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NAVY ASBESTOS IDENTIFICATION PROFICIENCY TESTING PROGRAM

FINAL REPORT TO LABORATORIES - TEST ROUND 117

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Prepared for the

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Section I - Summary of Test Round

The 117th test round of the Navy Asbestos Identification Proficiency Testing Program began with the distribution on 1 May 2012 of test samples to 44 laboratories enrolled for participation. Program enrollment has slowly decreased from a maximum of 103 laboratories in mid-1994, resulting principally from a series of base closures and the decommissioning of numerous ships. That trend continues, with enrollment currently at its lowest level since Test Round 17 in early 1987, when 52 laboratories were enrolled.

Each test set distributed consisted of four samples, three of which contained asbestos. The asbestos-containing lots were a spray-on insulation (Lot B), an RTI-formulated floor leveling compound (Lot C), and a fire door insulation (Lot D). Lot A, a cementitious exterior siding, did not contain asbestos. Complete reference analyses for the four sample lots are provided in Section II of this report.

Grading criteria as set by the Navy and Marine Corps Public Health Center (NAVMCPUBHLHCEN) require laboratories to identify and report all asbestos types present and to semiquantitate fibrous asbestos amounts of <1% (trace) or greater. The evaluation criteria include penalty points for semiquantitative errors. The threshold for failing a test round is 100 penalty points. A laboratory establishes a proficiency rating for each test round based on total penalty points and also establishes a program proficiency status based on its collective performance over a sliding window of four consecutive test rounds. Additional details regarding these changes are available on RTI's Web site for the program (<http://navy.rti.org>).

RTI's competency as a proficiency testing provider is continuously assessed against the requirements of ISO/IEC 17043: Conformity assessment – General requirements for proficiency testing (from the International Organization for Standardization/International Electrochemical Commission). To fully comply with those requirements, RTI routinely solicits information about the test methods and analytical techniques being employed by the program participants and provides a summary of those data, as well as a correlation with errors committed in the test round, in Section

VIII of each Final Report to Laboratories.

As expected, the test round proved to be of slightly above-average analytical difficulty, with 11 identification errors incurred on Lot D, a sample containing two asbestos types. All of the 30 participating laboratories classified all four samples correctly; moreover, 18 submitted results with no classification or asbestos identification errors. No false negative errors were incurred on Lots B, C, and D, and no false positive errors were incurred on Lot A.

Section II - Reference Laboratory Analyses

Two independent laboratories previously approved by NAVMCPUBHLHCEN for this contract provided reference analyses for the test materials. The results of these laboratories' analyses and of RTI's in-house analyses, listed by sample lot, are shown in Table 1. The laboratories agreed on the classification of all four sample lots and on the asbestos types in the three asbestos-containing (positive) sample lots. The correlation of asbestos percentages reported by the three laboratories was fair on the chrysotile contained in Lots B and C, and good on the chrysotile and amosite in Lot D.

Section III - Semiquantitation of Asbestos in Positive Sample Lots

The concentrations of asbestos in the three asbestos-containing (positive) lots were determined by visual estimation, point counting, and semiquantitative X-ray diffraction (XRD), each where applicable. Means and standard deviations were calculated for the point counts, XRD values, and visual estimates of gravimetric residues (if applicable) for the asbestos in each sample and were used to develop two-sided tolerance limits (acceptance ranges). Limits were chosen so as to have 99% confidence that 95% of the reported values would be deemed acceptable. The final acceptance range for a sample used the lowest value among the minimum values and the highest value among the maximum values for all techniques used. Semiquantitative results and acceptance ranges are shown in Table 2. The number of replicate analyses used in each calculation is also indicated.

