

CALIBRATION STANDARDS FOR COUNTING ASBESTOS
CONF-730518-3

LAWRENCE W. ORTIZ
HARRY J. ETTINGER
CHARLES I. FAIRCHILD

LA-UR-73-832

HEALTH DIVISION
LOS ALAMOS SCIENTIFIC LABORATORY
UNIVERSITY OF CALIFORNIA, LOS ALAMOS, NEW MEXICO*

ASBESTOS DUST HAS LONG BEEN RECOGNIZED AS AN INDUSTRIAL HAZARD RESULTING IN ASBESTOSIS, AND ON THE BASIS OF MORE RECENT INFORMATION, LUNG CANCER. INCREASING AWARENESS OF THIS SERIOUS OCCUPATIONAL RISK HAS RESULTED IN THE INSTITUTION OF AN EMERGENCY TEMPORARY STANDARD BY THE SECRETARY OF LABOR, CONCERNING HUMAN EXPOSURE TO ASBESTOS. THIS EMERGENCY STANDARD, PUBLISHED IN THE FEDERAL REGISTER AND EFFECTIVE AS OF JULY, 1972 -- DICTATES THAT THE MAXIMUM PERMISSIBLE OCCUPATIONAL EXPOSURE TO AIRBORNE ASBESTOS FIBERS SHALL NOT EXCEED FIVE (5) FIBERS LONGER THAN 5 μ M PER CUBIC CENTIMETER OF AIR. IN ADDITION, THE FEDERAL REGISTER FURTHER STIPULATES A REDUCTION IN THIS MAXIMUM PERMISSIBLE LEVEL TO 2 FIBERS/CC, BY JULY, 1976. THESE TLV'S ARE 8-HOUR TWA'S AS DETERMINED BY A MICROSCOPIC FIBER COUNTING PROCEDURE. THE STANDARD ALSO SETS A SHORT TERM MAXIMUM EXPOSURE LEVEL OF 10 FIBERS/CC.

TO ASSURE UNIFORM FIBER COUNTING PROCEDURES BY THE VARIOUS HEALTH PROTECTION AGENCIES MONITORING EXPOSURE TO ASBESTOS, A LABORATORY PROFICIENCY COUNTING PROGRAM, CALLED THE PROFICIENCY ANALYTICAL TESTING OR PAT PROGRAM, WAS INITIATED BY THE NATIONAL INSTITUTE FOR OCCUPATIONAL SAFETY AND HEALTH OR NIOSH. THIS PROGRAM WILL PERMIT

*Work supported by the National Institute for Occupational Safety and Health, Cincinnati, Ohio, Project R-072, and Under Contract W-7405-ENG-36 with the U.S. Atomic Energy Commission.

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QUANTITATIVE EVALUATION OF ASBESTOS MONITORING PROCEDURES BY VARIOUS DESIGNATED STATE AGENCIES, AS WELL AS HELP DEFINE THE VARIABLES INHERENT IN FIBER COUNTING. THE BASIS OF THIS STANDARDIZATION PROCESS IS A MICROSCOPIC FIBER COUNTING METHOD ORIGINALLY DEVELOPED BY THE U.S. PUBLIC HEALTH SERVICE. BRIEFLY, THIS TECHNIQUE INVOLVES AIR SAMPLING THROUGH A MEMBRANE FILTER, CLEARING THE MEMBRANE, AND COUNTING FIBERS LONGER THAN 5 μ m UNDER PHASE CONTRAST OPTICS AT MAGNIFICATIONS OF 400-430 X.

THE LOS ALAMOS SCIENTIFIC LABORATORY OR LASL -- AT THE REQUEST OF NIOSH -- DEVELOPED A TECHNIQUE FOR THE PREPARATION OF MULTIPLE MEMBRANE FILTER SAMPLES CONTAINING "IDENTICAL AND PREDICTABLE" CONCENTRATIONS OF ASBESTOS FIBERS, WITH CONSISTENT PARTICULATE BACKGROUNDS. THESE SAMPLES WERE TO BE REPRESENTATIVE OF SAMPLES OBTAINED FROM MANY WORK SITUATIONS.

