ANALYTICAL CAPABILITIES AND TEST METHODS

Rio Tinto Minerals
Analytical and Technical Services
Greenwood Village, CO
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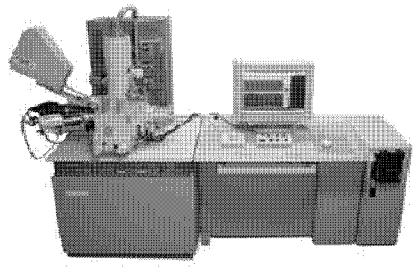
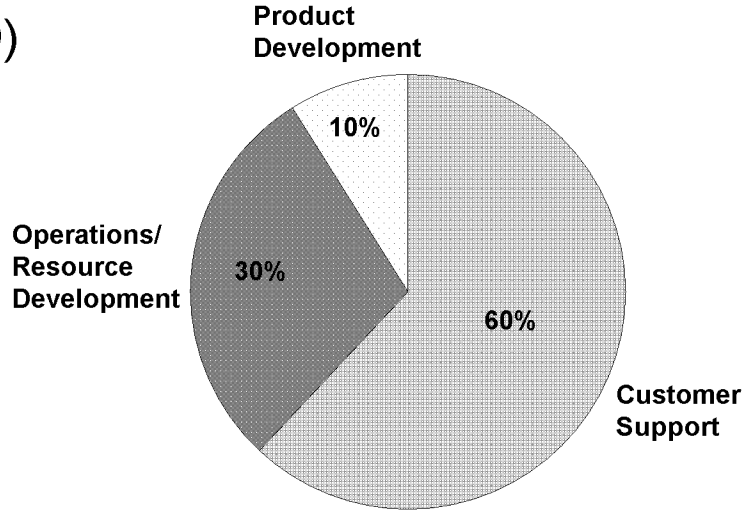
RTM Analytical Capabilities

- Microscopy/Spectral Analysis
  - Optical Microscopy: PLM, Dispersion Staining
  - Scanning electron microscopy (SEM)
  - Transmission electron microscopy (TEM)
  - X-ray diffraction (XRD)
  - Infra-red spectroscopy/microscopy (FT-IR)
  - Thermal gravimetric analysis (TGA)
  - Chemical analysis (EDS, XRF, LECO)



- Physical Testing
  - Particle size
    - Median (SediGraph, Coulter)
    - Top size (Hegman, Sieve)
  - Surface area
  - Color/Brightness (Minolta, GEB)
  - Other

