RESPIRABLE TALC

Methods Research Branch

Analytical Method

Analyte:	**Respirable Talc**	Method No.:	P&CAM 355
Matrix:	Air	Range:	200 - 4000 μg/m^3
Procedure:	Filter collection, low temperature ashing, Re-deposition on Ag filter, XRD	Precision:	$\overline{\%RSD}_I = 5.3$ (analytical) $\overline{\%RSD}_T = 34$ (sampling and analytical)
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1. Synopsis

A known volume of air is drawn through a 0.45-μm polyvinyl chloride filter.

The filter and sample are radio-frequency ashed; the residue is suspended in 2-propanol and co-deposited with α-Al_2O_3 on a silver membrane filter.

1.3 The samples are analyzed by X-ray diffraction.

2. Working Range, Sensitivity, and Detection Limit

The method was evaluated over the range 50 to 4000 μg/m^3, corresponding to 10 to 800 μg/filter for a 200-L air sample collected at 20 °C and 750 torr.

2.2 The instrumental sensitivity is 0.0053 μg/count for the 002 peak and 0.0083 μg/count for the 006 peak.

The detection limit is approximately 12 μg/filter for the 002 peak and 10 μg/filter for the 006 peak. The lowest analytically quantifiable level is 50 μg/filter for the 002 peak and 30 μg/filter for the 006 peak.

3. Interferences

A preliminary, qualitative scan of the sample is performed and indicates whether interferences are present.

Either the 002 or 006 peak of talc may be used for analysis to assist interfering peaks such as the 310 peak of tremolite (006 peak of talc).

4. Precision and Accuracy

The precision of the analytical method is 5.3% RSD, based on six samples at each of three concentration levels. The total precision of the sampling and analytical method is 34%, based on six samples at each of three concentration levels. The poor precision was caused by the inconsistent synthetic atmosphere generated during testing of the method.

The analytical method recovery was determined to be 1.00 at 160 μg per filter. In stability studies of generated samples, the mean of samples analyzed after fourteen days was the same as that of samples after one day, at the 95% confidence level. Details of the experiment are given in Reference 1.

5. Advantages and Disadvantages

The sampling device is small and portable, and the samples easily collected.

The analytical method requires a skilled operator and expensive equipment.

When no interferences or only avoidable interferences are present, the method is highly specific for talc.

Compared to the alternates of electron or optical microscopy, the method is rapid and very specific.

Occasionally (rarely) talc peaks may be obscured totally by interferences.

6. Apparatus

Sampling equipment, consisting of filter unit (0.45-μm polyvinyl chloride filter with backup pad, 37-mm diameter, in a plastic cassette with 10-mm nylon cyclone) and personal sampling pump capable of sampling at 1.5 - 2 L/min. The pump must be calibrated with a representative filter and cyclone in line, using a soap-bubble flowmeter or wet- or dry-test meter, and its flow rate must be known accurately to within ±5%.

Silver membrane filters, 25-mm diameter and 0.45-µm pore size: Selas Flotronics, Huntingdon Valley, PA 19006.

Vacuum filtration apparatus with modified filtering tower. The bore of the filtering tower should be sized to yield the largest sample surface that can be optimally exposed to the X-ray beam without being shadowed by the sample holder at the low angle (9.45 ° 2θ) of the 002 talc reflection.

X-ray diffractometer equipped with broad focus copper target X-ray tube, rotating sample holder, secondary beam monochromator, scintillation detector, pulse height analyzer and digital printout capability. The diffractometer parameters should be set to provide high intensity at the expense of resolution. In addition to higher counting rates this will improve statistics by allowing a greater fraction of the talc crystallites to contribute to the peaks.

Micro-pipets with disposable tips (Eppendorf, Oxford or equivalent). Micropipets should be calibrated gravimetrically using 2-propanol. A variety of sizes covering the range from 0.20 mL to 1.00 mL will be required.

Glassware (borosilicate)

6.6.1 Volumetric flasks, class A, 100-mL.

6.6.2 Beakers, 30-mL.

6.6.3 Erlenmeyer flask, wide mouth, tapered stopper, 125-mL.

Ultrasonic Agitator

Low Temperature Asher.

Magnetic Stirrer and Teflon coated stir bars

7. Reagents

2-propanol.

Hydrochloric acid, 38% (w/w).

Ammonium Hydroxide, 30% (w/w).

Dilute hydrochloric acid, 6 N. Add 50 mL HCl to 40 mL H_2O. Dilute to 100 mL.

7.5 α-Alumina (Alpha Micropolish, Buehler Ltd).

7.6 Talc, TA-99, MMD = 3.45 µm, MMAD = 5.5 µm, described in Reference 11.3.

Stock standard talc suspension; suspend 10 mg TA-99 talc in 100 mL 2-propanol in an Erlenmeyer flask containing a magnetic stir bar. Agitate ultrasonically for two minutes followed by two minutes of stirring.

Stock α-Al_2O_3 suspension; suspend 30 mg α-Al_2O_3 in 100 mL 2-propanol as described in Section 7.7.

