FILE NAME: Scotts (SCTS)

DATE: 1977 Dec 8

DOC#: SCTS021

DOCUMENT DESCRIPTION: Report - Scotts Turf Builder Field Tests

RECD DEC 9 1977

GRACE

Construction Products Division

PLAINTIFF'S EXHIBIT SCO-11

To: E. S. Wood

Date: December 8, 1977

From: F. W. Eaton

Subject: Field Report/
Scotts Turf Builder

cc: W. R. Hanlon
J. W. Wolter

Per your request, a field test report on the application of Scotts Turf Builder at Wagog Gardens Apartment complex is attached.

F. W. Eaton

FWE/cc

Attachment

Subject: Scotts Turf Builder Field Tests

Purpose: To determine user exposure to tremolite fiber during the application of Scotts Turf Builder at normal feed rate.

Test Location and Date: Nagog Gardens Apartment Complex, Acton, Massachusetts - November 14, & 15, 1977

I. Test Equipment and Material

1. New Model 35 Scotts spreader with guide markers.
2. Fifteen (15) bags of Scotts Turf Builder (5000 sq. ft. coverage/bag) received by Dr. H. C. Duecker - November 11, 1977 from O. M. Scott.
3. Thirty (30) lbs. flour.
4. 2" X 2" X 6' stakes for mounting area sampling pumps.
5. Seven (7) Bendix super sampler pumps Model BDX 31 calibrated November 3, 1977.
6. Cassettes with Millipore fibers cat. no: AAWP03700.
7. Miscellaneous items such a sampling thief, stop watch etc.

II. Test Procedure

A. General

Nagog Gardens was selected as the test site as it represents a typical residential area where lawns would be fertilized during the normal coarse of home upkeep. This site, with over 100,000 sq. ft. of grass area, would allow air sampling while fertilizing over an extended period of time.

The tests were conducted with two people - one filling spreader hopper and fertilizing and the other maintaining log sheets, time and pump calibration.

B. Area Samples (Refer to Attach Area Sketch)

Area sampling pumps were located as shown on the attached sketch with the sampling filter 5 ft. above ground level. Filters faced the following direction during the sampling period:

A-1 & 5	West
A-2, 6, & 9	East
A-3, 7, & 11	South
A-4, 8, & 12	North
A-10	South

Wind direction during the sampling period November 14, 1977, was variable W. - S.W. and southerly on November 15, 1977. Sampling pump flow rate was set for 1.6 l/min and checked at 30 minute intervals.

Area samples (A-1, 2, 3 & 4) were taken for 180 min. November 14, 1977, A.M. prior to application of Turf Builder. Samples A-5, 6, 7 & 8 were taken November 14, 1977, during the fertilizing period of 494, 496 and 488 Nagog Gardens. Samples A-9, 10, 11 & 12 were taken November 15, 1977, while fertilizing 492 and 490 Nagog Garden.

C. Application of Turf Builder

Two sampling pumps with filter cassettes located in the left and right breathing zone, were worn by the applicator during the sampling/fertilizing period. The applicator filled the spreader hopper to within 2" of the top and refilled when hopper was approximately 3/4 empty. Direction of fertilizing is shown on the attached sketch by broken lines. The applicator maintained a constant walking pace and took one 5 minute coffee break during the sampling period at 492 Nagog Garden. No breaks or rest periods were taken during the other sampling periods.

The lawn area fertilized was from the back of each building to the street/parking area except at 490 Nagog Garden, the area went 50 ft. west of the building. Filter cassettes and sampling period were changed for each apartment. During the 350 min. sampling period, 12-5000 sq. ft. bags of Turf Building were spread.