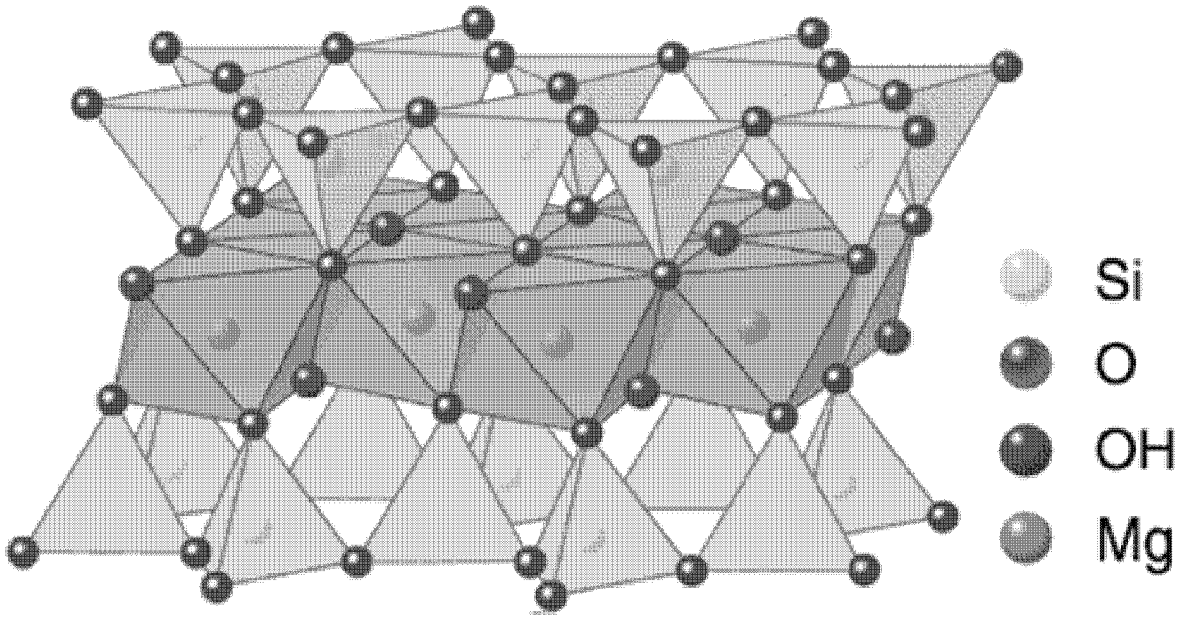


Crystal Structure of Talc

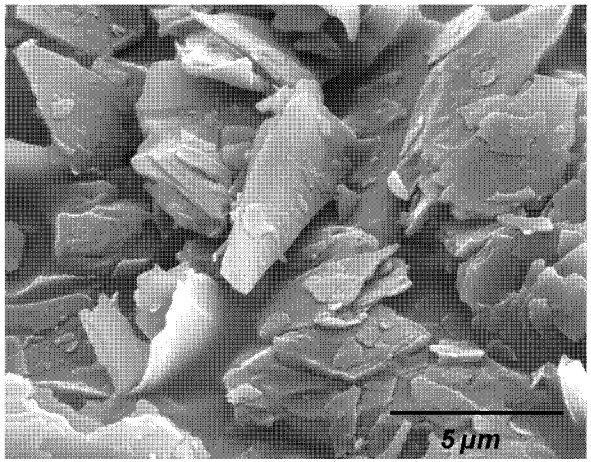
Tetrahedral Layer

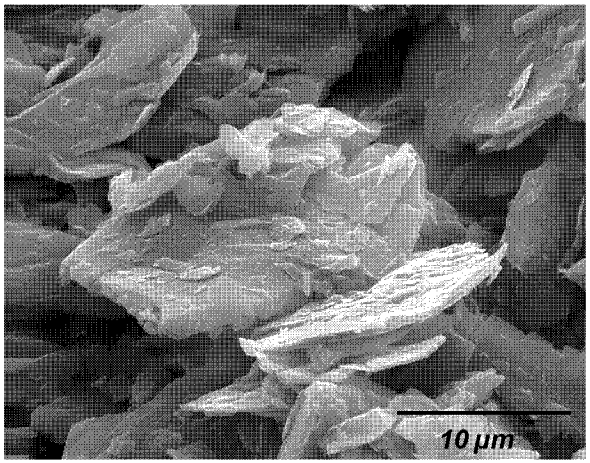
Octahedral Layer

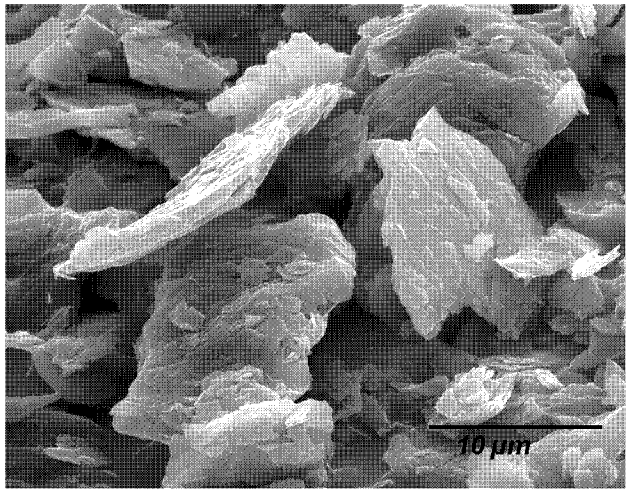
Tetrahedral Layer



RTM Talc – Guangxi Ore







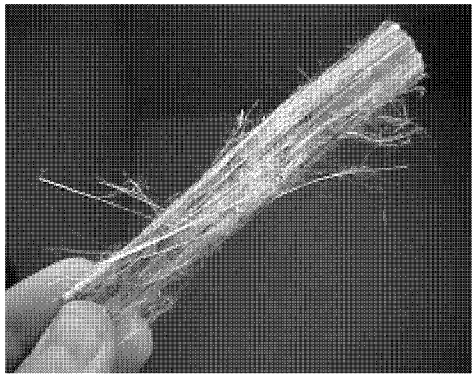
Asbestos encompasses six minerals with a unique, fibrous habit

Serpentine asbestos
95% of world production

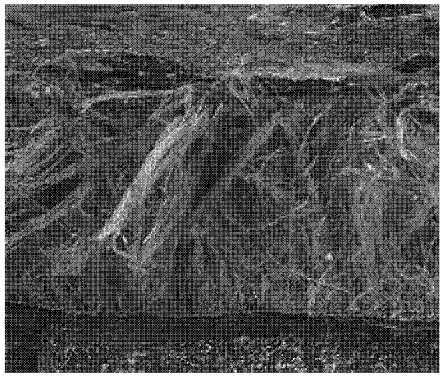
Chrysotile ("white asbestos")

Amphibole asbestos

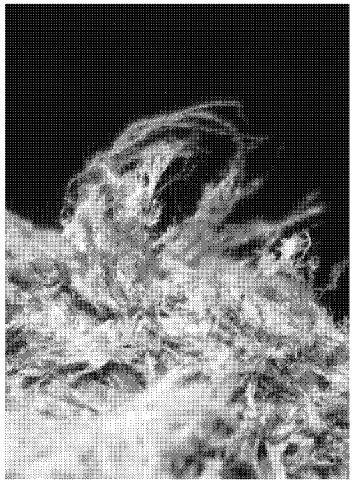
Amosite ("brown asbestos")



Crocidolite ("blue asbestos")

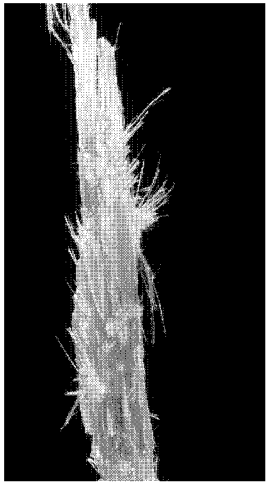


Tremolite asbestos*



Actinolite asbestos*

Anthophyllite asbestos*



Exposure to these minerals has been linked with asbestosis and mesothelioma

*** "Uncommon" asbestos**

The Six Regulated Asbestos Types:

Group	Chemical Composition	Morphology	
		Asbestiform	Non-Asbestiform
Serpentine	$3\ MgO \bullet 2\ SiO_2 \bullet 2\ H_2O$	Chrysotile	Antigorite, Lizardite
Amphibole	$7\ FeO \bullet 8\ SiO_2 \bullet H_2O$ $Na_2O \bullet Fe_2O_3 \bullet 3\ FeO \bullet 8\ SiO_2 \bullet H_2O$ $2\ CaO \bullet 5\ MgO \bullet 8\ SiO_2 \bullet H_2O$ $2\ CaO \bullet 3\ MgO \bullet 2\ FeO \bullet 8\ SiO_2 \bullet H_2O$ $7\ MgO \bullet 8\ SiO_2 \bullet H_2O$	Amosite Crocidolite Tremolite asbestos Actinolite asbestos Anthophyllite asbestos	Grunerite Riebeckite Tremolite Actinolite Anthophyllite