THE NEEDS OF NIOSH, COUPLED WITH THE INHERENT HEALTH HAZARD OF AIRBORNE ASBESTOS DUST DICTATED THAT A SIMPLE, EASILY CONTROLLABLE AND SAFE TECHNIQUE BE USED. THE APPROACH CHOSEN WAS THE FILTRATION OF MEASURED VOLUMES OF DILUTE LIQUID DISPERSIONS OF MILLED CHRYSOTIL ASBESTOS, AND Al_2O_3 THROUGH MEMBRANE FILTERS. THE RESULTANT DEPOSITS ON THE MEMBRANE FILTER SURFACE CAN THEN BE MONITORED BY THE USPHS FIBER COUNTING TECHNIQUE. CHRYSOTILE PRODUCED BY THE INTERNATIONAL UNION CONCERNING CANCER (UICC) WAS SELECTED AS THE BASIC MATERIAL BECAUSE, (1), CHRYSOTILE IS THE MOST WIDELY USED FORM OF ASBESTOS IN INDUSTRY, AND (2), UICC MATERIAL IS READILY AVAILABLE, WELL CHARACTERIZED, USED EXTENSIVELY IN MEDICAL RESEARCH, AND HAS BEEN PROPOSED AS A WORLDWIDE STANDARD FOR RESEARCH PURPOSES. THE UICC MATERIAL REQUIRES NO MILLING TO OBTAIN THE DESIRED RANGE OF

ASBESTOS FIBERS, AS DO OTHER SOURCE MATERIALS.

A BRIEF DESCRIPTION OF THE DISPERSION-FILTRATION TECHNIQUE FOLLOWS: ACCURATELY WEIGHED QUANTITIES OF MILLED CHRYSOTILE ARE DILUTED TO FIXED VOLUMES WITH REAGENT GRADE TOLUENE. THE ASBESTOS IS THEN DISPERSED THROUGHOUT THE LIQUID BY SONICATION (60 HZ) AND INTERMITTANT, VIGOROUS HAND SHAKING, UNTIL UNIFORM TYNDALL LIGHT SCATTER CONDITIONS EXIST. A UNIFORM DISPERSION HAS MANY SMALL FIBERS, BUT VERY FEW LARGE FIBROUS CLUMPS. SEPARATE DISPERSIONS OF Al_2O_3 PARTICLES ARE PREPARED IN SIMILAR FASHION.

THE LOADED FILTERS ARE THEN PREPARED BY SLOW, CONTROLLED VACUUM FILTRATION OF MEASURED VOLUMES OF THE PREPARED DISPERSIONS, THROUGH INDIVIDUAL MEMBRANE FILTERS. THE FILTRATION VOLUMES ARE ADJUSTED TO OBTAIN THE DESIRED FIBER AND PARTICULATE DEPOSIT CONCENTRATIONS ON THE MEMBRANE SURFACE. MULTIPLE FILTERS CONTAINING 4 LEVELS OF ASBESTOS CONCENTRATIONS RANGING FROM 500 TO 1700 FIBERS/MM², A RANGE WHICH IS TYPICAL OF INDUSTRIAL FIELD SAMPLING WERE PREPARED AND SENT TO NIOSH. SAMPLE WEDGES FROM EACH FILTER WERE RETAINED AT LOS ALAMOS.

QUALITY CONTROL WAS ACCOMPLISHED BY MULTIPLE COUNTERS, EACH COUNTING THE SAME WEDGES TAKEN FROM REPRESENTATIVE PORTIONS OF THE FILTERS. RULES FOR COUNTING FIBERS WERE CONSISTENT WITH THOSE SPECIFIED BY NIOSH.

A SAMPLE OF TYPICAL FIBER COUNT DATA OBTAINED ON FILTERS PREPARED BY THE DISPERSION-FILTRATION TECHNIQUE IS SHOWN IN THE FIRST SLIDE. THIS SLIDE ILLUSTRATES TYPICAL COUNT DATA OBTAINED ON SEPARATE FILTER SAMPLES CONTAINING FOUR (4) DIFFERENT ASBESTOS CONCENTRATIONS. THE SIGNIFICANT FEATURE ILLUSTRATED BY THESE RESULTS

ARE THE SPECIFIC LEVELS ACHIEVABLE BY THE DISPERSION/FILTRATION TECHNIQUE. AN ADDITIONAL EXAMPLE (SLIDE 2) OF COUNT DATA SUMMARIZES RESULTS OBTAINED ON TEN "IDENTICAL" FILTERS. THE COEFFICIENTS OF VARIATION, THAT IS, THE STANDARD DEVIATIONS DIVIDED BY THE MEAN, RANGE FROM ± 8 TO $\pm 36\%$.

