MCC

Materials Characterization Center

**Summary Report for the
Interlaboratory Round Robin
on the MCC-1 Static Leach
Test Method**

March 1982

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
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by Battelle Memorial Institute

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SUMMARY REPORT FOR THE
INTERLABORATORY ROUND ROBIN
ON THE MCC-1 STATIC LEACH
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J. W. Johnston
J. L. Daniel

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under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Richland, Washington 99352

EXECUTIVE SUMMARY

PERSPECTIVE FOR READING THIS REPORT

The MCC-1 Static Leach Test Method Round Robin (RR) was undertaken with several objectives in mind. It was envisioned as an intensive means of introducing the procedure to a large segment of the nuclear waste community, at a time when there was relatively little common ground among the participants. The RR also was expected to elicit feedback from participants regarding problem areas in the procedure, or inadequately controlled steps in the test. It provided information on the importance of an "experience factor" for new users of MCC-1, since only the Materials Characterization Center (MCC) staff who developed MCC-1 had any extensive prior experience with its use. In fact, several of the RR participants had never attempted the procedure until this time. Finally, and very importantly, the results of the round robin were to be used to estimate the precision to be expected between laboratories analyzing "identical" specimens as guidance in establishing procedures for quantitative comparison of the various nuclear waste materials and systems to be evaluated.

All of these objectives have been achieved with the MCC-1 RR. This report summarizes the statistics of the RR results, emphasizing the evaluation of the entire data set rather than concentrating on identification of "best" data, or including results from only the "experienced" or "qualified" laboratories. The MCC plans to use approved reference materials to verify that different laboratories can produce comparable data from unknown new candidate waste forms. Fundamental to the use of reference materials as a laboratory "certification" step is a clear statistical statement of what inter- and intra-laboratory precision is achievable under "ideal" test conditions, and what is a reasonable expectation for interlaboratory agreement on the standard materials test. In the absence of an interlaboratory study using the reference material, the RR results are the only source of information on interlaboratory precision.

One important factor which may have a significant impact on interlaboratory agreement in the MCC-1 RR is the uncertain homogeneity of the specimens tested. Considerable thought and care were invested in selection of the three

materials used for the tests. They were chosen because they were the best materials available at that time, representative of top candidate waste disposal forms, and available in sufficient quantity for multiple testing by all participating laboratories. "Certification" level homogeneity evaluation of the test materials was not attempted, but a careful limited analytical comparison indicated that composition differences between test pieces did not exceed 10 percent relative, and generally appeared to be substantially less than that.

All of these aspects of this RR test need to be kept clearly in mind when studying this report. The test results reflect a complex interplay of a large number of variables: 25 participating laboratories, 29 combinations of element-waste form-leachants, and a new multi-step test procedure requiring exercise of mechanical, chemical and analytical controls. In addition, all of the data were obtained from short-term leach tests, (≤ 28 days), a period during which the leach rate is highly time dependent. Considering the conditions, perhaps it is not surprising that a large variability is exhibited for many of the interlaboratory results.

Finally, it is important to remember that the current need is for the ability to discriminate between nuclear waste forms of broadly acceptable and unacceptable leaching stability, rather than for detailed characterization of small differences in materials of similar behavior. We are concerned as much with defining the precision with which we know these characteristics as we are in measuring the values themselves. From this report, a reasonably clear picture emerges regarding the level of agreement which should be expected from unrestricted application of MCC test procedures on a national or international scale. Clearly, this kind of information is essential as a basis for evaluating new waste form data submitted by individual laboratories. It should be used also for establishing meaningful MCC policies and procedures for obtaining handbook-quality information on all waste forms.

SUMMARY OF THE REPORT

This report has the results from the interlaboratory test (round robin) on the MCC-1 Static Leach Test Method conducted by the MCC over a period of

15 months, 1980-82. A total of 25 laboratories provided data in connection with the RR. All together these laboratories tested 769 waste form specimens using the 1980 draft of MCC-1. The specimens tested were approximately one gram wafers cut by the laboratory from samples provided by MCC: NBS borosilicate glass (a NBS Standard Reference Material normally used for refractive index measurements); core drillings of basalt from the Hanford Reservation; and MCC Type 76-68 simulated waste glass. These specimens were tested for 3, 7, 14, 28 days in ovens at 90°C using three different leachants. The resulting leachates were analyzed for elemental concentrations, and elemental mass losses were calculated (grams of element released into the leachant per square meter of geometric specimen surface area). The specimens were weighed before and after leaching to determine specimen mass loss.

Other important features of the RR were as follows:

- An analytical round robin (ARR) was conducted using simulated leachates, provided by the MCC, to estimate the range of analytical biases of the participating laboratories.
- Each participating laboratory tested two additional specimens of each waste form in each of the three leachants for 28 days. The resulting leachates were returned to the MCC for chemical analysis to provide another check for potential analytical biases.
- The written numerical and narrative report by which each laboratory's results were reported provided the only checks on how well the laboratories followed the specified MCC-1 procedure.

This summary report on the round robin has two major parts: Part I provides plots of the data (which are listed in an Appendix) and summary statistics so that the participating laboratories can compare performance with other laboratories. From these plots and summaries, each laboratory also can identify its problem areas. Part II discusses the statistical analysis of the data, and characterizes the precision of the MCC-1 Static Leach Test Method at the time the RR was conducted. The precision characterization indicates that the MCC and waste form community need to institute tighter control of MCC-1 and similar testing practices to reduce the impact of between-laboratory differences on statistical comparisons.

Based upon the normalized mass loss data (for the elements in the leachates from the waste forms) that had the best precision statistics, the precision of the MCC-1 Static Leach Test Method can be characterized as follows:

Precision Statement

When carefully applied by an experienced laboratory, the elemental loss (g/m^2) measured by the MCC-1 28-day static leach test can be expected to have a relative within-laboratory precision of 6 to 11 percent and a relative between-laboratory precision of 25 to 40 percent, provided the elemental mass loss is at least 5 g/m^2 .

The immediate practical implications of this statement are:

1. Measured loss of elemental mass per unit area must be at least 5 g/m^2 for meaningful comparisons of results within or between laboratories.
2. Within laboratory comparisons would be able to detect real differences between waste forms which are at least one-third of the consensus element mass loss averages obtained in the MCC-1 RR.
3. Between laboratory differences at least as large as the consensus average for MCC-1 RR would be detectable using statistical procedures for element-waste form-leachant combinations with a demonstrated relative precision of less than 35 percent.
4. Since in most combinations the between laboratory precision is greater than 35 percent, substantial improvement in demonstrated MCC-1 precision must be achieved to give statistical comparisons between laboratories the power to detect differences less than the consensus averages obtained in the MCC-1 RR.

Statisticians frequently recommend replication (testing a number of specimens at each test condition) to improve precision statistics. But replication can improve only within-laboratory precision. The MCC ARR indicated that within-laboratory imprecision accounts for less than 10 percent of the total imprecision, so that additional replication of specimens or chemical analyses

would not appreciably reduce the total imprecision. The major problem is with the biases between laboratories, which can be reduced only by eliminating overall procedural bias, or by having many laboratories test the candidate waste forms.

The MCC-1 RR results provide valuable information which can be applied to solving the problem of what to do about the observed between-laboratory differences. The interpretation by MCC of the significance of those differences impacts on many of the decisions MCC must make, e.g., comparing of two waste forms, determining whether a laboratory's application of a test procedure is acceptable for inclusion in the Handbook, or specifying a leach rate characteristic of a waste form or waste form package. A scientifically valid method for making these decisions based on statistical decision rules or estimation methods remains to be specified.

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FOREWORD

The U.S. Department of Energy established the Materials Characterization Center (MCC), located at the Pacific Northwest Laboratory, to assemble a standardized nuclear waste materials data base for use in research, systems and facility design, safety analyses, and waste management decisions. This centralized data base will be provided through the means of a Nuclear Waste Materials Handbook. The first issue of the Handbook was published in the fall of 1981 in looseleaf format so that it can be updated as additional information becomes available. To ensure utmost reliability, materials data appearing in the Handbook will be obtained by standard procedures defined in the Handbook and approved by an independent Materials Review Board (MRB) comprised of materials experts from Department of Energy laboratories and from universities and industry.

The first priority of the MCC has been to define a series of standardized leach test methods for solidified nuclear wastes. Although a large amount of leaching of solidified waste forms has been done over the past two decades, comparison of the leach-rate data has been difficult because many different, usually ill-defined, leach test procedures were being used. The goals of the MCC are to bring uniformity into the leach testing being done by waste form developers in the United States, and to develop data that can be used to predict the long-term leaching behavior of waste forms. To do this, five leach test methods involving only waste form specimens and leachant solutions have been defined (the differences in the tests involve temperature, specimen configuration and leachant flow rate); the next step will be to do five more complex systems tests that also include other elements, such as backfill materials and rocks, in order to more closely simulate repository conditions.

The reliability of the data generated by the MCC leach tests depends upon having a thorough knowledge of the precision and accuracy capabilities of the test methods. Interlaboratory (round robin) testing is a well-accepted technique for assessing the precision and accuracy capabilities of a test method. It is one of the techniques being used by the MCC.

A planning meeting for the first MCC round robin was held in Germantown, Maryland, on August 21, 1980. Attendees at the meeting are listed below. Plans for round robin testing of the MCC-1 Static Leach Test Method were discussed at the meeting and expressions of interest in participating in the round robin were solicited. There was excellent response and this report describes results of the round robin test that ensued. As a result of discussions at the MCC-sponsored "Workshop on Compositional and Microstructural Analysis of Nuclear Waste Materials" held in Seattle, November 11 and 12, 1980, it was also decided to hold an analytical round robin using synthetic leachates in conjunction with the MCC-1 Static Leach Test Method Round Robin.⁽¹⁾ Results of the analytical round robin are included in this report.

Attendees at the round robin planning meeting in Germantown, Maryland, were:^(a)

D. E. Clark	University of Florida
D. G. Coles	Lawrence Livermore Laboratory
D. Cronin	National Bureau of Standards
J. L. Daniel	Pacific Northwest Laboratory
K. F. Flynn	Argonne National Laboratory
H. Frederikse	National Bureau of Standards
H. W. Godbee	Oak Ridge National Laboratory
W. Greenhalgh	Westinghouse-Hanford
W. Haller	National Bureau of Standards
A. Johnson	Rockwell-Rocky Flats
K. S. Kim	Nuclear Regulatory Commission
H. M. Kingston	National Bureau of Standards
E. L. Lewis	Mound Facility
J. Mondel	National Bureau of Standards
J. E. Mendel	Pacific Northwest Laboratory
J. Merrill	Pacific Northwest Laboratory
R. D. Nelson	Materials Characterization Center
G. L. Ogle	Radian Corporation
R. A. Palmer	Rockwell-Hanford
R. C. Paule	National Bureau of Standards

(a) A meeting was also held at the National Bureau of Standards (NBS) on August 22, 1980, in which further discussion concerning the round robin took place between MCC and NBS staff.

L. Penberthy	Penberthy Electromelt
P. H. Permar	Savannah River Laboratory
T. C. Rains	National Bureau of Standards
D. G. Schweitzer	Brookhaven National Laboratory
C. W. Sill	EG&G-Idaho
J. H. Simmons	Catholic University
B. A. Staples	Exxon Nuclear Idaho Co.
S. A. Sterling	General Atomic
J. A. Stone	Savannah River Laboratory
D. M. Strachan	Pacific Northwest Laboratory
R. D. Walton, Jr.	Department of Energy
W. B. White	Pennsylvania State University
E. A. Wick	Nuclear Regulatory Commission
H. T. Yolken	National Bureau of Standards
R. Zeisler	National Bureau of Standards

1.0 INTRODUCTION

An interlaboratory test on the MCC-1 Static Leach Test Method was conducted by the Materials Characterization Center (MCC) over a period of 15 months, 1980-82. A total of 25 laboratories provided data in connection with this round robin (RR) testing. All together these laboratories tested 769 waste form specimens using the 1980 draft of MCC-1. The specimens tested were approximately one gram wafer's cut by the laboratory from samples provided by MCC: NBS borosilicate glass (a NBS Standard Reference Material normally used for refractive index measurements); core drillings of basalt from the Hanford Reservation; and MCC Type 76-68 simulated waste glass. These specimens were tested for 3, 7, 14, 28 days in ovens at 90°C using three different leachants. The resulting leachates were analyzed for elemental concentrations, and elemental mass losses were calculated (grams of element released into the leachant per square meter of geometric specimen surface area). The specimens were weighed before and after leaching to determine specimen mass loss. An analytical round robin (ARR) was also conducted using simulated leachates, provided by the MCC, to estimate the range of analytical biases of the participating laboratories.

Standard forms and instructions were sent to all MCC-1 RR participants (Appendix A). Each participant was to prepare test specimens from the sample materials supplied by the MCC according to the August 1980 draft of the MCC-1 Static Leach Test Method⁽²⁾ with certain modifications:

- Two additional specimens were to be run for 28 days in each leachant and the resulting leachates shipped to the MCC for analysis.
- Instead of a single blank, two blanks of each leachant were required.
- Instead of ethylene glycol, water was required as the coolant in preparing the RR specimens.

Nominal composition of these specimen types is given in Table 1.1. The times, leachants, and temperature specified for the MCC-1 RR are shown in Table 1.2.

TABLE 1.1. Nominal Composition of MCC-1 Round Robin Specimens(a)

Element Oxide	Composition, %								
	NBS Glass	S.D.	f _i	Basalt	S.D.	f _i	MCC 76-68	S.D.	f _i
Al ₂ O ₃	3.58	0.13		13.09	0.15		0.60	0.02	
B ₂ O ₃	16.85	0.06	0.053				8.81	0.23	0.0294
BaO	0.27	0.01		0.03			0.53	0.01	
CaO	0.14	0.11		9.47	1.04	0.074	2.36	0.07	
CdO							0.05		
Ce ₂ O ₃							0.90	0.02	
Cr ₂ O ₃				0.03			0.15	0.13	
Cs ₂ O							1.02		0.0103
Fe ₂ O ₃	0.10			10.76	0.77	0.082	9.40	0.26	
K ₂ O	7.15		0.066	0.45			0.13	0.07	
La ₂ O ₃							4.06	0.09	
Li ₂ O	0.46	0.05							
MgO				5.75	0.90		0.15	0.02	
MnO ₂				0.21			0.06		
MoO ₃							1.93	0.06	
Na ₂ O	2.68		0.0074	4.68		0.018	11.53	0.31	
Nd ₂ O ₃							1.38	0.04	
NiO							0.25	0.09	
P ₂ O ₃							0.74	0.09	
PbO	0.09	0.02							
Pr ₂ O ₃							0.48		
SiO ₂	66.52		0.327	46.85	1.48	0.238	40.84	1.22	0.186
SrO				0.24			0.42	0.01	0.0034
TiO ₂				1.64			3.03	0.08	
ZnO							4.72	0.12	
ZrO ₂				0.03	1.68		1.68	0.56	
TOTAL	97.84			93.23			95.22		

(a) Analysis of triplicate specimens by MCC laboratory using ICP method on dissolved samples. Standard deviation (S.D.) reflects both analytical precision and sample homogeneity.

f_i - Initial elemental mass fraction used for all normalized mass loss calculations (see p. 2.4).

TABLE 1.2. Test Matrix for Each Material (all tests at 90°C)

<u>Leachant</u>	<u>Number of Replicate Containers at Each Time Period</u>				
	<u>3 days</u>	<u>7 days</u>	<u>14 days</u>	<u>28 days</u> <u>Specimens</u>	<u>Blanks</u>
Silicate Water				3 + 2(a)	2
Pure Water	1	1	1	3 + 2(a)	2
Brine				3 + 2(a)	2

(a) Leachates from these tests were returned to MCC for analysis; all other leachates were analyzed by participants.

The instructions to the laboratories participating in the ARR are given in Appendix B. Only the simulated leachants (SR-1, 2, and 3) were used. Laboratories could use any method for sampling aliquots from the 100 ml bottles and their routine method of analyzing for the elements of interest.

The purpose of Part I of this report is to inform the participants of the MCC-1 RR where their results fell in relation to the other participants. Summary tables and figures are reported in Section 2, plots and tables of the individual mass loss data for each waste form specimen in Section 3, plots of the time sequence data in Section 4, and summaries of the narrative data in Section 5. All data,^(a) except for time-related data are for the 28-day leach tests. Each participating laboratory is identified only by a code assigned by MCC. A list of participating laboratories is given in Appendix C. The MCC laboratory identification code is S; each participant has been notified of his own laboratory's code also.

If any reported data or summary points disagree with original laboratory records, the contributor of the data is asked to advise the MCC of the error.

(a) The data are reported to a convenient number of digits for computer application without losing statistically meaningful digits. Summary statistics are reported to fewer digits. This practice follows the philosophy detailed by D. B. Delury in "Computations with approximate numbers," pp. 392-401 in NBS Sp. Pub. 300, Precision Measurement and Calibration, Statistical Concepts and Procedures, H.H. Ku, (ed), U.S. Government Printing Office, Washington, D.C., 1965.

Any explanations available for the large deviations from the consensus results exhibited in some instances also are requested. It is assumed that the readers of this report are familiar with the MCC-1 Static Leach Test Method.⁽²⁾

A summary of the statistical analyses and interpretations of the results are discussed in Part II of this report. Appendix D contains tabulations of the elemental mass loss, specimen mass loss and final pH data. Appendix E presents examples of application of the statistical summaries.

PART I. THE DATA

2.0 SUMMARIES OF THE DATA

The MCC-1 RR laboratory testing began in late 1980, and the last data were received in early 1982. The MCC provided each participant with bulk specimens of three materials to be tested by the MCC-1 Static Leach Test Method: natural basalt, clear glass and a simulated waste glass. In addition, participants received specimens of three different simulated leachate solutions prepared by MCC for independent testing of the laboratory's analytical procedures. The leaching test procedures were specified in detail, and the required test information was to be reported on the report sheets provided by MCC. Table 2.1 summarizes the specimen types, and gives a key to the abbreviations used for specimens, leachants and elements in the tables and plots in this report.

This report is based primarily on data received from 21 laboratories: 2 at universities, 6 in the nuclear industry, 10 at DOE national laboratories, and 3 laboratories outside the U.S. (Canada, Australia and Italy). Table 2.2 shows which laboratories (identified only by code) tested each material. Results from 4 additional laboratories (1 university and 3 foreign laboratories), received after the earlier data had been analyzed, are included in a few of the data tabulations.

Only six laboratories tested all three waste forms on the MCC-1 RR, and only four laboratories reported complete data for the analytical round robin (ARR). The RR required that each material be run in each of three leachants: 1) silica-saturated water solution (SiW), 2) deionized water (DIW), and 3) brine. Laboratory G only ran the MCC glass in DIW; all other laboratories ran all three leachants with each waste form it tested. Most participants also returned leachates (RL) from at least one of the materials tested in their laboratory. Currently, MCC computer files contain information on a total of 769 waste form specimens tested for the MCC-1 RR. The summaries are based on 667 specimens from the earlier reports, that is, excluding the results for the 102 specimens tested by the four "late laboratories."

TABLE 2.1. Key to Abbreviations Used in Tabulations and Plots

Specimens Tested

ARR - Analytical Round Robin Solutions
SL-1 simulated leachate from deionized water
SL-2 simulated leachate from deionized water
SL-3 simulated leachate from brine

BASALT, BAS - Natural basalt from Columbia Plateau

NBS - Clear glass provided by National Bureau of Standards

MCC - Simulated MCC waste glass type 76-68 manufactured by Penberthy (Lot 3)

RL - Leachates from tests of NBS, BAS or MCC specimens, returned by participant to PNL and analyzed by MCC laboratory

Leachant Solutions

Brine, BR - Saturated brine solution

DIW - Deionized water

SiW - SiO₂-saturated water

Elements Analyzed

Boron - B
Calcium - C or CA
Cesium - X or CS
Iron - F or FE
Potassium - K
Sodium - N or NA
Strontium - R or SR
Silicon - S or SI

TABLE 2.2. Laboratory Identification Codes and Results Received(a)

Laboratory Code Letter	ARR	MCC-1 On Waste Forms			
		NBS	BASALT	MCC	RL
Laboratories included in all data analyses					
A	a		x	x	x
B	a	x		x	x
C	x	x			x
D	x	x		x	x
F	x	x	x	x	x
G	a			s	x
H		x		x	x
I		x	x	x	x
J		x		x	x
K	a	x	x	x	x
L		x	x		x
M	a	x	x		x
O		x			x
P		x	x	x	
Q		x			
R		x			x
S (MCC)	x	x	x	x	x
T	a	x	x	x	x
U				x	x
W	a				
<u>X</u>	<u>—</u>	<u>x</u>	<u>—</u>	<u>—</u>	<u>—</u>
No. of Labs	21	11	17	9	17
Late laboratories included only in tabulations of data					
N	a	x		x	
V				x	x
Y				x	
<u>Z</u>	<u>—</u>	<u>x</u>	<u>—</u>	<u>x</u>	<u>—</u>
No. of Labs	<u>4</u>	<u>1</u>	<u>2</u>	<u>1</u>	<u>1</u>
TOTAL LABS	25	12	19	10	18

(a) x - complete data set reported
s - partial test data reported
a - average values only reported.

Table 2.3 gives the number of laboratories reporting data for each element-waste form-leachant combination of the MCC-1 RR. Five of the cells in Table 2.3 have a "(b)" for elements that were "blanked," that is, the blank concentration was greater than the concentration in the leachate from the specimen leach test. Two cells have "d" meaning that less than detection limit results were reported by most laboratories. These seven cells were removed from further analysis. The precision analysis for MCC-1 was based on the 29 element-waste form-leachant combinations ("cells"), each having triplicate results for 9 to 17 laboratories. Only silicon was determined in all nine waste form-leachant combinations ("materials"), and iron was measurable only for basalt in brine. There were 367 laboratory-element-material combinations in all.

The normalized elemental mass loss and specimen mass loss were calculated for the 28-day data by computer, using the following equations:

$$C_i = E_i - B_i \quad (2.1)$$

where, for each specimen leachate,

C_i is the blank corrected mg/l for element i ,

E_i is the mg/l for the specimen, reported by the laboratory,

$B_i = 1/2 (B_{i1} + B_{i2})$, the average of the two blanks run with the specimen.

Then for the normalized elemental mass loss,

$$NL_i = (0.1)[C_i/(f_i SA/V)] \quad (2.2)$$

where

NL_i is the normalized element mass loss in g/m^2 for element i ,

C_i is defined by Eq (2.1),

f_i is the mass fraction determined by MCC for element i in the waste form initially,

SA/V is the ratio of [the waste form surface area (in mm^2) to the leachant volume (in mm^3)] $\times 100$,

(0.1) is the factor required to convert the units used to g/m^2 .

TABLE 2.3. Number of Laboratories Whose Data Was Used for Each Element-Waste Form-Leachant Combination

Element	NBS			Basalt			MCC			Total Combinations ^(a)
	SiW	DIW	Brine	SiW	DIW	Brine	SiW	DIW	Brine	
Boron	17	17	17				11	12	11	85
Calcium				9	9	9				27
Cesium							11	12	11	34
Iron				d	d	9			9	
Potassium	17	17	(b)							34
Sodium	(b)	17	(b)	(b)	9	(b)				26
Strontium							12	13	12	37
Silicon	17	17	17	9	9	9	12	13	12	115
<u>No. of Combinations</u>										
Element-Leachant	3	4	2	2	3	3	4	4	4	
Element-Waste Form-Leachant	9			8			12			
TOTAL				29						367

(a) Does not include data from 4 late laboratories. Inclusion of those data would increase total number of data combinations submitted to 437.

(b) Blank concentration in leachant greater than concentration in specimen leachate.

d = Most laboratories reported "less than detection limit."

For the normalized specimen mass loss,

$$ML = (10^6) \text{ SPWTD/SA} \quad (2.3)$$

where

ML is the normalized mass loss in g/m^2 ,

SPWTD is the difference between the initial and final specimen weight in g,

SA is the geometric surface area in mm^2 ,

(10^6) converts the ratio to g/m^2 .

2.1 SUMMARY FOR MCC-1 ROUND ROBIN

Table 2.4 gives the summary statistics for each element-waste form-leachant combination. This table is based on the elemental mass loss (g/m^2) for the three replicate specimens analyzed by each laboratory. It gives the consensus element averages and the pooled relative within-laboratory and between-laboratory standard deviations. These statistics were obtained from individual one-way analyses of variance (AOV) on each of the 29 waste form-leachant-element combinations.

A consensus average is the overall arithmetic average of all the g/m^2 results from all laboratories for an element in a leachant type for a specific waste form, and includes from 27 (3 specimens times 9 laboratories) to 51 (3×17) results for each cell. None of the reported data was excluded except those for the "blank" or "detection limit" cells. S_r , the within laboratory standard deviation, is "an average of the repeatability found in the separate laboratories,"⁽³⁾ and is an indication of the amount of spread among replicates to be expected within a laboratory. S_L , the between laboratory standard deviation, is an estimate of the amount of spread among laboratory averages for the same element-waste form-leachant combination. In Table 2.4 these standard deviations are reported relative to the consensus averages so that they can be easily compared from cell to cell.