Talc:

Group	Chemical Composition
Talc	$3\ MgO \bullet 4\ SiO_2 \bullet H_2O$

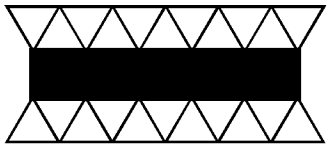
Each asbestiform mineral also has a non-asbestiform counterpart.

Talc vs. Serpentine – Edge view

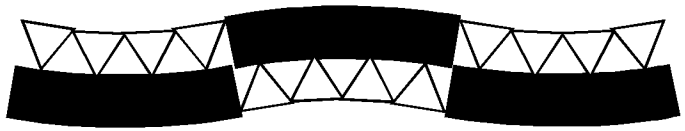
3-Layers

2-Layers

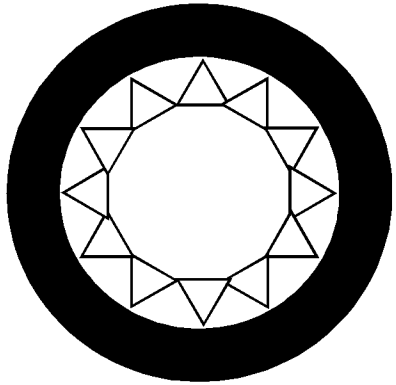
Talc



Lizardite



Antigorite

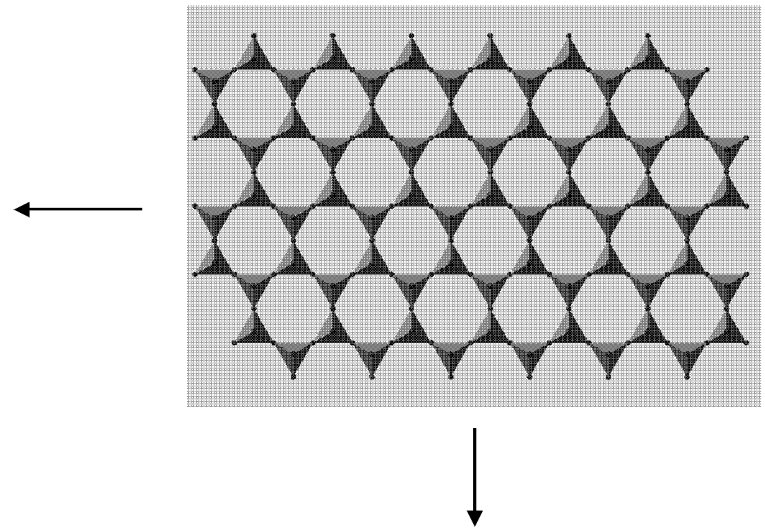


Chrysotile (asbestos)

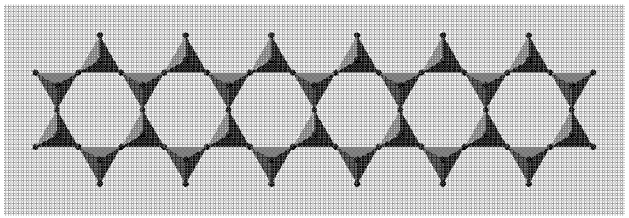
Talc and serpentine share the same elements, but the relative amounts and internal structures are different.

