PLAINTIFF'S EXHIBIT

UC-2620

Report of Investigations 8452

Chemical and Physical Characterization of Amosite, Chrysotile, Crocidolite, and Nonfibrous Tremolite for Oral Ingestion Studies by the National Institute of Environmental Health Sciences

By William J. Campbell, Charles W. Huggins, and Ann G. Wylie

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UNITED STATES DEPARTMENT OF THE INTERIOR
Cecil D. Andrus, Secretary

BUREAU OF MINES
Lindsay D. Norman, Acting Director

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CHEMICAL AND PHYSICAL CHARACTERIZATION OF AMOSITE, CHRYSOTILE, CROCIDOLITE, AND NONFIBROUS TREMOLITE FOR ORAL INGESTION STUDIES BY THE NATIONAL INSTITUTE OF ENVIRONMENTAL HEALTH SCIENCES

by

William J. Campbell,[1] Charles W. Huggins,[2] and Ann G. Wylie[3]

ABSTRACT

The Federal Bureau of Mines provided five test materials--amosite, short- and long-fiber chrysotile, crocidolite, and nonfibrous tremolite--to the National Institute of Environmental Health Sciences (NIEHS) for studies on the biological effects of orally ingested asbestos-related materials. An interagency agreement between the Bureau and NIEHS made possible this program to characterize these test materials. The Bureau in turn received support through contracts with the Ontario Research Foundation for the milling of amosite and crocidolite, and with the University of Maryland for microscopic measurements.

The five test materials, which were supplied in quantities of 960 to 1,200 pounds each, were characterized by chemical-instrumental analysis for elemental composition, surface area, density, X-ray diffraction, infrared spectroscopy, thermogravimetric analysis, mineralogy, and particle size analyses. Particle size distributions were emphasized because of the current theories relating adverse biological effects to length-width characteristics of durable elongated particles.

INTRODUCTION

Although there has been increased concern in the past 2 decades about the pathogenic effects of inhaled asbestos, relatively little attention has been given to the effects of ingested asbestos. In November 1973, a conference, cosponsored by the National Institute of Environmental Health Sciences (NIEHS) and the Environmental Protection Agency (EPA), was held in Durham, N.C., on

[1]Program coordinator, Particulate Mineralogy Unit, Avondale Research Center, Bureau of Mines, Avondale, Md.

[2]Morphology coordinator, Particulate Mineralogy Unit, Avondale Research Center, Bureau of Mines, Avondale, Md.

[3]Associate Professor, Department of Geology, University of Maryland, College Park, Md.

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the biological effects of orally ingested asbestos (5).[4] Following the conference, the Department of Health, Education, and Welfare Committee to Coordinate Toxicology and Related Programs formed a subcommittee to review data and to recommend future research needs. The subcommittee concluded that there were not sufficient data and proposed that specific studies be undertaken. Based on extensive reviews and comments on their research protocol, NIEHS, with EPA support, developed research contracts to study the effects of orally ingested asbestos in rats at the Hazleton Research Laboratories in Vienna, Va., and in hamsters at the Illinois Institute of Technology Research Institute (IITRI) in Chicago, Ill.

The data summarized in this report were obtained under interagency agreement 10-09-0070-598 between NIEHS and the Federal Bureau of Mines. The objective of the amended agreement was to provide and characterize chrysotile of long- and short-fiber lengths, amosite, crocidolite, and tremolite for the oral ingestion studies. The Bureau of Mines contracted in 1976 with the Ontario Research Foundation (ORF), Mississauga, Ontario, Canada, for the milling of the amosite and crocidolite, and with the Department of Geology, University of Maryland, for size distribution measurements by optical and electron microscopy. The Bureau provided the five test samples in quantities of approximately 1,000 pounds each to Dr. John Moore, NIEHS, Research Triangle Park, N.C.

Characterization data summarized in this report include chemical-instrumental analyses for major, minor, and trace constituents; X-ray diffraction; density; surface area; infrared spectrophotometry; differential thermal and thermogravimetric analyses; mineralogy; and particle size distribution. Many health scientists believe that particle size and shape is the dominant factor producing tumors in test animals; therefore, size-shape parameters are emphasized.

The Fine Particle Laboratory, IITRI, provided NIEHS with characterization data on the five samples. Questions regarding the IITRI characterization data and availability of the test materials should be directed to Dr. Moore.

ACKNOWLEDGMENTS

This interagency program was conducted under the coordination of Dr. Moore, NIEHS, whose advice and guidance are gratefully acknowledged. Sarkis G. Ampian and Robert A. Clifton, Jr., Division of Nonmetallic Minerals, Bureau of Mines, Washington, D.C., provided invaluable technical advice in selecting test materials; guidance in processing of amosite, crocidolite, and tremolite samples; and general assistance throughout the program. R. T. Vanderbilt Company, Inc., in Norwalk, Conn., supplied the milled tremolite sample, through arrangements by C. S. Thompson and Allan M. Harvey. William Reed and Gene Deardorff of the Office of Standards Reference Materials, National Bureau of Standards (NBS), Washington, D.C., blended the tremolite.

[4]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

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The Ontario Research Foundation (ORF) supplied chemical and size characteristics data throughout the milling program, and the assistance of R. W. Glass, D. R. Hollingbery, B. J. Ferguson, and E. Brown, all of ORF, is gratefully acknowledged. Numerous analytical personnel assigned to Avondale Research Center and student assistants from the Department of Geology, University of Maryland, provided most of the data summarized in this report. Their assistance is acknowledged in the sections of the report to which they contributed.

SAMPLES

Tremolite

Twelve hundred pounds of nonfibrous tremolite, containing approximately 25 percent serpentine plus minor amounts of talc and other minerals, was collected from veins at two levels in the Vanderbilt mine, Balmat, N.Y. This sample was milled by Vanderbilt to minus 325 mesh in a Wheeler mill.[5] Because of slight variations in the mineralogy of the samples at the two levels in the mines, the sample was blended, using the NBS sample preparation facilities. Blending was performed in a 10-cu-ft V blender (fig. 1), but because of the limited capacity of the blender, approximately 960 pounds was blended, using a four-stage blending procedure. The blended tremolite was repackaged in 38 virgin cardboard drums, containing about 25 pounds each. Homogeneity of the samples was verified by fluorescent X-ray spectrography for samples collected from six randomly selected drums, which were being filled for shipment to NIEHS. No significant differences were detected by the various characterization procedures.

Chrysotile

Two chrysotile test materials were purchased in quantities of approximately 1,000 pounds each. The short-fiber chrysotile, identified as COF-25, was purchased from the Union Carbide Corporation, Niagara Falls, N.Y. This sample was obtained from the New Idria serpentinite mass located in the southern part of the Diablo Range in southwestern San Benito and western Fresno Counties of California. The ore body is unusual because of the virtual absence of long fibers and the high chrysotile content of the ore. To check for homogeneity, samples of COF-25 were randomly selected from nine drums of the material shipped to NIEHS. No significant differences were detected in the nine lots by the various characterization procedures.

A broader range fiber-length test material, identified as Plastibest-20, was obtained from the Johns-Manville Jeffrey mine, Asbestos, Quebec, Canada. Plastibest-20 is a particularly clean grade of chrysotile used by the plastics industry. To check for homogeneity, samples of Plastibest-20 were collected from 10 drums of the test material supplied to NIEHS. No measurable differences in composition were observed, using the various characterization procedures.

[5]Reference to specific trade names or equipment does not imply endorsement by the Bureau of Mines.

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FIGURE 1. - Blending the milled tremolite in the National Bureau of Standards 10-cu-ft V blender.

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Amosite and Crocidolite

The Bureau purchased 4,000 pounds of amosite, identified as S-33, from the Atlas Asbestos Company, Montreal, Canada. This material is from a mine in the area known as Penge, in the Transvaal, South Africa. Not a proper mineral name, amosite is a term used to describe the material from Asbestos Mines of South Africa. It is composed primarily of grunerite asbestos, with a minor amount of actinolite asbestos. A previous Bureau publication gives a detailed discussion of habit descriptions for amphibole minerals (1).