III. Summary and Interpretation of Results

A. General

Laboratory analysis results by phase contrast illumination of sampled filters are as follows:

Sample No.	No. of Fields Read	Total Fibers Counted	Analysis (f/ml) (see note)
Area Samples			
A-1	100	0	<0.01
A-2	100	0	<0.01
A-3	100	0	<0.01
A-4	100	0	<0.01
A-5	100	0	<0.01
A-6	200	1	0.01
A-7	100	0	<0.01
A-8	200	1	0.01
A-9	100	2	0.02
A-10	100	0	<0.01
A-11	100	0	<0.01
A-12	100	0	<0.01

Sample No.	No. of Fields Read	Total Fibers Counted	Analysis (f/ml) (see note)
Applicator Samples			
TB-1	100	0	<0.04
TB-2	100	0	<0.04
TB-3	100	0	<0.03
TB-4	100	0	<0.03
TB-5	100	0	<0.04
TB-6	100	0	<0.04
TB-7	100	0	<0.03
TB-8	100	0	<0.03
TB-9	100	0	<0.02
TB-10	100	0	<0.02

Note:

1. Fiber Analysis Formula (f/ml)

   $f/ml = \frac{KxF}{VxT}$

   K = constant 342,000 (reticle calibration)
   F = no. fibers/field
   V = flow rate (ml/min)
   T = time (min)

2. Less than (<) value means, no fibers were observed in the counted fields. For statistical reason where no fibers are observed or counted, Grace plugs in one (1) fiber in the analysis calculation so that no answer is zero (0) f/ml. The < sign clarifies the quantitative value.

B. Area Samples

During the sampling period to collect background data (A1-5), the analysis showed no fibers. During the application of Truf Builder November 14, 1977, one (1) fiber was counted in 200 field on sample A-6 upwind and A-8 downwind of the test site. It should be pointed out that samples A-2, 6 & 9 were taken at the property boundry line between 494 and 496 Nagog Garden approximately 70 ft. in back of the apartments. Based on wind direction November 14, 1977, the fiber observed in sample A-6 came from the ambient air and not from the application of Turf Builder. Due to the wind direction shifting from west to southwest and sequence of fertilizing 494, 496 and 498 Nagog Garden, it is unlikely the fiber counted in sample A-8 was the result of the application of Truf Builder.

During the application of Turf Builder November 15, 1977, two (2) fibers were counted on sample A-9. Due to the sampling location, wind direction and fertilizing locations, the observed fibers are believed to be in the ambient air.

C. Turf Builder Applicator Samples (Personnel)

No fibers were observed or counted in the ten (10) applicator samples during the 350 minute test period.

Although basically meaningless, the time weighted average (TWA) user exposure for applying Turf Builder is <0.0076 f/ml, (Grace's consumer exposure time factor for applying lightweight lawn fertilizer is 2/8 or 2 hr. in an 8 hr. day).

IV. Conclusion

Based on approved membrane filter sampling and phase contrast illumination analytical techniques, the consumer is not exposed to fibers in the normal application of Turf Builder to lawns.

Based on sampling conditions listed in other sections of this report, it is assumed that fibers counted in area samples are from the ambient air and not related to the application of Turf Builder.

