottle.

(c) Mercuric chloride, saturated aqueous solution. Add approximately 10 grams of reagent grade $HgCl_2$ to a glass bottle for each 100 ml solution required. Add distilled water and shake the solution vigorously for approximately one minute.

Iron analyses, detn of, in asbestos

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(d) Diphenylamine indicator solution, 1% dissolved in concentrated sulfuric acid. Alternate preparation: To one gram of barium diphenylamine sulfonate in 100 ml water, add approximately 0.5 grams sodium sulfate. Acidify the solution with dilute sulfuric acid and heat gently on the hot plate. Stir occasionally to aid precipitation. Filter the hot solution through a close-textured filter paper catching the filtrate in an amber dropper bottle. Discard the paper and precipitate.

(e) Sulfuric-phosphoric acid mixture. While stirring, add 150 ml concentrated ACS reagent sulfuric acid to 500 ml cold distilled water. Follow this by adding, while stirring, 150 ml concentrated phosphoric acid. Dilute the mixture to approximately 1,000 ml with distilled water.

(f) Potassium dichromate, 0.1N solution. Weigh 4.904 grams of dried $K_2Cr_2O_7$. Dissolve in distilled water and dilute to one liter in a liter volumetric flask.

Standardize the solution as follows: Weigh to the nearest 0.1 mg approximately 0.2 grams high purity iron wire in a 400 ml beaker and add 20 ml concentrated hydrochloric acid. Attach cover glass and heat the solution gently until the iron is dissolved. Complete the standardization according to the following procedure beginning with the addition of stannous chloride and continuing throughout the procedure.

5. Procedure

(a) Weigh approximately 2 grams of the asbestos to three decimal places on the analytical balance and transfer to 400 ml beakers. Add 100 ml distilled water and 50 ml concentrated hydrochloric acid. Add cover glasses and boiling chips to the beakers and place on the hot plate. Boil gently for a minimum of one hour, adding more water as needed to maintain the volume essentially constant.

(b) To the hot solutions, add while stirring stannous chloride drop by drop until the solutions are colorless. Now add 3 to 5 drops in excess. Place the solutions in an ice bath for cooling. When cold, quickly add 20 ml saturated mercuric chloride solution while stirring vigorously (note 7a). Continue stirring for approximately 30 seconds. Place the beakers in the cold bath for 5 to 10 minutes.

(c) Dilute the solutions to approximately 250 ml with distilled water and add 15 ml of the sulfuric acid-phosphoric acid mixture. Add about 10 drops of the diphenylamine indicator solution. While stirring, titrate with 0.1N potassium dichromate until a permanent dark blue color is reached. This color should persist for 30 seconds.

6. Calculations

$$\text{Normality of } K_2Cr_2O_7 = \frac{\text{grams of pure iron taken}}{\text{ml titrant} \times 0.05585}$$

$$\% \text{ Iron (Fe)} = \frac{\text{ml} \times \text{Normality of } K_2Cr_2O_7 \times 5.585}{\text{grams sample}}$$

$$\% \text{ Iron Oxide } (Fe_2O_3) = \% \text{ Iron (Fe)} \times 1.43$$

7. Notes

A white feathery precipitate should appear at this point. If no precipitate is evident, not enough stannous chloride was added; if a dark precipitate appears, too much stannous chloride was added. If either of the latter two situations develops, the test must be repeated using a greater or lesser quantity of the stannous chloride.

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