Six thousand pounds of crocidolite, identified as ML-6 was purchased by the Bureau from Certain-Teed Company, Ambler, Pa. This sample was obtained from a mine in South Africa located near the Kalahari desert in the Kuruman Hills.

Air Jet Milling of Amosite and Crocidolite

A contract for air jet milling of the amosite and crocidolite was established in 1976 between the Bureau and ORF. Air jet milling was selected because this type of pulverizer would damage the amphibole crystalline structure the least.

A Jet-O-Mizer 0304F air jet mill and collection system was purchased from Fluid Energy and Equipment Company, Hatfield, Pa. The collection system was a 10-inch high-efficiency Jet-O-Clone cyclone collector constructed of 304 stainless steel, with duct work connecting the mill to a continuous-pulsing dust bag collector composed of 304 stainless steel structural parts and silicone-treated Dacron filter bags.

A blending table, approximately 5 by 8 feet, was used for simultaneous manual blending of six 100-pound bags of asbestos. The table was enclosed on three sides above the table surface, and a hood was positioned above the table. The rotary feeder, supplied by Fluid Energy, was capable of feed rates up to 130 pounds per hour.

The major safety considerations were to minimize exposure of fibers to operating personnel, and to contain all fibers within the processing area. The processing facilities and personnel equipment are discussed in detail in periodic reports provided by ORF to the Bureau.[6] Each operator wore a Scott air-pak safety mask with filtered breathing air supplied from an external source (fig. 2).

During the initial milling of the amosite fibers, severe erosion of the milling chamber occurred because of the highly abrasive action of the amosite, resulting in failure of the milling chamber (fig. 3), together with unacceptable contamination of the amosite with chromium and nickel from the stellite used for hard facing. Materials milled using the stellite hard facing were subsequently discarded.

[6]Inquiries about the ORF reports should be directed to W. J. Campbell.

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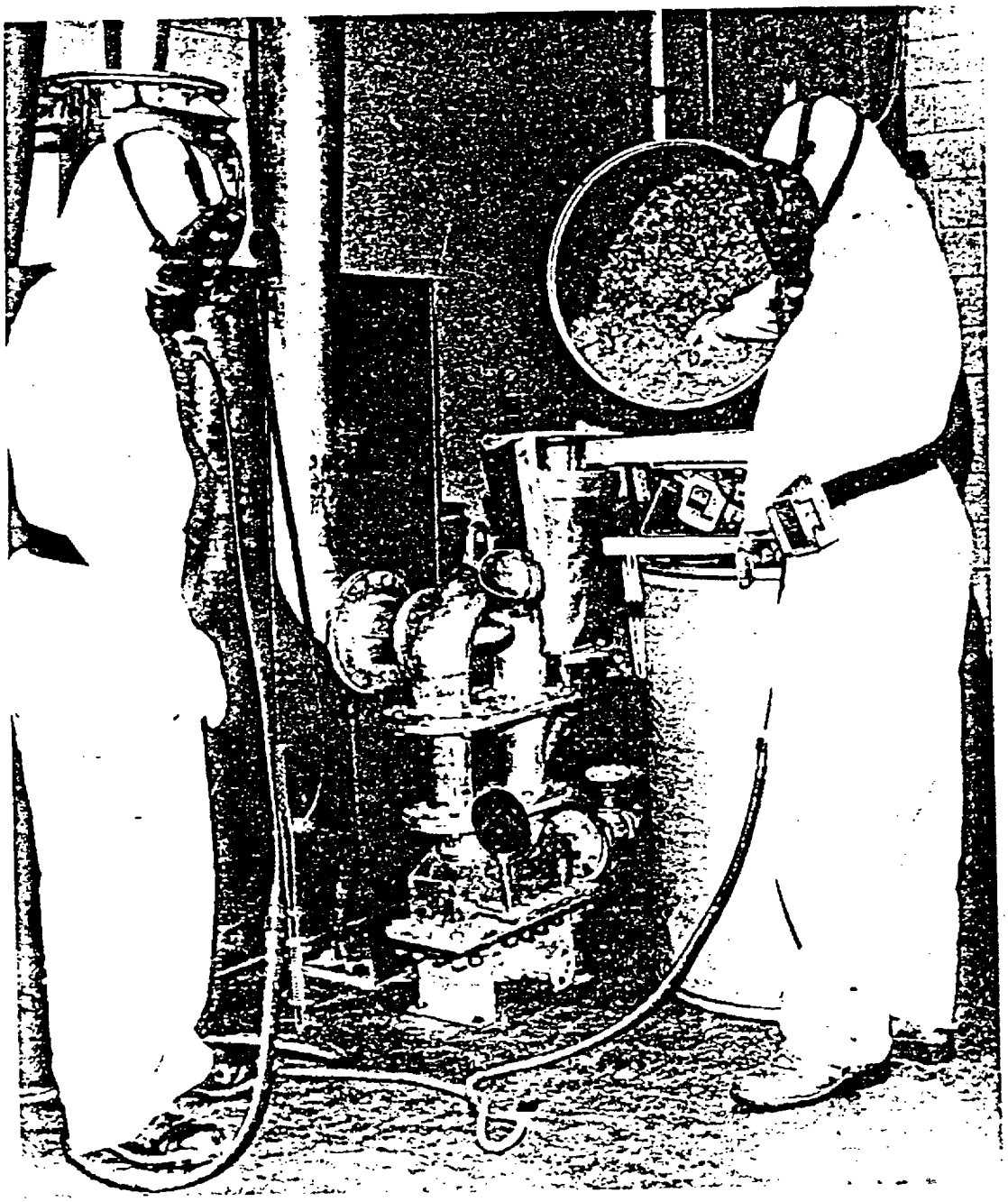


FIGURE 2. - Air jet milling at Ontario Research Foundation.

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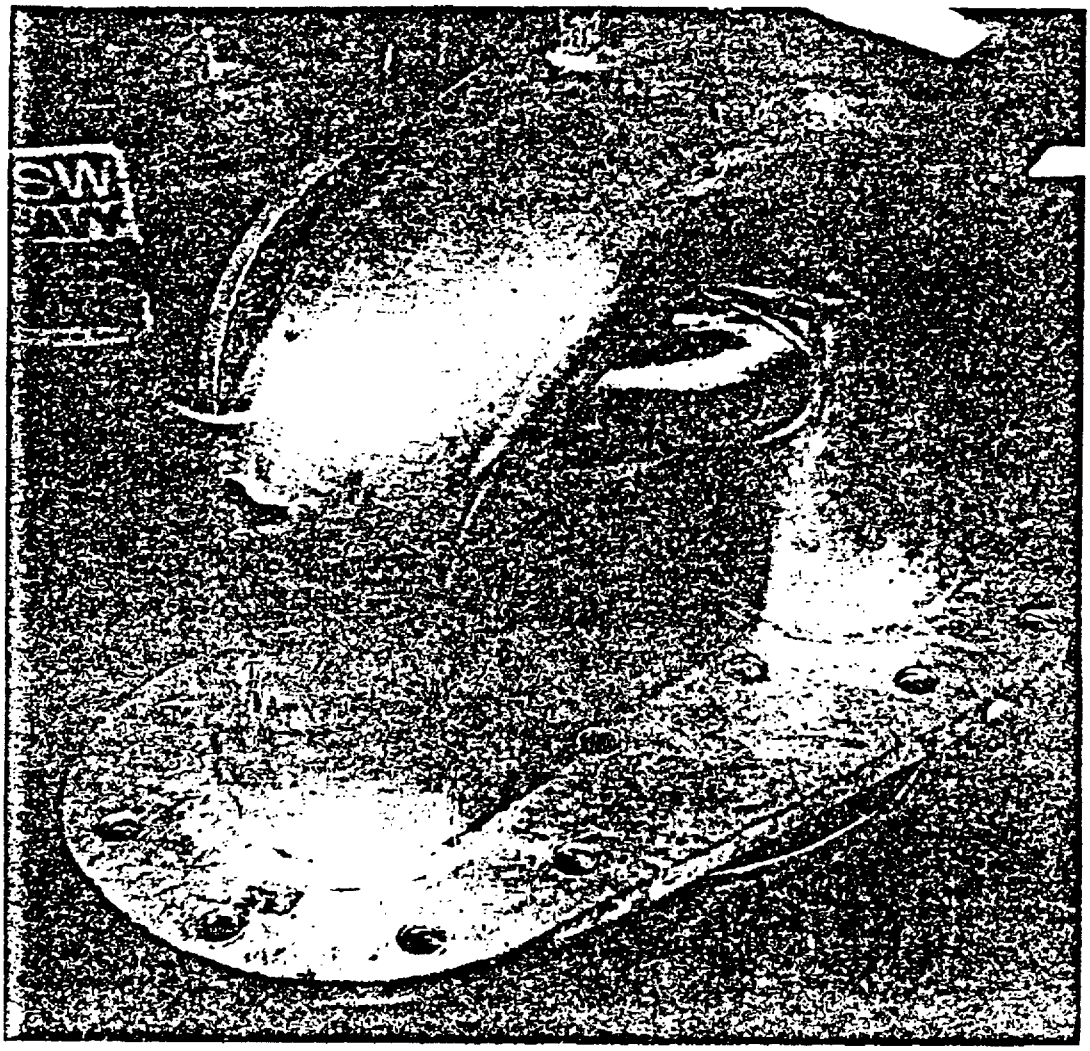