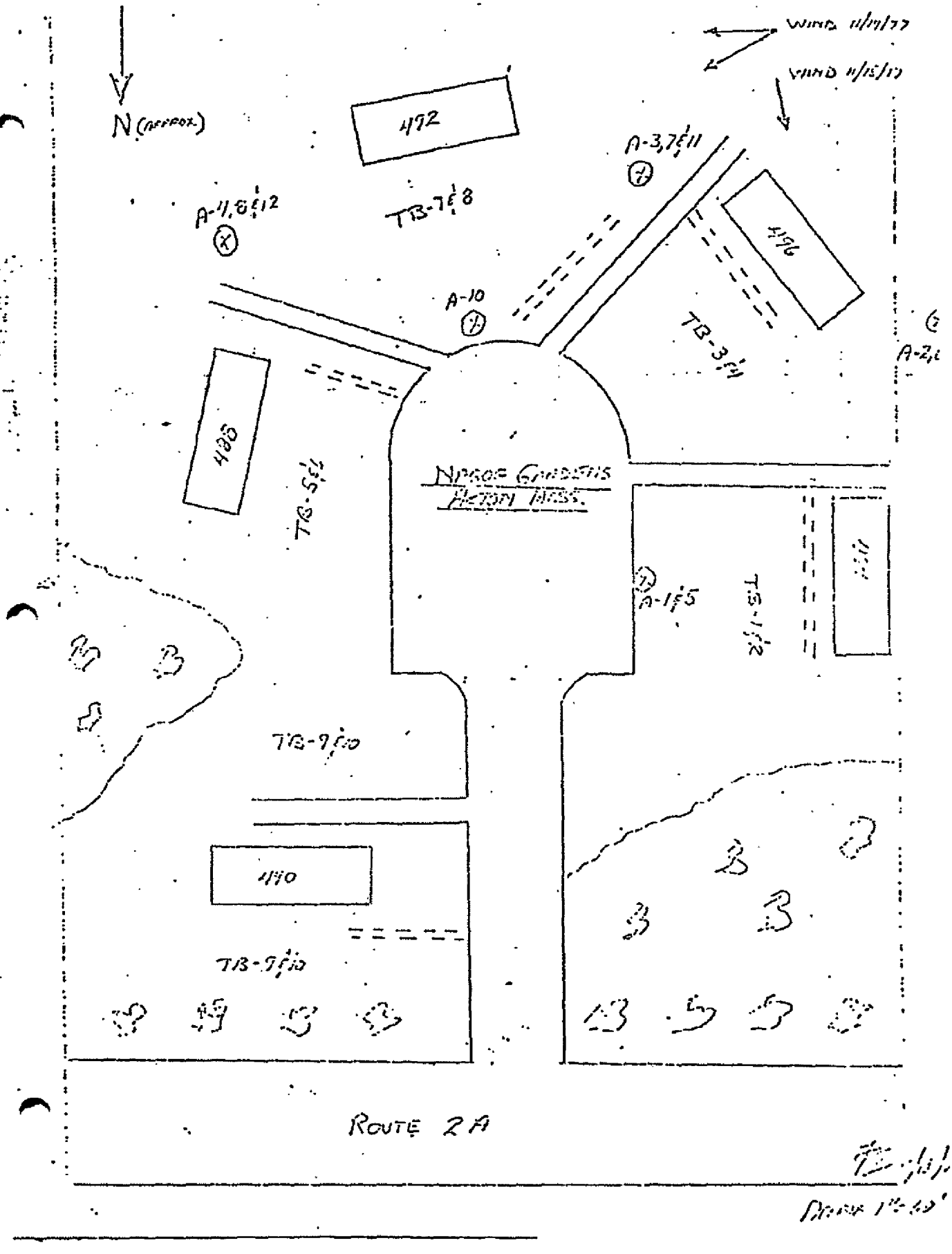
F. W. Eaton

FWE/cc

Attachment:

(1) Air Sampling Record Sheets (4)

(2) Area Sketch


WIND 11/9/77
WIND 11/15/77
N (APPROX.)
492
A-3, 7 & 11
A-11, 8 & 12
TB-7 & 8
496
A-10
TB-3 & 4
A-2, 6
486
TB-5 & 6
NAVAL GUIDANCE
TEST LABS.
A-1 & 5
TB-1 & 2
TB-9 & 10
490
TB-9 & 10
ROUTE 2A

SC-000094

STATE

AIR SAMPLING RECORD SHEET

CONFIDENTIAL

LOCATION: Nagoo Gardens/Eaton Dam SAMPLING CONDITIONS:

CONTAMINANT: Fibers

SAMPLING BY: F.W. Eaton/S. Lowry

DATE: 11/14/77

OUTSIDE: Cold, Clear, Light Wind

INSIDE DRAFT: _____

VISIBLE DUST: _____

HOUSEKEEPING: _____

Scott Tube Bundle Tests

Sample Number	Employee Name	Job Location and Description	Remarks	Pump Number	Pump Off	Pump On	Sampling Time	Flow Rate	Total Sampled Volume	Lab Evalu
A-1		Area Sampling	Prior To Removing Shafts	100-1	1015	1215	120	1.5		<0.
A-2		Tube Bundles To Load. See Attached		100-4	1340	1540	120	1.6		<0
A-3		Sheet For Specific Location & Duration		100-5	1130	1330	120	1.4		<0
A-4				100-8	1035	1235	120	1.6		<0
A-5		Area Sampling	During Tube Preparation	100-1	1010	1255	195	1.6		<0
A-6		Of Scott Tube	Bundles To Manufacturer.	100-4	1005	1305	195	1.5		0.0
A-7		Control Area, Shaft Loading & Packed		100-5	1010	1300	200	1.5		<0.
A-8		As Sample A-1 To A-4		100-8	1005	1305	200	1.5		0.