Talc vs. Amphibole: Top View

Sheet Silicate - Talc



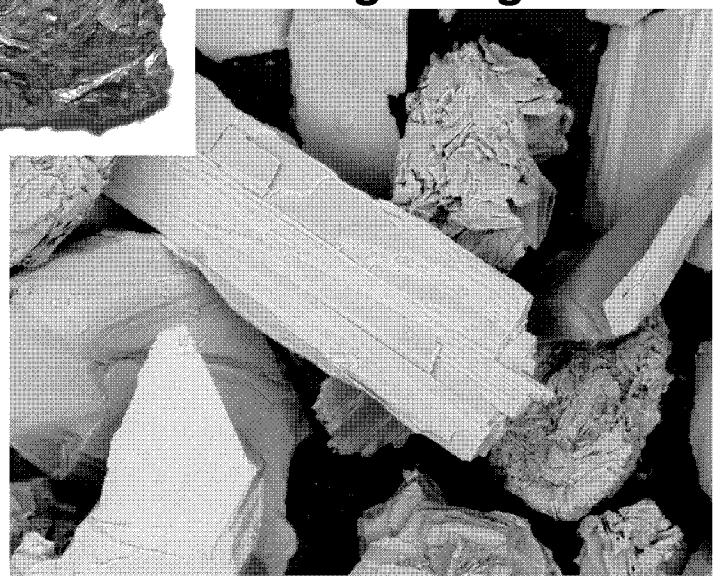
Chain Silicate - Amphibole



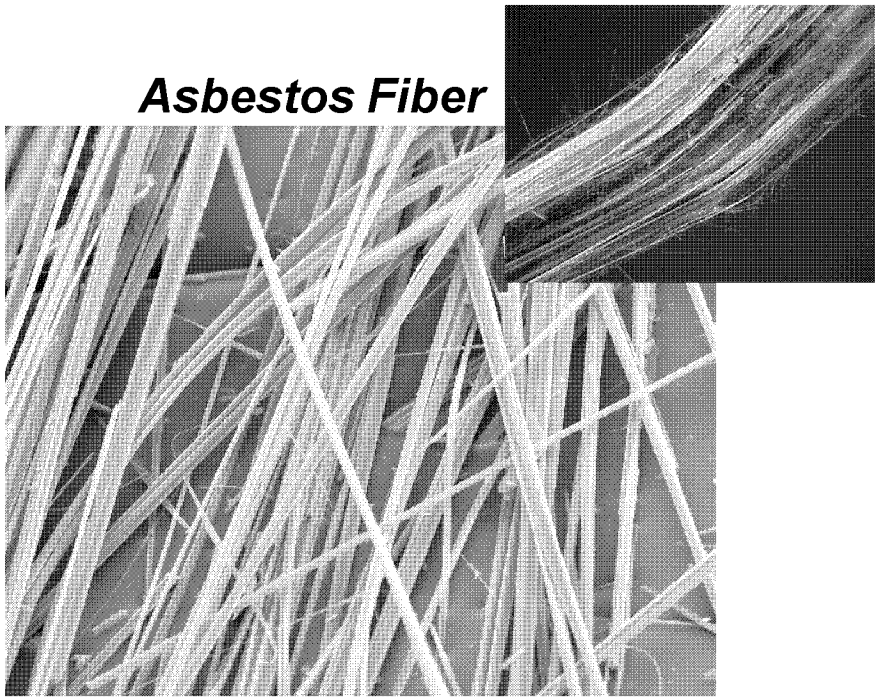
Sheet silicates are platy; chain silicates are naturally elongated (even non-asbestiform varieties).

Asbestiform Morphology

Cleavage Fragment



Asbestos Fiber



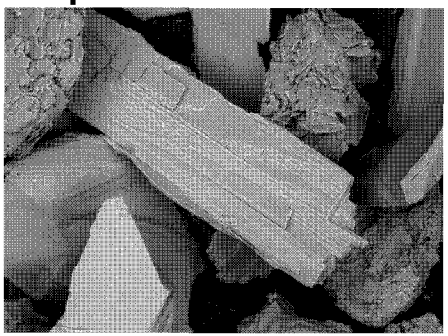
= Fractured Particle

= Growth Habit

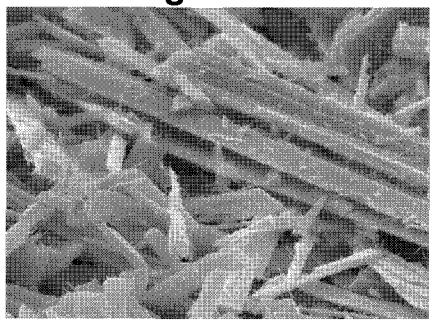
Cleavage fragments are not asbestos;
Grinding cannot produce asbestos; although grinding can produce "regulated fibers."