FIGURE 3. - Erosion of hard-surfaced stainless steel as a result of air jet milling of amosite.

To reduce contamination, CRF applied facing composed of a low-carbon, low-chromium-containing steel. Because of continuing erosion of the hard facing, it was necessary to dismantle and inspect the mill at regular intervals and to reface the worn areas as necessary. Crocidolite was passed through the mill twice to reduce the number of very long fibers in the final material. Amosite was processed by a single pass.

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Nickel contamination of milled amosite and crocidolite was insignificant when a facing of low-carbon steel was used; however, the chromium content increased from approximately 90 to approximately 170 ppm in both types of milled asbestos. Therefore, the chromium in the milled samples is present mineralogically and as a metallic contaminant.

Twelve hundred pounds each of milled amosite and crocidolite was packaged as 20-pound lots in virgin fiberboard containers and shipped to NIEHS. Random samples were selected from various drums for homogeneity tests and for characterization by the Bureau. No significant differences were noted for samples from various drums using the characterization techniques.

ELEMENTAL ANALYSIS

Optical Emission Spectrography[7]

Dilutions of 1:1, 1:9, and 1:49 were made for semiquantitative determinations on the five NIEHS test materials. The samples were prepared as follows:

1:1—One part of sample plus one part of high-purity graphite.
1:9—One part of the 1:1 dilution and four parts of graphite.
1:49—One part of the 1:9 dilution and four parts of graphite.

Fifteen and twenty milligrams of each diluted sample were then excited by direct current arc, using graphite electrodes in a prism and grating spectrograph, respectively. SA #1 plates were used in the grating spectrograph to record the spectra from 2400 to 3400 A, and 1-N plates were used in the prism spectrograph for spectra from 3400 to 9000 A. An argon-oxygen atmosphere was used to eliminate interference from CN bands in the prism spectrograph. Excitation conditions in both spectrographs were 8 amp for 60 sec, and then 15 to 17 amp for an additional 30 to 60 sec. The plates were racked at the end of the first 60 sec to detect the more volatile elements, then processed according to American Society for Testing and Materials E-115 specifications. Sample spectra were then compared with spectra on master plates prepared under identical conditions using various concentrations of 50 elements. Semiquantitative results for 22 elements are given in table 1.

[7]Optical spectrographic analyses by James B. Zink, chemist, Avondale Research Center, Avondale, Md.

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TABLE 1. - Semiquantitative optical emission spectrographic analyses of NIEHS test materials

Element	Concentration range, weight-percent				
	Amosite	Crocidolite	Chrysotile COF-25	Chrysotile Plastibest-20	Tremolite[1]
Iron	>10	>10	0.3 -3	1 -10	0.1 -1
Silicon	>10	>10	>10	>10	>10
Magnesium	1 -10	1 -10	>10	>10	>10
Calcium	.03 - .3	.3 - 3	.1 -1	.1 - 1	>5
Aluminum	.03 - .3	.03 - .3	.1 -1	.1 - 1	.1 -1
Chromium	.003 - .03	.01 - .1	.03 - .3	.03 - .3	ND
Copper	.001 - .01	.003- .03	.0003- .003	.0003- .003	.0003- .003
Manganese	.3 - 3	.03 - .3	.03 - .3	.03 - .3	.03 - .3
Nickel	.001 - .01	.001- .01	.03 - .3	.03 - .3	.0003- .003
Lead	.003 - .03	.01 - .1	ND	ND	.0003- .003
Tin	.0003- .003	ND	ND	ND	ND
Titanium	.003 - .03	.01 - .1	.0003- .003	.003 - .03	.003 - .03
Potassium	.03 - .3	.03 - .3	ND	.1 - 1	.003 - .03
Lithium	ND	.003- .03	ND	ND	.003 - .03
Sodium	.01 - .1	1 -10	.01 - .1	.1 - 1	.1 -1
Silver	.0001- .001	ND	ND	.0001- .001	ND
Cobalt	ND	ND	.003 - .03	.003 - .03	ND
Vanadium	ND	ND	.003 - .03	.003 - .03	.001 - .01
Boron	ND	ND	.0003- .003	.0003- .003	.0003- .003
Molybdenum	ND	ND	.0003- .003	ND	ND
Strontium	ND	ND	.0003- .003	.003 - .03	.03 - .3
Barium	ND	ND	ND	ND	.001 - .01

ND Indicates below limit of detection.
[1]Contains ~25 percent serpentine, talc, and other minerals.

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Chemical-Instrumental Analysis[8]

Major Elements

Major constituents were determined by the well-established silicate rock procedure described by Jeffery (7) in which silica is determined gravimetrically. Ammonia is used to precipitate the R_2O_3 group, followed by determination of calcium, magnesium, aluminum, and iron. Atomic absorption was used for iron concentrations of <2 percent, dichromate titration on a separate sample for >2 percent iron, and ferrous iron by titration also on a separate sample.

To check the accuracy of the determinations, samples of the U.S. Geological Survey W-1 standard dibasic rock (8) were analyzed with the test materials. A comparison between the published values and Bureau data is summarized in table 2. The Bureau values represent the range of eight individual determinations. Concentrations of the major elements in the five test materials are summarized in table 3.

TABLE 2. - Comparison of measured and accepted values for major elements in USGS standard dibasic rock W-1

Element	Concentration range, weight-percent	
	Accepted	Bureau of Mines
Aluminum.....	7.66- 8.28	7.91- 7.97
Calcium.....	7.71- 7.95	7.66- 7.84
Iron.....	7.57- 7.95	7.53- 7.85
Magnesium.....	3.78- 4.20	3.96- 4.12
Silicon.....	24.34-24.66	24.03-24.41
Titanium.....	.52- .76	.69- .75

Minor and Trace Elements

Minor elements were determined by a chemical dissolution-atomic absorption procedure. A 1-gram sample was decomposed by a $HF-HNO_3-HClO_4$ fusion, followed by dissolution of the essentially silica-free products in a dilute $HCl-HNO_3$ solution. The minor and trace elements were determined in the dilute acid solution by atomic absorption spectrophotometry. Two potential sources of analytical error, incomplete dissolution and contamination of the analytical solution, were minimized by adherence to accepted analytical practices. Reliability of the combined dissolution-atomic absorption procedure was checked, using the NBS Fly Ash SRM 1633 (table 4). A sample of the fly ash

[8]Chemical instrumental analyses were provided by R. C. Gabler, Jr., research chemist; R. L. Stoll and R. P. Lambert, student trainees in Chemical Engineering; and Louis Reichel, student trainee in Chemistry, Avondale Research Center, Avondale, Md.

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standard was analyzed with each set of test materials so that the ranges represent 5 to 10 individual analyses. Concentrations of trace and minor elements in the five NIEHS test materials are shown in table 3.