TABLE 2.4. Summary for all Element Data, MCC-1 RR

Element	NBS			Basalt			MCC		
	SiW	DIW	Brine	SiW	DIW	Brine	SiW	DIW	Brine
Consensus Averages ^(a) for Elemental Mass Loss (g/m ²)									
Boron	61.1	17.8	19.2				32.4	36.0	23.2
Calcium				0.7	2.7	30.8			
Cesium							23.8	27.8	22.6
Iron				d(c)	d(c)	1.0			
Potassium	45.0	11.9	(b)						
Sodium	(b)	13.3	(b)	(b)	9.9	(b)			
Strontium							0.8	1.8	16.5
Silicon	25.6	4.9	8.1	2.7	5.6	2.2	18.1	23.9	11.1
Pooled Within-Laboratory Relative Standard Deviation S _r (%)									
Boron	15.5	16.2	29.0				7.0	8.4	21.4
Calcium				13.7	38.6	16.3			
Cesium							5.0	5.3	6.5
Iron				d	d	38.0			
Potassium	11.3	22.3	(b)						
Sodium	(b)	33.5	(b)	(b)	9.9	(b)			
Strontium							19.2	16.2	19.2
Silicon	15.5	17.6	29.1	12.0	7.0	27.1	5.6	7.2	6.7
Between-Laboratory Relative Standard Deviation S _L (%)									
Boron	26.3	63.9	37.9				32.7	31.0	32.7
Calcium				67.2	64.3	51.7			
Cesium							64.6	42.9	87.9
Iron				d	d	103.8			
Potassium	30.2	65.3	(b)						
Sodium	(b)	90.4	(b)	(b)	76.8	(b)			
Strontium							69.3	59.7	48.0
Silicon	27.7	120.3	32.2	50.8	13.4	35.4	29.1	33.6	29.6

(a) Overall arithmetic average of all the g/m² results from all laboratories for an element in a leachant type for a specific waste form (each cell in Table 2.3).

(b) Blank concentration greater than concentration in leachate.

(c) "d" indicates that most laboratories reported "less than detection limit."

The within-laboratory relative precisions for the 20 laboratories reporting data for the MCC-1 RR range from 5 percent to almost 40 percent, and the between-laboratory from 13 percent to 120 percent.

2.2 SUMMARY FOR ARR

A large volume of each of the three simulated leachate solutions (SL-1, SL-2, and SL-3) was prepared and thoroughly mixed, so that each participant received an identical 100-ml aliquot of each solution. Reasonable care was taken to achieve the intended concentrations for each "leachate," but no attempt was made to provide a certified composition. Solutions were shipped in Teflon® screw-cap bottles.

Table 2.5 gives the summary statistics for the analyses of each element in each simulated leachate solution for the four full design laboratories (those laboratories which reported results for 2 to 4 replicate aliquots each day for 2 to 4 days as specified for the ARR). A "nested" AOV procedure, similar to that for the MCC-1 RR AOVs, was used. The notations sd(W) (within laboratory) and sd(L) (between laboratory) are used to differentiate the ARR indices of precision from the MCC-1 RR indices, and have the same definitions as S_r and S_L , respectively. Lab F had some grossly disparate values, and so was excluded from the calculations for some element-simulated leachate combinations.

For the ARR, the within-laboratory standard deviations for these 4 laboratories range from 1.5 to 14 percent, and the between-laboratory standard deviations from 7 to 53 percent, approximately half that of the MCC-1 RR's 20 laboratories.

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TABLE 2.5 Summary for all Element Data, ARR [For the Four Full Design Labs: C, D, F, S(MCC)]

<u>Consensus Averages (mg/l)</u>			
	<u>SL-1</u>	<u>SL-2</u>	<u>SL-3</u>
Boron	50.96	28.12	52.59
Cesium	5.62	5.54	8.75
Potassium	d(0.16)(b)	d(0.05)	19920.0
Sodium	87.07	41.79	22259.0
Strontium	4.55	2.71	4.78(a)
Silicon	19.60	d1(0.14)	21.08
<u>Pooled Within-Laboratory Relative Standard Deviation sd(W) (%)</u>			
Boron	1.48(a)	2.96(a)	3.41(a)
Cesium	7.15	5.25	5.70
Potassium	d	d	3.43
Sodium	4.86	4.07(a)	5.42
Strontium	2.95(a)	6.94	6.44(a)
Silicon	1.93(a)	d	14.35
<u>Between-Laboratory Relative Standard Deviation sd(L) (%)</u>			
Boron	7.82	25.68	14.57
Cesium	19.95	14.86	53.50
Potassium	d	d	7.88
Sodium	11.65	7.36	11.15
Strontium	21.96	16.46	16.34(a)
Silicon	11.48	d	25.43

(a) Lab F data excluded; see text.

(b) "d" indicates that most laboratories reported "less than detection limit."

3.0 PLOTS OF BASIC DATA

The plots in this section provide a visual comparison of the data provided by each participating laboratory. They are not intended to be used for quantification of differences, but rather to identify differences for subsequent correction or assignment of causes.

3.1 MCC-1 RR TRIPLICATE RESULTS

Figures 3.1 through 3.9 are computer plots of the data for the three replicate specimens of each waste form run by each laboratory in each leachant. These are the basic data used to characterize the precision of the MCC-1 procedure. Each figure is for one "material" (waste form-leachant combination). Each panel within a figure is for the elemental mass loss data, specimen mass loss or final pH. A vertical line connects the three results for each laboratory, and the laboratories identification letter appears above its results. Scaling factors are provided along with the element symbol at the bottom of each panel. For example, in Figure 3.1, for SiW in NBS glass, the first panel, for B, boron, has a scale factor of 60, meaning that the ordinate must be multiplied by 60. Thus, the points for Labs C and H would be slightly less than 90 g/m^2 . This scaling factor is indicated in the same way for all elements and mass loss (ML), but "pH 5-10" means that the pH scale runs from 5 to 10, each tic on the ordinate scale being 1 pH unit.

These data plots can be used to obtain a qualitative indication of the within- and between-laboratory variability for each panel, without reference to any scaling factor. For example, it is obvious in Figure 3.1 that, for boron, Labs D, H, I and O have practically no within-laboratory variability, Lab M has a broad spread, and the remaining laboratories a moderate spread. It is also obvious that Labs C and H averages would be about three times that of Lab K, and twice that of Lab O. Except for Lab M, the spread of the points over all laboratories is considerably greater than the spread within any one laboratory. It is also interesting to note that the elemental loss data usually have the same pattern as the specimen mass loss data (ML), both within and between laboratories.

Part II of this report quantifies the information in these plots using statistical analysis of the data to estimate the within- and between-laboratory components of variance.

The triplicate data for the late laboratories are listed in Tables 3.1 through 3.3. The interested reader can plot these values on Figures 3.1 through 3.9 using the scale factors in the figures.

FIGURE 3.1. Data Reported by MCC-RR Participants for Tests of NBS Glass in SiW (Laboratories are identified by code letter. See text for explanation of scale factors used; ordinate units are g/m^2 or pH units)

FIGURE 3.2. Data Reported by MCC-RR Participants for Tests of NBS Glass in DIW (Laboratories are identified by code letter. See text for explanation of scale factors used; ordinate units are g/m^2 or pH units)

FIGURE 3.3. Data Reported by MCC-RR Participants for Tests of NBS Glass in Brine (Laboratories are identified by code letter. See text for explanation of scale factors used; ordinate units are g/m^2 or pH units)

FIGURE 3.4. Data Reported by MCC-RR Participants for Tests of Basalt in SiW (Laboratories are identified by code letter. See text for explanation of scale factors used; ordinate units are g/m² or pH units)

FIGURE 3.5. Data Reported by MCC-RR Participants for Tests of Basalt in DIW (Laboratories are identified by code letter. See text for explanation of scale factors used; ordinate units are g/m^2 or pH units)

FIGURE 3.6. Data Reported by MCC-RR Participants for Tests of Basalt in Brine (Laboratories are identified by code letter. See text for explanation of scale factors used; ordinate units are g/m^2 or pH units)

FIGURE 3.7. Data Reported by MCC-RR Participants for Tests of MCC Glass in SiW (Laboratories are identified by code letter. See text for explanation of scale factors used; ordinate units are g/m² or pH units)

FIGURE 3.8. Data Reported by MCC-RR Participants for Tests of MCC Glass in DIW (Laboratories are identified by code letter. See text for explanation of scale factors used; ordinate units are g/m^2 or pH units)

FIGURE 3.9. Data Reported by MCC-RR Participants for Tests of MCC Glass in Brine (Laboratories are identified by code letter. See text for explanation of scale factors used; ordinate units are g/m^2 or pH units)

TABLE 3.1. NBS Glass Data for Late Laboratories

Laboratory	Leachant	Element g/m ²				Mass Loss	Final pH
		B	K	Na	Si		
N	SiW	30.26	42.39	-27.01	24.75	42.43	9.110
		27.01	42.28	55.87	22.44	32.56	8.980
		25.67	39.56	-13.57	20.27	31.58	9.090
Z	SiW	64.60		13.61	23.71	27.17	9.300
		69.81		13.51	22.32	27.50	9.100
		74.10		16.33	24.02	29.04	9.300
N	DIW	6.30	8.55	6.11	6.33	15.99	8.410
		35.06	37.05	45.46	23.07	37.47	8.720
		27.63	30.55	46.64	25.09	40.26	8.690
Z	DIW	13.92		2.16	4.73	5.76	8.000
		15.38		2.31	4.16	6.41	8.000
		15.77		2.29	4.11	5.66	7.900
N	Br	11.10	1765.91		10.18	21.00	6.220
		12.09	1707.93		3.63		6.210
		10.89	-611.89		7.06		6.360
Z	Br	15.37			5.97	8.71	5.800
		14.97			5.89	7.83	6.000
		14.82			5.91	8.55	5.900

TABLE 3.2. Basalt Data for Late Laboratories

<u>Laboratory</u>	<u>Leachant</u>	<u>Element g/m²</u>				<u>Mass Loss</u>	<u>Final pH</u>
		<u>Ca</u>	<u>Fe</u>	<u>Na</u>	<u>Si</u>		
V	SiW	0.37	0.04	0.00	5.46	10.84	9.600
		0.26	0.05	0.00	4.45	11.42	9.800
		0.52	0.09	-5.56	5.08	11.43	9.500
V	DIW	1.70	0.18	27.78	11.64	15.59	8.300
		1.84	0.43	33.33	10.00	14.28	8.100
		1.70	-0.02	50.00	6.81	15.64	8.400
V	Br	4.05	0.12		5.04	1.30	5.950
		4.86	0.13		4.29	2.33	5.850
		5.41	0.26		5.08	2.86	6.050

TABLE 3.3. MCC Glass Data for Late Laboratories

Laboratory	Leachant	Element g/m ²				Mass Loss	Final pH
		B	Cs	Sr	Si		
N	SiW	13.25		0.00	13.21	14.53	9.520
		15.97		0.00	15.05	27.19	9.720
		14.22		0.30	16.03	16.71	9.360
V	SiW	22.25	24.55	1.25	10.73	7.55	9.000
		21.05	24.50	1.13	9.85	7.23	9.000
		23.09	25.25	1.13	11.32	7.71	9.000
Y	SiW	27.33	0.00	1.06	33.23	10.95	9.800
		22.23	0.00	1.38	28.92	9.12	9.700
		26.99	0.00	0.94	31.99	11.20	9.800
Z	SiW	30.81		1.01	16.28	9.39	9.100
		30.04		0.97	14.45	9.00	9.200
		31.25		0.85	15.45	8.76	9.200
N	DIW	16.11		1.48	23.29	16.52	9.510
		17.31		0.90	24.62	21.63	9.890
		14.16		0.89	20.11	19.80	9.660
V	DIW	37.05	40.29	0.97	23.26	13.86	9.300
		35.49	39.32	1.12	22.43	12.55	9.600
		36.29	39.76	1.00	22.81	12.60	9.100
Y	DIW	27.13	0.00	2.65	34.25	12.78	9.500
		28.83	0.00	2.35	33.49	13.02	9.400
		24.06	0.00	3.53	30.38	11.96	9.400
Z	DIW	30.56		2.11	19.85	13.88	9.900
		37.53		1.36	24.04	14.22	9.500
		37.79		1.55	24.21	15.06	9.800
N	Br	21.58		47.15	13.54	5.09	6.830
		12.95		34.80	18.51	22.95	6.680
		18.40		48.00	13.79	15.03	6.840
V	Br	9.25		10.48	6.95	7.67	7.200
		9.61		10.88	7.22	6.49	7.200
		8.09		9.24	6.39	7.14	7.200
Y	Br	20.83	48.54	25.59	20.32	8.10	6.650
		21.17	48.54	27.06	21.18	8.06	6.540
		21.51	29.13	27.65	21.18	7.82	6.550
Z	Br	18.80		9.41	7.39	5.45	6.700
		17.82		9.73	7.41	5.01	6.600
		18.68		11.31	7.48	4.94	6.700

3.2 ARR PLOTS

The four laboratories [C, D, F and S(MCC)] that conducted the fully replicated sampling and analyses specified for the ARR reported concentrations (mg/l) of each element in each of the aliquots run on each day. For most elements and simulated leachates, the aliquot within-day variability was about the same as the day-to-day-variability within a laboratory. Figures 3.10, 3.11 and 3.12 for simulated leachates SL-1, SL-2 and SL-3 are plots of the element averages of the aliquots run on a particular day for each laboratory. Each panel within a figure is for a single element analyzed in the respective simulated leachate. The element and the scale factor used are given at the bottom of each panel; explanation of the plot format is the same as for the MCC-1 RR. Points are plotted with the laboratory letter code.

Laboratory differences from the consensus element averages for each simulated leachate are plotted in Figures 3.13 through 3.15, for all 11 laboratories that reported ARR element averages. The value plotted is the percentage difference of the laboratory average from the consensus average for the four full-design laboratories. It was calculated as:

$$\text{PERCENT DIFF} = 100 (\text{Lab Average} - \text{Consensus Average}) / \text{Consensus Average},$$

for each of the simulated leachates. The average concentrations reported by each of the 11 ARR participants are given in Table 3.4. The consensus averages are from the four "full-design" laboratories, as given in Table 2.5. The points are plotted with the element letter code, and the abscissa axis gives the laboratory's identification code. The vertical lines connect all elemental analyses from a single laboratory for that ARR leachate. A laboratory can identify its problem areas by locating its code on the x-axis and noting the larger deviations of its element codes from the horizontal zero line. For example, in Figure 3.13, Lab F is low for strontium (R), but not as low as Lab T. Lab M is high for all elements and, except for strontium (R), Lab S(MCC) is below the consensus average.

FIGURE 3.10. Comparison of Daily Averages for Analysis of Simulated Leachate SL-1 (Laboratories are identified by code letter. See text for explanation of scale factors used.)

FIGURE 3.11. Comparison of Daily Averages for Analysis of Simulated Leachate SL-2 (Laboratories are identified by code letter. See text for explanation of scale factors used.)

FIGURE 3.12. Comparison of Daily Averages for Analysis of Simulated Leachate SL-3 (Laboratories are identified by code letter. See text for explanation of scale factors used.)

FIGURE 3.13. Relative Differences of Participant Laboratory's Analysis from the Consensus Averages for Elements in Simulated Leachate SL-1 (%)

FIGURE 3.15. Relative Differences of Participant Laboratory's Analysis from the Consensus Averages for Elements in Simulated Leachate SL-3 (%)

TABLE 3.4. Laboratory Averages of Analyses of Simulated Leachates in the ARR

Specimen	Laboratory	Element Concentration (mg/l)					
		B	CS	K	NA	SI	SR
SL-1	A					19.38	5.07
	B	55.28		-0.01	74.51	19.59	4.91
	C	50.38	5.77	0.10	87.56	18.77	5.03
	D	48.82	5.03		79.95	17.50	4.78
	F	58.39	7.02	0.22	101.44	23.02	3.00
	G	43.00	4.50		91.00	20.00	5.00
	K	50.50	5.14	0.14	85.10	18.40	6.18
	M	53.10	7.50	0.09	90.02	20.80	4.70
	S	47.06	4.46		79.52	18.92	5.13
	T	195.00	5.10	0.08	82.80	18.90	1.38
	W	55.10	5.29	0.11	75.60	20.00	4.55
	Consensus Average ^(a)	50.96	5.62	--	87.07	19.60	4.55
SL-2	B	27.98		-0.01	36.95	-0.01	2.38
	C	25.77	6.52	0.05	42.19	0.19	2.48
	D	24.07	5.22		40.88	0.00	2.43
	F	40.17	5.69	0.06	46.00	0.17	3.42
	K	25.70	5.29	0.06	42.20	-0.01	2.85
	M	27.30	9.40	0.04	42.65	1.10	2.20
	S	23.45	4.62		38.80		2.55
	T	179.00	5.20	0.05	39.30		2.40
	W	26.50	5.46	0.05	41.79		2.71
	Consensus Average ^(a)	28.12	5.44	--	41.79	--	2.71
SL-3	B	91.96		18942.00	18152.00	21.35	4.69
	C	48.17	10.30	20069.00	20975.00	27.50	4.79
	D	48.40	4.30	4480.00	-1.75	20.53	3.63
	F	66.39	14.19	18272.00	25756.00	22.83	13.36
	K		2.56		20950.00	19.80	
	M	51.20	6.60	19757.60	17699.00	46.20	4.70
	S	48.65	4.72	21419.00	21456.00	15.22	5.35
	T	64.50	4.20	17700.00	21200.00	2.36	1.73
	W	52.80	7.19	18900.00	17900.00	25.00	1.85
	Consensus Average ^(a)	52.59	8.75	19920	22259	21.08	4.78

(a) Data from four laboratories; see Table 2.5.

3.3 COMPARISONS OF PARTICIPANT RESULTS WITH MCC DATA

The MCC-1 Static Leach Test Method is the result of extensive research and development activity by the MCC organization. As a consequence, the MCC has accumulated considerably more experience with application of the MCC-1 test procedure than the other participants in the round robin. A comparison of the MCC's results with the consensus average of all participants is made in Figure 3.16. The participant data used are the consensus averages for all 29 element-leachant-waste form combinations, as given in Table 2.4; the MCC data are the averages obtained for MCC's five specimens. All data for Figure 3.16 are tabulated in Table 3.5, in order of increasing consensus average concentration EL. The figure shows that all MCC values (except the calcium at 30 g/m²) fall near or slightly above the line of equality, indicating that all the consensus averages are equal or slightly higher than the MCC analyses. The interested reader can plot the averages for individual laboratories (taken from Tables 7.1 to 7.4) for direct comparison with MCC. Direct comparisons of the biases relative to the consensus average are given in Figures 7.1 through 7.8.

As a direct check for relative biases in MCC-1 RR results which were introduced by the analytical methods used by the participating laboratories, leachates obtained from two of the five specimens tested by MCC-1 at each laboratory for each waste form-leachant combination were sent to the MCC for analysis. The MCC used inductively coupled plasma (ICP) spectrography for all elements except cesium, for which atomic absorption was used. It is important to remember that the leachates sent to MCC for analysis contained all the sources of variability due to running the test at the participant's laboratory, plus any due to preparation for shipment and transit to MCC. The transportation contribution to variability is expected to be negligibly small. A comparison plot for each participating laboratory which returned leachates is given in Figures 3.17 through 3.32. In these figures, the laboratory value is shown by the vertical axis and the MCC value by the horizontal axis. A point above the line of equality means that the average of the three laboratory values was high relative to the average of the two MCC's values for the same laboratory's RLs.

FIGURE 3.16. Comparison of Participant's Consensus Averages with the Corresponding MCC Average (see Table 3.5)

TABLE 3.5. Comparison of MCC Test Averages with Participants
Consensus Averages for MCC-1 RR

Element Conc'n Rank	Waste Form, Leachant Tested ^(a)	Element Analyzed	Element Concentration Consensus Avg. (EL)	(g/m ²) MCC Avg. (MCC)	Conc'n Diff., MCC-EL
1	BAS, SiW	Ca	0.71	0.91	0.20
2	MCC, SiW	Sr	0.82	1.02	0.20
3	BAS, BR	Fe	1.00	0.33	-0.67
4	MCC, DIW	Sr	1.85	1.98	0.13
5	BAS, BR	Si	2.15	1.09	-1.06
6	BAS, DIW	Ca	2.69	1.54	-1.15
7	BAS, SiW	Si	2.72	2.22	-0.50
8	NBS, DIW	Si	4.89	2.64	-2.25
9	BAS, DIW	Si	5.59	5.46	-0.13
10	NBS, BR	Si	8.09	8.16	0.07
11	BAS, DIW	Na	9.86	10.16	0.30
12	MCC, BR	Si	11.09	6.89	-4.20
13	NBS, DIW	K	11.87	8.07	-3.80
14	NBS, DIW	Na	13.43	8.89	-4.54
15	MCC, BR	Sr	16.52	17.65	1.13
16	NBS, DIW	B	17.81	10.38	-7.43
17	MCC, SiW	Si	18.11	12.80	-5.31
18	NBS, BR	B	19.21	17.32	-1.89
19	MCC, BR	Cs	22.59	8.53	-14.06
20	MCC, BR	B	23.16	13.61	-9.55
21	MCC, SiW	Cs	23.79	21.16	-2.63
22	MCC, DIW	Si	23.91	18.82	-5.09
23	NBS, SiW	Si	25.61	23.18	-2.43
24	MCC, DIW	Cs	27.75	26.94	-0.81
25	BAS, BR	Ca	30.83	36.44	5.61
26	MCC, SiW	B	32.42	21.14	-11.26
27	MCC, DIW	B	36.05	25.87	-10.18
28	NBS, SiW	K	44.98	45.49	0.51
29	NBS, SiW	B	61.64	59.08	-2.56

(a) See Table 2.1.

FIGURE 3.17. Comparison of MCC-1 RR Test Averages of Lab A with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.18. Comparison of MCC-1 RR Test Averages of Lab B with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.19. Comparison of MCC-1 RR Test Averages of Lab C with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.20. Comparison of MCC-1 RR Test Averages of Lab D with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.21. Comparison of MCC-1 RR Test Averages of Lab F with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.22. Comparison of MCC-1 RR Test Averages of Lab H with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.23. Comparison of MCC-1 RR Test Averages of Lab I with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.24. Comparison of MCC-1 RR Test Averages of Lab J with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.25. Comparison of MCC-1 RR Test Averages of Lab K with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.26. Comparison of MCC-1 RR Test Averages of Lab L with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.27. Comparison of MCC-1 RR Test Averages of Lab M with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.28. Comparison of MCC-1 RR Test Averages of Lab O with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.29. Comparison of MCC-1 RR Test Averages of Lab R with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.30. Comparison of Lab S(MCC) "Triplicate MCC-1 RR Test" Averages with MCC's Two Designated "Returned Leachates" (All five tests and analyses conducted by the same MCC laboratories)

FIGURE 3.31. Comparison of MCC-1 RR Test Averages for Lab T with MCC Analysis of Corresponding Returned Leachate (RL)

FIGURE 3.32. Comparison of MCC-1 RR Test Averages for Lab U with MCC Analysis of Corresponding Returned Leachate (RL)

4.0 TIME SEQUENCE RESULTS FOR LEACH TEST IN DEIONIZED WATER

The MCC-1 RR procedure required running a single specimen of each waste form in DIW leachant for 3 days, another specimen for 7 days and a third specimen for 14 days, as well as the five specimens tested for 28 days. The concentration for the 3-, 7-, 14- and 28-day leachates analyzed by each laboratory are given in the tables and plots of this section.

The average of the three 28-day concentrations reported was used for the 28-day data point for each laboratory. Since the participating laboratories were not required to run blanks corresponding to the 3-, 7- and 14-day specimens, none of the concentration data (including the 28-day data) were adjusted for blanks in this comparison series. Therefore, the elemental mass loss calculation was

$$NL_i = 0.1E_i / (f_i SA/V) \quad (4.1)$$

where the notation is as defined for Equations 2.1 and 2.2. Since DIW was used, the blank concentrations should be small compared to concentrations of the corresponding elements in the leachates from specimens. It was assumed that the DIW blanks would not change appreciably over 28 days; so not correcting for blanks should not have a large impact on comparisons between laboratories over time.

There is a table and plot for each element-waste form combination, and for specimen mass loss. Iron for basalt was at the detection limit, so it is not included in the series. Data points for laboratories reporting very high concentrations were not included on some plots, and are so noted on the plot. The laboratory letter code is plotted at the point corresponding to the element concentration for each day analyzed.

The tables also report the consensus average in the next to bottom row; the averages with apparently anomalous data ("fliers") deleted appear in the bottom row. Data deleted are indicated in the first column of the bottom row by laboratory letter codes. These bottom-row averages (with apparently anomalous data deleted) are shown on the plots with open circles connected by a solid line. The MCC's data points (Lab S) are connected by a dashed line.

TABLE 4.1. Boron Concentration in DIW/NBS Glass Leachate (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
B	4.15	6.22	3.20	4.77
C	24.46	28.23	26.37	27.17
D	15.58	15.68	15.70	16.72
F	0.00	0.00	0.00	11.95
H	18.62	23.12	14.03	28.05
I	10.57	13.03	12.77	14.69
J	31.41*	39.04*	46.68*	53.18*
K	11.77	11.36	12.06	21.46
L	20.00	14.89	21.15	20.00
M	14.57	17.55	15.38	18.69
O	0.19	0.19	0.19	1.51
P	11.60	14.24	16.74	13.67
Q	12.72	14.40	15.15	16.71
R	10.06	12.91	10.88	12.75
S	11.02	13.24	10.43	10.57
T	90.00*	91.89*	94.15*	98.09*
X	7.38	13.15	15.07	15.61
DAY MEANS	17.30	19.36	19.41	22.68
WITHOUT T,J	7.79	9.72	9.02	11.80

TABLE 4.2. Potassium Concentration in DIW/NBS Glass Leachate (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
B	1.82	8.47	2.57	5.14
C	20.41	20.20	20.25	21.41
D	9.20	9.42	9.48	10.88
F	5.76	7.58	9.39	10.10
H	10.01	13.09	6.60	19.29
I	3.24	5.44	6.33	6.97
J	23.11*	28.14*	33.18*	36.54*
K	7.10	7.07	7.69	16.31
L	12.42	9.39	13.49	13.43
M	8.44	10.61	10.88	17.58
O	1.35	1.36	1.37	7.56
P	8.88	12.09	11.29	10.87
Q	8.76	10.28	11.12	12.63
R	6.97	9.11	8.39	9.02
S	7.47	15.30	7.88	8.21
T	6.74	7.71	9.03	9.88
X	6.07	8.45	8.62	9.53
DAY MEANS	8.69	10.80	10.44	13.26
WITHOUT J	7.79	9.72	9.02	11.80

*Data are out of the bounds chosen for plot.