Additional Comments: _____

Laboratory Evaluation By: [illegible]

Date: [illegible]

SC-000094

GRACE

CONFIDENTIAL

AIR SAMPLING RECORD SHEET

PLANT LOCATION Nagog Gardens / Acton, Mass SAMPLING CONDITIONS: HOUSEKEEPING:

CONTAINMENT FIBER OUTSIDE Cloudy - Cool

SAMPLING BY F.W. Eaton / G. Kinney INSIDE DRAFT

DATE: 11/15/71 VISIBLE DUST

Scotts Turf Builder Tests

Sample Number	Employee Name	Job Location and Description	Remarks	Pump Number	Pump Off	Pump On	Sampling Time	Flow Rate	Total Sampled Volume	Lab Eval
A-9				1	1310	1320	210	1.6		0.4
A-10	& Area	Sampling During the Application		2	1305	1315	210	1.6		<0
A-11	of Scotts Turf Builder. See Attached Sheet			4	1159	1325	205	1.6		<0
A-12	For Sample Location & Direction			5	1155	1320	205	1.6		<0

Additional Comments:

Laboratory Evaluation By: Paul Mahn

Date: 11-17-71

SC-000094

GTSAC

AIR SAMPLING RECORD SHEET

CONFIDENTIAL

SHIP LOCATION: NASA Goddard/Green Mtns
CONTAINMENT: Fiber
SAMPLING BY: F. Lafond / G. Lucy
DATE: 4/19/17

SAMPLING CONDITIONS:
OUTSIDE Cold Clear, Light Snow
INSIDE DRAFT
VISIBLE DUST

HOUSEKEEPING:

Scott Type Breathing Tanks — Time by Stop Watch

Note Ref	Employee Name	Job Location and Description	Remarks	Pump Number	Pump Off	Pump On	Sampling Time	Flow Rate	Total Sampled Volume	Lab Evaluation
-1	G. Lucy	Scrubbing Scott Tank Holder w/ Sponge		100-5 100-6		1519	60	1.6		< 0.
-2		Mask 35 Scrubbing w/ Bronze Brush		100-7		1519	60	1.6		< 0.
-3		Rinse - Setting Up, Mix Solution in				1520	20	1.6	< 0.03	< 0.0
-4		Rinsed w/ Clear Water, Scrubbing				1535	79	1.6	< 0.03	< 0.0
-5		Hard Corners Cannot Reach on Inner Fl.				1539	53	1.6	[illegible]	< 0.
-6		Light Corrosion, See Attached Sheet For				1539	53	1.6	[illegible]	< 0.
-7		Location of Recommended Tie Rod Samples								
-8		Recommended Wire Brushing At Light Rod Corrosion Areas								
		After 3 Min Ground Rod on TB-6 Flat								
		100-5 To 100-2,								

Additional Comments: 100-6 Last Sample; 100-7 Next

Laboratory Evaluation By: [illegible]

Date:

SC-000094

GRACE

CONFIDENTIAL

AIR SAMPLING RECORD SHEET

PLANT LOCATION NYAGG GARDEN CITY, N.Y. SAMPLING CONDITIONS: HOUSEKEEPING:

CONTAMINANT FIBER OUTSIDE CLOUDY - COOL

SAMPLING BY F. W. EATON INSIDE DRAFT

DATE: 11/15/77 VISIBLE DUST

TIMES BY STAT LATCH

Sample Number	Employee Name	Job Location and Description	Remarks	Pump Number	Pump Off	Pump On	Sampling Time	Flow Rate	Total Sampled Volume	Lab Evaluat
S-7	G. Haney	SPRAYING SCOTCH DUCBOARD W/ SCOTCH		100-6		1320	6.5	1.6		<0.02
S-8		MODEL 35 SPECTROMETER @ SAMPLING W.		100-7		1322	6.5	1.6		<0.0
		ASSEMBLY @ 4TH FLOOR ED								
S-9	F. Eaton	ASSEMBLY @ 4TH FLOOR ED.				1505	10'	1.6		<0.0
S-10						1505	10'	1.6		<0.0
-11		TESTS CONDUCTED @ 1765 W/ 1ST DUCT TO DETERMINE DURING TIME								
-12		350 MIN (SIX-SIXTY) SPRAYING/SAMPLING PERIOD, 10-SHEET MASS SPECTRO								
		WITH AN AVERAGE CONTENT OF 60.4% CT. SITE ATTACHED SHEET FOR								
-13		AREA CLOSED. IF ANY SAMPLE HAS AN ASBESTOS CONTENT OF LESS THAN 0.2%								
-14		MAY NOW LEAVE ROOMS CONTAINMENT FOR PURPOSES WITH THE SPECIFIED WORK HAS BEEN								

Additional Comments: GAVE OUT.