TABLE 3. - Chemical-instrumental analyses of NIEHS test materials

Oxides	Weight-percent				
	Amosite	Crocidolite	Chrysotile COF-25	Chrysotile Plastibest-20	Tremolite[1]
Al_2O_3.....	0.42	0.07	0.66	1.47	1.57
CaO.....	.48	1.04	.32	.05	11.26
FeO.....	34.61	18.32	ND	ND	ND
Fe_2O_3.....	2.24	18.62	2.02	2.93	.27
MgO.....	6.22	3.81	40.62	40.26	26.71
K_2O.....	.30	.07	ND	.08	.18
SiO_2.....	50.36	48.73	39.77	39.90	54.00
Na_2O.....	.03	4.43	.01	.04	.80
TiO_2.....	ND	.07	.03	.04	.03
Li_2O.....	ND	ND	ND	ND	.02
MnO.....	2.66	.08	.07	.06	.05
SnO.....	ND	ND	ND	ND	.01
SrO.....	ND	ND	ND	ND	.03
Bi_2O_3.....	ND	ND	ND	ND	.01
Cr_2O_3.....	.03	.02	.17	.06	ND
NiO.....	.01	ND	.17	.06	ND
Co_2O_3.....	ND	ND	.01	ND	ND
CO_2.....	.88	2.18	.78	.51	.78
H_2O^-.....	.15	.45	1.54	1.17	.24
H_2O^+.....	[2]2.30	2.10	12.69	12.81	3.73
Total	100.69	99.99	98.86	99.44	99.69

ND Not detected.
[1]Contains ~25 percent serpentine, talc, and other minerals.
[2]Corrected for structural water converted to hydrogen as a result of auto-oxidation of ferrous to ferric iron.

TABLE 4. - Comparison of measured and certified values for NBS Fly Ash SRM 1633

Element	Concentration range, ppm	
	Certified	Bureau of Mines
Barium.....	2,270- 3,070	2,500- 2,660
Bismuth....	47- 75	39- 58
Chromium...	129- 133	108- 140
Cobalt.....	38- 46	48- 64
Copper.....	123- 133	116- 140
Lead.....	66- 74	72- 87
Manganese..	486- 500	450- 510
Nickel.....	95- 101	90- 105
Potassium..	15,000-19,000	14,000-20,000
Zinc.....	190- 230	190- 210

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Water and Carbon Dioxide[9]

Water and carbon dioxide were determined gravimetrically using absorption tubes with a thermogravimetric analyzer. (See Thermographic Analysis section for details.) The inert carrier gas was purified by serial passage through (1) copper wire heated to 600° C, (2) a tube filled with ascarite (NaOH + asbestos), (3) an absorption tube filled with anhydrone ($MgClO_4$), and (4) a glass wool filter. Carbon dioxide and water released from the sample during the heating cycle, together with precleaned carrier gas, are passed through tubes filled with anhydrone and ascarite to collect the water and carbon dioxide, respectively. Two parallel sets of these absorption tubes were used: one set collected the water and carbon dioxide released from room temperature to 110° C; and the other set was for the temperature range 110° to 1,100° C. The analytical system also included backup tubes, filled with ascarite and anhydrone to collect any carbon dioxide and water that passed through the primary collectors.

The carbon dioxide and water values given in table 3 are from room temperature to 1,100° C. There were no significant differences in the values for carbon dioxide and water (+) over the range from room temperature to 1,100° C, compared with 110° to 1,100° C range. Weight loss data from the thermogravimetric analyzer were in excellent agreement with the absorption values.

SURFACE AREA[10]

Specific surface areas were determined by the flowing gas-B.E.T. method. In this procedure, nitrogen gas is adsorbed by the sample from a stream of nitrogen in helium, then the nitrogen is desorbed from the sample and measured. The quantity of nitrogen adsorbed per unit weight of sample is a function of the available surface area. Measurements were conducted on a Perkin-Elmer 212D Sorptometer and a Quantachrome QS-7 Quantasorb.

Temperature and time period must be adequate for complete degassing of the sample, but temperature must also be accurately selected so as not to alter the sample surface being measured. Tests conducted with the Quantasorb showed significant variations in the measured surface area values when degassing conditions were not carefully controlled. Table 5 summarizes degassing parameters for the five test materials and four surface area standards.

Table 6 compares values obtained with the two surface area instruments with recommended values for four surface area standards. The five NIEHS test materials were run in triplicate from 5 to 10 lots of each material; therefore, the values listed in table 7 represent the average of 15 to 30 independent measurements.

[9]Data provided by R. E. Brown, physical science technician, Avondale Research Center, Avondale, Md.

[10]Data provided by R. C. Gabler, Jr., research chemist; R. L. Stoll and R. P. Lambert, student trainees in Chemical Engineering; and Louis Reichel, student trainee in Chemistry, Avondale Research Center, Avondale, Md.

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TABLE 5. - Degassing parameters for surface area measurements[1]

Sample	Temperature, °C	Time, hr
Materials:		
Amosite	350	8
Chrysotile Plastibest-20	390	8
Chrysotile COF-25	390	8
Crocidolite	420	7
Tremolite	200	8
Standards:		
Aminco anatase	300	6
Aminco bone char	150	16
Aminco silica spheres	105	8
Reno alumina	200	16

[1]Samples degassed under flowing helium for Perkin-Elmer 212D Sorptometer and under 10^{-3} torr vacuum for Quantachrome QS-7 Quantasorb.

TABLE 6. - Comparison of surface area measurements on reference materials

Sample	Surface area, m^2/g		Relative error, percent
	Listed	Found	
Aminco anatase	10.2	[1]8.7 ± 0.4	14.9
		[2]10.3 ± .6	1.0
Aminco bone char	73.6	[1]64.6 ± 2.3	12.2
		[2]53.4 ± 5.0	27.4
Aminco silica spheres	23.6	[1]23.4 ± 2.1	.8
Reno Research Center alumina catalyst	163.4 ± 1.3	[2]159.7 ± 2.5	2.3

[1]Perkin-Elmer 212D Sorptometer.
[2]Quantachrome QS-7 Quantasorb.

TABLE 7. - Surface area values for NIEHS test materials

Sample	Specific surface area, m^2/g	
	Perkin-Elmer	Quantachrome
Tremolite[1]	5.2 ± 0.5	3.7 ± 0.4
Chrysotile COF-25	54.2 ± .9	54.3 ± 3.9
Chrysotile Plastibest-20	24.9 ± 2.2	20.2 ± .1
Amosite	4.0 ± .1	3.6 ± .3
Crocidolite	9.8 ± 1.0	10.4 ± .9

[1]Contains ~25 percent serpentine, talc, and other minerals.

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DENSITY[11]

Densities of the five test materials were determined by pycnometric measurements; ethanol was used as the liquid medium because its wetting characteristics are superior to those of water. Sample size ranged from approximately 0.5 to 1 gram for amosite, chrysotile (Plastibest-20), and crocidolite, to 2.5 grams for chrysotile (COF-25) and tremolite. Particle size and shape of the test materials were the critical parameters in determining the weight of sample placed in the 50-ml pycnometers.

Samples were dried for 1 hour at 115° C to remove adsorbed moisture. The pycnometers containing the test materials were placed under slightly negative pressure for several minutes to eliminate entrapped air. All pycnometers, with and without samples, were brought to volume slowly with ethanol, in a constant temperature bath of 20°±0.05°C.

The specific gravity of the samples relative to ethanol was calculated using the following equation:

$$\text{Specific gravity} = \frac{c-a}{(b-a) - (d-c)} \qquad (1)$$

where a = weight of empty pycnometer,
b = weight of pycnometer + ethanol,
c = weight of pycnometer + sample, and
d = weight of pycnometer + sample + ethanol.

The specific gravity values based on ethanol are converted to densities by multiplying by the ratio of densities of ethanol to water at 20° C.

There was a slight weight loss of the ethanol and water (for conversion to density) during the time required to accurately transfer and weight the pycnometers. This source of error was minimized by recording the weight versus time and extrapolating back to time zero. For each set of measurements, a control pycnometer with alcohol only was included to check the density of the alcohol. Absorption of moisture by the alcohol results in a slight increase in the specific gravity.