LABS T, J OUT OF BOUNDS

FIGURE 4.1. Boron Concentration in DIW/NBS Glass Leachate
(Plotted by Laboratory Code Letter)

LAB J OUT OF BOUNDS

FIGURE 4.2. Potassium Concentration in DIW/NBS Glass Leachate (Plotted by Laboratory Code Letter)

TABLE 4.3. Sodium Concentration in DIW/NBS Glass Leachate (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
B	3.65	8.37	5.13	13.90
C	20.39	22.01	22.99	25.11
D	12.50	12.69	12.77	14.30
F	10.81	13.51	16.21	18.47
H	16.23	21.71	10.86	32.51*
I	8.72	10.64	9.94	12.70
J	35.94*	32.29*	28.65*	44.32*
K	9.95	9.87	10.65	25.06
L	14.86	10.80	16.23	15.76
M	15.14	15.81	14.59	19.14
O	6.70	6.74	6.80	8.09
P	9.26	11.87	14.06	11.63
Q	12.09	12.56	15.15	14.42
R	7.99	12.26	25.26*	11.40
S	9.12	13.01	9.44	9.06
T	8.11	9.46	12.97	19.08
X	9.11	10.77	10.82	11.76
DAY MEANS	12.39	13.79	14.27	18.04
WITHOUT J	10.91	12.63	13.37	16.40

TABLE 4.4. Silicon Concentration in DIW/NBS Glass Leachate (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
B	0.43	0.40	0.40	0.90
C	8.24*	10.33*	9.07*	10.07*
D	0.65	0.94	1.35	1.60
F	1.10	1.62	2.63	10.70*
H	3.49	5.56	4.77	3.72
I	1.76	2.89	2.83	3.74
J	13.65*	17.98*	22.31*	24.74*
K	0.60	0.93	1.37	2.25
L	0.70	1.37	1.35	2.25
M	0.77	1.77	2.01	2.63
O	0.24	0.55	0.55	1.53
P	2.48	2.36	2.51	3.84
Q	1.53	2.21	2.03	2.65
R	1.33	1.33	2.29	2.58
S	2.78	4.13	2.57	2.72
T	1.68	2.45	3.73	3.75
X	1.40	2.28	2.87	3.01
DAY MEANS	2.52	3.48	3.80	4.86
WITHOUT J,C	1.40	2.05	2.22	3.19

*Data are out of the bounds chosen for plot.

LABS H,J,R OUT OF BOUNDS

FIGURE 4.3. Sodium Concentration in DIW/NBS Glass Leachate
(Plotted by Laboratory Code Letter)

LABS C,J OUT OF BOUNDS

FIGURE 4.4. Silicon Concentration in DIW/NBS Glass Leachate
(Plotted by Laboratory Code Letter)

TABLE 4.5. Mass Loss for NBS Glass in DIW (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
B	3.20	4.45	4.08	4.83
C	11.14*	12.72*	11.14*	13.58*
D	2.67	3.38	4.36	3.67
F	1.28	2.82	6.13	4.14
H	2.00	9.79	6.24	8.19
I	3.49	4.52	3.92	5.03
J	15.28*	19.18*	23.07*	25.76*
K	0.00	13.23	2.46	11.31*
L	3.33	3.29	4.24	5.02
M	2.75	4.24	3.25	5.57
O	0.00	0.44	0.45	1.05
P	4.02	4.43	4.66	5.34
Q	2.78	3.71	3.49	4.14
R	2.26	1.51	3.75	3.58
S	4.23	6.09	3.92	4.07
T	3.27	6.44	6.87	9.66
X	2.44	3.62	4.18	4.37
DAY MEANS	3.77	6.11	5.66	7.02
WITHOUT J	3.05	5.29	4.57	5.85

TABLE 4.6. Calcium Concentration in DIW/Basalt Leachate (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
A	1.000	2.054	5.797*	2.527
F	0.000	0.000	0.000	0.090
I	0.737	1.128	1.187	1.229
K	2.154	2.355	2.455	5.727*
L	1.689	3.363	1.230	3.812
M	2.720	2.189	2.811	5.095*
P	0.773	2.095	2.499	1.872
S	0.728	0.846	1.142	1.648
T	1.824	2.108	2.392	3.063
DAY MEANS	1.292	1.793	2.168	2.785

*Data are out of the bounds chosen for plot.

LABS C,J OUT OF BOUNDS

FIGURE 4.5. Mass Loss for NBS Glass in DIW
(Plotted by Laboratory Code Letter)

LABS A,K,M OUT OF BOUNDS

FIGURE 4.6. Calcium Concentration in DIW/Basalt Leachate
(Plotted by Laboratory Code Letter)

TABLE 4.7. Sodium Concentration in DIW/Basalt Leachate (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
A	2.78	3.72	6.11	6.81
F	7.22	11.11	11.11	13.89
I	5.34	7.37	8.32	10.63
K	9.19	9.63	13.28	18.69
L	6.11	8.88	8.89	14.45
M	11.01	10.33	11.11	17.11
P	5.61	7.50	8.72	9.89
S	5.02	5.53	7.78	10.85
T	3.89	5.00	7.22	8.70
DAY				
MEANS	6.24	7.67	9.17	12.34

TABLE 4.8. Silicon Concentration in DIW/Basalt Leachate (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
A	1.597	2.941	3.739	5.098
F	1.134	2.689	3.319	4.916
I	1.904	3.135	3.460	4.849
K	2.777	3.021	4.654	7.123
L	1.470	3.150	3.363	5.604
M	1.981	2.966	4.424	6.508
P	1.902	3.354	3.911	4.946
S	1.974	2.751	3.891	5.601
T	0.798	1.933	3.487	5.756
DAY				
MEANS	1.727	2.882	3.805	5.600

*Data are out of the bounds chosen for plot.

FIGURE 4.7. Sodium Concentration in DIW/Basalt Leachate
(Plotted by Laboratory Code Letter)

FIGURE 4.8. Silicon Concentration in DIW/Basalt Leachate
(Plotted by Laboratory Code Letter)

TABLE 4.9. Mass Loss for Basalt in DIW (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
A	4.6	6.2	7.2	6.6
F	0.0	6.1	3.5	5.6
I	3.2	3.9	3.2	7.1
K	-12.6	-12.4	-4.9	5.0
L	1.2	2.1	2.1	3.4
M	-3.5	-3.5	-4.8	-1.8
S	29.3	21.9	22.3	19.6
T	88.6*	9.1	-2.9	34.6
DAY				
MEANS	13.8	4.2	3.2	10.0
WITHOUT T	3.2	3.5	4.1	6.5

TABLE 4.10. Boron Concentration in DIW/MCC Glass Leachate (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
A	--	--	--	--
B	7.812	15.288	24.471	30.174
D	2.991	4.944	15.079	27.030
F	0.000	0.000	0.000	63.496*
G	34.678	26.466	14.655	30.924
H	3.980	5.520	--	28.884
I	7.900	13.417	17.376	30.736
J	16.667	25.575	35.032	48.818*
K	6.541	9.650	7.788	28.419
P	10.557	16.038	21.865	40.819*
S	6.666	10.104	15.774	26.316
T	157.482*	155.102*	169.047*	184.013*
U	10.332	15.970	22.300	36.696
DAY	22.134	24.839	31.217	48.027
MEAN				
WITHOUT T	9.83	13.00	17.43	35.67

*Data are out of the bounds chosen for plot.

LAB T OUT OF BOUNDS

FIGURE 4.9. Mass Loss for Basalt in DIW
(Plotted by Laboratory Code
Letter)

FIGURE 4.10. Boron Concentration in DIW/MCC Glass Leachate
(Plotted by Laboratory Code Letter)

TABLE 4.11. Cesium Concentration in DIW/MCC Glass Leachate (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
A	8.446	14.368	19.708	38.932
B	--	--	--	--
D	3.091	5.255	17.020	29.391
F	4.854	11.650	21.358	39.484
G	6.860	12.754	18.484	28.349
H	4.175	5.835	--	21.074
I	0.000	0.000	0.000	0.000
J	3.184	2.376	4.200	6.183
K	7.693	13.213	9.279	28.671
P	11.664	17.458	25.340	41.103
S	8.349	13.012	18.534	27.303
T	6.893	11.844	19.417	37.540
U	8.946	13.234	22.523	32.956
DAY MEAN	6.180	10.083	15.988	27.582

TABLE 4.12. Strontium Concentration in DIW/MCC Glass Leachate (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
A	4.9999	5.2941	5.2941	2.2745
B	4.6992	4.9942	4.1145	2.6578
D	2.5168	4.3933	5.2452	2.1408
F	0.0000	0.0000	0.0000	0.0000
G	4.1566	7.4303	4.4207	2.9277
H	3.7653	5.4213	--	3.2412
I	5.0195	5.1810	4.4080	1.8098
J	0.9470	0.9469	0.9470	0.9470
K	4.8889	6.1584	7.5688	3.7060
P	4.4172	4.9362	3.6167	0.8039
S	4.6470	5.0600	3.6452	1.9404
T	6.7647	7.9411	5.8823	1.2843
U	4.7651	4.6032	3.8567	2.0363
DAY MEANS	3.9682	4.7969	4.0832	1.9823
WITHOUT J,F	4.60	5.68	4.81	2.26

*Data are out of the bounds chosen for plot.

FIGURE 4.11. Cesium Concentration in DIW/MCC Glass Leachate
(Plotted by Laboratory Code Letter)

FIGURE 4.12. Strontium Concentration in DIW/MCC Glass Leachate (Plotted by Laboratory Code Letter)

TABLE 4.13. Silicon Concentration in DIW/MCC Glass Leachate (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
A	6.612	10.053	12.956	19.086
B	5.905	11.277	15.042	17.440
D	2.765	4.344	11.104	18.374
F	7.956	12.849	18.816	45.164*
G	5.427	11.952	15.623	26.759
H	3.796	5.299	--	18.427
I	6.113	9.799	12.376	19.370
J	13.231	19.782	24.977	33.059
K	5.923	8.274	7.623	19.540
P	9.043	13.212	16.555	27.636
S	6.182	8.711	12.628	19.078
T	4.731	8.817	13.924	26.362
U	6.750	9.283	13.177	19.334
DAY MEANS	6.495	10.281	14.567	23.818

TABLE 4.14. Mass Loss for MCC Glass in DIW Leachate (g/m²)

LABORATORY	TEST DURATION, DAYS			
	3	7	14	28
A	4.066	5.910	9.324	12.293
B	4.059	8.276	11.405	10.966
D	1.965	3.834	7.909	11.993
F	4.409	6.186	18.033	12.981
G	14.721	9.824	19.762	15.492
H	2.750	5.509	--	13.775
I	3.816	6.530	8.253	13.215
J	8.942	11.223	13.465	19.753
K	4.832	10.469	7.877	13.310
P	6.027	8.963	10.540	17.254
S	4.115	5.818	8.277	13.116
T	18.382	24.056*	9.808	31.356*
U	4.215	5.800	8.562	11.646
DAY MEANS	6.331	8.646	11.101	15.165

*Data are out of the bounds chosen for plot.

LAB F OUT OF BOUNDS

FIGURE 4.13. Silicon Concentration in DIW/MCC Glass Leachate (Plotted by Laboratory Code Letter)

FIGURE 4.14. Mass Loss for MCC Glass in DIW Leachate
(Plotted by Laboratory Code Letter)

The time sequence results for the four late laboratories are listed in Table 4.15.

TABLE 4.15. Time Sequence Results for Late Laboratories, g/m²

Element	Laboratory	Waste Form	Test Period, Days			
			3	7	14	28
Boron	N	NBS	8.1	10.8	14.6	23.0
	Z	NBS	12.6	16.2	16.2	15.2
	N	MCC	4.2	5.7	8.2	15.8
	V	MCC	10.7	15.7	21.5	36.4
	Y	MCC	6.5	10.9	16.0	26.8
	Z	MCC	7.8	15.8	24.5	35.4
Calcium	Y	BAS	0.9	1.1	1.5	2.1
Potassium	N	NBS	17.9	19.4	19.4	25.9
Cesium	V	MCC	12.1	19.4	24.8	39.8
Sodium	N	NBS	17.9	30.4	25.2	36.7
	Z	NBS	2.4	2.4	2.7	2.7
	Y	BAS	41.7	44.4	77.8	37.0
Strontium	N	MCC	3.8	3.7	3.0	1.1
	V	MCC	4.6	4.0	2.3	1.0
	Y	MCC	5.3	5.6	4.7	2.8
	Z	MCC	4.7	4.1	2.9	1.7
Silicon	N	NBS	5.1	8.7	11.7	18.2
	Z	NBS	2.9	3.7	4.9	4.5
	Y	BAS	2.6	4.0	6.1	9.5
	N	MCC	7.8	10.5	14.6	22.6
	V	MCC	8.0	11.6	14.5	22.8
	Y	MCC	10.8	15.9	24.1	32.7
	Z	MCC	6.4	11.2	17.7	23.0
Mass Loss	N	NBS	19.5	8.2	16.2	31.2
	Z	NBS	3.4	5.2	6.7	5.9
	Y	BAS	6.7	33.7	4.4	15.2
	N	MCC	13.1	14.4	16.5	19.3
	V	MCC	5.0	7.2	8.9	13.0
	Y	MCC	3.9	6.3	8.6	12.6
	Z	MCC	3.9	7.6	10.7	14.4

5.0 SUMMARY OF NARRATIVE DATA

The evaluation of a test procedure includes an assessment of how well the specified procedure was followed. This assessment will assist in providing direction for improving the procedure. Just as with numerical data, the value of the narrative data is dependent on the completeness and accuracy of the reported information.

5.1 SPECIMEN PREPARATION

The wafers used as specimens differed in method of cutting and surface preparation, and in their geometry. Three laboratories (C, J and M) used diamond drills to produce a cylindrical sample, which was then sectioned to produce wafers with height-to-diameter ratios of 0.30, 0.38 and 0.52. The other laboratories used 200- to 240-grit diamond cutting blades to section the sample into wafers. All laboratories removed the uncut surfaces from the as-received samples. There was great diversity in the sequence of cuts used to arrive at wafers of the specified geometric surface area of about 400 mm². Three laboratories (H, K and U) polished the specimens after cutting. The average surface area for the specimens prepared by each laboratory was between 346 and 442 mm², except for Lab F which had an average of 500 mm². The various cutting procedures produced specimens with aspect ratios (minimum dimension/maximum dimension) ranging from 0.04 (a wafer) to a 0.995 (a cube). Two laboratories (O and T) used ethylene glycol as the cutting fluid, the others used dionized water (6 laboratories), distilled water (5 laboratories), tap water (2 laboratories), or unspecified water (5 laboratories). Branson® ultrasonic cleaners were used by 14 laboratories (Models B-12, B-32, B-52, B-72 or B-220). Cleaning fluid was some form of alcohol, variously reported as ETOH, absolute ethenol, pure ethyl alcohol, or denatured ethyl alcohol. Two laboratories (A and P) precleaned the specimens with acetone. Various batching schemes were used for cleaning. Some laboratories reported difficulty in drying specimens to constant weight to get the initial specimen

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weight, particularly for the basalt specimen. Storage containers and storage times before start of tests varied widely. All laboratories reported using "clean" procedures in handling specimens, including lint-free gloves and tweezers of various types. Average specimen weights at the laboratories varied from 0.94 g to 1.5 g, except for Labs A and B (low at 0.34 g and 0.68 g), and Lab L (high at 1.65 g).

5.2 LEACHANT PREPARATION

Only two laboratories reported deviations from the directions for leachant preparation. Lab R used 247.5 g of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ (not 116.0 g MgCl_2) in the brine leachant. Lab U found that its "AR grade silicic acid would not completely dissolve in the recommended amount of NaHCO_3 solution"; instead, $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ was dissolved in water. Storage durations and time of leachant analysis varied considerably. The average chemical analysis and pH reported for each leachant is given in Table 5.1 along with the standard deviations.

5.3 TEST CONDITIONS

All laboratories used the Savillax® 60 ml capacity container with tight fitting lid. Specimens were suspended by tying various knots around the specimen and to the lid. Most laboratories (13) used Blue M® ovens. Other ovens were Fisher Scientific®, Bekel gravity convection, Valenti Thermostabil and NVC Australia Pty. Ltd. Most laboratories reported some check of the calibration of the temperature-monitoring equipment used. Fewer laboratories discussed the calibration of the other equipment used in the test, e.g., calipers, scales, and pipettes.

Perhaps of greater concern than the routine calibration and measurement control practices used by experienced laboratories are the broad variabilities in the elapsed time between preparation of leachant and specimen and starting

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TABLE 5.1. Analysis of Leachants by Participants (Leachant Averages and Standard Deviations, mg/l)

	SiW			DIW			Brine		
	N ^(a)	Ave	sd	N ^(a)	Ave	sd	N ^(a)	Ave	sd
B	17	0.161	0.364	17	0.132	0.361	14 ^(b)	1.412	1.52
Ca	8	0.072	0.114	8	0.076	0.112	7	1.014	1.164
Cs	8 ^(c)	0.110	0.229	8 ^(c)	0.108	0.226	7 ^(c)	0.520	0.672
Fe	8 ^(d)	0.006	0.007	8 ^(d)	0.006	0.007	8	0.867	0.967
K	16	0.782	1.27	16	0.145	0.220	15 ^(d)	20650	8722
Na	17 ^(d)	49	6	27	0.	0.	15 ^(d)	37214	17253
Sr	11 ^(d)	0.004	0.007	11	0.004	0.007	10	0.094	0.093
Si	18	22.76	7.02	18	0.143	0.275	18	3.596	6.32
pH	18	7.582	0.290	18	5.772	0.763	18	6.480	0.194
Specified pH	7.5 ± 0.3			---			6.4 ± 0.3		

(a) N is number of participating laboratories that reported leachant analyses.

(b) With Lab B value of 15.1 mg/l and Lab X value of 41 mg/l deleted.

(c) Lab I dl value of 41.4 deleted.

(d) Element-leachant combination corresponds to data cell not used in the analysis of the leachate data.

the test run, and the storage times of the leachates after completing the test procedure before they were analyzed by the participating laboratories or the MCC. These time periods are summarized graphically in Figure 5.1, where each laboratory is represented by a horizontal time line. The leach test run starts at day zero, and preparations and analyses precede or follow.

The time span between specimen and leachant preparation and the completion of in-house chemical analysis of the leachants and leachates ranged from 37 days (Labs Q and X) to 125 days (Lab I); up to 203 days (Lab T) elapsed before the RLs were returned and analyzed at MCC. Pre-analysis of the leachant

FIGURE 5.1. Scheduling of Steps in the MCC-1 Round Robin
(Time Lines by Laboratory)

was conducted just before the leach test was started by only 4 laboratories; 4 analyzed the leachant while the test was in progress, 8 analyzed after completion of the test (60 days later at Lab I), and 3 did not report a leachant pre-analysis date at all. Elapsed time between completion of the test run and analysis of the leachate also varied widely, from 2 days (Labs F and R) to 44 days (Lab M). Analysis of RLs was delayed even more in some cases (up to 153 days for Lab T) because of delays in shipping to MCC.

Clearly, scheduling of the important preparation and analytical steps in the MCC-1 RR varied widely among the participants. The ultimate effect of these procedural differences on test results may be difficult to determine specifically among the many other test variables.

5.4 ANALYTICAL METHODS

The chemical analysis methods used for RR solutions are summarized in Table 5.2. ICP (spectroscopy) was used in about two thirds of the 156 laboratories-element combinations, and for all elements but cesium. Atomic absorption (AA) was the next most used method (22%) followed by atomic emission (AE, 5%), colorimetry at (5%), and flame emission (FE, 3%). Other methods (silicon analysis, x-ray fluorescence, mass spectrometry, isotopic dilution, Technicon Auto-analyzer®, and neutron activation) were used by various laboratories. One laboratory did not analyze for boron, and another did not do cesium analyses.

® Auto-analyzer is a registered trade name of Technicon Industrial Systems, Terrytown, NY.

TABLE 5.2. Number of Laboratories Using Various Chemical Methods in Solution Analysis for Elements

Glass	Element	Analytical Method ^(a)						No Anal.	Total Labs
		ICP	AA	AE	Color	FE	Other		
NBS									
	B	13	1		2		1		17
	K	9	6	1		1			17
	Na	10	6			1			17
	Si	13	2		2				17
		45	15	1	4	2	1		68
Basalt									
	Ca	5	3	1					9
	Fe	6	3						9
	Na	4	3	1		1			9
	Si	7	1		1				9
		22	10	2	1	1			36
MCC 76-68 Glass									
	B	9			2		1	1	13
	Cs		6	3		1	2	1	13
	Sr	8	2	2			1		13
	Si	10	1		1		1		13
		27	9	5	3	1	5	2	52
Total Times Method Used		94	34	8	8	4	6	2	156
Frequency of Use (%)		60	22	5	5	3	4	1	100

(a) See text for method identification.

PART II. STATISTICAL ANALYSIS

The objectives of the statistical analysis of the MCC-1 RR Robin data were to

- estimate the precision of the MCC-1 procedure;
- determine sources of imprecision;
- isolate procedural steps that could be changed to improve precision.

Section 6 gives an example of the statistical method, using the data on the silicon results for NBS glass in silicon water (SiW). Section 7 gives the element-waste form-leachant summaries. Section 8 has the precision statements, allocation of total variability and other major results. Appendix E provides example of the application of the statistical summaries.

6.0 EXAMPLE OF METHODS USED FOR STATISTICAL ANALYSIS

The statistical analysis followed the suggestions given in ASTM E 691-79.⁽³⁾ A table for each element, similar to Table 6.1 for silicon provided the $p \times q$ table with n replicates per cell. Table 6.1 has $p = 20$ laboratories by $q = 9$ "materials" for a total of 180 cells, each with $n = 3$ replicates. This table has many missing cells, indicated by rows of asterisks, and three missing replicates, two for Lab H and one for Lab I. Table 6.1 shows, for example, that Labs A, G and U did not test NBS Glass and that Lab G only tested MCC Glass in DIW. Similar tables of the basic data used in the precision analysis are given in Appendix D.