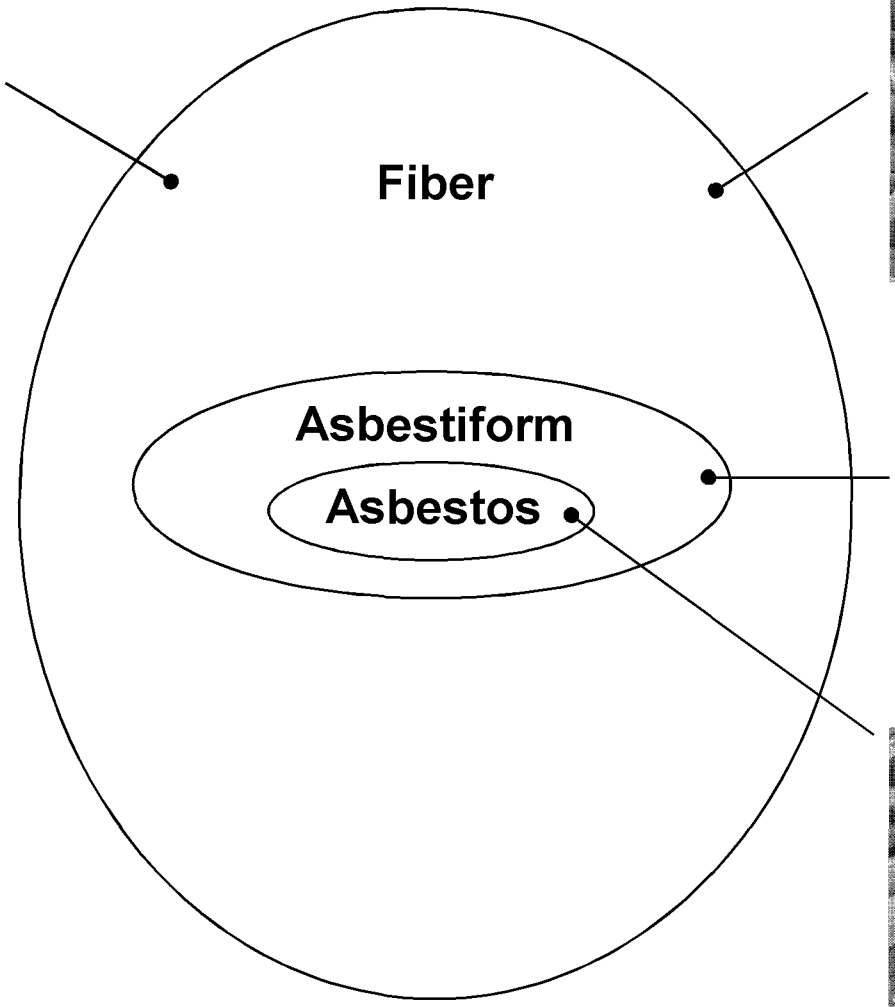
Elongated Mineral Particles

e.g. Cleavage Fragment Amphibole

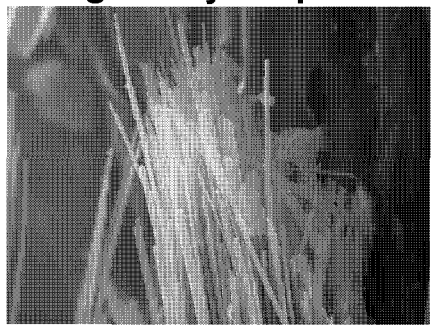


e.g. Wollastonite

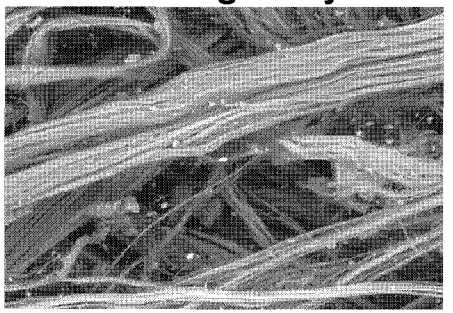




e.g. Libby Amphibole

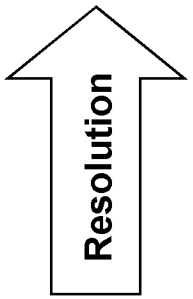


e.g. Chrysotile

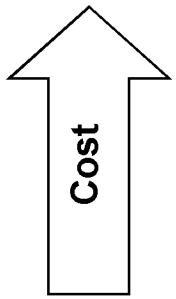


"Fiber" is a regulatory term, defined by aspect ratio and length – varies according to method/regulation.

RTM uses a combination of state-of-the-art techniques to test ore and products for asbestos



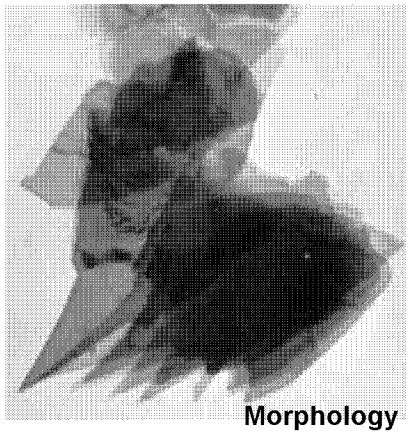
- **Microscopy Methodology**
  - TEM = Transmission Electron Microscopy
  - SEM = Scanning Electron Microscopy
  - PLM = Polarizing Light Microscopy
  - PCM = Phase Contrast (light) Microscopy

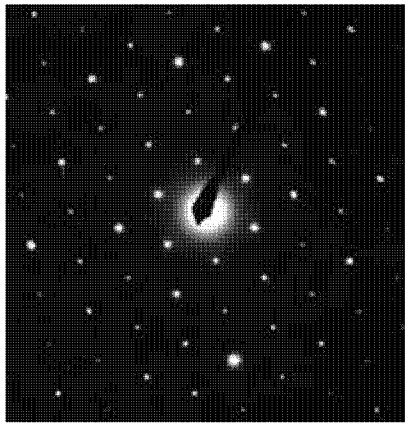


- **Non-microscopy Methodology**
  - XRD* = X-ray Diffraction

XRD cannot distinguish asbestiform from non-asbestiform varieties of the same mineral.