Table 1. Reference Analysis Results

Sample Lot	Sample Classification	Reference Analysis Laboratory 1	Reference Analysis Laboratory 2	RTI
A	-	1 ----- 50% Cellulose 49% Calcium carbonate and quartz binder 1% Paint Tra Talc and tremolite cleavage fragments	2 ----- 10% Cellulose 90% Gypsum, cement, and unspecified matrix	1, 2, 4 ----- 10% Cellulose 40% Calcium carbonate 50% Quartz
B	+	1, 3 ----- 6% Chrysotile Tra Actinolite Tra Cellulose 45% Vermiculite 49% Calcium sulfate	1, 3 ----- 3% Chrysotile 52% Vermiculite and clay 45% Gypsum and calcareous matrix	1, 3, 4 ----- 5% Chrysotile Tra Actinolite 45% Vermiculite, phlogopite, and sepiolite 50% Gypsum
C	+	1, 3 ----- 9% Chrysotile Tra Cellulose 66% Calcium carbonate and clay binder 25% Quartz aggregate	3 ----- 4% Chrysotile 96% Quartz, feldspars, aggregate, clay, matrix, and pigments Tra Cellulose	1, 2, 3, 4 ----- 5% Chrysotile Tra Cellulose 65% Gypsum and bassanite 30% Quartz, phlogopite, and pigments
D	+	1, 3 ----- 8% Amosite 2% Chrysotile 90% Calcium carbonate and calcium sulfate Tra Cellulose	1, 3 ----- 10% Amosite 1% Chrysotile Tra Crocidolite Tra Cellulose 55% Calcareous binder 34% Silica, clay, and matrix	1, 2, 3, 4 ----- 10% Amosite 1% Chrysotile 25% Calcite 64% Insoluble binder

Numerical Code for Analytical Techniques:

- 1 = Gravimetric reduction by acid dissolution
- 2 = Gravimetric reduction by ashing
- 3 = Point counting
- 4 = Qualitative and/or semiquantitative X-ray diffraction (XRD)

Table 2. Semiquantitative Means and Ranges, and Acceptance Ranges

	Lot B	Lot C	Lot D	
	Chrysotile	Chrysotile	Amosite	Chrysotile
<u>Point Count</u> Mean (%) Range (%) Replicates (#)	5.4 4.0 to 8.0 5	3.4 1.8 to 5.3 6	8.4 4.0 to 10.8 5	0.8 0.4 to 1.5 7
<u>Semiquantitative XRD</u> Mean (%) Range (%) Replicates (#)	4.9 4.5 to 6.0 10	8.2 6.9 to 9.4 8	Not applied	Not applied
<u>Visual Estimates</u> Mean (%) Range (%) Replicates (#)	Not applied	5.8 4.3 to 9.4 16	7.3 5.6 to 9.8 18	0.9 0.4 to 1.5 11
99/95 Acceptance Range (%)	Trace to 20, inclusive	Trace to 20, inclusive	Trace to 30, inclusive	0 to 5, inclusive

Section IV - Individual Laboratory Results

Please refer to the computer printout on page 6 for a tabulation of your individual laboratory results and a comparison of those results to the reference laboratories' values. The total penalty points, test round proficiency status, and test program status for your laboratory appear in the upper right-hand corner of the page, along with the number of false negatives, false positives, asbestos identification errors, and semiquantitation errors, where incurred.

Section V - Total Test Round Effort

Of the 44 laboratories enrolled, 30 submitted results of analyses for the test round, for a response rate of 68.2%. This rate is lower than the 81.8% average participation rate for the 37 test rounds conducted under this proficiency rating format. A laboratory not returning results was deemed nonproficient for the test round unless it had notified the NAVMCPUBHLTHCEN beforehand and received a waiver based on a qualifying

reason for nonparticipation. One laboratory was granted a waiver for this test round.

Tables 3 and 4 illustrate the total test round effort, as generated from the data submitted by participating laboratories. Table 3 shows the distribution of penalty points incurred by proficient (P) and nonproficient (NP) laboratories based on the grading criteria described on pages 7 and 8. A laboratory was rated NP for incurring 100 or more penalty points or for not participating. The total numbers of P and NP laboratories are also indicated.

For the test round, 29 laboratories (65.9% of the total enrolled, 96.7% of the total participating) were rated P. Of the 14 NP ratings assigned for the test round, 1 was incurred for a combination of identification and semiquantitation errors, and 13 were incurred for not submitting results.

Table 3. Distribution of Penalty Points Incurred

	Total Penalty Points Incurred	Number of Laboratories
Proficient Laboratories	0	18
	1 - 24	0
	25 - 49	0
	50 - 74	8
	75 - 99	3
Total Proficient Laboratories		29
Analytically Nonproficient Laboratories	100 - 124	0
	125 - 149	0
	150 - 199	1
	200 or more	0
Nonparticipants		13
Total Nonproficient Laboratories		14
Laboratories with Approved Waivers		1

One-page
“computer printout of graded results”
to be inserted here

Table 4 presents the total numbers of false negatives, false positives, and asbestos identification errors and the grading of asbestos semiquantitation, by sample lot. False negatives and false positives are denoted by “FN” and “FP,” respectively; asbestos identification errors are denoted by “ID”; and asbestos semiquantitation errors are denoted by “SQ.” Totals of all error types incurred for the test round are shown at the bottom of the table.