8. Procedure

Cleaning of Equipment.

8.1.1 Glassware. New glassware must be cleaned by soaking in hot, concentrated nitric acid, followed by thorough rinsing with distilled or deionized water. Then, after each use, the glassware should be washed with, in order, detergent solution, tap water, dilute nitric acid (soak 4 hours or longer), and distilled or deionized water.

8.1.2 Silver membrane filters. If the qualitative scan described in Section 3 indicates use of the 006 talc peak at 28.59 °2θ, it will be necessary to determine whether the silver filters contain any AgCl whose 111 peak at 27.83 ° 2θ interferes. If so, the AgCl may be leached from the filters by soaking in concentrated NH_4OH for two hours followed by two rinses in 2-propanol and air drying.

Collection and Shipping of Samples

8.2.1 Assemble the filter in the cassette and close firmly to ensure that a seal is made around the edge of the filter (the filter is supported by a cellulose backup pad). Apply a shrinkable cellulose band to the outside of the cassette.

8.2.2 Attach nylon cyclone to cassette and remove cap. Remove the plugs from the cassette and attach to the personal sampling pump by means of flexible tubing. Clip the cassette, face down, to the worker's lapel. Air should not pass through any hose or tubing before entering the cassette.

8.2.3 Take the sample at an accurately known flow rate as close as possible to 1.7 L/min. A sample size of 200-L is recommended. Check the pump frequently during sampling to ensure that the flow rate has not changed. (If sampling problems preclude the accurate measurement of air volume, discard the sample.) Record the sampling time, flow rate, and ambient temperature and pressure.

8.2.4 With each batch of ten samples or less, submit one filter as a blank. Blanks should be from the same lot used for sampling, and should have been subjected to step 8.2.1.

8.2.5 Ship the cassettes so as to prevent excessive vibration or jarring to the cassette.

Analysis of Samples

8.3.1 Open the cassette filter holder and carefully remove the filter with tweezers and transfer it to a 30-mL glass beaker.

8.3.2 Ash filter in low temperature asher for 45 minutes.

8.3.3 Add 2 mL 6 N HCl to residue; agitate ultrasonically for 30 seconds and let stand for 3 minutes.

8.3.4 Turn on the magnetic stirrer under the α-Al_2O_3 suspension and stir for one minute. Turn the stirrer off and immediately withdraw 2 mL of the suspension using a micropipet with disposable tip and transfer to the sample beaker. Agitate ultrasonically for 2 minutes.

8.3.5 Mount silver membrane filter under filtering tower. With vacuum off, add approximately one half inch 2-propanol to the filtering tower. Pour the contents of the sample beaker into the tower with a minimum of splashing and apply vacuum. As the liquid level in the tower permits, rinse the beaker into it with 2-propanol. As the level in the tower approaches the filter, wash down the sides of the tower with 2-propanol from a squeeze bottle being careful not to shoot the stream directly into the liquid. Add no more 2-propanol after the filter surface is exposed.

8.3.6 Let air continue to be pulled through the filter until the sample is dry before releasing the clamp and removing the filter.

8.3.7 Analyze the talc peak selected in Section 3 by step scanning the peak and integrating the counts. Count the background on each side of the peak for one half the time used for peak integration, and add the counts from each side to obtain an average background. The difference between the integrated peak counts and the average background counts is the net peak counts. Matrix correction is recommended(11.2).

9. Calibration and Standardization

Prepare working standards by pipetting aliquots of the stock standard suspension onto polyvinyl chloride filters. Working standards should contain 0, 20, 50, 100, 200, 400, and 800 µg talc per filter, i.e., 0.20 mL, 0.50 mL, 1.00 mL, 2.00 mL, 4.00 mL, and 8.00 mL stock suspension. Subject all standards to the same procedure as the samples, steps 8.3.3 - 8.3.7.

Using a least squares linear regression (or higher order fit), generate calibration equations from the net peak counts and prepared concentrations (μg talc) of the standards.

10. Calculations

10.1 From the calibration equations obtained in Section 9, convert the sample net peak counts to μg talc/sample (W).

For personal sampling pumps with rotameters only, the following volume correction should be made.

$$V = \frac{f \cdot t}{1000}\left(\frac{P_1}{P_2} \quad \frac{T_2}{T_1}\right)^{\frac{1}{2}}$$

where

V = the corrected volume (cubic meters) of air sampled

f = flow rate sample (liters/min)

t = sampling time (min)

P_1 = pressure during calibration of sampling pump (mm Hg)

P_2 = pressure of air sampled (mm Hg)

T_1 = temperature during calibration of sampling pump (K)

T_2 = temperature of air sampled (K).

Calculate the talc concentrations (C) in the air sample ($\mu g/m^3$) using the formula:

$$C = \frac{W}{V}$$

11. References

Backup Data Report for Respirable Talc, prepared under NIOSH Contract 210-79-0060.

P&CAM 259, NIOSH Manual of Analytical Methods, Volume 5 (NIOSH Publication 79-141).

Data Summary for Talc (TA-99), prepared under NIOSH Contract 210-75-0043.

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