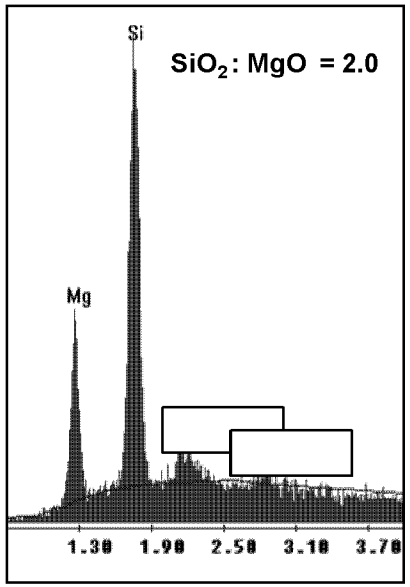
Transmission Electron Microscopy



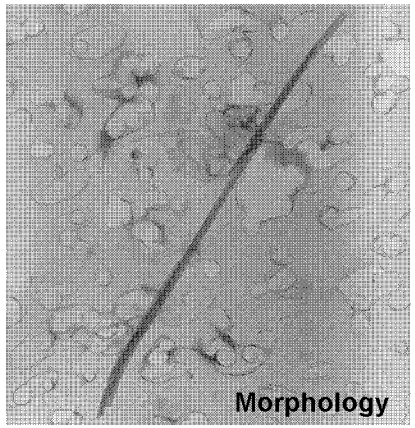


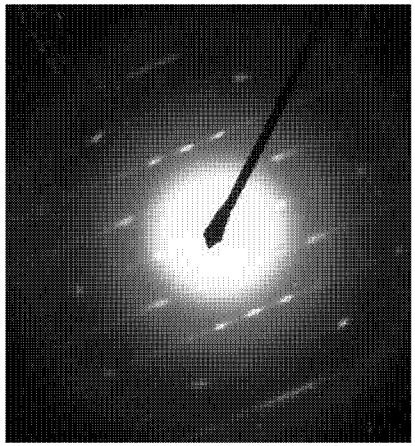
Electron Diffraction

Talc



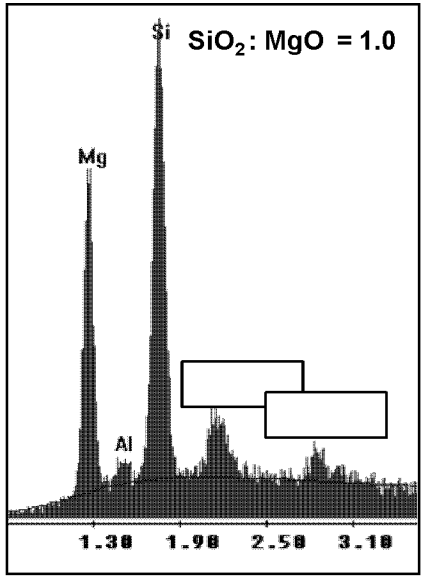
EDS Elemental Analysis





Electron Diffraction

Serpentine Asbestos (Chrysotile)



EDS Elemental Analysis

TEM analysis is most sensitive technique; utilizes morphology, elemental analysis, and diffraction.

*CTFA J4-1 vs. USP standard**

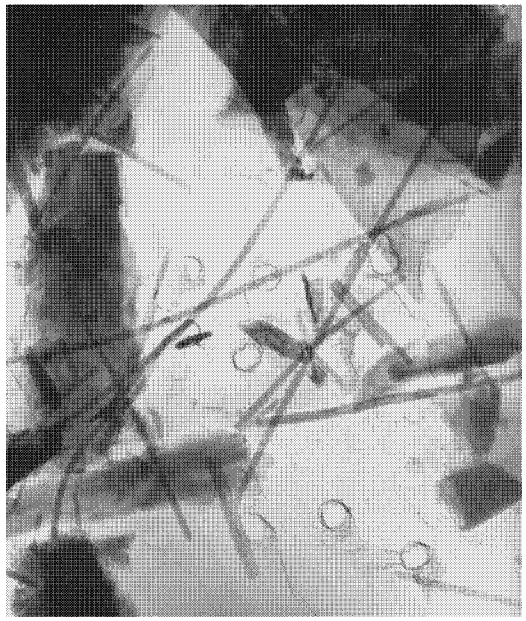
(Asbestos Analysis in Talc for Personal Care Products)

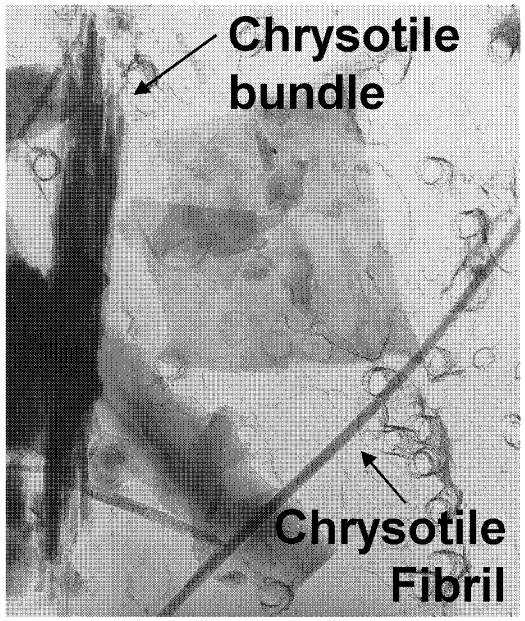
- **Both** – initial evaluation by XRD (Note: asbestiform vs. non-asbestiform cannot be distinguished by XRD).
- **Both** – further analysis if "suspect phases" are detected by XRD. CTFA requires PLM (mineralogy + morphology); USP requires optical microscopy (morphology only). Note: PLM is required for most other "bulk" asbestos analyses.
- **CTFA** – includes morphology criteria for asbestos + counting rules for individual fibers; **USP** – includes general morphology definition, but no counting rules for individual fibers.
- **CTFA** – used for amphibole asbestos only; **USP** used for amphibole asbestos and chrysotile (although chrysotile is often below the resolution limit of both XRD and PLM).

The most comprehensive strategy is CTFA (or USP) for amphibole followed by TEM for chrysotile

*CTFA = Cosmetics, Toiletries, and Fragrance Association (currently Personal Care Products Council – PCPC); USP = United States Pharmacopeia

Johnson & Johnson / RTM protocol - goes beyond USP





These chrysotile fibers are detected by TEM only; none would be detected by USP (or by CTFA used alone). The J&J / RTM protocol detects *all* fibers and reduces risk of liability.

Johnson & Johnson / RTM Historical Specification (since 1988):

- **CTFA J4-1 (for amphibole)**
  - XRD followed by PLM if XRD is positive
  - Includes morphological criteria for asbestos
  - Minimum fiber counted: >5 µm length, 5:1 aspect ratio; maximum fiber counted: 30 µm length, 3 µm width
- **J & J Internal Method TM7024* (chrysotile)**
  - Based on Kremer and Millette (1990), “A Standard TEM Method for Identification and Quantification of Asbestiform Minerals in Talc”.
  - Minimum fiber counted: >1 µm length; 3:1 aspect ratio

This strategy is the basis for a comprehensive RTM program for all products (begun in 2001).