Table 4. Sample Classification, Asbestos Identification, and Semiquantitation Errors, by Sample Lot

Sample Lot	Asbestos Content	Number of Errors, by Type			
		FN	FP	ID	SQ
A	-	----	0	----	----
B	+	0	----	1	2
C	-	0	----	0	4
D	+	0	----	11	6
Total Errors Incurred		0	0	12	12

FN = False Negative

FP = False Positive

ID = Asbestos Identification Error

SQ = Asbestos Semiquantitation Error

The following evaluation criteria were used to assign these sample classification and asbestos identification errors:

<u>Proficiency Grading Criterion</u>	<u>Penalty Points</u>
Failing to submit analysis results (without waiver granted)	Automatic NP
Reporting asbestos in a blank sample (FP)	100
Failing to report asbestos in a positive sample (FN)	100
Reporting incorrect asbestos type (ID)	45/type
Failing to report a second asbestos type (ID)	50
Failing to report a third asbestos type (ID)	25

Reporting trace asbestos in a blank sample or trace incorrect asbestos type(s) in a positive sample	No penalty
Failing to report trace asbestos when RTI QC confirms it in all samples (SQ)	25/type
Failing to report trace asbestos when RTI QC does not confirm it in all samples	No penalty
Per sample, first asbestos semiquantitation outside acceptance range (SQ)	20
Per sample, second asbestos semiquantitation outside acceptance range (SQ)	10
Per sample, third asbestos semiquantitation outside acceptance range (SQ)	5

Section VI - Special Analysis Instructions and Explanations

The following observations are provided concerning trends or patterns seen in classification and identification errors for each of the four test sample lots.

Overall, the positive (asbestos-containing) samples in Test Round 117 generated expected numbers of asbestos classification and identification errors, based on a comparison with analytical performance on past similar samples. There were no classification errors, resulting in a classification error rate of 0.0%, which is lower than the historic classification error rate of 3.7% for the 37 test rounds conducted under the current four-sample format. There were no false negatives, resulting in a false negative error rate of 0.0%, which is lower than the historic 3.3% false negative error rate over the 37 test rounds conducted under the current four-sample format. No false positive errors were incurred, resulting in a false positive error rate of 0.0%. This rate is much lower than the historic 5.1% false positive error rate over the 37 test rounds conducted under the current four-sample format.

There were 12 identification errors, resulting in an identification error rate of 13.3%. This rate is higher than the historic 9.7% identification error rate over the 37 test rounds conducted under the current four-sample format. One of the identification errors was incurred on the Lot B spray-on insulation that contains 5% chrysotile and a trace amount of actinolite. This error was incurred by a laboratory that reported 1% tremolite for the sample and did not report any chrysotile. This sample contains a trace amount of actinolite, which has very similar optical properties to tremolite. The sample contains

enough chrysotile to be easily detected; however, the chrysotile fibers and bundles may be obscured by the white, fine-grained matrix material. Eleven of the identification errors were incurred on the Lot D fire door insulation, which contains 10% amosite and 1% chrysotile in a non-friable calcareous matrix. Eleven laboratories did not report any chrysotile for this sample. The probable reason for not reporting chrysotile is that the Lot D sample contains two asbestos types, amosite and chrysotile, and the amosite is much more plentiful and more evident than the chrysotile. Also, the chrysotile may be obscured by the Lot D sample's fine-grained white matrix material.

A conscientiously applied internal quality control program, in conjunction with the use of time-proven difficult samples (low-asbestos-percentage samples, asbestos look-alikes, and so on) and the use of sample reduction by low-temperature and/or acid dissolution, where appropriate, should greatly minimize the repetition of avoidable analytical errors such as those incurred this test round on all sample lots.

Test Round 117 samples were chosen such that the test round posed an above average analytical challenge. Laboratories should prepare for more challenging test rounds. Semiquantitative calibration standards containing any of the six asbestos types in a variety of real-world matrices and mine-grade samples of chrysotile, amosite, and crocidolite are available from RTI at http://www.rti.org/files/Asbestos_Order_Form.pdf.

Section VII - Electronic Submittal of Analysis Results

Beginning with Test Round 69, RTI made available a Web site for use by program participants to upload analysis results to RTI, download final reports from RTI at the conclusion of the test round, acquire general information about the program, and communicate contact information changes to RTI. In Test Round 117, 29 laboratories (96.7%) of the participating laboratories took advantage of the convenience of the Web site to submit analysis results.