Laboratory Evaluation By: Diane Hale

Date: 11-17-77

SC-000094

CAMBRIDGE

C O N F I D E N T I A L

TO: H. C. Duecker

DATE: November 29, 1977

FROM: Julie C. Yang

SUBJECT: Turf Builder Analysis

CC: F. W. Eaton
R. C. Ericson
L. G. Feldman
W. R. Hanlon
D. M. Kirven
R. M. Vining
J. W. Wolter
E. S. Wood
File: 70-974

SUMMARY

Turf Builder from O. M. Scott, which has been tested at Nagog Gardens, Acton, on November 14 and 15, was analyzed for the tremolite content.

All the slow-releasing fertilizer was removed by repeated boiling water extraction. Turf Builder was found to be composed of approximately one-third vermiculite and two-thirds fertilizer. The vermiculite present was a mixture of S.A. and Libby vermiculite, judging from the color appearance.

No detectable tremolite ($< 0.02\%$) was found in the Turf Builder product by x-ray method.

BACKGROUND INFORMATION

Fifteen bags of Turf Builder (coverage 5000 sq.ft./bag) were received from O. M. Scott Company. A field test was conducted for respirable fiber content evaluation at Nagog Gardens in Acton on November 14 and 15 (T&A 67629).

Two composite samples from tests made on the two separate days were submitted for analysis.

EXPERIMENTAL

1. Extraction

Because of the saturation of slow-releasing fertilizer on vermiculite, the material (about a 200 g sample) had to be leached with fresh boiling water repeatedly (a total of 12 liters of water used - leached for six times), and then a carefully quartered sample was extracted in a Soxhlet extractor.

To: H.C.Duecker
From: J.C.Yang

Turf Builder Analysis
Date: November 29, 1977

Total water soluble (average of two determinations):

1) from leaching	62.30
2) from Soxhlet extraction	2.99
	65.29%
Total vermiculite and others	34.71%

2. Tremolite Analysis

The wet vermiculite from Step 1 was dried in the oven, then floated. The heavy particle portion is 37.7% (average of 2) of the sample which corresponds to 37.7 x 34.71 = 12.96% of the total sample.

The heavy particle portion was ground to -100 mesh and x-rayed. No detectable tremolite was found by x-ray diffraction method. Since the limit of detection by x-ray method is 0.2%, the limit for total tremolite present was less than 12.96 x 0.2% = 0.025% of the total sample.

3. Sieve Analysis

The leached vermiculite was sized. The particle size distribution is listed as follows:

	Cumulative % Retained	Spec. (L-4)
+30M	20.45	15-65
-30 +50M	72.5	60-90
-50 +100M	92.9	90-100
-100 +200M	97.9	---
-200M	100.0	---

4. Bulk Density

The bulk density of the vermiculite after water extraction treatment was 23.85 PCF. The sorption of fertilizer and granulation process had broken down some of the particles and also densified the product.

5. Visual Observation

The color appearance indicated the vermiculite used in the Turf Builder is a mixture of Libby and S.A. vermiculite.

To: H.C.Duecker
From: J.C.Yang

Turf Builder Analysis
Date: Nov. 29, 1977

CONCLUSIONS AND COMMENTS

The analysis of Turf Builder showed the product is composed of one-third vermiculite and two-thirds fertilizer, approximately. By color appearance it is a mixture of S.A. and Libby vermiculite, and no detectable tremolite was found in the product ($<0.025\%$).

Julie C. Yang

JCY:mlr

GRACE

Construction Products Division

PROCEDURE

FOR

FIBER COUNTING

W. R. GRACE & CO.

I. EQUIPMENT NEEDED FOR SLIDE PREPARATION

1. Mounting medium - see Appendix for preparation.

2. Ross Optical Lens Tissue - this lens tissue is preferred because of its superior cleaning qualities. It may be purchased from Ladd Research Industries, P.O. Box 901, Burlington, Vermont 05401. A dozen boxes at 1000 sheets each should last some time.