New proposed ASTM methodology for asbestos analysis in talc

- Current work item for ASTM committee D22.07 (Sampling and Analysis of Asbestos)
- RTM (J. Pier) is participating in development
- Will likely include electron microscopy
- Portions will likely be based on ASTM D5756 (Standard test method for TEM analysis of asbestos in dust)

Significant debate exists over amphibole fiber definitions

- **Variation between:**
  - Mineralogical definitions
  - Regulatory fiber definitions
  - Definitions based on health effects
  - Definitions used for litigation

"Short-list" of existing methods show variation in definitions

Fiber Definition		Method	Analysis Type
Aspect Ratio	Fiber Length		
>3 : 1	5 µm	NIOSH 7400 NIOSH 7402 OSHA ID-160 OSHA ID-191 EPA 600/M4-82-020 1982 ISO 14966 EU Directive 83/477/EEC* German BIA CTFA J4-1	PCM – Air TEM – Air PCM – Air PLM – Bulk PLM – Bulk SEM – Air PCM – Air SEM – Mineral Dust XRD/PLM – Talc
>5 : 1	0.5 µm	EPA 40 CFR Part 763 (AHERA) EPA 600/J-93/167 ISO 10312 & 13794 ASTM D 6281 ASTM D 5755, 5756 & 6480 AWWA 2570	TEM – Air TEM – Dust (carpet) TEM – Air TEM – Air TEM – Dust TEM – Water
(>20 : 1)	5 µm	EPA 600/R-93/116 1993 NIOSH 9002 ISO (draft) USP 31 NF26	PLM – Bulk PLM – Bulk PLM/SEM/TEM – Bulk XRD/PLM – Talc

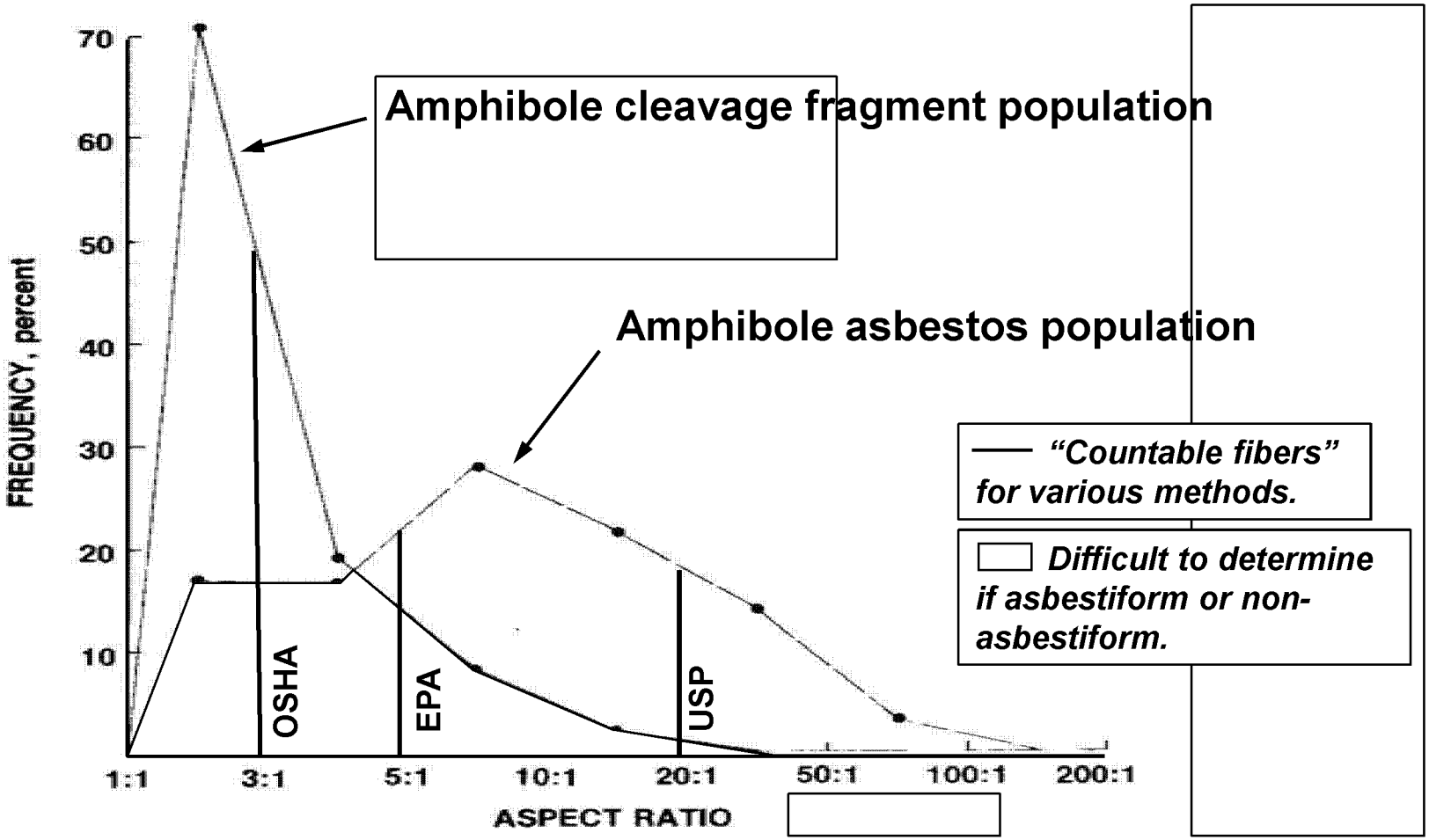
Asbestiform Morphology – "Wylie Definition" – applies to "statistical population"

- mean aspect ratio of 20:1 or greater for fibers longer than 5 um,
- very thin fibrils, usually less than 0.5 um in width, and
- two or more of the following:
  - parallel fibers occurring in bundles,
  - fiber bundles displaying splayed ends.
  - fibers in the form of thin needles,
  - matted masses of individual fibers, and/or fibers showing curvature."