The reliability of the pycnometer method was checked using powdered silicon and zinc samples; the pycnometer-determined values listed in table 8 are in good agreement with the literature values. Densities of the NIEHS test materials are listed in table 9. The relatively high standard deviation for the crocidolite is possibly related to the large variation in particle size for crocidolite, compared with the other test materials.

[11]Data provided by R. C. Gabler, Jr., research chemist; R. L. Stoll and R. P. Lambert, student trainees in Chemical Engineering; and Louis Reichel, student trainee in Chemistry, Avondale Research Center, Avondale, Md.

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TABLE 8. - Comparison of measured and literature densities for silicon and zinc

Sample	Density, g/cm^3	
	Literature	Measured
Silicon....	2.33 ± 0.01	2.33 ± 0.06
Zinc.....	7.14	7.13 ± .036

TABLE 9. - Densities of NIEHS test materials

Material	Density, g/cm^3	Standard deviation, g/cm^3
Tremolite[1].....	2.907	± 0.010
Chrysotile COF-25.....	2.577	± .022
Chrysotile Plastibest-20..	2.607	± .016
Amosite.....	3.350	± .026
Crocidolite.....	3.32	± .08

[1]Contains ~25 percent serpentine, talc, and other minerals.

BENZENE-EXTRACTED ORGANICS[12]

The organic content of the NIEHS materials was determined by the method described by Gibbs and Hui (4). Twenty to fifty grams of each sample was weighed into extraction thimbles of sintered glass, placed in a Soxhlet extraction apparatus, and extracted with redistilled benzene for 8 hours. The solvent was then distilled to low bulk, transferred to a platinum dish, evaporated to dryness under vacuum, and the residue weighed. Table 10 shows that the extractable organic content of the tremolite sample was significantly lower than the four asbestos samples.

[12]Data provided by W. F. Lowry, supervisory chemist, Avondale Research Center, Avondale, Md.

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TABLE 10. - Benzene-extracted organics in NIEHS test materials

Sample	Organics extracted, weight percent				
	Amosite	Crocidolite	Chrysotile COF-25	Chrysotile Plastibest-20	Tremolite[1]
1.....	0.029	0.051	0.018	0.008	0.003
2.....	.024	.036	NA	.012	.006
3.....	.012	.034	.016	.011	.005
4.....	.034	.042	.028	.013	.001
5.....	.014	NA	.027	.011	.003
6.....	.020	NA	.041	.008	.001
7.....	.012	NA	NA	.013	NA
8.....	NA	NA	NA	.007	NA
9.....	NA	NA	NA	.017	NA
10.....	NA	NA	NA	.009	NA
Average	.021	.041	.026	.011	.003

NA Not analyzed.
[1]Contains ∿25 percent serpentine, talc, and other minerals.

X-RAY DIFFRACTION[13]

Powder X-ray diffraction data were obtained with a Philips Electronics wide-angle X-ray diffractometer, using a theta-compensating slit and an AMR focusing monochromator. The gas proportional detector was coupled to a solid state linear amplifier and single-channel analyzer. The scans were made at 1/4° 2θ/min, with a 0.2-mm receiving slit, ratemeter setting of 3-percent standard deviation, a multiplier value of 1 x 10^3, using CuKα radiation at 40 kv, 20 ma.

X-ray diffraction charts were run for six to nine samples of each test material. Two theta values were located by measuring the diffraction peak centers. These measured values were corrected, where necessary, from calibration data obtained with the silicon standard run immediately after each sample. X-ray diffraction data for the five test materials are listed in tables 11 through 15. Accessory minerals noted in these tables are discussed in the Mineralogy section of this report.

[13]Data provided by C. W. Huggins, research chemist; M. A. Cargill and K. M. Reeve, physical science aids, Avondale Research Center, Avondale, Md.

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TABLE 11. - Powder X-ray diffraction data, chrysotile COF-25

hkl	2θ	d_{obs}	d_{lit}[1]	I	Other minerals
002	12.01	7.368	7.35	100	
([2])	18.59	4.772	([2])	4	Brucite.
020	19.84	4.474	4.58	13	
004	24.32	3.659	3.66	100	
([2])	29.37	3.040	([2])	3	Calcite.
130	ND	ND	2.66	ND	
201	34.56	2.595	2.594	13	
$20\bar{2}$	35.61	2.521	2.549	11	
202	36.76	2.444	2.456	15	
203	ND	ND	2.282	ND	
$20\bar{4}$	ND	ND	2.215	ND	
204	43.07	2.100	2.096	3	
008	49.92	1.826	1.829	5	
206	52.32	1.748	1.748	3	
060	60.15	1.538	1.536	28	
$0\cdot\bar{0}\cdot10$	63.61	1.462	1.465	5	
$40\bar{2}$	72.02	1.311	1.317	10	
0·0·12	78.51	1.218	([3])	3	

ND Not detected.
[1]Values from PDF card 10-381.
[2]Other mineral.
[3]Not listed on card.

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TABLE 12. - Powder X-ray diffraction data, chrysotile Plastibest-20

hkl	2θ	d_{obs}	d_{lit}[1]	I	Other minerals
(2)	11.18	7.913	(2)	12	Apophyllite.
002	12.05	7.344	7.36	>100	
(2)	18.62	4.765	(2)	28	Brucite.
(2)	19.84	4.474	(2)	18	Apophyllite.
020	ND	ND	4.58	ND	
(2)	20.77	4.276	(2)	5	Quartz?
(2)	22.36	3.975	(2)	4	Apophyllite.
004	24.32	3.659	3.66	>100	
(2)	26.64	3.346	(2)	9	Quartz?
(2)	27.46	3.247	(2)	3	
(2)	27.93	3.194	(2)	2	Albite?
(2)	29.45	3.032	(2)	2	Calcite.
(2)	30.09	2.969	(2)	2	Magnetite.
130	ND	ND	2.66	ND	
201	34.52	2.598	2.594	10	
20$\bar{2}$	35.43	2.533	2.549	15	
(2)	35.95	2.498	(2)	8	Apophyllite.
202	36.82	2.440	2.456	19	
(2)	37.89	2.374	(2)	4	Brucite.
20$\bar{3}$	ND	ND	2.282	ND	
20$\bar{4}$	ND	ND	2.215	ND	
204	43.06	2.100	2.096	4	
008	49.90	1.827	1.829	7	
(2)	50.81	1.796	(2)	2	Brucite.
206	ND	ND	1.748	ND	
(2)	56.96	1.616	(2)	4	Magnetite.
060	60.26	1.535	1.536	30	
(2)	62.50	1.485	(2)	3	Brucite or magnetite.
0·0·10	63.57	1.463	1.465	6	
(2)	68.27	1.373	(2)	3	Brucite.
40$\bar{2}$	71.97	1.311	1.317	7	
0·0·12	78.44	1.219	(3)	4	

ND Not detected.
[1]Values from PDF card 10-381.
[2]Other Mineral.
[3]Not listed on card.