3. 10 gross of 75 x 25 mm precleaned glass slides with one end frosted. Fisher Scientific supplies these as item 12-552.

4. 1 case of 22 x 22 mm no. 1.5 (0.16 to 0.19 mm thick) cover glasses. Fisher item 12-541B.

5. 2 curved forceps. Fisher item 8-875.

6. 2 dissecting knife handles. Fisher item 8-913.

7. 6 dissecting knife blades. Fisher item 8-916D.

8. 1 package of 125 mm x 3 mm stirring rods. Fisher item 11-380A.

9. Pencil.

10. Small spatula or knife (aside from the one used to cut the filter paper) to cut the tape around the cassette.

11. Quarter or nickel for prying open cassette.

II. SLIDE PREPARATION

1. Place clean lens tissue on desk.

2. Wipe dissecting knife, forceps, and 1 stirring rod end with another piece of lens tissue using a separate section of the tissue for each instrument. After wiping each instrument, it should be placed on the tissue in step (1). The wiping tissue should be thrown away.

3. Place a second piece of clean lens tissue on the desk.

4. Take hold of a glass slide by its edges at the frosted end and hold it up to the light. Remove any visible dirt by wiping with lens tissue. Put the slide down on the clean tissue in step (3) frosted side up, and replace the cover on the box of remaining slides.

5. Take hold of a cover glass by its edges, hold it up to the light checking for cleanliness and, again, remove any visible dirt by wiping with lens tissue. Place it on the clear edge of the glass slide so that it overhangs enough so that one of its edges is resting on the lens paper. Close the cover on the remaining cover glasses.

6. Cut the sealing tape on the air sample cassette with a spatula, knife, or fingernail.

7. Loosen the cassette by prying around the edges with a coin (nickel or quarter should do) but do not remove the cover yet.

8. Place a drop of mounting medium (see Appendix for preparation) in the center of the slide. This should be done with the stirring rod which comes with the Balsam bottle. The first drop should be permitted to fall back into the bottle; the second drop should fall on the slide (never touch the slide with this rod). Always place the cover on the bottle after you are finished putting the drop on the slide.

9. Pull one edge of the drop of mounting fluid, on the slide, out so as to get a pear shaped drop. This is done with the clean stirring rod on the tissue from step (2). After doing this, the stirring rod should be wiped dry with the center of another piece of lens tissue and replaced in its original position. Don't discard the cleaning tissue yet.

10. Remove the top of the sample cassette completely.

11. Holding the cassette containing the filter paper firmly on the desk with one hand, cut a pie shaped segment from the filter paper with an arc of about one cm. using the dissecting knife. The cuts should be made starting from the filter paper's center and extending out to its edge. Wipe both sides of the scalpel blade carefully with a clean portion of the paper used to wipe the rod in step (9) and replace the knife in its original position. (Wipe the blade pulling the cutting edge toward yourself rather than pushing it toward you. The latter technique results in rather bloody cuts.) Again, don't discard the cleaning tissue.

12. Remove the pie shaped filter segment from the cassette using the curved forceps and place it on the drop of mounting medium so that the drop and segment shapes correspond. The paper should be grasped by its circumferential edge with the forceps, to minimize disturbing the dust on the paper's surface. Replace the cassette cover.

13. Place the cover glass over the sample. Wipe the forceps clean with a clean portion of the paper from step (11), and now discard the latter. Apply a gentle pressure on the cover glass using the convex curve of the forceps for about 30 seconds. Then put the forceps back in their original position.

14. On the frosted end of the slide write in pencil, using a three-line format, giving sample identification, date of slide preparation, and your initials. Put the slide aside on a piece of lens tissue.

15. Repeat the pressure described in step (13) two more times - once after each of the next two slide preparations. This should clear the central portion of the filter paper leaving only the three corners of the original pie visible if the size of the mounting medium drop was correct.