Each column of data in Table 6.1 (and in the Appendix) was analyzed using components of variance analysis of variance (AOV). An example of an AOV analysis is given in Table 6.2 based on data in the first column of Table 6.1. The AOV table at the top separates the total variability in the data into the components due to between-laboratory variability and that due to within laboratory

TABLE 6.1. Waste Form-Leachant Combination

LAB	WASTE FORM LEACHANT COMBINATION								
	NBS.SI	NBS.DI	NBS.BR	BAS.SI	BAS.DI	BAS.BR	MCC.SI	MCC.DI	MCC.BR
A	*****			3.73950	5.16765	2.39454	16.8280	19.1398	11.9892
	*****			3.65546	4.99958	2.35252	16.1828	18.4946	12.4194
	*****			3.57143	5.12563	2.31050	16.7204	19.6237	12.3120
	21.6793	0.8752	4.8248	*****	*****	*****	17.1893	16.4999	12.6861
B	33.5482	0.8154	2.4426	*****	*****	*****	20.4045	16.4826	11.3794
	23.5474	0.9664	4.9530	*****	*****	*****	19.8714	19.3340	10.3670
	43.9902	9.7566	9.3361	*****	*****	*****	*****	*****	*****
C	33.6947	8.2335	10.5292	*****	*****	*****	*****	*****	*****
	30.5968	12.1663	9.5826	*****	*****	*****	*****	*****	*****
	25.0035	1.4117	15.2869	*****	*****	*****	13.3205	18.5791	12.8280
D	24.7101	1.5303	3.6767	*****	*****	*****	14.0897	17.8484	12.9949
	24.4043	1.8041	3.8975	*****	*****	*****	13.0925	18.5864	15.1715
	41.4053	10.7026	9.9687	4.91557	4.91613	2.22689	29.5161	48.9289	7.4735
F	41.4067	10.7030	9.9694	4.70568	4.91597	2.18487	30.7515	43.2830	6.2357
	41.7560	10.7038	10.3058	4.91615	4.91560	2.26891	29.5161	43.2812	8.0863
	*****	*****	*****	*****	*****	*****	24.0288	*****	*****
G	*****	*****	*****	*****	*****	*****	28.3811	*****	*****
	*****	*****	*****	*****	*****	*****	27.8787	*****	*****
	26.9211	*****	7.4715	*****	*****	*****	18.8347	18.3435	8.0092
H	32.5399	3.1838	1.8926	*****	*****	*****	19.7978	18.1130	8.0674
	*****	3.9032	2.9161	*****	*****	*****	18.6361	17.7781	8.7743
	24.7063	3.5033	6.4552	2.45019	5.28167	1.45918	14.5203	19.6804	8.0173
I	24.2359	3.8671	6.8950	2.35133	4.40700	1.44184	14.6865	*****	8.6479
	24.1562	3.7927	6.8838	2.32786	4.78568	1.61496	13.0975	19.3543	8.3801
	23.5292	23.4605	8.9817	*****	*****	*****	24.4797	32.0699	16.4853
J	30.9052	24.0532	7.6762	*****	*****	*****	24.4528	29.6831	16.3160
	25.1498	26.6848	7.1252	*****	*****	*****	24.6673	37.3934	16.3586
	12.1369	1.0785	11.9852	2.85699	6.44878	2.95542	16.1924	18.8789	13.9576
K	8.2617	3.1491	12.8213	2.38649	6.83116	2.60225	15.7481	19.1112	12.7307
	5.5540	2.4904	12.5884	2.80653	8.04387	2.37421	14.5288	20.5710	11.7204
	27.0336	1.9582	9.6733	1.59842	5.63156	2.43636	*****	*****	*****
L	25.4923	1.4675	0.9777	2.05975	5.46474	1.76642	*****	*****	*****
	27.3528	3.3301	9.2266	1.84874	5.71554	2.39322	*****	*****	*****
	12.3639	2.4648	8.5856	1.96218	6.34874	2.26261	*****	*****	*****
M	25.9694	2.7095	3.3869	1.31513	6.34454	1.20798	*****	*****	*****
	7.0000	2.6789	9.8089	1.12185	6.78151	4.47689	*****	*****	*****
	20.7549	1.2844	9.0108	*****	*****	*****	*****	*****	*****
O	20.8093	1.2787	8.9709	*****	*****	*****	*****	*****	*****
	20.9480	1.2814	9.0918	*****	*****	*****	*****	*****	*****
	26.3716	4.6231	8.1651	0.96591	4.98468	1.09379	17.1954	27.3646	11.9801
P	23.5046	2.9242	8.0387	0.67194	4.68929	1.05068	16.5096	28.3671	11.5025
	23.8218	3.9774	8.2506	0.79832	5.10595	1.26171	17.8451	27.0960	10.7527
	23.5491	2.6215	8.2493	*****	*****	*****	*****	*****	*****
Q	23.2211	2.3799	8.8595	*****	*****	*****	*****	*****	*****
	*****	*****	8.8641	*****	*****	*****	*****	*****	*****
	26.0728	2.3222	2.2659	*****	*****	*****	*****	*****	*****
R	32.4569	2.8323	4.7435	*****	*****	*****	*****	*****	*****
	30.6129	2.3418	3.0597	*****	*****	*****	*****	*****	*****
	23.3333	2.8836	8.0256	2.22581	5.61692	1.00815	13.2854	19.1720	7.2581
S MCC	23.3390	2.6682	8.2416	2.14286	5.57081	1.00840	12.4683	18.7664	7.4243
	22.9000	2.5642	8.1059	2.14286	5.57215	0.96594	13.1695	19.2333	6.7994
	36.4679	2.3790	15.2599	5.10504	5.58823	3.52941	24.5520	26.7204	16.7204
T	26.9878	4.8930	14.9235	3.76050	5.25210	3.57143	21.4785	25.4839	15.4301
	31.2691	3.9755	15.6269	5.10504	6.42857	3.82353	21.3172	26.8817	16.2903
	*****	*****	*****	*****	*****	*****	14.0635	19.0627	8.1288
U	*****	*****	*****	*****	*****	*****	10.9358	19.4967	8.0890
	*****	*****	*****	*****	*****	*****	12.0522	19.4458	7.5744
	24.5482	3.6103	8.0248	*****	*****	*****	*****	*****	*****
X	22.4209	2.7136	8.1153	*****	*****	*****	*****	*****	*****
	22.2724	2.6940	8.4156	*****	*****	*****	*****	*****	*****

TABLE 6.2. AOV of Variance for Si-NBS Glass-SiW Leachant Combination

	<u>DF</u>	<u>SS</u>	<u>MS=SS/DF</u>	<u>F-Ratio</u>	<u>V. Comp.</u>	<u>Standard Deviation</u>
Between Labs:	16	2609.4	163.1	10.42	50.1493	7.0816
Within Labs:	<u>33</u>	<u>516.4</u>	15.6		<u>15.6485</u>	<u>3.9558</u>
TOTAL	49	3125.7			65.7978	8.1116

<u>Laboratory</u>	<u>N</u>	<u>Mean</u>	<u>Standard Deviation</u>
B	3	26.26	6.38
C	3	36.09	7.01
D	3	24.71	0.30
F	3	41.52	0.20
H	2	29.73	3.97
I	3	24.37	0.30
J	3	26.53	3.88
K	3	8.65	3.31
L	3	26.63	0.99
M	3	15.11	9.78
O	3	20.84	0.10
P	3	24.57	1.57
Q	3	23.53	0.30
R	3	29.71	3.29
S	3	23.19	0.25
T	3	31.57	4.75
X	3	<u>23.08</u>	<u>1.27</u>
MEAN:		25.570	
POOLED Standard Deviation: 3.96			

variability. The degrees of freedom (DF) are an indication of the amount of data and number of averages going into the variance estimates. (The "NBS.SI" column of Table 6.1 has 50 observations: 3 replicates times 17 laboratories minus the one missing observation for Lab H.) The details of the one-way components of variance AOV calculations and theory can be found in any basic statistical methods textbook. The SS column contains the sum of squared deviations of the data from the laboratory averages for the "Within Labs" row, and of the laboratory averages from the consensus average in the "Between

Labs" row. The mean square (MS) column gives the variance estimates and the F-Ratio column gives the statistic for testing the null hypothesis that all the laboratory averages are the same, within the limits of the within laboratory variability. An F-ratio greater than 1.97 indicates that the laboratory averages are different, implying statistically significant relative biases between laboratories. The "Between Labs" MS is composed of both sources of variability. A further calculation is required to separate the variance components (V.Comp) so that each is on a per chemical analysis basis. This calculation is

$$\text{Between Labs V.Comp} = n_o (\text{Between Labs MS} - \text{Within Labs MS})$$

where n_o would be 1/3 if there were no missing data, and is a weighted average of the number of replicates per laboratory when there are missing data. Taking the square root of the variance components gives the precision statistics in the standard deviation (sd) column.

The within laboratory standard deviation is the index of the precision within laboratories, S_r . It is equivalent to "root mean squaring," or pooling, the individual laboratory standard deviations given in the second part of Table 6.2. The between laboratory standard deviation is the index of between laboratory precision, S_L . The TOTAL standard deviation is the per analysis reproducibility index, S_R . The statistics shown in Table 6.2 are for silicon from NBS Glass in SiW only. A similar AOV was done for each element-waste form-leachant combination (29 in all) to characterize the precision of the MCC-1 Static Leach Test Method.

The second part of Table 6.2 contains the number of observations (N), the mean and within laboratory standard deviation for each laboratory. The heterogeneity of within-laboratory variances is obvious from the Standard Deviation column, where the smallest standard deviation is 0.10 for Lab 0 and the largest is 9.78 for Lab M.^(a) These extremes represent the true range of variability

(a) Lab 0 reported identical concentration (mg/l) for its three replicates so that the 0.10 g/m² standard deviation is due to the variability in the SA/V ratio.

in this RR and they were allowed to have their full impact on the precision indices since heterogeneity of within-laboratory variances is to be expected in very broad-based interlaboratory tests. The goal here is to get meaningful summary indices of precision. We are not using tests of hypotheses (despite our pointing out the usual meaning of the F-Ratio in Table 6.2) so that the basic assumption of homogeneity of variances required for using the AOV to test hypotheses is not so important.^(a)

(a) A recent article⁽⁴⁾ reviewed the statistical problems of estimating variance components when heterogeneous error variances are present. The AOV procedure was only slightly worse than the other unbiased estimators based on a Monte Carlo study of the variance of the "between laboratory" variance estimators.

7.0 ELEMENT SUMMARIES

The RR data are summarized by element in two ways in this section. The tabular summary gives the laboratory averages, the consensus average and the precision statistics for each waste form-leachant combination that was analyzed for the element. The precision statistics from these tables provide the basis for characterizing the precision of the MCC-1 test procedures. The graphical summary shows the bias, relative to the consensus average, for each laboratory that analyzed the element for the waste form-leachant combination. These plots show how extreme values affected the consensus average and the precision statistics, as well as where the data for a specific laboratory fell in relation to the consensus average and the other laboratories.

7.1 SUMMARY STATISTICS BY ELEMENT

Table 7.1 is the statistical summary for a single element--silicon--for each of the three waste forms in each of the three leachants. The table can be understood by reference to the first column, which contains the results from Table 6.2. The MEAN for each laboratory is given, followed by $\overline{EL}$, the consensus average for the element in the waste form-leachant combination heading the column. The averages for the Late Laboratories come next. These averages are followed by several indices of precision defined as follows:

- S_r - Within-laboratory precision
- S_L - Between-laboratory precision
- $S_r\%$ - Relative within-laboratory precision, $100(S_r)/\overline{EL}$
- $S_L\%$ - Relative within-laboratory precision, $100(S_L)/\overline{EL}$
- S_R - Reproducibility index, the combined within- and between-laboratory precision
- $S_R\%$ - Relative reproducibility index, $100(S_R)/\overline{EL}$
- $I(R)$ - Reproducibility limit, $(2\sqrt{2})(S_R) = 2.83(S_R)$
- $I(R)\%$ - Relative reproducibility limit, $100[I(R)/\overline{EL}]$

TABLE 7.1. Summary for Silicon--All Waste Forms and Leachants, g/m²

Lab	Mean Concentration of Silicon from Waste Form • Leachant Combination								
	NBS.SI	NBS.DI	NBS.BR	BAS.SI	BAS.DI	BAS.BR	MCC.SI	MCC.DI	MCC.BR
A	---	---	---	3.6554	5.0976	2.3525	16.577	19.086	12.240
B	26.258	0.885	4.073	---	---	---	19.155	17.438	11.477
C	36.093	10.052	9.815	---	---	---	---	---	---
D	24.705	1.582	7.620	---	---	---	13.500	18.337	13.664
F	41.522	10.703	10.081	4.8457	4.9158	2.2268	29.927	45.164	7.265
G	---	---	---	---	---	---	---	26.762	---
H	29.730	3.543	4.093	---	---	---	19.089	18.078	8.283
I	24.366	3.721	6.744	2.3764	4.8247	1.5053	14.101	19.517	8.348
J	26.528	24.732	7.927	---	---	---	24.533	33.048	16.386
K	8.650	2.239	12.464	2.6833	7.1079	2.6439	15.489	19.520	12.802
L	26.626	2.251	6.625	1.8356	5.6039	2.1986	---	---	---
M	15.111	2.617	7.260	1.4663	6.4915	2.6491	---	---	---
O	20.837	1.281	9.024	---	---	---	---	---	---
P	24.565	3.841	8.151	0.8120	4.9266	1.1353	17.183	27.609	11.411
Q	23.529	2.615	8.657	---	---	---	---	---	---
R	29.714	2.498	3.356	---	---	---	---	---	---
S	23.190	2.705	8.124	2.1705	5.5866	0.9941	12.974	19.057	7.160
T	31.574	3.749	15.270	4.6568	5.7563	3.6414	22.449	26.362	16.146
U	---	---	---	---	---	---	12.350	19.335	7.930
X	23.080	3.005	8.185	---	---	---	---	---	---
EL	25.570	4.850	8.086	2.7225	5.5901	2.1497	18.111	23.906	11.093

Late
Labs(a)

N	22.487	18.166	6.956	---	---	---	14.765	22.674	15.280
V	---	---	---	---	---	---	10.636	22.831	6.850
Y	---	---	---	4.999	9.481	4.803	31.379	32.706	20.896
Z	23.352	4.332	5.924	---	---	---	15.391	22.700	7.427

Index

S _r	3.96	0.85	2.35	0.33	0.39	0.58	1.01	1.72	0.74
S _L	7.08	5.83	2.61	1.38	0.75	0.76	5.27	8.03	3.28
S _r %	15.5	17.6	29.1	12.0	7.0	27.1	5.6	7.2	6.7
S _L %	27.7	120.3	32.2	50.8	13.4	35.4	29.1	33.6	29.6
S _R	8.11	5.90	3.51	1.42	0.84	0.96	5.36	8.21	3.36
S _R %	31.7	121.5	43.4	52.2	15.1	44.6	29.6	34.4	30.3

Reproducibility Limit

I(R)	22.94	16.67	9.93	4.02	2.39	2.71	15.16	23.22	9.50
I(R)%	89.7	343.7	122.8	147.5	42.7	271.3	83.7	97.1	85.7

(a) Data not included in calculation of consensus averages or precision indices.

The reproducibility limit^(a) is the reproducibility standard deviation (S_R) multiplied by $\sqrt{2}$, (since the reproducibility statistic is to be used to judge a difference between the test results for two laboratories^(b)) and by 2 (since an approximate 95 percent confidence level is desired). The interpretation of $I(R)$ is that "there is reason to question one or both of the test results" when "the difference in absolute value of two test results obtained in different laboratories on the same material....is larger than $I(R)$."⁽³⁾ Therefore, $I(R)$ defines an "interval of agreement" delimited by 0 and $I(R)$. That is, any absolute difference in the test results obtained at two laboratories which is less than $I(R)$ is within the variability expected for the method. If the absolute value of the difference is greater than $I(R)$ the results should be questioned. For example, the silicon-NBS-SiW combination has an $I(R)$ of 22.94 g/m². If, at some future date, one laboratory got 25.57 g/m² and another got 3.05 g/m² for silicon from NBS Glass in SiW, the difference would be 22.52, and the conclusion would be that the results are in agreement within the precision expected of the method. The relative reproducibility limit $I(R)\%$ is a convenient statistic for "grading" the MCC-1 procedure on its precision for the various materials. It is not intended to be used in constructing "confidence limits," since it is too easy to forget that $I(R)$ pertains to a difference once it is made relative to a number and multiplied by 100. $I(R)\%$ assumes a difference between two test results as large as the consensus average. If the relative limit is greater than 100% then results below the detection limit would be in agreement with results at the consensus average. At the other extreme, results at the consensus average and twice the consensus average would not be outside the variability expected given the precision of the method. Note that in Table 7.1 for silicon, only basalt in DIW (5.BAS.DI) has a relative reproducibility limit less than 83%.

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- (a) ASTM E691-79 calls this the "reproducibility interval," a definition which might lead to some confusion since one number cannot define an interval. The interval of interest is the interval on the real line defined by the test results obtained by two laboratories on the same material.
- (b) The variance of a difference is the sum of the variances of the terms in the difference if their covariance is zero.

The wide reproducibility limits shown here result in part from the inclusion of all results reported, regardless of their relationship to the other laboratories' data, the consensus average, or the results obtained by an "accepted reference laboratory" such as the MCC. In practice, if the experienced chemist were evaluating this set of data from 20 laboratories, he would be justified in questioning those data which stand conspicuously apart from the bulk of the results (by inspection of a plot such as Figures 3.1-3.9, for example), and would exclude the "fliers" in his initial calculations of the statistics for the procedure. However, if the chemist was given the data from just two or three of the same laboratories "blind" (e.g., Labs F, X and O), he would have no basis for judging which data to accept, in the absence of any reference analyses or consensus average.

Silicon was the only element that was specified for analysis in all waste form-leachant combinations. The same format used in Table 7.1 is used for presenting the results for the other elements, but more than one element is summarized in each table. The best relative reproducibility limit for the other elements in Tables 7.2 through 7.4 is 86.4 percent for boron from NBS Glass in SiW.

TABLE 7.2. Summary for Boron and Calcium

Lab	Mean Concentration from Waste Form • Leachant Combination								
	Boron			Calcium			Boron		
	N.SI	N.DI	N.BR	B.SI	B.D	B.B	P.SI	P.DI	P.BR
A	---	---	---	0.2702	2.1216	11.148	---	---	---
B	57.912	4.113	13.819	---	---	---	37.590	29.675	29.078
C	84.113	27.092	23.932	---	---	---	---	---	---
D	69.842	16.688	17.280	---	---	---	22.971	26.971	24.620
F	69.930	11.949	19.873	0.0000	0.0900	6.036	58.956	63.496	14.832
G	---	---	---	---	---	---	---	30.083	---
H	86.335	28.011	17.454	---	---	---	32.695	28.689	15.770
I	74.069	14.510	14.713	0.7373	1.1794	29.222	24.440	30.684	15.203
J	64.244	53.182	11.918	---	---	---	39.876	48.818	31.698
K	15.195	21.409	19.158	1.1423	5.7190	50.531	25.910	28.303	17.960
L	73.492	19.958	15.154	0.7752	3.8119	20.180	---	---	---
M	37.786	18.672	18.738	1.5405	4.9594	48.905	---	---	---
O	42.228	1.317	9.279	---	---	---	---	---	---
P	60.088	13.668	19.740	0.6815	1.8720	29.302	29.251	40.801	28.561
Q	69.471	16.834	19.386	---	---	---	---	---	---
R	61.544	12.701	13.407	---	---	---	---	---	---
S	61.112	10.574	17.264	0.9421	1.6348	36.049	21.356	26.282	13.607
T	65.660	19.481	42.138	0.2702	2.7927	46.126	35.513	42.176	37.414
U	---	---	---	---	---	---	28.106	34.807	25.996
X	67.764	15.612	33.375	---	---	---	---	---	---
EL	61.644	17.805	19.213	0.7066	2.6868	30.833	32.424	36.048	23.158

Late
Labs(a)

N	27.648	23.000	11.358	---	---	---	14.481	15.860	17.642
V	---	---	---	---	---	---	22.130	36.274	8.983
Y	---	---	---	0.385	1.747	4.774	25.515	26.672	21.173
Z	69.504	15.024	15.055	---	---	---	37.701	35.291	18.435

Index

S _r	9.54	2.89	5.57	0.097	1.04	5.02	2.27	3.02	4.95
S _L	16.24	11.38	7.27	0.475	1.73	15.93	10.61	11.16	7.58
S _r %	15.5	16.2	29.0	13.7	38.6	16.3	7.0	8.4	21.4
S _L	26.3	63.9	37.9	67.2	64.3	51.7	32.7	31.0	32.7
S _R	18.83	11.74	9.16	0.484	2.01	16.70	10.85	11.57	9.05
S _R %	30.6	66.0	47.6	68.6	75.0	54.2	33.5	32.1	39.1

Reproducibility Limit

I(R)	53.26	33.22	25.90	1.370	5.70	47.23	30.69	32.71	25.60
I(R)%	86.4	186.5	134.8	193.9	212.1	153.2	94.6	90.8	110.5

(a) Data not included in calculation of consensus averages or precision indices.

TABLE 7.3. Summary for Potassium, Iron and Cesium

Lab	Mean Concentration from Waste Form • Leachant Combination								
	Potassium			Iron			Cesium		
	N.SI	N.DI	N.BR	B.S	B.D	B.B	P.S	P.D	P.B
A	---	---	---	0.59756	0.59756	0.6300	31.326	32.621	26.343
B	32.374	3.479	---	---	---	---	---	---	---
C	55.533	19.934	---	---	---	---	---	---	---
D	48.854	10.885	---	---	---	---	24.827	29.392	---
F	44.524	9.494	---	0.00000	0.00000	0.0000	54.368	39.484	61.405
G	---	---	---	---	---	---	---	28.353	---
H	53.222	19.115	---	---	---	---	1.752	21.072	0.000
I	66.909	6.788	---	0.20819	-0.02394	0.1340	0.000	0.000	0.000
J	49.912	36.274	---	---	---	---	11.510	5.426	12.445
K	13.649	16.223	---	-0.08072	1.21084	0.5052	24.895	28.509	48.666
L	51.010	13.431	---	0.02684	0.65468	2.5198	---	---	---
M	14.126	3.363	---	0.04186	0.37967	3.0142	---	---	---
O	45.214	7.558	---	---	---	---	---	---	---
P	47.288	10.870	---	0.01483	0.07601	0.9764	29.509	41.103	19.734
Q	56.226	12.882	---	---	---	---	---	---	---
R	49.200	9.023	---	---	---	---	---	---	---
S	46.818	8.213	---	0.02519	0.02519	0.4877	21.144	27.283	9.127
T	52.042	7.467	---	0.02519	0.13821	0.7317	34.446	37.540	28.155
U	---	---	---	---	---	---	27.891	32.995	20.034
X	44.183	9.515	---	---	---	---	---	---	---
EL	44.976	11.868	---	0.10619	0.33980	0.9999	23.788	27.749	22.591

Late
Labs(a)

N	41.41	25.38	---	---	---	---	---	---	---
V	---	---	---	---	---	---	24.76	39.79	---
Y	---	---	---	0.06	0.19	0.17	0.0	0.0	42.07
Z	---	---	---	---	---	---	---	---	---

Index

S _r	5.10	2.65	b	d1	d1	0.38	1.19	1.46	1.46
S _L	13.57	7.74				1.03	15.37	11.91	19.85
S _r %	11.3	22.3				38.0	5.0	5.3	6.5
S _L %	30.2	65.3				103.8	64.6	42.9	87.9
S _R	14.50	8.19				1.10	15.41	12.00	19.90
S _R %	32.2	69.0				109.6	64.8	43.2	88.1

Reproducibility Limit

I(R)	41.00	23.15				3.10	43.60	33.93	56.28
I(R)%	91.2	195.1				309.9	183.3	122.3	249.2

(a) Data not included in calculation of consensus averages or precision indices.

TABLE 7.4. Summary for Sodium and Strontium

Lab	Mean Concentration from Waste Form • Leachant Combination								
	Sodium						Strontium		
	N.S	N.D	N.B	B.S	B.D	B.B	P.S	P.D	P.B
A	---	---	---	-17.037	4.925	---	1.4607	2.2450	28.627
B	274.19	11.060	---	---	---	---	0.4113	6.6257	19.082
C	-18.35	24.210	---	---	---	---	---	---	---
D	-0.89	14.057	---	---	---	---	0.4543	2.1405	22.043
F	40.54	14.414	---	-44.443	12.111	---	0.0000	0.0000	2.937
G	---	---	---	---	---	---	---	2.9278	---
H	794.87	8.820	---	---	---	---	1.4229	3.1064	15.078
I	16.90	12.697	---	-31.614	10.625	---	0.7681	1.4299	17.764
J	-95.41	43.499	---	---	---	---	0.0000	0.0000	3.943
K	27.33	24.484	---	-34.112	18.445	---	1.5160	3.4886	16.983
L	45.04	15.762	---	-35.202	14.448	---	---	---	---
M	-14.18	14.279	---	-29.074	15.111	---	---	---	---
O	94.09	8.089	---	---	---	---	---	---	---
P	-43.43	11.633	---	-65.996	9.8501	---	0.6469	0.8039	29.401
Q	0.00	13.517	---	---	---	---	---	---	---
R	64.39	9.845	---	---	---	---	---	---	---
S	19.81	9.060	---	-17.589	10.794	---	1.0685	1.8815	17.649
T	75.45	-20.268	---	-74.166	-7.685	---	0.5077	1.2843	8.823
U	---	---	---	---	---	---	1.5310	2.0365	15.874
X	25.38	11.644	---	---	---	---	---	---	---
EL	63.72	13.430	---	-38.804	9.860	---	0.8156	1.8547	16.517
Late Labs(a)									
N	5.097	32.736	---	---	---	---	0.100	1.090	43.318
V	---	---	---	---	---	---	1.172	1.029	10.199
Y	---	---	---	-1.851	37.037	---	1.127	2.843	26.764
Z	14.484	2.252	---	---	---	---	0.940	1.673	10.149
Index									
S _r	d1	4.50	b	d1	0.97	d1	0.157	0.30	3.17
S _L		12.14			7.58		0.565	1.11	8.09
S _r %		33.5			9.9		19.2	16.2	19.2
S _L %		90.4			76.8		69.3	59.7	49.0
S _R		12.95			7.64		0.587	1.15	8.69
S _R %		96.4			77.5		71.9	61.8	52.6
Reproducibility Limit									
I(R)		36.63			21.60		1.659	3.24	24.58
I(R)%		272.7			219.1		203.4	174.9	148.8

(a) Data not included in calculation of consensus averages or precision indices.

7.2 STANDARDIZED BIAS

The standardized bias is an indicator of the magnitude of the bias at laboratory i for each "material" (element-waste form-leachant combination j). It is defined as

$$e_{ij} = \frac{\overline{EL}_{ij} - \overline{EL}_j}{s_{rj} \sqrt{\frac{1}{3} \frac{p-1}{p}}}$$
$$= \frac{\text{Lab } i\text{'s Mean} - \text{Consensus Average.}}{\text{sd(Lab Mean)}}$$

The denominator of e_{ij} is the pooled standard deviation of the within-laboratory replicates adjusted for the number of replicates (usually 3) and the number of laboratories (p) for the "material" combination. The standardized bias is thus in within-laboratory standard deviation units, denoted by S_r^1 .

In Figures 7.1 through 7.8, (one figure for each element statistically analyzed for the MCC-1 RR), there is a panel for each waste form-leachant combination with each laboratory's letter code plotted above the standardized bias scale. A vertical line separates the plot into positive and negative relative bias zones. For example, in Figure 7.1 for silicon from NBS Glass in SiW, Lab K has a negative bias which is about 7.5 times the pooled within-laboratory standard deviation of the laboratory average, while Lab F has a positive bias of about the same magnitude. For NBS Glass in DIW, it appears that Labs J, F and C pull the consensus average to the right.

The plots of Figures 7.1 through 7.8 can be used to make judgments about which laboratories should look for causes of relative bias. In the absence of relative bias, it would be expected that values for e_{ij} would follow a normal distribution with a mean of zero and a standard deviation of unity. This is obviously not the case, since most of the e_{ij} are outside the interval $(-2, +2)$, the approximate interval within which 95 percent of the e_{ij} should fall if there were no relative bias.

FIGURE 7.1. Standardized Bias (e_{ij}) for Silicon. (Letters plotted are laboratory identification code.)

(a)

(b)

FIGURE 7.2. Standardized Bias (e_{ij}) for Boron. (Letters plotted are laboratory identification code.)

FIGURE 7.3. Standardized Bias (e_{ij}) for Potassium. (Letters plotted are laboratory identification code.)