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TABLE 13. - Powder X-ray diffraction data, tremolite

hkl	2θ	d_{obs}	d_{calc}[1]	I	Other minerals
(2)	8.80	10.048	(2)	4	Mica.
(2)	9.44	9.368	(2)	9	Talc.
020	9.82	9.010	9.025	9	
110	10.52	8.376	8.417	>100	
(2)	12.13	7.296	(2)	35	Serpentine.
130	17.44	5.085	5.085	8	
-111	18.21	4.872	4.877	4	
200	18.65	4.758	4.758	35	
040	19.68	4.511	4.512	11	
220	21.14	4.202	4.209	94	
-131	22.94	3.876	3.875	4	
(2)	24.37	3.652	(2)	29	Serpentine.
131	26.37	3.379	3.381	18	
240	27.24	3.273	3.274	74	
(2)	27.60	3.231	(2)	4	Anthophyllite.
310	28.59	3.122	3.124	>>100	
(2)	29.44	3.033	(2)	12	Calcite.
-151 221	30.40	2.940	2.940 2.940	30	
330	31.92	2.804	2.806	>100	
-331	32.79	2.731	2.731	22	
151	33.11	2.705	2.706	36	
061	34.60	2.592	2.592	10	
-202	35.44	2.532	2.533	9	
(2)	35.96	2.497	(2)	2	Calcite.
-261 311	37.37	2.406	2.410 2.410	4	
350	37.75	2.382	2.383	24	
-351	38.52	2.337	2.336	30	
-421	38.77	2.322	2.321	30	
-171	39.17	2.299	2.298	11	
-312	39.65	2.273	2.274	5	
331	39.96	2.256	2.255	2	
-242	40.82	2.211	2.209	2	
132 261	41.79	2.161	2.166 2.162	25	
440	42.95	2.105	2.104	2	
202	44.34	2.042	2.044	7	
351 -402	44.95	2.016	2.017 2.014	22	
370	45.32	2.001	2.001	13	
-371	45.98	1.973	1.973	2	
-422 190	46.24	1.963	1.965 1.962	6	
421	47.13	1.928	1.928	4	
510	48.08	1.892	1.893	90	

See footnotes at end of table.

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TABLE 13. - Powder X-ray diffraction data, tremolite--Continued

hkl	2θ	d_{obs}	d_{calc}[1]	I	Other minerals
-461	48.48	1.877	1.877	2	
-191	48.84	1.864	1.865	6	
530	50.33	1.813	1.815	50	
-512	52.38	1.746	1.745	5	
-551	53.29	1.718	1.718	3	
-282	54.49	1.684	1.684	7	
-133			1.684		
461	55.73	1.649	1.650	60	
480	56.21	1.636	1.637	20	
1·11·0	56.97	1.616	1.617	12	
600	58.18	1.586	1.586	42	
620	59.16	1.561	1.562	12	
2·10·1			1.561		
-571	59.36	1.557	1.557	8	
402			1.555		
-602	60.40	1.532	1.533	21	
422			1.532		
-263	61.28	1.512	1.512	5	
-482	61.74	1.502	1.502	25	
551			1.502		
-2·10·2	63.27	1.469	1.470	6	
442			1.470		
3·11·0	63.86	1.457	1.457	5	
4·10·0	64.86	1.438	1.438	85	
-661			1.438		
-533	65.55	1.424	1.425	3	
-591	66.87	1.399	1.399	10	
621			1.399		

[1]Calculations based on the following unit cell dimensions: a = 9.837A; b = 18.049A; c = 5.277A; β = 104.69°. These dimensions were calculated by least-square refinement of the 54 2θ values for tremolite, assuming a space group of C2/m.

[2]Other mineral.

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TABLE 14. - Powder X-ray diffraction data, amosite

hkl	2θ	d_{obs}	d_{calc}[1]	I	Other minerals
(2)	7.37	11.994	(2)	5	
(2)	8.76	10.094	(2)	10	Mica.
020	9.70	9.118	9.156	8	
110	10.66	8.299	8.325	>100	
130	17.38	5.099	5.111	2	
200	18.99	4.673	4.673	28	
040	19.41	4.573	4.578	7	
(2)	20.90	4.250	(2)	4	Quartz.
220	21.34	4.164	4.162	26	
150	26.16	3.406	3.410	2	
(2)	26.44	3.371	(2)	8	Mica.
(2)	26.68	3.341	(2)	23	Quartz.
240	27.27	3.270	3.270	50	
310	29.08	3.071	3.071	>>100	
(2)	30.61	2.921	(2)	2	Mica.
330	32.26	2.775	2.775	33	
061	34.03	2.634	2.636	6	
260	35.12	2.555	2.555	4	
-202	35.69	2.156	2.153	3	
(2)	36.59	2.456	(2)	2	Mica.
350	37.89	2.374	2.373	4	
400	38.49	2.339	2.337	7	
261	41.01	2.201	2.200	8	
(2)	42.49	2.127	(2)	1	
440	43.44	2.083	2.081	3	
370	45.24	2.004	2.003	2	
510	48.97	1.860	1.860	7	
312	50.17	1.818	1.817	3	
530	51.02	1.790	1.787	4	
550	55.12	1.666	1.665	12	
1·11·0	56.12	1.639	1.639	15	
-403 -621	59.27	1.559	1.560 1.562	26	
0·12·0	60.69	1.526	1.526	11	
640	63.31	1.469	1.475	6	
462 -642 -661	66.42	1.407	1.408 1.407 1.406	14	
710	70.67	1.333	1.332	12	
730	72.42	1.305	1.304	5	
4·12·0	74.19	1.278	1.278	5	
751	82.48	1.169	1.170	6	

[1]Calculations based on the following unit all dimensions:
a = 9.551A; b = 18.312A; c = 5.344A; β = 101.88°.
[2]Other mineral.

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TABLE 15. - Powder X-ray diffraction data, crocidolite

hkl	2θ	d_{obs}	d_{calc}[1]	I	Other minerals
([2])	9.48	9.328	([2])	4	Talc?
020	9.91	8.931	9.002	5	
110	10.61	8.341	8.369	>100	
([2])	12.33	7.178	([2])	1	Chlorite?
200	18.80	4.721	4.726	2	
040	19.72	4.502	4.501	25	
([2])	20.90	4.250	([2])	2	Quartz.
$\bar{1}31$	22.92	3.879	3.876	2	
$\bar{2}21$	24.42	3.645	3.650	1	
131	26.11	3.412	3.412	15	
150	26.50	3.362	3.364	6	
([2])	26.71	3.337	([2])	17	Quartz.
240	27.36	3.259	3.259	18	
310	28.76	3.104	3.104	>100	
221	30.12	2.966	2.966	4	
([2])	30.83	2.900	([2])	6	Epidote?
330	32.10	2.788	2.789	20	
151	32.92	2.720	2.719	28	
061	34.52	2.598	2.596	10	
260	35.47	2.531	2.533	10	
400	38.00	2.367	2.363	3	
$\bar{3}51$	38.85	2.318	2.318	10	
$\bar{1}71$ $\bar{4}21$	39.18	2.299	2.294 2.294	4	
270	39.88	2.260	2.259	3	
360	41.56	2.173	2.173	13	
$\bar{3}61$	42.48	2.128	2.131	2	
$\bar{4}41$ 440	43.15	2.096	2.098 2.092	2	
202	43.60	2.075	2.075	2	
351	44.71	2.026	2.024	8	
370	45.47	1.994	1.993	3	
$\bar{2}81$ $\bar{3}71$	46.28	1.961	1.961 1.961	2	
$\bar{2}62$ 421	46.90	1.937	1.938 1.934	1	
431 510	48.38	1.881	1.881 1.880	3	
$\bar{1}91$	48.89	1.862	1.861	4	
441	50.23	1.816	1.813	4	
530	50.61	1.803	1.803	8	
512 $\bar{3}32$	53.04	1.726	1.727 1.726	2	

See footnotes at end of table.

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TABLE 15. - Powder X-ray diffraction data, crocidolite--Continued

hkl	2θ	d_{obs}	d_{calc}[1]	I	Other minerals
133	53.91	1.700	1.701	2	
$\bar{3}13$	54.44	1.685	1.685	3	
550	54.94	1.671	1.673	3	
501 461	55.50	1.655	1.654 1.653	15	
480	56.38	1.631	1.630	8	
$\bar{6}01$ $\bar{1}\cdot11\cdot0$	57.01	1.615	1.617 1.613	18	
$\bar{6}21$ $\bar{1}53$	57.95	1.591	1.592 1.591	3	
600 $\bar{1}33$	58.57	1.575	1.575 1.574	8	
$\bar{5}71$	60.01	1.541	1.543	2	
551	61.66	1.504	1.503	14	
481 153 $\bar{4}42$	62.61	1.483	1.486 1.486 1.482	3	
$\bar{5}13$	64.00	1.454	1.453	6	
572 $\bar{6}42$ $\bar{4}\cdot10\cdot1$	65.03	1.434	1.438 1.435 1.434	5	
661	65.50	1.424	1.423	15	
631 313 590	68.08	1.377	1.379 1.378 1.374	5	
333 $\bar{7}10$	69.77	1.347	1.347 1.346	9	
$\bar{7}51$ $\bar{7}40$	73.06	1.295	1.296 1.294	8	
573 $4\cdot12\cdot0$ 750	74.86	1.268	1.268 1.267 1.265	2	

[1]Calculations based on the following unit cell dimensions in angstroms: a = 9.730; b = 18.004; c = 5.331; B = 103.69 degrees. These dimensions were calculated by least-square refinement of the 49 2θ values for crocidolite, assuming a space group of C2/m.