16. A new lens tissue should be set out for the preparation of each new slide (step 3).

17. The slide is ready for reading 0.5 hour after preparation. But waiting for 0.5 to 1 day is better, because there is some early graininess to a fresh slide which makes it more difficult to read. Slides should be read within 5 days after preparation to prevent any difficulties from possible crystallization of the mounting medium.

III. MICROSCOPE PRELIMINARIES

This section is written in general terms since the procedures are dependent on the make and model of the microscope used.

The Porton reticle should be calibrated against a slide micrometer. Ours has been adjusted to 50 microns to a side, resulting in a counting field area of 0.0025 mm^2.

Place a sample slide on the stage as for counting. Then:

1. Focus on some particulates on the filter paper area of the slide.
2. Adjust the light source for maximum brightness.
3. Examine the phase rings for correct overlap. Adjust if necessary.
4. Close the illuminator iris and adjust the substage condenser until the image of the iris opening is sharp and on the verge of changing colors (blue to red or vice versa).
5. Open the illuminator iris until the field of view is just filled with light.
6. Adjust the fine focus to get the best image.

I have found that a single check of the phase rings per day is sufficient unless the objectives are changed to some other power. This usually disrupts ring alignment in our scope (as do the vibrations of a passing freight train) and step 3 above must be repeated. Adjustment of the substage condenser is more variable and can easily be checked for each slide with our microscope.

IV. COUNTING

After the preliminaries of section III have been completed, the slide is ready to be counted. We count 100 fields. However, if time is available, more fields should be counted to get a statistically more reliable figure, especially if the fiber counts are low. The 100 fields are selected at random in ten sweeps across the filter paper area with ten stops during each sweep. We stay away as best we can from the edges, the apex, and the arc of the original pie shaped segment.

Only those fibers in the left hand square of the Porton reticle are counted. This square constitutes the counting field. A decision should be made prior to counting concerning fibers which cross the boundaries of the square field. Two adjacent sides of the square should be chosen so that fibers crossing those sides are counted while fibers crossing the other two sides are ignored even if a portion of them lies within the field.

Only those fibers greater than 5 microns in length are counted. Lengths may be estimated by comparison with the hash marks along the left side of the Porton field square. (Based on our calibration, the distance between the closest marks in our microscope is conveniently set at 5 microns.) The circles along the bottom edge of reticle may also be used. Only those fibers whose length to diameter ratio (aspect ratio) is greater than 3 should be counted. Do not count fibers whose diameter is greater than 3 microns, and do not count as fibers crystals or slivers of material. Crystals can be particularly deceptive if an edge is visible and the refractive index of the crystal is a near match for that of the mounting medium (a good example of this problem is perlite).

V. CALCULATION OF RESULTS

The calculation of the number of fibers/ml. of air samples is done as follows:

(1) the sampling area of Millipore filter paper AAWP03700 is 855 mm^2 (we have checked this and found it to be correct). However, you should doublecheck this to make sure for your filter paper.

(2) divide the total sampling surface area of the filter paper (855 mm^2) by the area of one counting field to get the total number of possible fields the filter paper may be divided into (in our case this is 855 ÷ .0025 = 342,000).

(3) multiply the result in step (2) by the average number of fibers found per field to get the total number of fibers on the whole filter disc.

(4) multiply the total time air was sampled by the pump (in minutes) by the flow rate through the pump (in ml./minute).

(5) divide the result in (3) by the result of (4) to get the final answer in terms of fibers per ml. of air.

These calculations are summarized by the following formula and example.

$$\text{fibers per ml. of air} = \frac{K \times F}{V \times T}$$

where, T = sampling time in minutes
V = air flow rate through pump in ml. per min.
F = average number of fibers per field
K = total sampling filter disc surface area divided by area within one counting field (both surface areas should be calculated using the same units, i.e., cm^2 or mm^2, etc., and only the actual sampling area of the filter paper should be included and not that covered by the upper plastic rim of the cassette).

Example:

T = 15 minutes
V = 1.5 liters/min. or 1500 ml./min.
F = 6 fibers/40 fields = 0.15 fiber/field
K = 855 mm^2 ÷ .0025 mm^2 = 342,000

$$\frac{342{,}000 \times 0.15}{1500 \times 15} = 2.28 \text{ fibers per ml. of air}$$

APPENDIX

1. Add 15 ml. of dimethyl phthalate to 15 ml. of diethyl oxalate and stir thoroughly to get complete mixing (use very clean equipment throughout to avoid contamination).