FIGURE 7.4. Standardized Bias (e_{ij}) for Sodium. (Letters plotted are laboratory identification code.)

FIGURE 7.5. Standardized Bias (e_{ij}) for Calcium. (Letters plotted are laboratory identification code.)

FIGURE 7.6. Standardized Bias (e_{ij}) for Iron. (Letters plotted are laboratory identification code.)

FIGURE 7.7. Standardized Bias (e_{ij}) for Cesium, (Letters plotted are laboratory identification code)

FIGURE 7.8. Standardized Bias (e_{ij}) for Strontium, (Letters plotted are laboratory identification code)

8.0 MAJOR RESULTS

The subsections to follow contain

- overall summary statements of the precision and how they were arrived at
- the allocation of total variability into procedural and analytical components as well as within and between lab components
- other major results.

8.1 PRECISION STATEMENTS

Statements of the precision of the measurement method are the fundamental goal of an interlaboratory test. Tables 7.1 through 7.4 provide a set of 29 element-waste form-leachant summaries. The essential information for characterizing the precision of the data from this RR for the MCC-1 28-day static leach test is contained in the $\overline{EL}$, S_r and S_L . These statistics were summarized in Table 2.4, where the relative standard deviations are listed.

The actual standard deviations in g/m^2 were used in straight-line fits (linear regression) to the equation

$$sd = a + b \overline{EL} \quad (8.1)$$

in order to "smooth" the summary statistics. In the above equation sd stands for a set of standard deviations, and $\overline{EL}$ for the corresponding set of element averages. The $\overline{EL}$, S_r and S_L are given in Table 8.1 for all 29 combinations, in the rank order of the element average.

TABLE 8.1. Statistics Used in Linear Fits of $sd = a + b \overline{EL}$

Element Conc'n Rank	Waste Form, Leachant Tested	Element Analyzed	Consensus Average, $\overline{EL}$	Standard Deviation, s.d.		s.d. Group			
				Within Lab (S_r)	Between Lab (S_L)	S_r		S_L	
						Low	Hi	Low	Hi
1	BAS, SiW	Ca	0.71	0.097	0.48	x		x	
2	MCC, SiW	Sr	0.82	0.157	0.57	x		x	
3	BAS, BR	Fe	1.00	0.380	1.03	x		x	
4	MCC, DIW	Sr	1.85	0.300	1.11	x		x	
5	BAS, BR	Si	2.15	0.580	0.76	x		x	
6	BAS, DIW	Ca	2.69	1.040	1.73		x	x	
7	BAS, SiW	Si	2.72	0.330	1.38	x		x	
8	NBS, DIW	Si	4.85	0.850	5.83	x			x
9	BAS, DIW	Si	5.59	0.390	0.75	x		x	
10	NBS, BR	Si	8.09	2.350	2.61		x	x	
11	BAS, DIW	Na	9.86	0.970	7.58	x			x
12	MCC, BR	Si	11.09	0.740	3.28	x		x	
13	NBS, DIW	K	11.87	2.650	7.74		x		x
14	NBS, DIW	Na	13.43	4.500	12.14		x		x
15	MCC, BR	Sr	16.52	3.170	8.09		x		x
16	NBS, DIW	B	17.81	2.890	11.38		x		x
17	MCC, SiW	Si	18.11	1.010	5.27	x		x	
18	NBS, BR	B	19.21	5.570	7.27		x	x	
19	MCC, BR	Cs	22.59	1.460	19.85	x			x
20	MCC, BR	B	23.16	4.950	7.58		x	x	
21	MCC, SiW	Cs	23.79	1.190	15.37	x			x
22	MCC, DIW	Si	23.91	1.720	8.03	x		x	
23	NBS, SiW	Si	25.57	3.960	7.08		x	x	
24	MCC, DIW	Cs	27.75	1.460	11.91	x			x
25	BAS, BR	Ca	30.83	5.020	15.93		x		x
26	MCC, SiW	B	32.42	2.270	10.61	x		x	
27	MCC, DIW	B	36.05	3.020	11.16	x		x	
28	NBS, SiW	K	44.98	5.100	13.57		x	x	
29	NBS, SiW	B	61.64	9.540	16.24		x	x	

The coefficients a and b are least squares estimates, obtained by minimizing the sum of squares of residuals:

$$\sum_{j=1}^n \epsilon_j^2 = \sum_{j=1}^n (sd_j - \hat{sd}_j)^2 \quad (8.2)$$

where

$$\hat{sd}_j = a + b \overline{EL}_j. \quad (8.3)$$

Then
$$\frac{\hat{sd}}{\overline{EL}} = \frac{a}{\overline{EL}} + b,$$

and if $a = 0$,
$$\frac{\hat{sd}}{\overline{EL}} = b.$$

In our application the intercept, a, is the smoothed value for the sd when the element average is extrapolated down to zero. The coefficient, b, is the slope of the line, which is the best estimate of the consensus relative standard deviation (coefficient of variation) over the range where the intercept no longer has a significant effect.

The data from Table 8.1 used in the linear fits are plotted in Figures 8.1 and 8.2, as scatterplots of S_r and S_L versus element averages $\overline{EL}$. In both those figures the overall trend is of increasing S_r or S_L with increasing $\overline{EL}$. In both cases two lines are required to describe the structures, which comprise a high sd group and a low sd group. Group membership is indicated by "x" in the "sd Group" columns in Table 8.1 for S_r and S_L . Table 8.2 gives the statistics for the linear fits used to describe the four groups of sds, compared to the fits to all the S_r or S_L . The four groups are plotted by respective symbols in Figure 8.3, using the same points as in Figures 8.1 and 8.2.

FIGURE 8.1. Within-Laboratory Standard Deviations for all Elements.
(Letters plotted are element codes.)

FIGURE 8.2. Between-Laboratory Standard Deviations for all Elements.
(Letters plotted are element codes.)

TABLE 8.2. Comparison of Fits to $sd = a + b \overline{EL}$

	sd	N	a	b	Fit ($R^2\%$)	Plot Symbol (Fig. 8.3)
Within Lab	S_r	29	0.2928	0.1182	62.6	
Low	S_r	17	0.1873	0.0610	88.1	A
High	S_r	12	1.5577	0.1164	78.9	B
Between Labs	S_L	29	2.294	0.2991	62.2	
Low	S_L	19	0.5640	0.2792	97.8	C
High	S_L	10	4.207	0.4115	58.5	D

FIGURE 8.3. Straight-Line Fits to the Four sd Groups

The fits for all the material combinations ($N = 29$) account for only about two-thirds of the total variability for S_r and S_L , as indicated by the $R^2\%$. When the standard deviations are divided into high and low groups, the $R^2\%$ shows significant improvement, except for the high S_L group. The lack of improvement for the high S_L group is due to the "D" point at $S_L = 20$ on Figure 8.3 (cesium-MCC-Brine; see Figure 8.2 and Table 8.1). Figure 7.7 shows that there is a broad spread of relative laboratory biases for cesium mass loss, and therefore no basis for deletion of any specific "outlying" laboratory data point. The obvious conclusion is that much better control-of-measurement methods for cesium in brine is required if cesium is to be used to compare results from two different laboratories, even if within-laboratory variability is acceptable. In fact, this same comment applies to the 20 element-waste form-leachant combinations with relative reproducibility limits, $[I(R)\%]$, greater than 100 in Tables 7.1 through 7.4.^(a)

A summary of this discussion of the precision achieved in this RR of MCC-1 is shown in Figure 8.4. Here the values of the slopes of the linear fits (Figure 8.3), multiplied by 100, appear as dashed horizontal lines providing a "floor" to the smoothed relative precisions located above these lines. The sharp increase in relative sd as element concentration decreases is frequently observed as measured values approach the analytical detection limits. But note that the curves in Figure 8.4 "take off" between 20 g/m^2 and 5 g/m^2 , depending on the sd group, whereas detection limits for this RR were usually equivalent to less than 0.1 g/m^2 . In these terms, the "detection limit" demonstrated by the MCC-1 RR was about 5 g/m^2 for the easier-to-test elements and materials, but as high as 20 g/m^2 for the harder-to-test combinations.

Based on the RR data and the statistics discussed above, the following summary precision statements can be made:

- These precision statements apply only to elemental mass losses greater than 5 g/m^2 . Precision decreases rapidly at lower concentrations.

(a) See Appendix E for another example of the interrelationships of the statistics in these figures and tables.

FIGURE 8.4. Smoothed Relative Precision of MCC-1

- Based on the set of element-waste form-leachant combinations with the lower imprecision, the relative precision for the MCC-1 procedure could be as good as 6 to 11 percent for within-laboratory data and 25 to 40 percent for between-laboratory component.
- Based on the combinations with the higher imprecision, the relative precision of the MCC-1 procedure could be as bad as 11 to 25 percent for within-laboratory data, and 40 to 75 percent for between-laboratory components, with elemental mass loss greater than 10 g/m² required.

The practical conclusions from these statements are as follows:

1. Elemental mass per unit area (g/m^2) losses must be at least 5 g/m^2 for any meaningful comparisons of results within a laboratory or between laboratories.
2. A relative between-laboratory sd of 35 percent means that, at a 95 percent confidence level, a test value at the consensus average for one laboratory would not be judged different from a detection level value obtained by another laboratory. The relative standard deviation between laboratories, $S_L\%$, was greater than 35 for 18 of the 29 element-waste form-leachant combinations studied.
3. Comparison of waste forms by tests within a single laboratory appears advisable, to avoid the greater relative biases between laboratories.
4. These RR data raise questions about how a "standard waste form" could be used for verification of test laboratory performance.

8.2 ALLOCATION OF TOTAL VARIABILITY

The ARR, conducted in conjunction with the MCC-1 RR, provided the data for allocating the total variability of the MCC-1 procedure into analytical and procedural components as well as within- and between-laboratory components. Due to considerations of comparability of data, only boron and silicon (two major matrix elements), and cesium and strontium (two waste element stand-ins), were included in the allocation calculations.

The ARR data were subjected to the same kind of analysis as the MCC-1 data. Fits to Equation 8.1 were made to each of the four groups of data:

	<u>ARR</u>	<u>MCC-1</u>
Within	$\text{sd}(W), \overline{EL}$	$(S_r), \overline{EL}$
Between	$\text{sd}(L), \overline{EL}$	$(S_L), \overline{EL}$

The resulting equations were used to calculate smoothed estimates of the standard deviations at $\overline{EL} = 20$. The resulting standard deviations were then

squared to give mean square (variance) estimates. The total variance of concern, MST, is estimated by

$$MST = (S_r)^2 + (S_L)^2.$$

Since

$$(S_r)^2 = [sd(PW)]^2 + [sd(W)]^2$$

$$[sd(PW)]^2 = (S_r)^2 - [sd(W)]^2.$$

Similarly,

$$[sd(PB)]^2 = (S_L)^2 - [sd(L)]^2$$

where the terms are defined by the table on Page 8.9 (unnumbered), and the procedural components are designated by

$$[sd(PW)]^2 \text{ for within laboratories}$$

and

$$[sd(PB)]^2 \text{ for between laboratories.}$$

These constructed variances were then divided by MST and multiplied by 100 to give the percentages in Table 8.3. The fits used and the allocation calculations are given in Table 8.4. Figure 8.5 shows the resulting smoothed characterization. The difference between the A and C lines is the within-laboratory procedural contribution, and that between B and D lines is the between-laboratory procedural contribution.

TABLE 8.3. Percentage Allocation of Total Variability
at $\bar{E}L = 20 \text{ g/m}^2$

Source	Analytical	Procedural	A & P
Within-Laboratory	1	4	5
Between-Laboratory	<u>29</u>	<u>66</u>	<u>95</u>
Within and Between	30	70	100

TABLE 8.4. Construction and Allocation of Total Variability

Fit	Data Sets (B, Cs, Sr, Si)			
	<u>sd(W)</u>	<u>sd(L)</u>	<u>(S_r) Low</u>	<u>(S_L) Low</u>
N	10	11	14	15
a	0.1656	1.1696	0.1667	0.5560
b	0.02142	0.1100	0.06151	0.2772
R ² %	71.9	56.2	87.2	97.1
Plot Letter	A	B	C	D
sd(20)	0.5940	3.3696	1.3959	6.0900
MS(20)	0.3528	11.3542	1.9513	37.0881
Total Reproducibility MS			39.0394	
Procedural MS			1.5985	25.7329

Note that the between-laboratory analytical imprecision, $sd(L)$ in Figure 8.5, is greater than the between-laboratory total, (S_L) , for element averages of about four or less. This may be due only to the imprecision of the fits, or it may be an artifact of high $sd(L)$ points which raised the intercept. Only the low (S_L) group from MCC-1 (Table 8.1, Figure 8.3) was used, to maximize the impact of the analytical components of the allocation.

FIGURE 8.5. Fits Used in Allocation of Total Variability

It is obvious that within-laboratory variability makes a minor contribution to the total variability of MCC-1. Practically, this means that the statistical method of using replications of specimens, which impacts procedural imprecision, will not be effective here. Replication affects only within-laboratory variability, a relatively small component of the overall imprecision.

The fact that the between-laboratory analytical component accounts for 29 percent of the total variability shows that the analytical methods used had significant relative biases. Figure 8.6 shows how the allocation varies over the range of element averages in the study. The within-laboratory components account for about 5 percent over the entire range. The relative biases in analytical methods account for a minimum of 20 percent of the total variability and substantially more for element averages below 20 g/m².

FIGURE 8.6. Composition of Smoothed Total Variability.

This analysis of the total variability also provides a "typical best case" characterization of the overall relative precision demonstrated by MCC-1 RR, since an elemental mass loss of 20 g/m^2 is used, which is near the overall average elemental mass loss obtained in MCC-1. Also, only the low S_r and S_L MCC-1 groups were used.

In Table 8.5, the overall relative precision of 31.2 percent implies that an $\overline{EL}$ of 20 g/m^2 would have a standard deviation of 6.24 g/m^2 . This means that results on the same material from two laboratories could be different by as much as

$$2\sqrt{2} (6.24) = 17.6 \text{ g/m}^2$$

and still be judged statistically the same with approximately 95 percent confidence.

TABLE 8.5. Relative Precisions at $\overline{EL} = 20 \text{ g/m}^2$, Percent

<u>Source</u>	<u>Analytical</u>	<u>Procedural</u>	<u>A & P</u>
Within-Laboratory	3.0	6.3	7.0
Between-Laboratory	16.8	25.4	30.4
Within and Between	17.1	26.1	31.2

8.3 OTHER MAJOR RESULTS

8.3.1 Ranking of "Materials"

The standardized biases plotted in Figures 7.1 to 7.8 provide a basis for overall rankings of the element-waste form-leachant combinations on their susceptibility to bias. The standard deviation of these biases for the laboratories that measured an element in a waste form-leachant combination are indicators of the spread of the biases over the laboratories for the combination. The greater the standard deviation the greater the relative differences among laboratories indicating that the element-leachant-waste form combination was more difficult to control for bias.

Table 8.6 ranks the 29 "materials" (element-waste form-leachant combinations) based on the magnitude of the $sd(e_{ij})$. This table confirms that, while the other elements are spread throughout the rankings, cesium is the most bias-prone element. But cesium also has the best within-laboratory relative precision, with $S_r\%$ of 6.5% or smaller. Both the average g/m^2 cesium and the pooled within-laboratory standard deviations are near the middle of the reported values, so the high standard deviation of the standardized bias is not an artifact of unusually low within-laboratory standard deviations (see Figure 8.1). Since the " 2σ limits" on the between laboratory bias for cesium are 30 to 50 times the within-laboratory precision, cesium analyses do not appear to be useful for comparing results from two different laboratories.

TABLE 8.6. Ranking of 29 "Materials" for Bias and Comparison of Within Laboratory Variability

Rank $sd(e_{ij})$	Element	Waste Form	Leachant	$sd(e_{ij})$ Value	N Labs	Rank $S_r\%$	$S_r\%$	$\overline{EL}$	S_r
1	Si	BAS	Br	2.01	9	24	27.1	2.15	0.58
2	Si	NBS	Br	2.23	17	26	29.1	8.09	2.35
3	B	NBS	Br	2.55	17	25	29.0	19.21	5.57
4	B	MCC	Br	2.97	11	22	21.4	23.16	4.95
5	B	NBS	SiW	3.24	17	14	15.5	61.64	9.54
6	Ca	BAS	DIW	3.24	9	29	38.6	2.69	1.04
7	Si	NBS	SiW	3.29	17	15	15.5	25.57	3.96
8	Si	BAS	DIW	3.64	9	6	7.0	5.59	0.39
9	Sr	MCC	Br	4.74	12	20	19.2	16.52	3.17
10	Na	NBS	DIW	4.79	17	27	33.5	13.43	4.5
11	K	NBS	SiW	4.80	17	11	11.3	44.98	5.1
12	Fe	BAS	Br	5.08	9	28	38.0	1.00	0.38
13	K	NBS	DIW	5.21	17	23	22.3	11.87	2.65
14	Ca	BAS	Br	5.93	9	18	16.3	30.83	5.02
15	Sr	MCC	DIW	6.56	13	16	16.2	1.85	0.30
16	Sr	MCC	SiW	6.58	12	21	19.2	0.82	0.16
17	B	MCC	DIW	6.70	12	9	8.4	36.05	3.02
18	B	NBS	DIW	7.03	17	17	16.2	17.81	2.89
19	Si	BAS	SiW	7.84	9	12	12.0	2.72	0.33
20	Si	MCC	Br	8.08	12	5	6.7	11.09	0.74
21	Si	MCC	DIW	8.40	13	8	7.2	23.91	1.72
22	B	MCC	SiW	8.54	11	7	7.0	32.42	2.27
23	Ca	BAS	SiW	9.09	9	13	13.7	0.71	0.097
24	Si	MCC	SiW	9.49	12	3	5.6	18.11	1.01
25	Si	NBS	DIW	12.01	17	19	17.6	4.85	0.85
26	Na	BAS	DIW	14.36	9	10	9.9	9.86	0.97
27	Cs	MCC	DIW	15.46	12	2	5.3	27.75	1.46
28	Cs	MCC	Br	24.77	10	4	6.5	22.59	1.46
29	Cs	MCC	SiW	25.68	11	1	5.0	23.77	1.19

The least bias prone "material" is silicon from basalt in brine. For this combination, "2 σ limits" on the potential bias are only four times the within-laboratory precision. The next best "material" is silicon from NBS glass in brine; silicon for MCC in brine is down the list at rank 20.

Figure 8.7 is a plot of the $sd(e_{ij})$ for all "materials" except the off-scale cesium-from-MCC points. Note that there is no consistent pattern for leachants from waste form to waste form. Silicon (S) had the greatest range of laboratory biases for DIW when NBS glass was the waste form. But, for basalt the SiW produced the greatest biases, and for MCC 76-68 glass, the silicon biases were all high. Boron (B) followed silicon for NBS glass, but with not so dramatic a peak at DIW. But for MCC glass, boron bias declined from SiW to DIW to brine. Sodium (N) for NBS glass had a relatively low range of bias; but, for basalt, the bias was almost as bad as for cesium from MCC glass. Such diverse behavior, and the fact that different laboratories are included in different combinations (see the N laboratories column of Table 8.6), makes rankings of elements, waste forms or leachants difficult to interpret adequately. The most meaningful summary of the bias problem is given in Figure 8.7 and Table 8.6. Pooled results are given in Table 8.7.

Another summary of interest is given in Table 8.8 although, again, these results cannot reflect the true complexity of the situation. This two-way table has leachants for rows and waste forms for columns. The marginals include the $sd(e_{ij})$ upon which the marginal rankings are based, both with and without cesium. NBS glass in brine has rank one, but only silicon and boron results were used for this combination. MCC glass in silicon water ranks last when cesium is included and seventh when the cesium data are removed. Without the cesium data, basalt in deionized water ranks last, due to the high biases for sodium at this combination. When cesium is included, MCC glass ranks last among the waste forms, and silicon water among the leachants. Without cesium, basalt produces the greatest bias of the waste forms, and deionized water of the leachants.

This tabulation may be indicative of the effects of waste form homogeneity and of complexity, on the MCC-1 results. NBS glass is a relatively simple glass and probably the most homogeneous. MCC glass is the most complex composition, and neither it nor basalt has been tested in detail for homogeneity.

FIGURE 8.7. Standard Deviations of Standardized Biases. (Elements plotted by letter symbol; cesium is not included.)

TABLE 8.7. Rankings of Elements over Waste Forms,
Leachants and Laboratories

Rank $sd(e_{ij})$	Element	$sd(e_{ij})$	N	$sd(e_{ij})$	
				Low	High
1	Potassium	4.93	34	3.24	9.09
2	Iron	5.08	9	---5.08---	
3	Boron	5.32	85	2.55	8.54
4	Strontium	5.87	37	4.74	6.58
5	Calcium	6.28	27	3.24	9.09
6	Silicon	7.10	115	2.01	12.01
7	Sodium	8.98	26	4.79	14.36
8	Cesium	21.47	33	15.46	24.77

TABLE 8.8. Rankings of Waste Forms and Leachants
over Elements and Laboratories

	Waste Form			$sd(e_{ij})$		Rank	
	NBS	BAS	MCC	With Cs	W.O. Cs	With Cs	W.O. Cs
<u>Leachant</u>							
SiW	2 (2) ^(a)	5 (8)	9 (7)	9.67	6.28	3	2
DIW	4 (6)	6 (9)	7 (5)	8.43	7.59	2	3
Brine	1 (1)	3 (3)	8 (4)	8.33	4.31	1	1
<u>With Cs</u>							
$sd(e_{ij})$	5.62	7.03	11.9	8.79	6.36		
Rank	1	2	3				
<u>W.O. Cs</u>							
$sd(e_{ij})$	5.62	7.03	6.91				
Rank	1	3	2				

(a) Parenthesized rankings are for data with cesium data omitted.

8.3.2 Ranking of Laboratories

Since not all laboratories ran all waste forms, nor all specified elements, the rankings by laboratory suffer from being based on the different combinations which each individual laboratory did run. For example, if a laboratory did not try to measure cesium it had no opportunity to be charged with a bias for cesium. Since cesium has by far the greatest bias, rankings are provided both with and without the cesium data in Table 8.9. Cesium was reported for MCC glass only, and what appears as a test bias could be due to cesium heterogeneity in the MCC glass.

Table 8.9 gives a ranking for silicon, since this element was analyzed by all laboratories and had precision statistics representative of all other elements. The rankings in Table 8.9 were based on the average of the absolute values of the standardized biases for each laboratory. The number of standardized biases, e_{ij} , used is given under the column, "No. of Materials." If the average were based on the actual, rather than the absolute, values of the e_{ij} the positive and negative biases would tend to cancel out, and we would not have a good indicator of the bias problem at the laboratory.

The three columns of rankings in Table 8.9 do not agree very well. For example, Lab A ranks sixth based on the silicon data only but drops to fourteenth in the ranking based on all elements except cesium. At the other extreme, Lab D gained seven rank places when all but cesium data were used instead of just silicon. For all three rankings, Labs Q and X have the first two positions, and Labs F and J are last. Since cesium results were generally questionable, the most meaningful of the three rankings is that for the standardized biases without cesium. As the "Change Due to Cesium" column shows, removing cesium affected only three laboratories significantly. The last two columns give the averages of the absolute standardized biases upon which the last two rankings were based.

TABLE 8.9. Rankings of Laboratories

Lab	No. of Materials	Rankings of Ave $ e_{ij} $			Change ^(a) Due to Cesium	Ave $ e_{ij} $	
		Si Only	All Elements	Without Cs		With Cs	Without Cs
A	17	6	13	14	-1	5.1	4.5
B	18	9	10	10	0	(b)	3.6
C	19	17	12	15	-3	*	4.5
D	20	15	6	8	-2	3.0 ^(c)	3.1
F	29	19	19	19	0	10.8	7.7
G	4	5	7	13	-6	3.4	4.3
H	21	11	18	11	+7	6.6	3.7
I	29	8	16	6	+10	6.0	2.8
J	21	20	20	20	0	11.2	9.7
K	29	14	15	16	-1	5.9	5.2
L	17	3	4	5	-1	*	2.6
M	17	12	14	17	-3	*	5.4
O	9	7	9	9	0	*	3.5
P	29	4	5	3	+2	3.0	2.2
Q	9	2	1	1	0	*	1.5
R	9	10	3	4	-1	*	2.3
S	29	13	8	7	+1	3.5	3.1
T	29	16	17	18	-1	6.3	5.7
U	12	18	11	12	-1	4.5	4.2
X	9	1	2	2	0	*	1.7

(a) Improvement (+) or decline (-) in ranking without cesium from ranking with cesium.

(b) Lab B tested MCC glass, but did not analyze for cesium.

(c) Lab D tested MCC glass, but did not analyze for cesium in brine.

* Lab did not test MCC glass.

The last two columns can be used to gauge the overall performance of a laboratory relative to the typical within-laboratory precision for the materials. The best laboratory, Lab Q, (which tested only NBS glass) had an average bias 1.5 times the within-laboratory precision on the average; Lab J had an average bias 9.7 times the typical within-laboratory precision when cesium was excluded, and 11.2 times when cesium was included. The without-cesium rankings can be used by a participating laboratory to see how it performed relative to other laboratories on an overall basis. Table 2.1 lists the waste forms tested by each laboratory.

9.0 REFERENCES

1. Daniel, J. L., D. M. Strachan, J. W. Shade, and M. T. Thomas. 1981. Materials Characterization Center Workshop on Compositional and Microstructural Analysis of Nuclear Waste Materials--Summary Report, PNL-3882, Pacific Northwest Laboratory, Richland, Washington.
2. Materials Characterization Center. 1981. "MCC-1 Static Leach Test Method." In Nuclear Waste Materials Handbook--Waste Form Test Methods, DOE/TIC-11400, Department of Energy, Washington, D.C. (Assigned Distribution.)
3. American Society for Testing and Materials. 1980. "Standard Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods, ANSI/ASTM E 6.91-79. In Annual Book of ASTM Standards, Philadelphia, Pennsylvania.
4. Rao, P.S.R.S., Jack Kaplan, and W. G. Cochran. 1981. "Estimators for the One-Way Random Effects Model with Unequal Error Variances." Journal of the American Statistical Association, 76: 89-97.