[2]Other mineral.

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INFRARED SPECTROSCOPY[14]

Samples were prepared for infrared spectroscopy by using a procedure similar to that described by Coles (2). Eight drops of isopropyl alcohol were added to a 50-mg sample in an agate mortar and hand-ground until dry. Five milligrams of this ground sample was blended with 1,000 mg of potassium bromide in a Wig-L-Bug for 1 min. Of this mixture, 175 mg was pressed into a 13-mm-diam disk, using a Perkin-Elmer evacuation die.

The resultant clear disk was scanned from 4,000 to 200 cm^{-1}, using a Perkin-Elmer 225 infrared grating spectrophotometer at the following instrument settings:

scale factor = 4;
scan speed, fast = 2.0;
pen traverse time = 3 sec;
suppression = 2.0;
slits, manual = 0;
program = 5;
gain = 0.20;
response = 1.0; and
intensity = 8.0.

Infrared spectra for the five test materials are shown in figures 4 through 8. The doublet hydroxyl modes were 3,690 and 3,640 cm^{-1} for the two chrysotile samples, COF-25 and Plastibest, and for the approximately 25 percent serpentine in the tremolite sample. Hydroxyl modes were 3,635 and 3,615 cm^{-1} for amosite and crocidolite respectively.

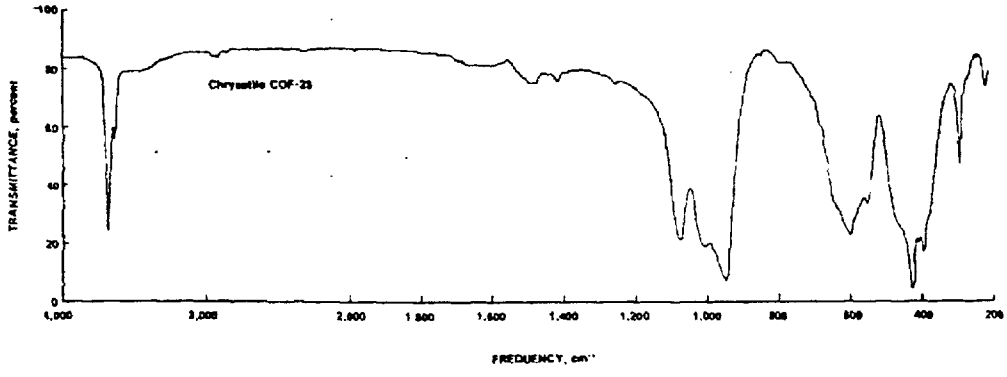


FIGURE 4. - Infrared spectra of chrysotile COF-25.

[14]Data provided by C. W. Huggins, research chemist, and M. A. Cargill, physical science aid, Avondale Research Center, Avondale, Md.

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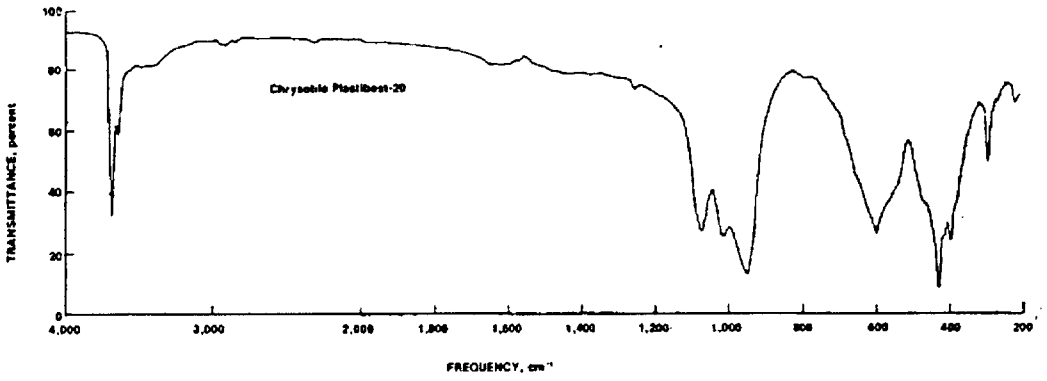


FIGURE 5. - Infrared spectra of chrysotile Plastibest-20.

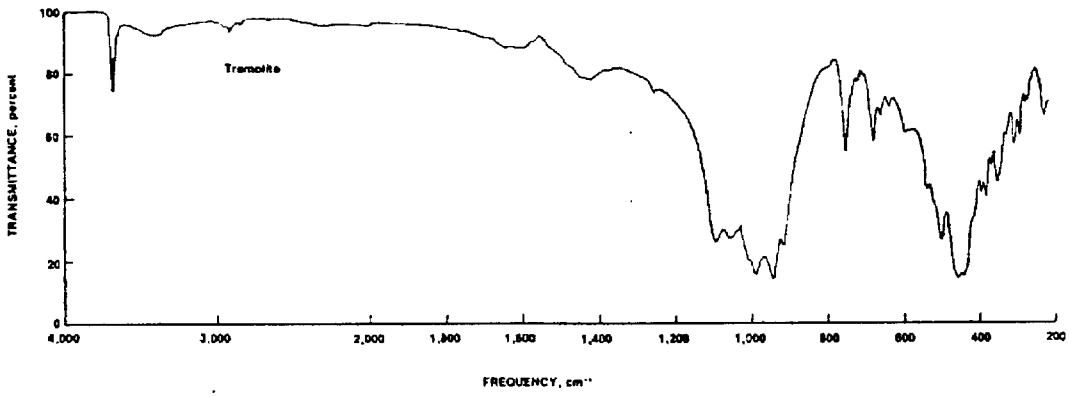


FIGURE 6. - Infrared spectra of tremolite-serpentine-talc.

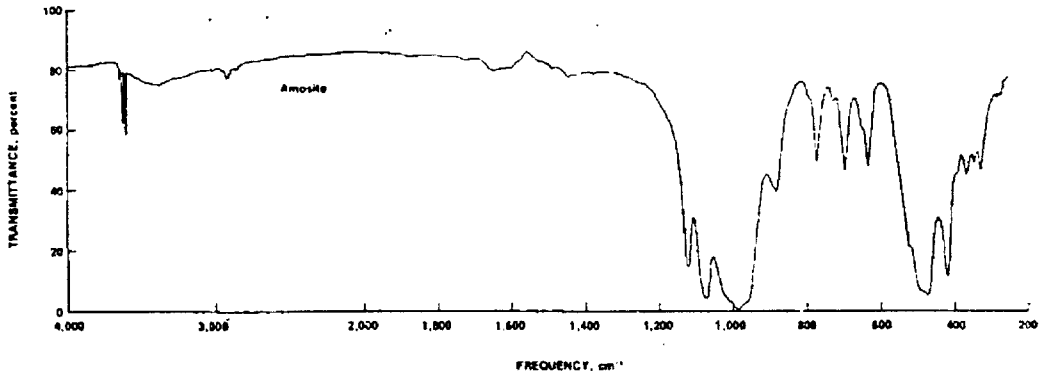


FIGURE 7. - Infrared spectra of amosite.