2. Weigh out 1 g. of Millipore AASP03700 filters (these are the same as the filters used to collect the dust samples) and place these in a Balsam bottle (Fisher Cat. No. 3-082).

3. Add 20 ml. of the solution prepared in Step (1) to the Balsam bottle and stir the contents briefly. Repeat the brief agitation once a day for about three or four days by which time the filter paper will have dissolved leaving a slightly cloudy solution ready for use as the mounting medium.

4. Always cover the Balsam bottle when not preparing or using its contents.

* * * * * * *

Dec. 13, 1977

GRACE

Construction Products Division

PROCEDURE FOR TREMOLITE ANALYSIS OF SCOTT'S TURF BUILDER

I. Solutioning of Fertilizer Coating

A 200 g representative sample was boiled in 2 l. water for 30 minutes, then filtered immediately to avoid a partial precipitation of the fertilizer.

The procedure was repeated six times, then the solid that remained was filtered and then dried in an oven at 110°C overnight, and weighed.

If most of the fertilizer was not removed, a solid cake will form after drying, the solutioning procedure should be repeated. Otherwise, the solid will appear fluffy in individual grains or small aggregates.

II. Soxhlet Extraction

The last trace of the fertilizer present was removed by Soxhlet Extraction.

A sample was weighed carefully on an analytical balance to 4 decimal places (approximately 12 g by weight). It was placed in a 33 x 94 mm Whatman extraction thimble and then in a Soxhlet Extractor fitted with a 24/40 500 ml round-bottom flask in a heating mantle. 300 ml of distilled water was used for extraction, and extraction was carried out for 8 hours. Duplicate runs were made.

When the extraction was completed, the solid residue was dried in the thimble in an oven, overnight, at 110°C and weighed.

III. Particle Size Distribution

The solid residues from Step II were combined and analyzed for particle size distribution. Sieves used were 30M, 50M, 100M, 200M and pan.

IV. Flotation of Vermiculite

The fractions from Step IV were recombined and mixed thoroughly. The solid (about 20 gram total) was then transferred to a 1 l. beaker to which 600 ml of water was added. The mixture was agitated by a spatula and allowed to stand for about a minute, then floated.

It was observed that all the light, well-expanded vermiculite will float on top of the beaker, whereas the partially or unexpanded vermiculite, rocks and tremolite sank to the bottom of the beaker due to substantial density difference.

PROCEDURE FOR TREMOLITE ANALYSIS OF SCOTT'S TURF BUILDER (cont.)

Caution: The flotation should be carried out in the time described; prolonged standing will cause the vermiculite to absorb water and sink slowly.

The vermiculite was then collected in a Buchner funnel, dried in the oven overnight and weighed. The sink portion was also collected in the same manner.

V. Tremolite Analysis

The heavy particle portion was pulverized in a Spex mill until all the material passed 100 mesh screen. The resultant powder was subjected to quantitative x-ray analysis.

A calibration curve of tremolite in a matrix of vermiculite and associated minerals up to 15% tremolite was prepared, based on the diffraction peak intensity at $2\theta = 10.55°$. Triplicate runs were made for each sample.

The detection limit of tremolite in a sample by this technique is 0.2%.

Tremolite in the vermiculite portion was analyzed by the same method.

VI. Calculations

(1) Total water solubles = Amount solubilized by water boiling - Step I + Amount solubilized by Soxhlet Ext. - Step II

(2) $$\text{Total solids} = \frac{\text{Wt. of sample} - \text{wt. of water soluble}}{\text{wt. of sample}}$$

(3) $$\text{Tremolite content} = \left(\begin{array}{c}\text{\% of Tremolite in the heavy particle portion determined by x-ray method}\end{array} \times \frac{\text{wt. of heavy particle}}{\text{wt. of total sample}}\right) + \left(\begin{array}{c}\text{\% of Tremolite in the Vermiculite portion}\end{array} \times \frac{\text{Wt. of Vermiculite}}{\text{Wt. of Total Sample}}\right)$$

SCHEMATIC DIAGRAMS FOR TREMOLITE ANALYSIS

Tremolite Determinations in SCOTT'S TURF BUILDER