SPECIFICALLY, THE VARIATION BETWEEN THESE TEN SAMPLES, COUNTED BY 5 LASL COUNTERS IS $\pm 17\%$. IN THIS IDEALIZED SITUATION, EACH OF THE 5 COUNTERS USED THE SAME MICROSCOPE AND ALL COUNTED THE SAME FILTER WEDGES. REPETITION OF THIS EXPERIMENT AT A LATER DATE PRODUCED A VARIATION BETWEEN 4 COUNTERS OF $\pm 20\%$. EXTENSIVE FIBER COUNT DATA FROM THIS QUALITY CONTROL PROGRAM DEMONSTRATES THAT UNDER IDEAL CONDITIONS (I.E., SEVERAL EXPERIENCED COUNTERS COUNTING THE SAME WEDGES AND USING THE SAME OPTICS) VARIATIONS BETWEEN COUNTERS AS LARGE AS $\pm 50\%$ ARE OBSERVED, WITH TYPICAL VARIATION ON THE ORDER OF $\pm 30\%$. MOREOVER, LIMITED UNPUBLISHED DATA CONTRIBUTED BY COUNTING FACILITIES PARTICIPATING IN THE NIOSH PAT PROGRAM INDICATES THAT LARGER VARIATIONS MAY BE EXPECTED WHEN ADDITIONAL VARIABLES ARE INTRODUCED. THESE VARIABLES INCLUDE COUNTERS OF VARIED EXPERIENCE; DIFFERENT SAMPLE WEDGES; DIFFERENT OPTICS; VARIED LEVELS OF SKILL IN LABORATORY AND MICROSCOPY TECHNIQUES; THICKNESS OF BOTH SLIDE AND COVER SLIP; RELATIVE SIZE OF COUNTING FIELD; RELATIVE PARTICULATE AND/OR FIBER CONCENTRATION; AND QUALITY OF OPTICAL MOUNT. ALL THESE ARE SIGNIFICANT FACTORS WHICH AFFECT FIBER COUNT RESULTS. OF ALL VARIABLES, THE TWO MOST IMPORTANT ARE THE QUALITY OF MICROSCOPE AND ATTITUDE OF COUNTER.

A MAJOR REASON FOR LARGE VARIATIONS IN COUNT LIES IN THE FACT THAT FIBERS COLLECTED ON MEMBRANE FILTERS DO NOT ALWAYS APPEAR AS

DISTINCT ENTITIES. THE NEXT SLIDE SHOWS A TYPICAL SAMPLE OF UICC CHRYSOTILE WITH AN Al_2O_3 BACKGROUND. A LARGE PERCENTAGE OF FIBER DIAMETERS, ARE NEAR THE LIMIT OF OPTICAL RESOLUTION. THIS PHOTOGRAPH PROVIDED A MAGNIFICATION OF APPROXIMATELY 500 X. FURTHER DIFFICULTY IS CAUSED BY THE FACT THAT ALL FIBERS DO NOT LIE IN THE SAME FOCAL PLANE, NECESSITATING A CAREFUL 3-DIMENSIONAL SEARCH OF EACH COUNTING FIELD.

A SINGLE EXPERIMENT WAS CONDUCTED IN AN ATTEMPT TO QUANTITATE THIS BUILT-IN VARIABLE OF THE MICROSCOPIC FIBER-COUNTING TECHNIQUE. FOUR EXPERIENCED COUNTERS EACH COUNTED TWO SEPARATE WEDGES FROM REPRESENTATIVE FILTERS PREPARED BY THE DISPERSION/FILTRATION TECHNIQUE. IN COUNTING THESE WEDGES, EACH COUNTER COUNTED THE IDENTICAL OPTICAL FIELD AND RECORDED HIS COUNTS WITHOUT INFORMING THE OTHER COUNTERS OF HIS RESULTS. THE NEXT SLIDE SUMMARIZES THESE DATA OBTAINED UNDER EXTREMELY IDEALIZED CONDITIONS. THAT IS, SEVERAL EXPERIENCED COUNTERS, USING THE SAME OPTICS, EACH COUNTED IDENTICAL VIEWING FIELDS. VARIATIONS OF $\pm 18\%$ AND $\pm 22\%$ ARE OBSERVED, WHICH IS CONSISTENT WITH THE VARIATIONS PREVIOUSLY NOTED BETWEEN "IDENTICAL SAMPLES" PREPARED FOR THE NIOSH PAT PROGRAM. THIS INDICATES THE CONSISTENCY OF THE PREPARATION TECHNIQUE AND SUGGESTS A MINIMUM ERROR WHICH SHOULD BE CONSIDERED WHEN COMPARING COUNT DATA.