Section VIII - Test Method/Analytical Technique Summary

Since Test Round 99, RTI has solicited information about the test methods and analytical techniques employed by the program participants on the samples for that test

round. Test method/technique combinations known by RTI to be in use by bulk asbestos laboratories are listed in Table 5. The method/technique combinations are numbered as they were in the instructions for data entry for this test round. RTI correlated false negative, false positive, asbestos identification, and asbestos semiquantitation errors to the method/technique used.

Table 5. Cited Test Methods and Techniques

Legend	Method	Qualitative Technique	Quantitative Technique
1 EPA INT / PTCT	EPA Interim Method (1982)	Polarized Light Microscopy	Point Counting
2 EPA INT / EVE			Equivalent Visual Estimation
3 EPA INT / GRAV			Gravimetric Reduction
4 EPA INT / XRD		X-Ray Diffraction	Standards Comparison
5 EPA REV / PTCT	EPA Revised Method (1993)	Polarized Light Microscopy	Point Counting
6 EPA REV / CVE			Calibrated Visual Estimation
7 EPA REV / GRAV			Gravimetric Reduction
8 EPA REV / XRD		X-Ray Diffraction	Standards Comparison
9 EPA REV / AEM		Analytical Electron Microscopy	Visual Estimation
10 NYS / PTCT	New York State Method 198.1	Polarized Light Microscopy	Point Counting
11 NYS / TEM	New York State Method 198.4	Transmission Electron Microscopy	Visual Estimation
12 NIOSH / VE	NIOSH Method 9002	Polarized Light Microscopy	Visual Estimation
13 NIOSH / XRD	NIOSH Method 9000	X-Ray Diffraction	Standards Comparison
14 OSHA / VE	OSHA Method D-191	Polarized Light Microscopy	Visual Estimation
15 OTHER	Method Not Specified Above		

Because of the relatively small number of errors of any type, presentation of these comparative data on a per-sample basis is of limited value and practicality to participating laboratories. Therefore, RTI presents the error type/frequency data for each test method used in cumulative terms for all samples in the test round. These data are provided in Table 6.

The absence of a particular method/technique combination from Table 6 means that no laboratory opted to use that combination. The last column presents the average error points per analysis. The number in each row of this column was calculated by summing the number of false negatives and false positives times 100, the number of identification errors times 50, and the number of semiquantitation errors times 20, and dividing that sum by the total number of analyses using that method. This weighting reflects the relative penalty point values for these error types in the actual grading criteria for the program.

Table 6. Analysis of Errors and Asbestos Semiquantitation, by Test Method/Technique Used

Test Round 117										
# Method/Technique Cited by Laboratory	Total No. of Analyses	FN	FN Error Rate (%)	FP	FP Error Rate (%)	ID	ID Error Rate (%)	SQ	SQ Error Rate (%)	Avg. Error Points per Analysis
2 EPA INT / EVE	12	0	0.0	0	0.0	2	16.7	3	25.0	13.3
6 EPA REV / CVE	40	0	0.0	0	0.0	4	10.0	4	10.0	7.0
7 EPA REV / GRAV	4	0	0.0	0	0.0	0	0.0	0	0.0	0.0
12 NIOSH / VE	56	0	0.0	0	0.0	6	10.7	5	8.9	7.1
15 OTHER	8	0	0.0	0	0.0	0	0.0	0	0.0	0.0

Two methods that use polarized light microscopy (PLM) for qualitative analysis and

visual estimation or calibrated visual estimation for quantitative analysis – the 1993 EPA Revised Method and NIOSH Method 9002 – were the ones most often cited by the laboratories this round. Of those two test methods, the lowest average penalty points per analysis were incurred by laboratories using the 1993 EPA Revised Method. However, the difference in average penalty points between the two methods was only 0.1 points per laboratory – indicating essentially a negligible difference between the two methods for this round. An additional observation worth noting is the relative use of test methods. The purpose of this section is to provide some minimum statistical correlation of the submitted data and analysis errors to the methods and techniques from which they are derived. The value of these assessments will no doubt become more meaningful as trends or patterns are revealed over the course of many test rounds.

Section IX - Schedule for Test Round 118

The following schedule for Test Round 118 of the Navy Asbestos Identification Proficiency Testing Program has been agreed upon by the NAVMCPUBHLHCEN and RTI:

1 August 2012	RTI distribution of test samples to participating laboratories
3 September 2012	Deadline for RTI receipt of laboratory results of analyses
1 October 2012	RTI distribution of “Final Report to Laboratories – Test Round 118” to participants

END OF REPORT