No methods specify how many fibers constitute a "population."

References:
- ***USP 31 NF26***
- *Wylie, A.G. "Discriminating Amphibole Cleavage Fragments from Asbestos: Rationale and Methodology"*
- *OSHA 1992 Final Asbestos Standard, Intro to 29 CFR Parts 1910 and 1926, Occupational Exposure to Asbestos, Tremolite, Anthophyllite and Actinolite, Section 4 – Mineralogical Considerations.*
- *Federal Register Part II, Department of Labor, Mine Safety and Health Administration, 30 CFR Parts 56, 57, and 71.*

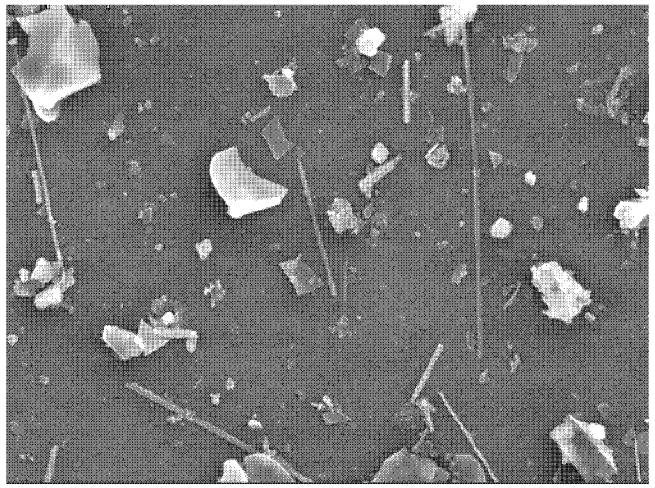
Distribution of <u>Amphibole</u> Fiber Aspect Ratios:



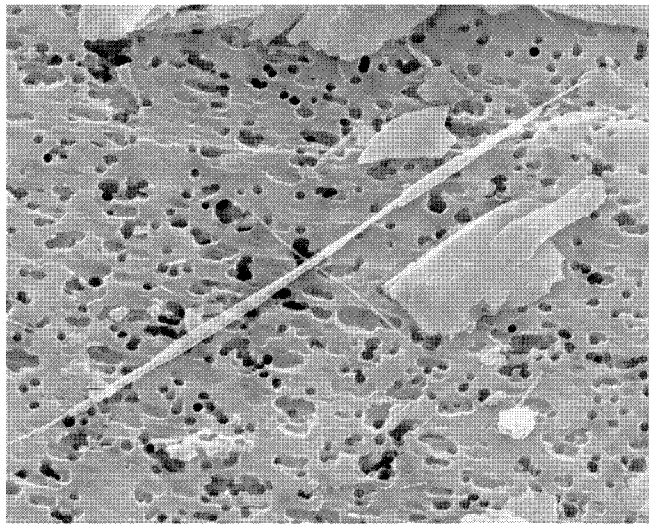
-From US Bureau of Mines Report No. IC 8751, "Selected Silicate Minerals and their Asbestiform Varieties"

Asbestos as a possible trace contaminant in talc

- If present, only a few fibers (or fibrils) are observed; statistical "population" characteristics cannot be applied to individual fibers/fibrils.
- Difficult to determine if individual fibers were originally associated with a bundle (may be disaggregated from milling and/or sample prep).
- If amphibole asbestos present, it is likely an "uncommon" type (lower mean aspect ratios; no curvature).
- "Countable" particles may become airborne (detectable on a personal air monitor), and *regulated* even if origin is non-asbestiform.
- "Many outside labs will err on the side of caution when only a few suspect fibers are detected.



Tremolite in dust (on filter prep)



Chrysotile in talc (on filter prep)

RTM closely monitors issues / trends …

- NIOSH “Roadmap” for asbestos
- IARC reviews
- Korean talc issue
- China developments
- PCPC/CIR/FDA
- IMA and Eurotalc
- ASTM and ISO
- Judgment against “industrial talc” (2008) – supplier exits from the market
- Congressional bills have proposed expanding asbestos “definitions.”
- Lack of consistent definitions - listed as high priority at ASTM Johnson Asbestos Conference
- Crayon issue (2000) – leading labs disagree on results
- “Naturally occurring asbestos” (NOA) – leading labs disagree on results
- Non-regulated amphibole at Libby – a major health issue

RTM monitors varying global regulatory requirements

- **Airborne exposure to fibers (definitions based on size, aspect ratio)**
  - OSHA, EU Directives – 0.1 fibers per cubic centimeter of air
  - MSHA – 0.1 fibers per cubic centimeter of air (recently changed from 2)
  - EPA – abatement clearance comparison
- **Fiber content (size not limited)**
  - EPA – 1% Asbestos Containing Material
  - OSHA, WHMIS, GHS – 0.1% carcinogen labeling requirement
  - UK, France – "Detectable"
- **Testing methodologies to measure fibers are often not consistent**

- ***RTM Objectives***
  - Take the most conservative approach to keep *all* fibers out of products using the most comprehensive and sensitive test methods
  - Continue to ensure that talc is safe (has been used in the most sensitive markets for over a century)!
  - Continue to ensure that global government authorities deem our products are safe
  - Maintain programs that decrease liability and maintain reputation for RTM and its customers
  - Diligently ensure employees safety
  - Continually monitor current issues / trends
  - Influence test method development and regulations
  - Continue to participate in regulatory discussion (IARC, NTP, ASTM, ISO, etc.)

Beyond the safety of our workers and product end-users, limiting liability to Rio Tinto and our customers is the driver behind our programs