THE IMPORTANCE OF THE QUALITY OF MICROSCOPIC OPTICS IS DIRECTLY RELATED TO THE INTRINSIC NATURE OF THE MINERAL ASBESTOS (ESPECIALLY CHRYSOTILE). IT IS WELL KNOWN THAT SAMPLES OF AIRBORNE ASBESTOS MAY CONTAIN MANY FIBERS OF A SIZE WHICH LIE WELL BELOW THE LIMIT OF RESOLUTION OF EVEN THE BEST OPTICAL MICROSCOPES (ESPECIALLY AT 400 TO 500 X). SAMPLES OF ASBESTOS CONTAINING INDIVIDUAL FIBERS

.025 μm - .03 μm

WITH DIAMETERS AS SMALL AS 250 - 300 ANGSTROMS HAVE BEEN MEASURED USING THE ELECTRON MICROSCOPE. THE NEXT SLIDE SUMMARIZING COMPARATIVE FIBER COUNTS OBTAINED USING ELECTRON MICROSCOPY (EM) VERSUS OPTICAL MICROSCOPY (OM) ILLUSTRATES A SIGNIFICANT FEATURE. THE FINAL ENTRY IN THIS TABLE IS OF SPECIFIC INTEREST. THESE FIBER COUNT RATIOS (EM/OM) OF 22 OR 23 INDICATE A POSSIBLE MAGNITUDE OF ERROR ASSOCIATED WITH COUNTS OBTAINED BY OPTICAL MICROSCOPY WHEN TOTAL FIBER CONCENTRATIONS ARE ASSESSED. THESE COMPARATIVE COUNTS WERE OBTAINED FROM A SINGLE FILTER SAMPLE PREPARED BY THE DISPERSION/FILTRATION TECHNIQUE. ELECTRON MICROSCOPE COUNTS WERE OBTAINED BY TRANSFERRING A PORTION OF THE TEST FILTER ONTO EM GRIDS, PHOTOGRAPHING RANDOM FIELDS AND COUNTING THE TOTAL AREAS PRESCRIBED. OPTICAL MICROSCOPE COUNTS WERE OBTAINED FROM A DIFFERENT SECTION OF THE SAME TEST FILTER BY THE CONVENTIONAL PHASE CONTRAST MICROSCOPIC FIBER COUNTING TECHNIQUE. AS NEAR AS PRACTICAL, THE SAME COUNTING CRITERIA APPLIED TO EACH TECHNIQUE.

AN IMPORTANT ASPECT, WHICH SHOULD NOT BE IGNORED, IS THAT THE RELATIVE HEALTH HAZARD ASSOCIATED WITH SMALL FIBERS IS AT PRESENT UNKNOWN. IN FACT, THESE SMALL FIBERS ARE MORE APT TO BE "RESPIRABLE", AND MAY CONSTITUTE A GREATER HAZARD.

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IN CONCLUSION:

EVALUATION OF THE DISPERSION/FILTRATION TECHNIQUE INDICATES THAT THE METHOD WILL PRODUCE ASBESTOS LOADED MEMBRANE FILTERS IN A CONTROLLABLE AND PREDICTABLE FASHION. SAMPLES PREPARED BY THIS TECHNIQUE CAN BE USED TO EVALUATE THE PROFICIENCY OF PERSONNEL RESPONSIBLE FOR FIBER COUNTING.

MINIMUM ERRORS INHERENT IN THE "STANDARD" FIBER-COUNTING TECHNIQUE HAVE BEEN QUANTITATED AND INDICATE THAT MICROSCOPIC FIBER COUNTING MAY UNDERESTIMATE THE POTENTIAL RISK OF HUMAN EXPOSURES TO ASBESTOS DUST. EVEN UNDER IDEAL CONDITIONS MICROSCOPIC FIBER COUNTING MAY BE TERMED AS INADEQUATE, SINCE THE VARIABLES IN THE TECHNIQUE TEND TO REDUCE THE NUMBER OF FIBERS COUNTED AND THUS, AGAIN, UNDERESTIMATE THE ACTUAL EXPOSURE.