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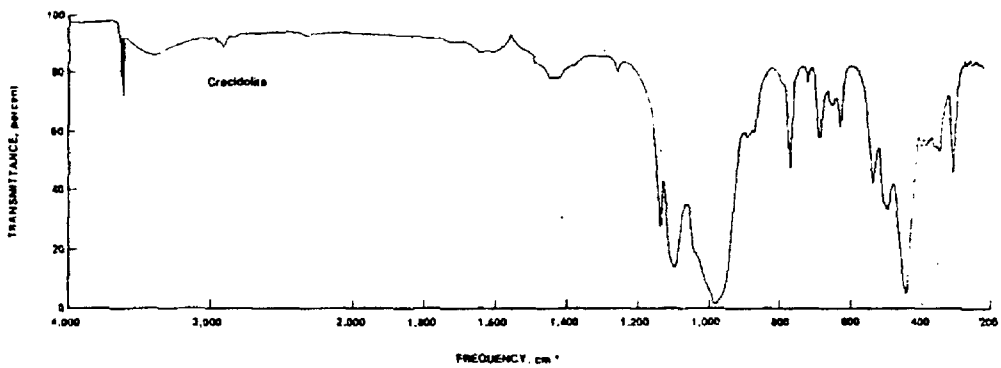


FIGURE 8. - Infrared spectra of crocidolite.

Both serpentine and amphibole minerals exhibit strong absorption in the 900 to 1,200 cm^{-1} region, which is attributable to Si-O stretching vibrations, and in the 300 to 600 cm^{-1} region, as a result of Si-O chain and ring vibrations.

THERMOGRAVIMETRIC ANALYSIS[15]

All thermogravimetric analyses (TGA) were conducted, using either a Cahn RG or RH electrobalance contained in Cahn glass vacuum bottles. The more sensitive RG electrobalance was equipped with a standard Cahn 9-mm-diam platinum stirrup-pan that holds approximately a 20-mg sample. A Cahn mullite Flowthru tube, 16-mm ID, enclosed the sample and the platinum suspension wire. Sample heating was achieved with a 1-1/4-inch-ID by 6-inch-long 1,500° C platinum-wound furnace, fabricated by the Bureau.

The larger capacity RH electrobalance was fitted with a Cahn Vycor Flowthru tube with a 48-mm ID. Sample crucibles were fabricated from either platinum or high-purity recrystallized alumina. The majority of TGA runs in the RH electrobalance consisted of sample weights of 1 gram contained in platinum crucibles of 5-cm^3 capacity. The TGA carbon dioxide and/or water absorption studies used 5-gram samples in a 15-cm^3 alumina crucible, placed in a 25-cm^3 platinum crucible. The suspension wire and stirrup arrangement consisted of 0.005-inch-diam platinum wire. A SATEC SF-15 three-zone Kanthal-wound furnace, 2.5-inch-ID by 10 inches long of split construction, was used up to 1,200° C.

Automatic temperature programming for both the RG and RH electrobalance was accomplished by linking each furnace with separate, but identical, Theta Industries No. 7200 SCR systems, equipped with platinum versus platinum-10 percent rhodium-control thermocouples located near the heating elements. Average heating rates were 5° C per min for all TGA runs except for TGA-absorption tests that employed heating rates of 12° C/min.

[15]Data provided by R. E. Brown, physical science technician, Avondale Research Center, Avondale, Md.

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Measuring thermocouples, composed of platinum versus platinum-10-percent-rhodium, were introduced internally through a T located at the bottom of the Flowthru tubes and were centrally positioned one-fourth-inch from the base of the sample containers. An electronic ice point compensator was used to convert reference temperatures to 0° C. The output of the electrobalance control unit was fed into a Cahn No. 7300 automatic range expander to amplify the signal by a factor of 10. Temperatures versus weight change measurements were recorded on the X and Y axis, respectively, of a Hewlett-Packard No. 7054A X-Y recorder.

Flowthru tube joints were fitted with vacuum-tight seals for connection to mechanical vacuum pumps, gas-drying chambers, gas inlets and/or outlets, flowmeters, pressure gauges, etc. After weighing the sample on the pan or crucible, the system was evacuated down to 50 microns, then filled with either nitrogen, helium, or air. This evacuation-gas fill procedure was repeated two more times, totaling three, to insure removal of any residual gas and/or vapors from previous runs. An additional bakeout step was performed at 110° C for TGA-absorption runs. Gases were introduced through the side port of the flowthru tube and allowed to exit through the lower port. Gasflow rates at ambient pressure were 5 cm^3/min and 50 cm^3/min, for the RG and RH electrobalance respectively, during normal runs. Faster flow rates of 0.5 l/min at reduced pressures of 650 torr, were employed for TGA-absorption tests conducted in the RH electrobalance.

Thermogravimetric analysis-absorption tests were designed to obtain gravimetric water and carbon dioxide values, along with the weight loss versus temperature thermograms. These tests involved insertion of a gas-train package between the exit end of the flowthru tube and a vacuum source. The gas-train package was composed of two identical sets of absorption bottles, with seven bottles per set. Heavy-walled rubber tubing was used to connect the seven bottles of each set in series. The first three bottles of each set contained magnesium perchlorate to absorb water, the next three bottles contained ascarite to absorb carbon dioxide, and the seventh bottle contained magnesium perchlorate plus ascarite for backflow protection. Bottles 1 and 4 were the primary absorption vessels, bottles 2 and 5 were used for backup protection, whereas bottles 3 and 6 were used for possible temperature and pressure corrections. Both sets were connected in parallel via two-way valves connected at each end, which made it possible to change the bottles without interrupting the gasflow. Water and carbon dioxide data for the NIEHS materials are listed in table 3.

Typical thermogravimetric scans for the five test materials are shown in figures 9 through 13. The two chrysotiles and the tremolite were heated in an airflow of 50 ml/min. Amosite and crocidolite were heated in a nitrogen flow of 50 ml/min. The large weight loss between 550° and 650° C, (shown in fig. 9-11) is a result of the dehydroxylation of serpentine. Crocidolite lost water at approximately 600° to 700° C in neutral atmosphere, amosite dehydroxylated between 600° and 800° C in nitrogen. Tremolite did not lose its combined water until approximately 1,000° C was exceeded.

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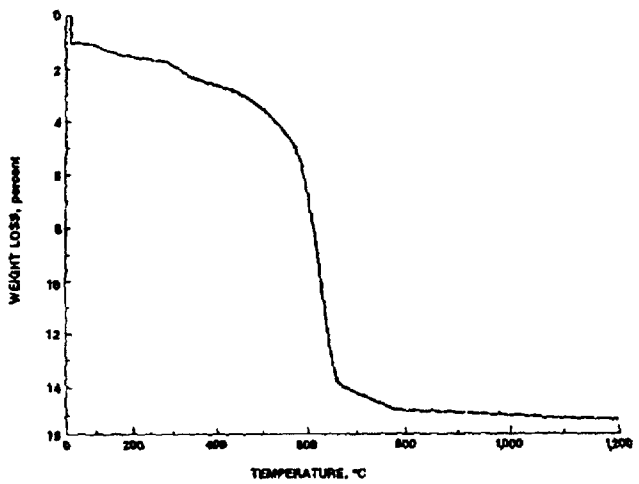


FIGURE 9. - Thermogravimetric scan in air for chrysotile COF-25.

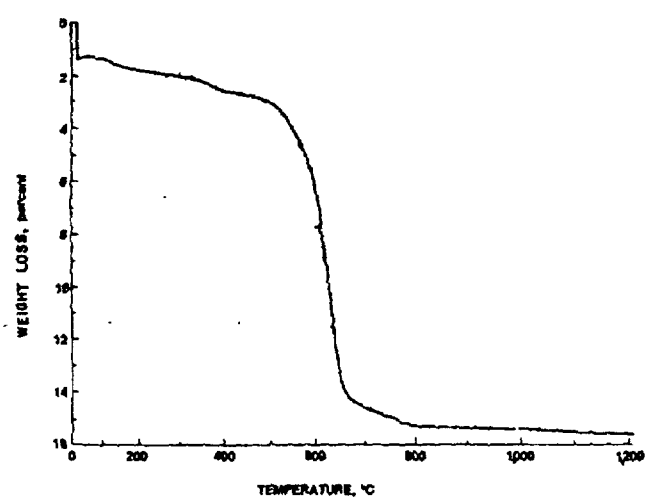


FIGURE 10. - Thermogravimetric scan in air for chrysotile Plastibest-20.

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