10. ACKNOWLEDGMENTS

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APPENDIX A

INSTRUCTIONS TO ROUND ROBIN PARTICIPANTS

November 26, 1980

This letter contains standard forms and instructions for submitting your MCC-1 Round Robin results. Under separate cover you will receive:

- Sample material (NBS glass,^(a) basalt, and/or 76-68) as agreed by telephone
- 18 Teflon containers for each different sample material, plus 6 Teflon containers for blanks
- Teflon monofilament for specimen suspension in the contains.

As agreed at the planning meeting in Germantown, MD, August 28, you, as a Round Robin participant, are to prepare test specimens with a surface area of approximately 400 mm² from the sample materials and leach them in leachants that you prepare. The procedure to be followed is that given on pp. 17-26 in "MC-1 Static Leach Test," August 1980, with the modifications described below.

Test Matrix. You will perform the "Required Test Conditions" given at the bottom of Page 21. For the special purposes of the Round Robin you are also being asked to run 2 additional specimens for 28 days in each leachant. This will result in 6 additional leachates (i.e., 2 silicate water, 2 DI water, and 2 brine) for each sample material you test. These 6 additional leachates are to be shipped to the MCC for analysis.

Blanks. Instead of the single blank of each leachant required at the bottom of Page 21, two blanks of each leachant are required for the Round Robin. You may apply these blanks to more than one material, if the material specimens and the blanks are run concurrently.

Specimen Preparation. The procedure in Section 6.3 requiring that ethylene glycol be used as the coolant during specimen cutting has been changed. Use water as the coolant for preparing the Round Robin specimens.

Archive Specimens. Prepare 3 extra specimens of each sample material you run for the Round Robin. Save these specimens so that they can be examined later, if such examination proves necessary.

Calibrations and Standards. Section 4.7 of the draft procedure has been expanded to require that the methods used to calibrate balances, thermocouples, micrometers, etc. be reported along with the test results. Please use the "6.0 Supplementary Information" pages in the standard form package for this purpose.

(a) The NBS glass is being sent directly to you from Washington, D.C.

For your information, it may become a required part of the MCC-1 procedure that the specimen of a standard material with a certified leach rate will have to be run each time the procedure is used. We have about 75 pounds of the 76-68 glass being supplied for the Round Robin which, depending on the results of the Round Robin, may become an interim standard material until the Independent Measurement Standard Laboratory (IMSL) can supply final standard materials.

You may be curious about how the Materials Review Board (MRB) review of the MCC-1 procedure is coming along. The members of the "Panel on Procedures" on the MRB have examined the August 1980 draft, and the MCC and the members of the panel will hold an all-day meeting in Chicago on October 31 for formal review of the procedure. Preliminary indications are that the mechanics of the test will be approved as they stand in the original draft, except for the changes noted above in specimen cutting fluid and documentation of calibration, and perhaps some other minor changes. If there are any changes resulting from the October 31 meeting that will affect the Round Robin I will let you know immediately.

The MCC is very gratified that you are participating in the MCC-1 Round Robin. It represents an important commitment on your part to assist in defining the precision and reproducibility of the leaching of waste forms. I apologize for the delay in getting the sample materials out to you. Many of you have already told me that this makes the December 31 deadline for return of data impossible to meet. It remains urgent that we complete the Round Robin as soon as possible, however, so please give the Round Robin as much priority as you can. The new deadline for receipt of data is January 31. Any data received after that day will not be included in the first report of Round Robin results.

Please call me if you have any questions at any time. Good luck.

Sincerely,

John Mendel
Staff Scientist

Enclosure

INSTRUCTIONS FOR REPORTING MCC-1 ROUND ROBIN TEST RESULTS

- A. Use only the standard "Report of Test Results" forms provided by the MCC. Fill out with ink. A separate set of forms (16 Pages) is provided for each of the sample materials, NBS glass, basalt and 76-68.
- B. The top and bottom of each page are standard and give the following information:
- "Test Procedure." This space has been filled out for you. The test procedure is the MCC-1 Round Robin. The reference material is also identified in this space.
 - All of the work you do on the first round robin with a given material will have one "Test No.," as follows:

xxx/MCC-1/y

where xxx identifies the laboratory, e.g., LLL, ORNL, etc., MCC-1 is the test procedure and "y" is 1,2 or 3 for NBS glass, basalt and 76-78, respectively.

- The pages are numbered consecutively. The total should include all pages of supplementary information.
 - Each page should be signed and dated when it is filled out.
- C. Mail the completed "Report of Test Results" to:
- J. E. Mendel
Battelle
Richland, WA 99352
- D. Special Instructions for Each Page:

1.0 Cover Page

This page identifies the responsible investigator. Space is provided for comments on the test, and for discussion of problems encountered, deviations from the test procedure, etc.

Table 1.1 Sample Composition

The nominal composition of the sample materials provided by the MCC for the Round Robin is given in this table. Space is provided for chemical analyses of the sample, if performed by the participating laboratory. Such analyses are optional and are not a required part of the round robin.

Table 2.1 Specimen Description

This space gives details of specimen preparation.

Figure 2.1 Sample Dissection to Prepare Test Specimen

Prepare a rough drawing to illustrate how the specimens were obtained from the sample material provided by the MCC.

3.0 Test Conditions

This page describes the test conditions. Except for some differences in the constant temperature equipment, these should be the same for all participants. Use Section 1.2 on the Cover Page to describe any deviations from the test conditions.

Table 4.1 Leachant Analyses

It is not necessary to analyze for all the constituents listed, but at a minimum the analyses must include pH, the major constituents used in preparation of the leachant, and the constituents that will be analyzed in the leachate.

Table 5.1 through 5.6a, Leachate Analyses

The last two columns in these tables contain the data that will be analyzed statistically, i.e., the mean results and the standard deviation. After statistical analysis they will be the major data output from the Round Robin. Tables 5.1a, 5.5a and 5.6a are for the two additional 28-day specimens in each leachant that have been added to the MCC-1 procedure only for the round robin. These tables will be completed by the participants and the MCC jointly, since the participants will not analyze these two leachates; instead they will return these two leachates (for each sample material) to the MCC for analysis. Participants should provide mass loss information and analyses of the blanks. The following ions must be analyzed in the leachates and blanks.

<u>NBS Glass</u>	<u>Basalt</u>	<u>76-68</u>
Si	Si	Si
B	Fe	B
K	Na	Sr
Na	Ca	Cs

6.0 Supplementary Information

These pages are to be used for reporting the results of post-leach specimen analysis. These analyses (SEM, ESCA, etc.) are not required as part of the round robin but are encouraged. These pages can also be used for further comments carried over from Section 1.2 of the Cover Page and should be used to describe methods for calibration of measurement equipment.

APPENDIX B

INSTRUCTIONS TO ANALYTICAL ROUND ROBIN PARTICIPANTS

November 26, 1980

To: MCC-1 Round Robin Participants and Analytical
Methods Workshop Participants

From: John Mendel and Lee Daniel

Subject: Analytical Methods Round Robin

One of the most important conclusions of the recent workshop on "Compositional and Microstructure Analysis" in Seattle, was that there is an urgent need for an analytical methods round robin, either before or concurrent with the MCC-1 leach test round robin. Because the leach test development is a critical step in the MCC program, and the MCC-1 round robin is an important part of that development, you should continue without delay on your MCC-1 tests as previously agreed, using the specimens sent to you earlier for that purpose. However, in addition we are sending under separate cover a set of analytical round robin reference solutions and solids to be used for evaluating MCC-1 test data, and to assist you in checking your own laboratory's success in obtaining accepted concentration values for components of homogeneous reference specimens.

We recommend that you conduct the analyses on these analytical round robin reference specimens immediately upon receipt, concurrent with your MCC-1 leach tests. Then submit your analytical round robin data to us immediately upon completion; we will evaluate and compare laboratory results as quickly as possible, and return the information for your review and possible use during completion of your MCC-1 test data. Prompt turn-around is especially important for the reference solutions analyses. This is because the MCC-1 Leach test round robin depends heavily on good solution analysis, but solids analysis is only recommended, not required, for that round robin.

We consider this analytical methods round robin an important integral part of the leach test methods development; demonstration of analytical capabilities is fundamental to interlaboratory correlation of leach test results in the MCC program. Analyses of these reference specimens by all participating laboratories will allow early identification of strengths, weaknesses and limitations, for the long-term benefit of everyone involved.

Attachments

DIRECTIONS FOR USE OF ANALYTICAL ROUND ROBIN
REFERENCE SOLUTIONS AND SOLIDS

1. Analyze for elemental composition, using the procedures normally applied in your laboratory to leach-test solutions and simulated nuclear waste solids. (See attached list of specimens.) The value of this round robin depends on application of your normal methods to these reference samples, without addition, deletion or changes in steps of your procedure.
2. Record and report all results obtained (including duplicates, "flyers," re-runs, etc.).
3. Repeat the analyses on at least three different days, analyzing the specimens each day as new and separate samples. Three to five replicates each day are recommended. Report the data obtained for each day separately. The simulated Leachates (SL-1, -2, -3) have first priority, the Accelerated Leach Test Leachate (LAT-1) second priority, and the solids (PB-242) lowest priority as far as timing is concerned. Data from analyses of each priority group should be submitted to John Mendel as soon as completed.
4. Include the following information and data in your analytical reports:
 - a. All results for all the analyses conducted, identified by date, laboratory, and method used.
 - b. Your summary of the analyses for each reference specimen (i.e., probable "correct" concentration you would report for each element in each specimen). Include your best estimate of standard deviation or other expression of estimated error interval for a single determination, preferably based on an independent determination from your own standards, etc.
 - c. Brief description of analytical methods, including specimen preparation, sampling procedure, and possible influencing factors. If you use a microbeam technique for the solid samples, note the location of sites analyzed, surface selection or preparation, etc.

5. Send all analytical data as soon as possible to:

John Mendel
Staff Scientist
Battelle Northwest
P.O. Box 999
Richland, WA 99352

6. A summary of analyses submitted by all participating laboratories (identified for purposes of that summary only by coded key letters) will be distributed to all participants as soon as enough returns have been received for meaningful comparisons.

LIST OF ANALYTICAL ROUND-ROBIN REFERENCE SPECIMENS PROVIDED

SOLUTIONS: (Analyze for minimum of 10 elements: B, Ce, Cs, K, Mg, Mo, Na, Nd, Si, Sr, + any others present significantly above detection limit).

A. Simulated Leachates

SL-1 (Water Leach)

SL-2 (Water Leach)

SL-3 (Brine Leach)

B. Accelerated Leach Test Leachate

ALT-1

SOLIDS: (Analyze for all detectable components)

PB-242 Simulated waste glass (2 wafers, ~6 grams each) (Identified as marked)

APPENDIX C

LABORATORIES PARTICIPATING IN MCC-1 ROUND ROBIN

LABORATORIES PARTICIPATING IN MCC-1 ROUND ROBIN

Argonne National Laboratory
Brookhaven National Laboratory
Cassacia Laboratory, Italy
Catholic University
Chalk River Laboratory, Canada(a)
EG&G Idaho
Exxon Nuclear Idaho
General Atomics
Hahn-Meitner Institute Laboratory, West Germany
Lawrence Livermore National Laboratory
Lucas Heights Laboratory, Australia
Marcoule Laboratory, France
Materials Characterization Center
Oak Ridge National Laboratory
Pacific Northwest Laboratory A
Pacific Northwest Laboratory B
Pacific Northwest Laboratory C
Pennsylvania State University
Radian Corporation
Rockwell Hanford Operations
Rockwell Science Center
Savannah River Laboratory A
Savannah River Laboratory B
Studsvik Laboratory, Sweden
University of Florida

(a) ARR only.

APPENDIX D

TABLULATION OF MASS LOSS AND FINAL pH DATA

APPENDIX D

TABULATION OF MASS LOSS AND FINAL pH DATA

Tabulations of the data for the 28-day tests are given in this appendix for each of the eight elements (g/m^2), specimen mass loss (g/m^2), and the final pH of the leachates. The form of tabulation is the same as for the silicon data of Table 6.1 except that the appendix tables contain the results for the MCC's analysis of the two RLs, as well as the results for the three leachates analyzed by the participating laboratory. Each laboratory has five "cases," (rows of data); the first three rows, coded "Ø" under "ANLAB," are for the leachates analyzed by the participating laboratory, the next two rows, coded "1" under "ANLAB," are for the returned leachates analyzed by the MCC. The elemental mass losses are adjusted for the blank leachate measured by the participating laboratory, and normalized by the mass fractions, f_i , and surface area to volume ratios, SA/V , as given by the calculation on page 2.4.

Silicon was analyzed for in each waste form-leachant combination, as shown in Table D.1. These combinations are indicated by the heading for each column using an appropriate combination of letters for each of the nine combinations. A "-1.000" is used to indicate missing data, i.e., data not reported for the laboratory because it did not test the waste form, the sample was lost, or leachates were not returned to MCC. Occasionally the g/m^2 for the MCC analysis of returned leachates is negative, indicating that MCC got a lower analytical result (mg/l) than the blank reported by the participating laboratory.

Table D.2 has the boron data, for NBS and MCC glass specimen, and the calcium data for basalt specimens. Table D.3 has the potassium in NBS glass, iron in basalt, and cesium in MCC glass.

Table D.4, for sodium and strontium has some interesting anomalies. A high sodium blank, combined with a low mass fraction, f_i , for NBS in silicon water resulted in extremely variable data in the first data column. Lab R, in cases 76 to 80, provides a good example of how this variability occurred. The mass fraction for sodium in NBS glass was 0.0074. The relevant data and calculations for Lab R are (using Equation 2.2 notation):

Specimen	ANLAB	E_i	C_i	C_i/f_i	$100(SA/V)mm^{-1}$	$(0.1C_i/f_i)/(SA/V)$
		Na, mg/l	Na-BL	(Na-BL)/0.0074		Na, g/m ²
21	0	51.1	-0.7	-94.59	0.9987	-9.472
22	0	61.1	9.2	1243.24	1.0002	124.300
23	0	57.6	5.8	783.78	1.0005	78.339
24	1	55.3	1.6	216.22	0.9989	21.645
25	1	53.4	3.5	472.97	1.0006	47.269
Blank 1	0	51.3				
Blank 2	0	52.3				
Blank	Ave	51.8				

Differences in the last two decimal places in the calculated and tabulated g/m² results are due to different rounding procedures in the hand calculation and computer calculations. The important points to note are that the blank corrected mg/l values go negative for one specimen and are less than 15 percent of the measured leachant values for the other specimens, and that the division by 0.0074 effectively multiplies these relatively small values by 135. The surface area-to-volume ratio was to be 0.01 mm⁻¹ but for computer calculation the actual SA/V ratios were multiplied by 100 so that they were near to unity. In these units a factor of 0.1 was needed to make the final conversion to g/m².

The reader can now understand how negative mass losses (implying mass gains) appeared and why there were such large swings in reported g/m² values for sodium from NBS glass in silicon water. It was this combination of low f_i and high blanks that resulted in the decision to remove the sodium in silicon water for NBS glass results from further study. The deionized water

had low Na blank values, so was included despite broad swings between laboratories and one laboratory (T, Case 86-90) having negative g/m^2 values. Since the brine leachant was high in sodium, about 35,000 mg/l , almost all laboratories had negative g/m^2 values for sodium, so these NBS glass and basalt data were excluded from the study and are coded as missing data (-1.000) in Table D.4. All sodium g/m^2 data for basalt in silicon water (data column 4) were also negative and excluded from further study. The only usable sodium data were for NBS glass and basalt in deionized water. The data for strontium in MCC 76-68 glass did not exhibit the problems encountered with sodium, despite having an f_i of 0.0034, the smallest mass fraction of any of the eight elements studied.

Table D.5 has the specimen mass loss data. Although the specimens for the returned leachates are identified in this table, the pre- and post-test weights were provided by the participating laboratory. The MCC did not receive any of these specimens. The results for some laboratories indicate mass gains in the form of negative values in Table D.5.

Table D.6 has the final pH of the leachate, measured after the specimen was removed from the leach container at the end of the test.

Only the results for the specimens and by the participating laboratories are plotted in Figures 3.1 to 3.9. The element averages for the three leachants analyzed by the participating laboratories are plotted against the average for the laboratories' two RLs analyzed by the MCC in Figures 3.17 to 3.32.

The triplicate data for the late laboratories were listed in Tables 3.1 through 3.3.

TABLE D.1. Elemental Mass Loss for Silicon, g/m²

CASE	TEST LAB	ANALYSIS LAB	SILICON								
			1.NBS.SI	2.NBS.DI	3.NBS.BR	4.BAS.SI	5.BAS.DI	6.BAS.BR	7.MCC.SI	8.MCC.DI	9.MCC.BR
1	1.	0.	-1.000	-1.000	-1.000	3.739	5.168	2.395	16.828	19.140	11.989
2	1.	0.	-1.000	-1.000	-1.000	3.655	5.000	2.353	16.183	18.495	12.419
3	1.	0.	-1.000	-1.000	-1.000	3.571	5.126	2.311	16.720	19.624	12.312
4	1.	1.	-1.000	-1.000	-1.000	3.739	5.336	2.836	16.720	20.753	14.928
5	1.	1.	-1.000	-1.000	-1.000	3.908	5.042	3.025	16.774	20.000	15.108
6	2.	0.	21.679	0.875	4.825	-1.000	-1.000	-1.000	17.189	16.500	12.686
7	2.	0.	33.548	0.815	2.443	-1.000	-1.000	-1.000	20.405	16.483	11.379
8	2.	0.	23.547	0.966	4.953	-1.000	-1.000	-1.000	19.871	19.334	10.367
9	2.	1.	24.277	0.454	4.035	-1.000	-1.000	-1.000	22.341	22.056	13.307
10	2.	1.	10.664	1.209	2.262	-1.000	-1.000	-1.000	27.370	18.470	12.500
11	3.	0.	43.990	9.757	9.336	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
12	3.	0.	33.695	8.233	10.529	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
13	3.	0.	30.597	12.166	9.583	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
14	3.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
15	3.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
16	4.	0.	25.003	1.412	15.287	-1.000	-1.000	-1.000	13.320	18.579	12.828
17	4.	0.	24.710	1.530	3.677	-1.000	-1.000	-1.000	14.090	17.848	12.995
18	4.	0.	24.404	1.804	3.898	-1.000	-1.000	-1.000	13.093	18.586	15.171
19	4.	1.	26.550	1.789	2.380	-1.000	-1.000	-1.000	14.462	17.587	10.974
20	4.	1.	24.526	1.270	1.525	-1.000	-1.000	-1.000	15.222	18.941	10.758
21	6.	0.	41.405	10.703	9.969	4.916	4.916	2.227	29.516	48.929	7.473
22	6.	0.	41.407	10.703	9.969	4.706	4.916	2.185	30.751	43.283	6.236
23	6.	0.	41.756	10.704	10.306	4.916	4.916	2.269	29.516	43.281	8.086
24	6.	1.	30.794	3.211	8.501	5.000	4.706	0.168	20.539	21.883	11.237
25	6.	1.	30.795	3.456	8.378	5.420	4.705	0.168	19.894	22.151	11.505
26	7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	24.029	-1.000
27	7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	28.381	-1.000
28	7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	27.879	-1.000
29	7.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	23.912	-1.000
30	7.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	23.349	-1.000
31	8.	0.	26.921	-1.000	7.471	-1.000	-1.000	-1.000	18.835	18.344	8.009
32	8.	0.	32.540	3.184	1.893	-1.000	-1.000	-1.000	19.798	18.113	8.067
33	8.	0.	-1.000	3.903	2.916	-1.000	-1.000	-1.000	16.636	17.778	8.774
34	8.	1.	23.353	3.429	1.662	-1.000	-1.000	-1.000	13.181	15.630	7.793
35	8.	1.	-1.000	2.801	2.458	-1.000	-1.000	-1.000	12.771	15.639	7.561
36	9.	0.	24.706	3.503	6.455	2.450	5.282	1.459	14.520	19.680	8.017
37	9.	0.	24.236	3.867	6.895	2.351	4.407	1.442	14.686	-1.000	8.648
38	9.	0.	24.156	3.793	6.884	2.328	4.786	1.615	13.098	19.354	8.380
39	9.	1.	21.107	3.524	-1.992	3.125	5.347	0.540	15.246	20.487	8.714
40	9.	1.	20.887	3.712	7.475	3.085	5.390	0.371	15.232	20.149	8.869
41	10.	0.	23.529	23.460	8.982	-1.000	-1.000	-1.000	24.480	32.070	16.485
42	10.	0.	30.905	24.053	7.676	-1.000	-1.000	-1.000	24.453	29.683	16.316
43	10.	0.	25.150	26.685	7.125	-1.000	-1.000	-1.000	24.667	37.393	16.359
44	10.	1.	24.146	20.267	2.187	-1.000	-1.000	-1.000	19.990	26.119	5.510
45	10.	1.	23.533	23.908	1.055	-1.000	-1.000	-1.000	19.945	29.398	4.543
46	11.	0.	12.137	1.078	11.985	2.857	6.449	2.955	16.192	18.879	13.958
47	11.	0.	8.262	3.149	12.821	2.386	6.831	2.602	15.748	19.111	12.731
48	11.	0.	5.554	2.490	12.588	2.807	8.044	2.374	14.529	20.571	11.720
49	11.	1.	3.460	1.604	6.945	-2.137	6.233	-0.022	13.317	14.925	5.972
50	11.	1.	3.849	1.384	7.290	-1.273	6.109	-0.022	-0.777	18.252	6.141

TABLE D.1. (contd)

CASE	TEST LAB	ANALYSIS TEST	SILICON								
			1.NBS.SI	2.NBS.DI	3.NBS.BR	4.BAS.SI	5.BAS.DI	6.BAS.BR	7.MCC.SI	8.MCC.DI	9.MCC.BR
51	12.	0.	27.034	1.958	9.673	1.598	5.632	2.436	-1.000	-1.000	-1.000
52	12.	0.	25.492	1.468	0.978	2.060	5.465	1.766	-1.000	-1.000	-1.000
53	L 12.	0.	27.353	3.330	9.227	1.849	5.716	2.393	-1.000	-1.000	-1.000
54	12.	1.	27.326	1.524	4.583	3.405	5.542	0.000	-1.000	-1.000	-1.000
55	12.	1.	29.771	2.770	0.000	3.364	7.232	0.000	-1.000	-1.000	-1.000
56	13.	0.	12.364	2.465	8.586	1.962	6.349	2.263	-1.000	-1.000	-1.000
57	13.	0.	25.969	2.709	3.387	1.315	6.345	1.208	-1.000	-1.000	-1.000
58	M 13.	0.	7.000	2.679	9.809	1.122	6.782	4.477	-1.000	-1.000	-1.000
59	13.	1.	14.196	1.514	-11.950	0.975	6.925	-16.376	-1.000	-1.000	-1.000
60	13.	1.	20.985	2.446	-11.338	1.313	6.160	-15.766	-1.000	-1.000	-1.000
61	15.	0.	20.755	1.284	9.011	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
62	15.	0.	20.809	1.279	8.971	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
63	O 15.	0.	20.948	1.281	9.092	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
64	15.	1.	23.089	1.648	2.766	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
65	15.	1.	24.475	1.672	2.933	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
66	16.	0.	26.372	4.623	8.165	0.966	4.985	1.094	17.195	27.365	11.980
67	16.	0.	23.505	2.924	8.039	0.672	4.689	1.051	16.510	28.367	11.502
68	P 16.	0.	23.822	3.977	8.251	0.798	5.106	1.262	17.845	27.096	10.753
69	16.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
70	16.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
71	17.	0.	23.818	2.847	8.864	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
72	17.	0.	23.549	2.621	8.249	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
73	Q 17.	0.	23.221	2.380	8.859	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
74	17.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
75	17.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
76	18.	0.	26.073	2.322	2.266	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
77	18.	0.	32.457	2.832	4.743	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
78	R 18.	0.	30.613	2.342	3.060	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
79	18.	1.	30.048	3.009	3.703	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
80	18.	1.	29.996	2.646	5.661	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
81	19.	0.	23.333	2.884	8.026	2.226	5.617	1.008	13.285	19.172	7.258
82	19.	0.	23.339	2.668	8.242	2.143	5.571	1.008	12.468	18.766	7.424
83	S 19.	0.	22.900	2.564	8.106	2.143	5.572	0.966	13.169	19.233	6.799
84	19.	1.	23.957	2.617	8.209	2.059	5.404	1.135	12.527	18.025	6.476
85	19.	1.	22.355	2.480	8.213	2.521	5.153	1.345	12.533	18.907	6.481
86	20.	0.	36.468	2.379	15.260	5.105	5.588	3.529	24.552	26.720	16.720
87	20.	0.	26.988	4.893	14.924	3.761	5.252	3.571	21.478	25.484	15.430
88	T 20.	0.	31.269	3.976	15.627	5.105	6.429	3.824	21.317	26.882	16.290
89	20.	1.	32.187	3.997	12.691	3.592	6.807	0.714	21.532	25.753	11.559
90	20.	1.	33.104	4.618	12.080	4.517	7.041	0.714	20.027	27.366	11.559
91	21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	14.064	19.063	8.129
92	21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	10.936	19.497	8.089
93	U 21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	12.052	19.446	7.574
94	21.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	15.620	20.500	7.938
95	21.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	15.669	20.552	7.820
96	24.	0.	24.548	3.610	8.025	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
97	X 24.	0.	22.421	2.714	8.115	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
98	24.	0.	22.272	2.694	8.416	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
99	24.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
100	24.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000