THESE LIMITATIONS SHOULD BE CONSIDERED IN CONNECTION WITH THE PROPOSED REDUCTION FROM 5 FIBERS/CC TO 2 FIBERS/CC IN 1976. CONCURRENTLY, IT IS APPROPRIATE TO CONSIDER THE DEVELOPMENT OF ALTERNATE METHODS TO PROVIDE MORE ACCURATE MONITORING OF ASBESTOS EXPOSURE. MOREOVER, THE COURSE OF DEVELOPMENT OF ACCURATE METHODS DEPENDS UPON THE PARALLEL INVESTIGATION OF THE MECHANISM OF FIBROUS PNEUMOCONIOSES. UNTIL IMPROVEMENTS IN MONITORING METHODS ARE REALIZED, INVOLVED AGENCIES AND INDIVIDUALS SHOULD BE AWARE OF THE UNCERTAINTIES IN THE PRESENT METHOD.

ASBESTOS COUNT DATA SERIES 149 - 152
 SAMPLE AND FIBERS/MM² FILTER AREA

COUNTER	15-A	15-B	15-C	15-D
1	706	961	1255	2294
2	863	1255	1216	1882
3	1000	1627	2216	3039
4	470	627	804	1353
AVERAGE:	760	1118	1373	2142
σ :	227	426	598	711
COEF. VAR.:	0.30	0.38	0.44	0.33



SLIDE 1

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ASBESTOS COUNT DATA - SERIES 125
INDIVIDUAL FIBER COUNT DATA - TEN "IDENTICAL"
COUNTER AND FIBERS COUNTED/MM² FILTER A

SLIDE 2

SAMPLE	A	B	C	
1	1207	1414	1172	
2	1586	1690	966	
3	1448	1448	896	:
4	1310	1828	1310	:
5	1793	1724	1310	:
6	1586	1759	1552	:
7	1586	1724	966	:
8	1793	1655	1310	:
9	1241	1655	1276	:
10	1414	1690	1103	]
AVERAGE:	1496	1659	1186	]
σ :	209	131	204	
COEF. OF VAR:	0.14	0.08	0.17	0
MEAN OF FIVE (5) LASL COUNTERS: 1496				
STANDARD DEVIATION (σ): 203				
AVERAGE COEF. OF VAR. 0.19				

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ADJUSTED COUNT DATA - SERIES 123
 INDIVIDUAL FIBER COUNT DATA - TEN "IDENTICAL" FILTERS
 COUNTER AND FIBERS COUNTED/MM² FILTER AREA

	B	C	D	E
7	1414	1172	1621	1828 - 1
6	1690	966	1931	1552 - 2
8	1448	896	2621	1690 - 3
0	1828	1310	2069	1517 - 4
3	1724	1310	2655	1724 - 5
6	1759	1552	1310	1069 - 6
5	1724	966	1103	1241 - 7
3	1655	1310	1103	1345 - 8
1	1655	1276	1517	1276 - 9
1	1690	1103	1000	1207 - 10
1	1659	1186	1693	1445
1	131	204	610	254
	0.08	0.17	0.36	0.18

COUNTERS: 1496

STANDARD DEVIATION (σ): 203

0.19

2

10.



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VICO CHRYSOTILE "B"
with
 AL_2O_3 PARTICULATE EXHIB.



SLIDE 3

GAF 15684

ASBESTOS COUNT DATA

FOUR COUNTERS EACH COUNTING THE SAME TWENTY FIVE FIELDS

COUNTERS	SAMPLE AND FIBERS/CM ² FILTER AREA	
	A	B
1	1034	1966
2	862	2276
3	1000	1862
4	1414	2759
	<u>4310</u>	<u>8863</u>
Avg.	$\div 4 = 1078$	$\div 4 = 2216$
	236	403
COEF. OF VAR.	+22%	+18%


 los alamos
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 at the University of California

SLIDE 4

12.

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COMPARATIVE FIBER COUNTS

ELECTRON MICROSCOPY VS OPTICAL MICROSCOPY

<u>COUNTING MODE</u>	<u>SAMPLE AND FIBERS/CM² FIBER AREA</u>		
<u>OPTICAL MICROSCOPY</u>	<u>A</u>	<u>B</u>	
AVERAGE OF FOUR (4) LASL COUNTERS:			
FIBERS >5 μ m:	1078	2216	
TOTAL FIBERS:	1079	2218	
<u>ELECTRON MICROSCOPY</u>			
FIBERS >5 μ m:	3695	7162	
TOTAL FIBERS:	25261	49684	
*Em/om	FIBERS >5 μ m:	3.4	3.2
Em/om	TOTAL FIBERS	23.4	22.4

$$*Em/om = \frac{\text{FIBER COUNTS OBTAINED BY ELECTRON MICROSCOPY}}{\text{FIBER COUNTS OBTAINED BY OPTICAL MICROSCOPY}}$$



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