TABLE D.2. Elemental Mass Loss for Boron and Calcium, g/m²

CASE	TEST LAB	ANALYSIS LAB	BORON			CALCIUM			BORON		
			1.B.N.SI	2.B.N.DI	3.B.N.BR	4.CA.B.S	5.CA.B.D	6.CA.B.B	7.B.M.SI	8.B.M.DI	9.B.M.BR
1	1.	0.	-1.000	-1.000	-1.000	0.270	2.108	13.149	-1.000	-1.000	-1.000
2	1.	0.	-1.000	-1.000	-1.000	0.270	2.230	9.730	-1.000	-1.000	-1.000
3	A 1.	0.	-1.000	-1.000	-1.000	0.270	2.027	10.568	-1.000	-1.000	-1.000
4	1.	1.	-1.000	-1.000	-1.000	-0.057	1.730	15.878	-1.000	-1.000	-1.000
5	1.	1.	-1.000	-1.000	-1.000	-0.088	1.595	14.324	-1.000	-1.000	-1.000
6	2.	0.	52.259	3.296	26.377	-1.000	-1.000	-1.000	33.066	28.463	36.914
7	2.	0.	67.252	4.433	5.651	-1.000	-1.000	-1.000	39.848	27.087	26.892
8	B 2.	0.	54.226	4.613	9.432	-1.000	-1.000	-1.000	39.856	33.477	23.430
9	2.	1.	47.743	1.980	-28.288	-1.000	-1.000	-1.000	31.326	30.230	-22.280
10	2.	1.	13.819	4.165	-31.305	-1.000	-1.000	-1.000	36.770	23.818	-23.299
11	3.	0.	86.496	25.481	23.193	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
12	3.	0.	80.706	23.238	23.412	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
13	C 3.	0.	85.139	32.559	23.193	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
14	3.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
15	3.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
16	4.	0.	70.165	16.272	31.596	-1.000	-1.000	-1.000	22.605	27.556	24.704
17	4.	0.	69.928	16.362	9.107	-1.000	-1.000	-1.000	24.125	25.972	22.038
18	D 4.	0.	69.436	17.432	11.137	-1.000	-1.000	-1.000	22.185	27.386	27.120
19	4.	1.	63.875	14.874	9.577	-1.000	-1.000	-1.000	22.446	23.808	21.527
20	4.	1.	61.535	15.960	7.894	-1.000	-1.000	-1.000	23.575	26.401	23.954
21	6.	0.	66.035	11.320	20.753	0.000	0.135	6.351	57.823	64.631	15.307
22	6.	0.	67.925	9.434	19.434	0.000	0.135	6.351	57.821	61.229	14.284
23	F 6.	0.	69.833	15.095	19.434	0.000	0.000	5.405	61.224	64.628	14.907
24	6.	1.	64.526	12.678	21.319	0.632	1.594	21.823	28.608	30.717	25.341
25	6.	1.	66.981	13.454	22.923	0.732	1.743	22.025	27.247	31.088	25.510
26	7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	25.843	-1.000
27	7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	31.337	-1.000
28	G 7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	33.070	-1.000
29	7.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	33.637	-1.000
30	7.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	33.178	-1.000
31	8.	0.	86.384	-1.000	21.537	-1.000	-1.000	-1.000	31.972	29.781	21.369
32	8.	0.	86.287	27.971	16.372	-1.000	-1.000	-1.000	35.458	28.256	2.231
33	H 8.	0.	-1.000	28.052	14.456	-1.000	-1.000	-1.000	30.654	28.032	23.712
34	8.	1.	57.661	23.111	14.758	-1.000	-1.000	-1.000	23.544	22.653	17.779
35	8.	1.	-1.000	22.946	14.550	-1.000	-1.000	-1.000	22.262	22.520	17.742
36	9.	0.	74.351	13.403	13.897	0.860	1.357	32.190	25.020	30.799	14.572
37	9.	0.	74.732	14.553	14.966	0.609	0.952	26.575	24.687	-1.000	15.722
38	I 9.	0.	73.126	15.574	15.277	0.743	1.229	26.902	23.615	30.571	15.316
39	9.	1.	75.387	14.487	19.309	0.742	1.030	40.667	24.550	30.282	21.185
40	9.	1.	66.251	13.959	19.656	0.621	1.192	32.527	25.105	29.931	21.903
41	10.	0.	64.898	47.796	17.642	-1.000	-1.000	-1.000	41.124	46.837	36.426
42	10.	0.	70.427	52.418	10.472	-1.000	-1.000	-1.000	37.009	42.374	27.719
43	J 10.	0.	57.408	59.332	7.641	-1.000	-1.000	-1.000	41.498	57.246	30.949
44	10.	1.	63.005	50.748	-0.283	-1.000	-1.000	-1.000	39.061	39.117	-2.211
45	10.	1.	55.454	54.717	-1.000	-1.000	-1.000	-1.000	38.740	45.578	-3.912
46	11.	0.	20.309	21.684	17.700	1.314	4.908	63.684	27.011	27.107	18.293
47	11.	0.	14.276	20.020	20.564	1.149	3.534	48.944	26.238	27.256	17.605
48	K 11.	0.	11.000	22.523	19.211	0.964	8.715	38.966	24.482	30.548	17.983
49	11.	1.	8.110	13.111	18.628	0.580	2.898	20.261	22.082	18.478	20.142
50	11.	1.	8.700	14.330	20.337	1.028	2.374	22.482	10.399	24.578	20.496

TABLE D.2. (contd)

CASE	TEST LAB	ANALYSIS LAB	BORON			CALCIUM			BORON		
			1.B.N.SI	2.B.N.DI	3.B.N.BR	4.CA.B.S	5.CA.B.D	6.CA.B.B	7.B.M.SI	8.B.M.DI	9.B.M.BR
51	12.	0.	74.245	19.217	17.754	0.852	3.636	19.455	-1.000	-1.000	-1.000
52	12.	0.	67.043	24.485	8.860	0.771	4.029	20.425	-1.000	-1.000	-1.000
53	L 12.	0.	79.190	16.173	18.850	0.703	3.771	20.661	-1.000	-1.000	-1.000
54	12.	1.	73.454	25.785	18.661	0.406	1.904	15.274	-1.000	-1.000	-1.000
55	12.	1.	77.790	16.450	11.145	0.392	4.327	16.753	-1.000	-1.000	-1.000
56	13.	0.	23.962	21.000	22.708	1.419	3.851	51.203	-1.000	-1.000	-1.000
57	13.	0.	76.226	19.509	12.519	1.581	6.486	46.392	-1.000	-1.000	-1.000
58	M 13.	0.	13.170	15.509	20.991	1.622	4.541	49.122	-1.000	-1.000	-1.000
59	13.	1.	28.623	24.698	4.575	0.897	2.571	17.473	-1.000	-1.000	-1.000
60	13.	1.	66.358	14.453	2.406	0.368	3.230	29.740	-1.000	-1.000	-1.000
61	15.	0.	42.062	1.321	9.266	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
62	15.	0.	42.172	1.315	9.225	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
63	O 15.	0.	42.453	1.318	9.349	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
64	15.	1.	56.415	10.090	16.972	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
65	15.	1.	58.123	9.842	18.656	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
66	16.	0.	54.337	13.468	20.189	0.545	1.690	28.414	28.992	40.078	28.890
67	16.	0.	62.855	14.928	19.047	0.676	1.305	32.440	28.266	42.510	28.224
68	P 16.	0.	63.073	12.610	19.985	0.824	2.621	27.053	30.496	39.817	28.571
69	16.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
70	16.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
71	17.	0.	76.653	16.319	18.764	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
72	17.	0.	68.844	16.930	20.641	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
73	Q 17.	0.	70.099	16.738	18.754	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
74	17.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
75	17.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
76	18.	0.	59.972	12.642	11.713	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
77	18.	0.	63.660	13.336	16.427	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
78	R 18.	0.	61.000	12.126	12.082	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
79	18.	1.	61.850	12.921	12.933	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
80	18.	1.	61.933	12.848	16.897	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
81	19.	0.	65.828	11.609	17.166	0.936	1.703	35.802	22.081	26.429	13.605
82	19.	0.	63.202	10.151	18.019	0.978	1.634	31.284	20.771	25.776	13.614
83	S 19.	0.	54.306	9.962	16.608	0.912	1.567	41.062	21.217	26.643	13.602
84	19.	1.	60.386	10.153	18.392	0.947	1.365	30.480	20.745	24.636	13.600
85	19.	1.	51.677	10.033	16.419	0.784	1.419	43.581	20.878	25.856	13.611
86	20.	0.	72.453	17.689	33.962	0.270	2.703	41.622	40.215	40.816	37.415
87	20.	0.	67.925	26.038	37.736	0.270	2.973	45.676	35.034	45.578	44.218
88	T 20.	0.	56.604	14.717	54.717	0.270	2.703	51.081	31.293	40.136	30.612
89	20.	1.	-22.075	-63.378	-277.925	0.101	2.527	46.892	-117.007	-103.401	-532.653
90	20.	1.	-14.528	-61.604	-280.189	0.001	2.521	52.297	-118.707	-100.000	-532.313
91	21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	28.574	35.360	25.714
92	21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	28.193	33.937	28.999
93	U 21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	27.553	35.125	23.275
94	21.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	23.495	26.589	9.975
95	21.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	23.496	27.277	8.246
96	24.	0.	65.607	16.846	35.769	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
97	24.	0.	68.980	15.050	28.998	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
98	X 24.	0.	68.708	14.941	35.359	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
99	24.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
100	24.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000

TABLE D.3. Elemental Mass Loss for Cesium, Iron and Potassium, g/m²

CASE	TEST LAB	ANALYSIS LAB	POTASSIUM			IRON			CESIUM			
			1.K.N.SI	2.K.N.DI	3.K.N.BR	4.FE.B.S	5.FE.B.D	6.FE.B.B	7.CS.M.S	8.CS.M.D	9.CS.M.B	
1	A	1.	0.	-1.000	-1.000	-1.000	0.598	0.598	0.890	31.650	33.204	27.670
2		1.	0.	-1.000	-1.000	-1.000	0.598	0.598	0.159	30.680	32.330	25.922
3		1.	0.	-1.000	-1.000	-1.000	0.598	0.598	0.841	31.650	32.330	25.437
4		1.	1.	-1.000	-1.000	-1.000	0.033	0.011	1.451	19.126	22.136	-1.000
5		1.	1.	-1.000	-1.000	-1.000	0.034	0.010	1.573	19.806	21.748	-1.000
6	B	2.	0.	28.744	3.630	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
7		2.	0.	38.683	3.030	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
8		2.	0.	29.697	3.780	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
9		2.	1.	41.153	1.817	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
10		2.	1.	16.350	3.042	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
11	C	3.	0.	57.568	19.353	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
12		3.	0.	53.081	17.540	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
13		3.	0.	55.951	22.910	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
14		3.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
15		3.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
16	D	4.	0.	48.743	10.744	-1.000	-1.000	-1.000	-1.000	24.441	30.430	-1.000
17		4.	0.	49.366	10.545	-1.000	-1.000	-1.000	-1.000	26.209	28.357	-1.000
18		4.	0.	48.455	11.366	-1.000	-1.000	-1.000	-1.000	23.833	29.391	-1.000
19		4.	1.	49.516	8.949	-1.000	-1.000	-1.000	-1.000	20.194	27.253	-1.000
20		4.	1.	46.714	9.753	-1.000	-1.000	-1.000	-1.000	19.369	29.126	-1.000
21	F	6.	0.	44.014	9.242	-1.000	0.000	0.000	0.000	52.427	40.780	62.139
22		6.	0.	44.773	10.000	-1.000	0.000	0.000	0.000	55.338	38.838	60.186
23		6.	0.	44.787	9.243	-1.000	0.000	0.000	0.000	55.340	38.836	61.891
24		6.	1.	54.619	9.151	-1.000	0.073	0.035	-1.341	28.643	33.401	-4.855
25		6.	1.	57.955	9.864	-1.000	0.072	0.038	-1.463	25.439	32.816	-1.942
26	G	7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	25.071	-1.000
27		7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	29.977	-1.000
28		7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	30.013	-1.000
29		7.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	38.990	-1.000
30		7.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	37.120	-1.000
31	H	8.	0.	52.034	-1.000	-1.000	-1.000	-1.000	-1.000	1.744	20.514	0.000
32		8.	0.	54.411	18.900	-1.000	-1.000	-1.000	-1.000	1.749	21.321	0.000
33		8.	0.	-1.000	19.331	-1.000	-1.000	-1.000	-1.000	1.766	21.383	0.000
34		8.	1.	40.938	16.757	-1.000	-1.000	-1.000	-1.000	23.219	23.619	17.567
35		8.	1.	-1.000	17.219	-1.000	-1.000	-1.000	-1.000	22.190	23.550	16.309
36	I	9.	0.	68.917	5.019	-1.000	0.573	-0.019	0.085	0.000	0.000	0.000
37		9.	0.	69.442	6.062	-1.000	0.032	-0.030	0.183	0.000	-1.000	0.000
38		9.	0.	62.370	9.285	-1.000	0.019	-0.022	0.134	0.000	0.000	0.000
39		9.	1.	52.254	9.514	-1.000	0.010	-0.033	0.305	-3.403	-3.398	-1.000
40		9.	1.	52.164	9.678	-1.000	0.020	-0.037	0.244	-3.400	-3.405	-1.000
41	J	10.	0.	53.046	33.031	-1.000	-1.000	-1.000	-1.000	11.219	6.694	9.102
42		10.	0.	53.333	36.637	-1.000	-1.000	-1.000	-1.000	11.219	4.659	13.523
43		10.	0.	43.360	39.154	-1.000	-1.000	-1.000	-1.000	12.094	4.926	14.711
44		10.	1.	51.286	34.729	-1.000	-1.000	-1.000	-1.000	34.273	39.632	11.854
45		10.	1.	49.313	36.248	-1.000	-1.000	-1.000	-1.000	36.911	42.350	10.884
46	K	11.	0.	17.373	12.363	-1.000	-0.088	1.136	0.712	26.709	29.508	49.751
47		11.	0.	12.656	17.140	-1.000	-0.076	0.329	0.236	25.108	25.466	48.231
48		11.	0.	10.917	19.167	-1.000	-0.078	2.167	0.567	22.870	30.554	48.018
49		11.	1.	7.103	7.760	-1.000	-0.100	0.202	1.503	11.128	21.230	11.695
50		11.	1.	7.615	8.633	-1.000	-0.091	0.072	1.680	24.941	27.339	9.673

TABLE D.3. (contd)

CASE	TEST LAB	ANALYSIS LAB	POTASSIUM			IRON			CESIUM		
			1.K.N.SI	2.K.N.DI	3.K.N.BR	4.FE.B.S	5.FE.B.D	6.FE.B.B	7.CS.M.S	8.CS.M.D	9.CS.M.B
51	12.	0.	51.515	12.430	-1.000	0.015	0.598	3.414	-1.000	-1.000	-1.000
52	12.	0.	48.461	16.663	-1.000	0.035	0.793	1.465	-1.000	-1.000	-1.000
53	L 12.	0.	53.056	11.201	-1.000	0.030	0.573	2.681	-1.000	-1.000	-1.000
54	12.	1.	57.547	18.012	-1.000	0.016	0.000	-0.244	-1.000	-1.000	-1.000
55	12.	1.	60.575	11.526	-1.000	0.016	0.155	0.488	-1.000	-1.000	-1.000
56	13.	0.	0.515	-1.106	-1.000	0.032	0.234	3.287	-1.000	-1.000	-1.000
57	13.	0.	30.848	-1.212	-1.000	0.032	0.624	2.921	-1.000	-1.000	-1.000
58	M 13.	0.	11.015	12.409	-1.000	0.062	0.280	2.835	-1.000	-1.000	-1.000
59	13.	1.	23.955	19.727	-1.000	0.007	0.013	1.267	-1.000	-1.000	-1.000
60	13.	1.	40.167	-0.424	-1.000	0.013	0.343	1.899	-1.000	-1.000	-1.000
61	15.	0.	45.036	7.576	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
62	15.	0.	45.154	7.542	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
63	O 15.	0.	45.455	7.558	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
64	15.	1.	46.667	8.026	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
65	15.	1.	47.590	7.980	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
66	16.	0.	42.926	10.603	-1.000	0.015	0.038	0.977	30.373	39.862	17.462
67	16.	0.	50.216	12.215	-1.000	0.014	0.034	0.854	27.386	43.311	22.324
68	P 16.	0.	48.723	9.792	-1.000	0.015	0.156	1.099	30.769	40.136	19.417
69	16.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
70	16.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
71	17.	0.	60.857	12.125	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
72	17.	0.	56.708	13.026	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
73	Q 17.	0.	55.745	12.739	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
74	17.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
75	17.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
76	18.	0.	45.209	9.313	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
77	18.	0.	52.871	9.219	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
78	R 18.	0.	49.523	8.537	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
79	18.	1.	46.414	9.054	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
80	18.	1.	48.000	8.902	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
81	19.	0.	51.061	9.201	-1.000	0.026	0.022	0.549	21.738	27.553	9.709
82	19.	0.	47.436	7.697	-1.000	0.030	0.049	0.549	20.652	27.510	10.298
83	S 19.	0.	41.960	7.742	-1.000	0.020	0.005	0.366	21.044	26.787	7.377
84	19.	1.	46.234	7.972	-1.000	0.013	0.020	0.000	21.728	26.072	7.376
85	19.	1.	40.758	7.724	-1.000	0.024	0.033	0.183	20.628	26.782	7.868
86	20.	0.	56.538	5.810	-1.000	0.122	0.366	0.732	36.349	38.835	29.126
87	20.	0.	53.053	9.780	-1.000	0.122	0.000	0.610	33.010	36.893	27.184
88	T 20.	0.	46.538	6.811	-1.000	0.122	0.049	0.854	33.981	36.893	26.155
89	20.	1.	48.356	9.379	-1.000	0.055	0.095	-0.366	33.689	37.767	19.320
90	20.	1.	54.417	10.311	-1.000	0.100	0.640	-0.732	31.845	41.650	30.583
91	21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	28.327	33.156	20.013
92	21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	27.647	32.454	20.012
93	U 21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	27.700	33.257	20.078
94	21.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	25.642	30.769	15.071
95	21.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	25.750	31.811	15.053
96	24.	0.	43.050	10.961	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
97	24.	0.	44.913	8.750	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
98	X 24.	0.	44.587	8.836	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
99	24.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
100	24.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000

TABLE D.4. Elemental Mass Loss for Sodium and Strontium, g/m²

CASE	TEST LAB	ANALYSIS LAB	SODIUM						STRONTIUM		
			1.NA.N.S	2.NA.N.D	3.NA.N.B	4.NA.B.S	5.NA.B.D	6.NA.B.B	7.SR.M.S	8.SR.M.D	9.SR.M.B
1	1.	0.	-1.000	-1.000	-1.000	-11.667	5.111	-1.000	1.382	2.235	33.235
2	1.	0.	-1.000	-1.000	-1.000	-16.111	4.500	-1.000	1.618	2.235	24.116
3	A 1.	0.	-1.000	-1.000	-1.000	-23.333	5.167	-1.000	1.382	2.265	28.529
4	1.	1.	-1.000	-1.000	-1.000	-33.333	3.722	-1.000	1.353	2.176	24.175
5	1.	1.	-1.000	-1.000	-1.000	-45.556	3.722	-1.000	1.529	2.088	27.059
6	2.	0.	80.957	7.824	-1.000	-1.000	-1.000	-1.000	0.500	2.884	21.670
7	2.	0.	592.988	3.918	-1.000	-1.000	-1.000	-1.000	0.352	3.171	19.133
8	B 2.	0.	148.649	21.440	-1.000	-1.000	-1.000	-1.000	0.382	1.822	16.443
9	2.	1.	143.038	0.324	-1.000	-1.000	-1.000	-1.000	0.441	1.590	27.060
10	2.	1.	132.324	2.213	-1.000	-1.000	-1.000	-1.000	0.176	2.527	25.588
11	3.	0.	-7.527	23.819	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
12	3.	0.	-20.358	21.718	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
13	C 3.	0.	-27.166	27.095	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
14	3.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
15	3.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
16	4.	0.	-1.338	13.507	-1.000	-1.000	-1.000	-1.000	0.527	2.019	27.206
17	4.	0.	0.000	14.216	-1.000	-1.000	-1.000	-1.000	0.290	2.388	19.903
18	D 4.	0.	-1.338	14.450	-1.000	-1.000	-1.000	-1.000	0.547	2.014	19.023
19	4.	1.	63.673	13.106	-1.000	-1.000	-1.000	-1.000	1.088	3.243	29.485
20	4.	1.	55.824	13.203	-1.000	-1.000	-1.000	-1.000	0.859	2.206	29.121
21	6.	0.	40.539	13.513	-1.000	-44.441	12.223	-1.000	0.000	0.000	2.941
22	6.	0.	40.541	14.864	-1.000	-44.443	12.222	-1.000	0.000	0.000	2.941
23	F 6.	0.	40.553	14.865	-1.000	-44.446	12.221	-1.000	0.000	0.000	2.930
24	6.	1.	-136.481	7.972	-1.000	-118.333	7.888	-1.000	1.500	1.235	-316.190
25	6.	1.	-110.811	8.109	-1.000	-121.667	8.388	-1.000	1.706	1.294	-316.176
26	7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	2.921	-1.000
27	7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	2.929	-1.000
28	G 7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	2.933	-1.000
29	7.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	0.915	-1.000
30	7.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	0.836	-1.000
31	8.	0.	738.676	-1.000	-1.000	-1.000	-1.000	-1.000	1.160	3.415	16.541
32	H 8.	0.	851.070	8.861	-1.000	-1.000	-1.000	-1.000	1.163	2.801	17.793
33	8.	0.	-1.000	8.779	-1.000	-1.000	-1.000	-1.000	1.947	3.103	10.901
34	8.	1.	552.353	4.457	-1.000	-1.000	-1.000	-1.000	0.809	2.783	16.246
35	8.	1.	-1.000	3.272	-1.000	-1.000	-1.000	-1.000	0.807	2.657	17.702
36	9.	0.	20.146	12.748	-1.000	-26.872	11.252	-1.000	0.720	1.132	17.350
37	9.	0.	13.938	12.939	-1.000	-35.777	9.955	-1.000	0.758	-1.000	17.930
38	I 9.	0.	16.631	12.405	-1.000	-32.195	10.670	-1.000	0.826	1.728	18.013
39	9.	1.	24.380	12.816	-1.000	-43.619	12.206	-1.000	0.721	1.308	26.314
40	9.	1.	8.250	13.508	-1.000	-26.987	12.209	-1.000	0.720	1.369	25.312
41	10.	0.	-133.024	35.800	-1.000	-1.000	-1.000	-1.000	0.000	0.000	-0.478
42	10.	0.	-98.031	45.800	-1.000	-1.000	-1.000	-1.000	0.000	0.000	-0.473
43	J 10.	0.	-55.196	48.899	-1.000	-1.000	-1.000	-1.000	0.000	0.000	12.781
44	10.	1.	-23.718	42.684	-1.000	-1.000	-1.000	-1.000	-0.294	-0.206	30.940
45	10.	1.	4.662	46.203	-1.000	-1.000	-1.000	-1.000	-0.294	-0.412	29.471
46	11.	0.	27.342	28.416	-1.000	-24.344	17.353	-1.000	1.375	4.085	17.468
47	11.	0.	36.294	17.412	-1.000	-39.786	18.592	-1.000	1.684	3.457	16.451
48	K 11.	0.	18.356	27.626	-1.000	-38.208	19.419	-1.000	1.489	2.925	17.031
49	11.	1.	8.834	11.098	-1.000	-144.634	12.574	-1.000	1.512	7.496	26.012
50	11.	1.	-3.443	12.426	-1.000	-112.858	12.742	-1.000	0.472	3.486	27.555

TABLE D.4. (contd)

CASE	TEST LAB	ANALYSIS LAB	SODIUM						STRONTIUM		
			1.NA.N.S	2.NA.N.D	3.NA.N.B	4.NA.B.S	5.NA.B.D	6.NA.B.B	7.SR.M.S	8.SR.M.D	9.SR.M.B
51	12.	0.	67.568	14.872	-1.000	-33.371	14.448	-1.000	-1.000	-1.000	-1.000
52	12.	0.	27.014	18.914	-1.000	-33.348	14.451	-1.000	-1.000	-1.000	-1.000
53	L 12.	0.	40.560	13.500	-1.000	-38.889	14.448	-1.000	-1.000	-1.000	-1.000
54	12.	1.	94.547	21.330	-1.000	-1.220	12.769	-1.000	-1.000	-1.000	-1.000
55	12.	1.	94.546	13.662	-1.000	-1.220	15.567	-1.000	-1.000	-1.000	-1.000
56	13.	0.	-34.054	13.649	-1.000	-30.444	14.944	-1.000	-1.000	-1.000	-1.000
57	13.	0.	67.027	15.946	-1.000	-35.278	14.222	-1.000	-1.000	-1.000	-1.000
58	M 13.	0.	-75.541	13.243	-1.000	-21.500	16.167	-1.000	-1.000	-1.000	-1.000
59	13.	1.	-45.135	17.297	-1.000	-51.889	14.685	-1.000	-1.000	-1.000	-1.000
60	13.	1.	-3.243	8.649	-1.000	-54.736	13.000	-1.000	-1.000	-1.000	-1.000
61	15.	0.	93.723	8.108	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
62	15.	0.	93.968	8.072	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
63	O 15.	0.	94.595	8.089	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
64	15.	1.	6.757	9.135	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
65	15.	1.	13.604	8.999	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
66	16.	0.	-30.208	11.730	-1.000	-69.688	9.632	-1.000	0.617	0.852	29.389
67	16.	0.	-39.896	12.800	-1.000	-65.801	9.241	-1.000	0.706	0.735	29.404
68	P 16.	0.	-60.210	10.370	-1.000	-62.500	10.678	-1.000	0.617	0.824	29.412
69	16.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
70	16.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
71	17.	0.	3.375	12.166	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
72	17.	0.	11.495	13.509	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
73	Q 17.	0.	-11.484	13.526	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
74	17.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
75	17.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
76	18.	0.	-9.471	10.349	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
77	18.	0.	124.306	9.669	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
78	R 18.	0.	78.343	9.518	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
79	18.	1.	21.645	9.656	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
80	18.	1.	47.268	9.130	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
81	19.	0.	35.135	10.329	-1.000	-17.214	9.558	-1.000	1.000	1.853	17.647
82	19.	0.	10.813	8.392	-1.000	-23.333	13.105	-1.000	1.147	1.968	17.659
83	S 19.	0.	13.510	8.459	-1.000	-12.222	9.720	-1.000	1.059	1.824	17.643
84	19.	1.	18.928	8.678	-1.000	-20.000	9.442	-1.000	1.118	2.321	19.110
85	19.	1.	16.216	8.577	-1.000	-25.000	9.000	-1.000	0.759	1.912	16.184
86	20.	0.	273.649	-27.697	-1.000	-61.944	-8.056	-1.000	0.406	1.059	8.824
87	20.	0.	-47.973	-7.095	-1.000	-91.944	-8.611	-1.000	0.559	1.412	8.824
88	T 20.	0.	0.676	-26.014	-1.000	-68.611	-6.389	-1.000	0.559	1.382	8.824
89	20.	1.	-97.973	-19.211	-1.000	-90.833	-3.611	-1.000	0.559	1.147	19.118
90	20.	1.	-84.459	-23.716	-1.000	-100.278	-1.662	-1.000	0.735	1.147	22.059
91	21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	1.509	1.950	15.712
92	21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	1.555	2.115	16.226
93	U 21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	1.528	2.045	15.687
94	21.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	1.583	1.953	13.385
95	21.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	1.631	1.725	14.854
96	24.	0.	24.166	13.614	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
97	24.	0.	32.046	10.745	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
98	X 24.	0.	19.950	10.574	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
99	24.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
100	24.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000

TABLE D.5. Specimen Mass Loss Data, g/m²

CASE	TEST LAB	ANALYSIS LAB	SPECIMEN MASS LOSS, g/m ²								
			1.NBS.SI	2.NBS.DI	3.NBS.BR	4.BAS.SI	5.BAS.DI	6.BAS.BR	7.MCC.SI	8.MCC.DI	9.MCC.BR
1	1.	0.	-1.0000	-1.0000	-1.0000	5.7143	6.8558	3.7825	10.9049	12.4413	11.5839
2	1.	0.	-1.0000	-1.0000	-1.0000	4.5346	5.6931	4.1262	10.3529	12.6492	10.9524
3	A 1.	0.	-1.0000	-1.0000	-1.0000	5.2506	7.3286	3.8278	10.7981	11.7925	11.6667
4	1.	1.	-1.0000	-1.0000	-1.0000	4.6683	6.9712	4.9065	10.9524	13.0332	10.8491
5	1.	1.	-1.0000	-1.0000	-1.0000	4.8662	6.4133	3.7915	10.8434	12.9930	11.9816
6	2.	0.	30.6435	5.3205	8.7252	-1.0000	-1.0000	-1.0000	12.4346	10.7061	7.9142
7	2.	0.	30.8108	4.3356	5.5176	-1.0000	-1.0000	-1.0000	12.0331	10.6737	5.7336
8	B 2.	0.	32.0093	4.8438	9.8568	-1.0000	-1.0000	-1.0000	12.7186	11.4908	5.8236
9	2.	1.	35.3559	7.4493	7.5389	-1.0000	-1.0000	-1.0000	12.7877	13.8145	5.2344
10	2.	1.	15.8340	4.3093	7.9960	-1.0000	-1.0000	-1.0000	14.1637	10.2524	5.4375
11	3.	0.	35.9494	13.3838	9.5718	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
12	3.	0.	34.8052	11.2245	9.6203	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
13	C 3.	0.	35.8247	16.0804	10.0756	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
14	3.	1.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
15	3.	1.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
16	4.	0.	30.5076	3.4815	14.3392	-1.0000	-1.0000	-1.0000	8.2587	11.9900	7.8000
17	4.	0.	30.3258	3.6750	4.5161	-1.0000	-1.0000	-1.0000	7.8571	11.2376	7.0927
18	D 4.	0.	30.3218	3.8404	3.9348	-1.0000	-1.0000	-1.0000	7.7506	12.7543	6.7164
19	4.	1.	29.5739	3.8025	5.0125	-1.0000	-1.0000	-1.0000	8.6250	10.9774	7.3935
20	4.	1.	28.8665	3.1888	4.3719	-1.0000	-1.0000	-1.0000	8.6902	11.7750	6.7574
21	6.	0.	37.9328	3.5585	10.0030	4.2489	5.1442	7.6586	12.6201	13.2349	3.9190
22	6.	0.	36.0490	4.6175	9.8991	3.8110	6.4702	6.1950	13.2445	12.3886	3.4067
23	F 6.	0.	36.2891	4.2414	9.5694	5.5322	5.2372	7.4153	12.3671	13.3261	4.4811
24	6.	1.	33.5815	4.0821	11.1029	2.4067	4.7111	8.6044	12.0278	12.4035	3.6671
25	6.	1.	34.1737	3.7115	10.0144	2.4845	3.1345	9.5404	12.2123	13.0590	4.2854
26	7.	0.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	14.2771	-1.0000
27	7.	0.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	16.1058	-1.0000
28	G 7.	0.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	16.1249	-1.0000
29	7.	1.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	17.4162	-1.0000
30	7.	1.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	18.4114	-1.0000
31	8.	0.	30.7962	9.2408	18.2363	-1.0000	-1.0000	-1.0000	13.7236	15.8471	3.7695
32	8.	0.	39.9397	5.8008	10.3145	-1.0000	-1.0000	-1.0000	5.0033	13.7256	4.5410
33	H 8.	0.	-1.0000	9.4953	15.7264	-1.0000	-1.0000	-1.0000	15.4114	11.7632	6.5200
34	8.	1.	39.9397	9.4953	10.3145	-1.0000	-1.0000	-1.0000	5.0033	13.7655	3.7695
35	8.	1.	-1.0000	5.8008	15.7264	-1.0000	-1.0000	-1.0000	13.7236	11.7292	6.5200
36	9.	0.	28.2663	4.7121	9.0743	4.3155	8.8701	-4.2153	9.2631	13.8124	5.3571
37	9.	0.	28.5736	5.3522	8.8940	3.0808	6.4477	-3.1004	9.5021	12.4672	5.4352
38	I 9.	0.	28.3838	5.0434	8.8170	3.9856	6.0520	-4.4061	9.7082	13.3807	7.4796
39	9.	1.	29.8168	5.1408	8.6592	3.1513	5.8709	-5.5690	8.9677	13.8458	7.0061
40	9.	1.	27.9804	4.7807	8.6734	3.2655	5.8169	-6.0417	9.0572	12.2677	5.6144
41	10.	0.	30.5191	24.2822	10.4582	-1.0000	-1.0000	-1.0000	15.1485	21.6526	10.2742
42	10.	0.	34.2209	25.5716	10.1818	-1.0000	-1.0000	-1.0000	14.5542	17.2678	9.2285
43	J 10.	0.	28.3547	27.4108	9.9130	-1.0000	-1.0000	-1.0000	15.9208	20.3985	8.9543
44	10.	1.	28.7091	25.2153	10.2405	-1.0000	-1.0000	-1.0000	14.7423	18.4964	8.7256
45	10.	1.	30.4627	28.9061	10.3420	-1.0000	-1.0000	-1.0000	14.6286	23.8925	8.7256
46	11.	0.	18.1578	5.2386	20.3335	-12.5919	17.3671	-19.9586	2.6559	13.3536	7.8354
47	11.	0.	13.1655	12.9159	21.4184	-19.7560	2.5143	-19.8871	7.6679	13.3554	8.0908
48	K 11.	0.	5.0309	15.7497	12.3842	-17.3173	-4.9686	-10.0038	10.3340	13.2230	5.2784
49	11.	1.	12.5717	7.8264	12.3695	-20.1425	-10.1156	-9.9793	8.0313	7.8947	7.8219
50	11.	1.	12.7405	7.6884	12.9836	-4.9668	-2.4957	-17.4086	13.0137	15.7315	7.8219

TABLE D.5. (contd)

CASE	TEST LAB	ANALYSIS LAB	SPECIMEN MASS LOSS, g/m ²								
			1.NBS.SI	2.NBS.DI	3.NBS.BR	4.BAS.SI	5.BAS.DI	6.BAS.BR	7.MCC.SI	8.MCC.DI	9.MCC.BR
51	12.	0.	31.1429	4.4565	10.3864	-0.3128	3.5507	-5.4670	-1.0000	-1.0000	-1.0000
52	12.	0.	29.8527	5.1067	2.3444	0.1129	3.4128	-7.4525	-1.0000	-1.0000	-1.0000
53	L 12.	0.	31.8668	5.4983	10.1585	-0.3604	3.1147	-6.7184	-1.0000	-1.0000	-1.0000
54	12.	1.	30.4632	4.9697	9.6487	0.5002	2.5283	-7.1305	-1.0000	-1.0000	-1.0000
55	12.	1.	32.3287	4.6875	3.3373	-0.4139	3.1198	-6.4024	-1.0000	-1.0000	-1.0000
56	13.	0.	12.4069	5.2239	9.7744	4.0000	-0.7519	15.7500	-1.0000	-1.0000	-1.0000
57	13.	0.	41.3965	5.7357	3.7594	6.4838	1.2500	17.7500	-1.0000	-1.0000	-1.0000
58	M 13.	0.	8.0000	5.7644	11.0000	-7.2500	5.0000	14.5000	-1.0000	-1.0000	-1.0000
59	13.	1.	17.2932	5.0125	3.5088	8.0000	4.9628	18.2500	-1.0000	-1.0000	-1.0000
60	13.	1.	27.0000	4.7500	3.5000	3.2828	5.2500	21.2121	-1.0000	-1.0000	-1.0000
61	15.	0.	13.8249	0.8889	3.1180	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
62	15.	0.	12.5828	1.1062	4.4346	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
63	O 15.	0.	13.4884	1.1601	2.4719	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
64	15.	1.	14.0000	1.1211	4.5977	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
65	15.	1.	12.9754	1.3453	3.5955	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
66	16.	0.	30.0801	6.1734	8.0818	-1.0000	-1.0000	-1.0000	10.8192	16.7768	7.6145
67	16.	0.	30.2313	4.6725	8.1384	-1.0000	-1.0000	-1.0000	10.5090	17.8356	6.5147
68	P 16.	0.	30.9114	5.1652	7.9485	-1.0000	-1.0000	-1.0000	11.2198	17.1249	6.5174
69	16.	1.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
70	16.	1.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
71	17.	0.	29.4748	4.4791	11.2882	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
72	17.	0.	29.1982	3.9689	9.0369	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
73	Q 17.	0.	29.0575	3.9739	10.0149	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
74	17.	1.	29.3030	4.7310	9.7209	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
75	17.	1.	28.2735	4.9945	10.7916	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
76	18.	0.	27.8549	3.0033	0.2509	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
77	18.	0.	32.0754	4.2424	3.2687	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
78	R 18.	0.	31.3927	3.4967	0.5053	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
79	18.	1.	33.0359	3.4802	1.5317	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
80	18.	1.	30.4808	1.7472	3.0320	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
81	19.	0.	28.9368	4.4974	8.7337	18.9117	22.1787	4.8312	9.6710	13.2197	4.1335
82	19.	0.	28.7675	3.9499	9.1691	12.8299	20.8719	8.2543	8.4471	12.8020	5.2168
83	S 19.	0.	26.2893	3.7583	8.6936	19.0920	15.6659	14.4255	8.7444	13.3294	4.3125
84	19.	1.	28.4025	3.9990	9.0466	22.0261	19.0100	9.0316	8.7339	12.6417	3.7629
85	19.	1.	26.2494	3.7342	8.8146	28.7509	18.8919	14.7348	8.4746	13.2385	3.5211
86	20.	0.	35.4067	9.5491	18.2464	11.4286	46.6321	4.1558	18.6732	17.4528	29.4964
87	20.	0.	38.2212	11.1399	21.9858	41.3098	51.2077	-37.0647	39.9478	41.1168	11.5479
88	T 20.	0.	36.0382	8.3123	14.8437	21.2500	5.5276	14.5679	42.6799	36.3196	39.3564
89	20.	1.	20.7792	0.9479	16.8646	26.9767	1.2225	5.0725	16.8269	32.6923	15.4217
90	20.	1.	36.7647	11.0024	14.6699	-5.5838	3.2828	0.2336	17.1429	16.8269	24.5000
91	21.	0.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	8.1890	11.6328	6.6104
92	21.	0.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	8.2308	11.6137	6.6947
93	U 21.	0.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	8.2227	11.6917	6.2485
94	21.	1.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	8.5519	11.7688	6.0913
95	21.	1.	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	8.5131	12.0884	6.2485
96	24.	0.	26.5967	4.9603	9.4288	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
97	24.	0.	25.9184	4.0959	10.2459	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
98	X 24.	0.	26.1590	3.9868	9.5599	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
99	24.	1.	29.2495	6.0524	9.4126	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000
100	24.	1.	27.2137	3.4373	9.6159	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000	-1.0000

TABLE D.6. Final pH Data, pH Units

CASE	TEST LAB	ANALYSIS LAB	FINAL pH, pH UNITS								
			1.NBS.SI	2.NBS.DI	3.NBS.BR	4.BAS.SI	5.BAS.DI	6.BAS.BR	7.MCC.SI	8.MCC.DI	9.MCC.BR
1	1.	0.	-1.000	-1.000	-1.000	9.200	7.900	5.700	9.400	9.300	6.900
2	1.	0.	-1.000	-1.000	-1.000	9.200	7.800	5.700	9.670	9.300	6.900
3	A 1.	0.	-1.000	-1.000	-1.000	9.200	8.000	5.800	9.700	9.300	7.000
4	1.	1.	-1.000	-1.000	-1.000	9.400	7.900	5.700	9.500	9.400	6.900
5	1.	1.	-1.000	-1.000	-1.000	9.400	8.000	5.800	9.400	9.300	6.900
6	2.	0.	9.200	5.200	6.300	-1.000	-1.000	-1.000	8.400	8.200	7.300
7	2.	0.	9.700	5.300	5.900	-1.000	-1.000	-1.000	8.600	8.200	7.000
8	B 2.	0.	9.400	6.200	6.500	-1.000	-1.000	-1.000	8.500	8.200	7.000
9	2.	1.	9.000	5.400	6.400	-1.000	-1.000	-1.000	8.400	8.200	6.900
10	2.	1.	9.000	5.700	6.200	-1.000	-1.000	-1.000	8.600	8.100	6.900
11	3.	0.	8.800	8.350	5.800	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
12	3.	0.	8.800	8.400	5.800	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
13	C 3.	0.	8.800	8.400	5.800	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
14	3.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
15	3.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
16	4.	0.	8.400	5.800	6.300	-1.000	-1.000	-1.000	8.700	8.400	6.500
17	4.	0.	8.400	5.800	6.300	-1.000	-1.000	-1.000	8.700	8.400	6.500
18	D 4.	0.	8.400	5.800	6.300	-1.000	-1.000	-1.000	8.700	8.400	6.500
19	4.	1.	8.400	5.800	6.300	-1.000	-1.000	-1.000	8.700	8.400	6.500
20	4.	1.	8.400	5.800	6.300	-1.000	-1.000	-1.000	8.700	8.400	6.500
21	6.	0.	9.300	8.100	6.400	9.000	8.100	6.400	9.600	9.500	7.000
22	6.	0.	9.100	8.000	6.400	9.200	7.700	6.400	9.600	9.600	7.100
23	F 6.	0.	9.100	8.100	6.400	9.000	8.100	6.400	9.600	9.600	7.100
24	6.	1.	9.100	7.700	6.400	9.100	7.900	6.400	9.600	9.600	7.100
25	6.	1.	9.100	8.200	6.400	9.100	7.800	6.400	9.500	9.600	7.100
26	7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	8.830	-1.000
27	7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	8.920	-1.000
28	G 7.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	8.900	-1.000
29	7.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	8.950	-1.000
30	7.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	8.940	-1.000
31	8.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
32	8.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
33	H 8.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
34	8.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
35	8.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
36	9.	0.	8.915	8.052	6.272	9.177	8.236	6.656	9.475	9.407	6.807
37	9.	0.	8.884	8.101	6.307	9.110	8.346	6.469	9.386	-1.000	6.876
38	I 9.	0.	8.909	8.026	6.305	9.116	8.371	6.652	9.445	9.261	6.824
39	9.	1.	8.917	8.000	6.288	9.126	8.473	6.591	9.400	9.329	6.774
40	9.	1.	8.882	7.999	6.312	9.203	8.327	6.650	9.413	9.335	6.850
41	10.	0.	9.180	8.570	6.100	-1.000	-1.000	-1.000	9.890	9.600	6.610
42	10.	0.	9.270	8.640	6.090	-1.000	-1.000	-1.000	9.850	9.520	6.490
43	J 10.	0.	9.000	8.630	6.040	-1.000	-1.000	-1.000	10.070	9.570	6.510
44	10.	1.	8.990	8.480	6.090	-1.000	-1.000	-1.000	9.800	9.490	6.520
45	10.	1.	8.950	8.500	6.080	-1.000	-1.000	-1.000	9.750	9.500	6.580
46	11.	0.	7.800	5.300	6.100	7.000	5.300	5.900	8.200	7.700	6.100
47	11.	0.	7.700	5.300	6.000	7.300	5.300	5.900	8.300	7.700	6.200
48	K 11.	0.	7.700	5.300	6.000	7.400	5.600	5.900	8.300	7.700	6.200
49	11.	1.	7.600	5.400	6.100	7.300	5.700	5.200	8.300	7.200	6.100
50	11.	1.	7.500	5.600	6.000	7.300	5.300	5.900	8.300	7.600	6.300

TABLE D.6. (contd)

CASE	TEST LAB	ANALYSIS LAB	FINAL pH, pH UNITS								
			1.NBS.SI	2.NBS.DI	3.NBS.BR	4.BAS.SI	5.BAS.DI	6.BAS.BR	7.MCC.SI	8.MCC.DI	9.MCC.BR
51	12.	0.	9.000	7.710	6.370	9.180	8.360	6.300	-1.000	-1.000	-1.000
52	12.	0.	8.760	8.000	5.050	9.240	8.650	6.660	-1.000	-1.000	-1.000
53	L 12.	0.	8.980	8.160	6.300	9.250	8.150	6.280	-1.000	-1.000	-1.000
54	12.	1.	8.830	7.900	6.410	9.240	8.220	6.420	-1.000	-1.000	-1.000
55	12.	1.	8.920	8.000	5.700	9.240	8.590	6.670	-1.000	-1.000	-1.000
56	13.	0.	8.980	7.570	6.270	9.220	7.220	5.040	-1.000	-1.000	-1.000
57	13.	0.	8.930	7.570	3.700	9.120	6.810	5.500	-1.000	-1.000	-1.000
58	M 13.	0.	9.030	7.600	6.410	9.150	6.970	5.340	-1.000	-1.000	-1.000
59	13.	1.	8.970	6.830	3.450	9.150	7.080	6.060	-1.000	-1.000	-1.000
60	13.	1.	8.900	7.590	4.130	9.380	7.030	5.630	-1.000	-1.000	-1.000
61	15.	0.	8.900	7.160	6.110	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
62	15.	0.	8.850	7.250	6.220	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
63	O 15.	0.	8.890	7.190	6.220	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
64	15.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
65	15.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
66	16.	0.	9.190	8.340	6.110	9.480	7.200	5.950	9.610	9.540	6.550
67	16.	0.	9.030	7.670	6.070	9.380	7.640	6.160	9.560	9.540	6.590
68	P 16.	0.	9.050	8.420	6.130	9.330	6.880	5.900	9.630	9.530	6.570
69	16.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
70	16.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
71	17.	0.	9.000	7.700	6.300	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
72	17.	0.	8.900	7.600	6.300	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
73	Q 17.	0.	8.900	7.600	6.200	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
74	17.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
75	17.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
76	18.	0.	9.500	6.900	6.600	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
77	18.	0.	9.700	7.100	5.800	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
78	R 18.	0.	9.600	7.100	6.300	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
79	18.	1.	9.600	7.100	6.100	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
80	18.	1.	9.600	7.000	5.700	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
81	19.	0.	8.930	7.500	6.190	9.040	7.270	5.720	9.510	9.470	6.760
82	19.	0.	8.980	7.580	6.150	9.100	7.270	5.730	9.490	9.510	6.670
83	S 19.	0.	8.960	7.560	6.150	9.010	7.280	5.710	9.500	9.480	6.670
84	19.	1.	9.000	7.620	6.160	9.190	7.480	5.780	9.560	9.470	6.750
85	19.	1.	8.990	7.570	6.160	8.900	7.530	5.430	9.450	9.410	6.730
86	20.	0.	9.300	7.800	6.000	10.000	7.400	5.700	10.100	9.500	6.800
87	20.	0.	9.000	7.900	6.200	9.900	7.700	5.800	9.800	9.500	6.900
88	T 20.	0.	9.200	7.900	6.100	10.100	7.400	5.700	9.800	9.500	6.900
89	20.	1.	9.100	8.000	6.000	9.800	7.800	5.700	9.900	9.500	6.900
90	20.	1.	9.100	8.100	6.000	10.000	7.700	5.500	9.700	9.500	6.900
91	21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	9.280	9.000	6.640
92	21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	9.280	9.030	6.660
93	U 21.	0.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	9.300	8.990	6.680
94	21.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	9.310	9.060	6.640
95	21.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	9.290	8.960	6.620
96	24.	0.	9.000	7.600	5.800	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
97	24.	0.	8.700	7.500	5.700	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
98	X 24.	0.	8.700	7.600	5.700	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
99	24.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000
100	24.	1.	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000

APPENDIX E

EXAMPLE OF APPLICATION OF THE STATISTICAL SUMMARIES

APPENDIX E

EXAMPLE OF APPLICATION OF THE STATISTICAL SUMMARIES

As a follow-on to the "worst case" cesium example on Page 8.7, a "good case" example is provided by the silicon point (S) at $\overline{EL} = 18$ and $S_L = 5.3 \text{ g/m}^2$ in Figure 8.2. From Table 8.1, we see that this point is for MCC-76-68 Glass in silicon water. Figure 7.1, in the plot for MCC glass in SiW, shows 7 laboratories with relative biases ranging down to $10 S'_r$ units below the consensus average and 5 ranging up to $21 S'_r$ units above it. (Remember S'_r is the standard deviation of laboratory mean, so that about 95 percent of the values plotted would be expected to be within $\pm 2 S'_r$ if there were no laboratory bias.) Lab F does appear to pull the consensus average slightly to the right. Figure 3.21, comparing Lab F's values with the MCC's analysis of RLs, shows that Lab F's value of 30 g/m^2 was about 10 g/m^2 greater than the MCC's value of about 20 g/m^2 . But then, the MCC was about 5 g/m^2 below the consensus average in this case. (See Table 3.4, where the MCC's value is about 13 g/m^2 , and Figure 7.1 where the MCC, Lab S, is about $8 S'_r$ units below the consensus average.) Other facts pertaining to Lab F in this combination are as follows:

- Lab F's Si blank was only 2.3 mg/l , whereas the average blank was 19.6 mg/l .
- Lab F's leachant analyses were consistent with the low blank level.
- A blank of 2.3 mg/l implies a correction of about 1.2 g/m^2 to the elemental loss data. The average blank correction would be 10.5 g/m^2 .
- From the ARR on SL-1, a silicon water, Lab F had an average of 23.0 versus a consensus average of 19.6 mg/l , a nonsignificant bias of 3.4 when compared to the between-laboratory sd of 2.25 mg/l . (In g/m^2 units, such an analytical bias would account for about 2 g/m^2 .)

Since the MCC used Lab F's low blank to correct the RLs analyses, MCC's results for the returned leachates would be at least as large as Lab F's if Lab F did not have an analytical or procedural bias, unless MCC was biased 10 g/m^2 low on its Si analysis of Lab F's RLs. (The MCC may be biased low by as much as 5 g/m^2 .) If we exclude a low blank as the cause of Lab F's high value, then we are left with an analytical or procedural bias as the cause. The ARR results do not indicate a significant analytical bias. The low blank and leachant values point to leachant preparation as a possible contributing cause, but do not exclude excessively rough specimens or other procedural artifacts.

If Lab F is removed from this example case, the consensus average drops about 1 g/m^2 , from 18.11 to 17.04 g/m^2 . S_r increases only slightly (from 1.01 to 1.03, due to a loss of degrees of freedom), but S_L decreases 1.36 g/m^2 from 5.27 to 3.91 g/m^2 , or relative to the consensus average, from 29.1 to 23.0 percent.

This example is by no means an isolated case in the RR results. A similar procedure can be followed by each laboratory using the numerical data in this report and its own laboratory data on the blanks and leachants to evaluate possible sources of poor precision statistics. A participating laboratory conducting such an investigation also would be able to use its own records and previous experience to help identify sources of bias.

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