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MCC
Materials Characterization Center

Meeting on Impact Testing of Waste Forms

Summary Report

October 1981

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

Pacific Northwest Laboratory
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MATERIALS CHARACTERIZATION CENTER

MEETING ON IMPACT TESTING OF WASTE FORMS

SUMMARY REPORT

M. D. Merz
D. Atteridge
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U.S. Department of Energy
under Contract DE-AC06-76RL0 1830

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1.0 SUMMARY

A meeting was held on March 25-26, 1981 to discuss impact test methods for waste form materials to be used in nuclear waste repositories. The purpose of the meeting was to obtain guidance for the Materials Characterization Center (MCC) in preparing the MCC-10 Impact Test Method to be approved by the Materials Review Board. The meeting focused on two essential aspects of the test method, namely the mechanical process, or impact, used to effect rapid fracture of a waste form and the analysis technique(s) used to characterize particulates generated by the impact.

1.1 RECOMMENDATIONS

- The impact test for waste forms should produce fracture of the waste form tested to provide comparative data on amount of respirable fines and surface area increase. Load or energy to fracture was not considered relevant to selection of waste form or to risk assessment.
- Testing of waste-form-filled, scaled canisters will be necessary to relate intrinsic properties such as fracture strength and fracture toughness to full scale impact behavior. Such testing was considered advanced relative to current experience, and MCC-10 will not include such testing.
- The impact test method should specify a particular pre-impact potential energy (height and weight). This energy should be selected on the basis of producing fracture in the "strongest" waste form.
- The selection of an axially loaded cylinder rather than a diametrally loaded cylinder was preferred on the basis of uniformity of stress in the specimen volume. However, the selection of specimen loading technique may depend on reproducibility.
- Respirable fines should be measured by sampling airborne particles, if possible, to obtain data on aerodynamic diameters of particles generated.

- Sieving analysis of particles generated will be appropriate to obtaining surface area increases.

1.2 CURRENT STATUS OF MCC-10 IMPACT TEST METHOD

The MCC has done further testing to develop the MCC-10 Impact Test Method since the March, 1981 meeting and a summary of the testing and the bearing on MCC-10 will be given here. The following conclusions on the test method have been made:

- Testing with a 795-kg (1750-lb) weight falling on a glass, 1.9-cm-diameter x 4.1-cm-length, axially loaded cylinder from 30.5 cm has indicated that about 7 to 8% of the available kinetic energy is absorbed by the specimen, or approximately 12 joules/cm^3. The reproducibility of loading rate was 5% and of peak loads was 7%. In these tests, the weight was arrested after fracture to prevent further comminution of the specimen. Tests on smaller, 1.3-cm-diameter x 3.2-cm-length cylinders also were reproducible and higher energy per unit volume was absorbed by the smaller specimens, approximately 15 joules/cm^3. The smaller specimen size will be specified in MCC-10. Specimen modulus matching end supports, or compliance pieces, will not be required.

- Currently, the MCC anticipates that 230 kg (500 lb) will be specified in MCC-10 rather than the larger 770-kg weight. Testing is in progress to assure reproducible loading rates and a relatively constant amount of absorbed energy in the energy range corresponding to 230 kg dropped from 30.5 cm. Results to date also indicate absorption of approximately 15 joules/cm^3 for these conditions for the smaller size pyrex glass specimens.

- A cyclone separator attached to the collection chamber was used to collect airborne respirable fines at the time of impact. The particles retained in the chamber were analyzed by sieving and aerosol generation, with respirable fines collected with a cyclone separator. Approximately 50% of the fraction of respirable fines less than 3.5 μm aerodynamic diameter remained in the collection chamber

and was not collected by the cyclone at the time of impact. Agglomeration and trapping prevented all potentially airborne particles from leaving the chamber. As a result of these findings, the MCC-10 Impact Test Method will specify collection in a closed chamber with analysis of collected particles by sieving and aerosol generation for respirable fraction. No sampling of airborne particles will be done at the time of impact.

- The MCC-10 Impact Test Method will specify a loading rate capability for the test fixture and foundation. Attainment of loading rate must be confirmed by the experimenter before testing can be done. A suitable method will be to instrument a relatively rigid steel cylinder with strain gages to confirm adequate loading rate. A load cell is not anticipated as necessary equipment for performing tests by MCC-10.

2.0 INTRODUCTION

The Materials Characterization Center has responsibility for developing test methods to characterize materials to be used in nuclear waste repositories. The test methods and data must be approved by the Material Review Board prior to publication in the Nuclear Waste Materials Handbook.

To develop test methods, the MCC solicits input from experts and persons familiar with waste disposal needs by holding workshops and meetings. For the purpose of developing an impact test method for waste forms, the MCC held meetings on impact testing on March 25-26, 1981 at Pacific Northwest Laboratory (PNL). The MCC encouraged comments on the draft MCC-10 Impact Test Method and encouraged open discussions to further select test parameters.

2.1 PURPOSE OF MEETING

The MCC held the Impact Test Meeting to solicit scientific opinion on the proposed MCC-10 Impact Test Method. The participants were asked to contribute to the discussion along the following lines, which were anticipated by the MCC to be the main issues.

1. Review presented information and contribute information on impact of waste forms.
2. Review and comment on a proposed MCC-10 Impact Test Method and the bases for selection of various proposed test parameters and techniques.
3. Reveal to MCC any important additional criteria for impact testing that have been overlooked or are not included in the scope of MCC-10, relevant to either the impact conditions or fines analysis.
4. Provide comments and additional information to the MCC on whether the proposed MCC-10 will rank waste forms in a way that corresponds to possible accidents and large scale impact and whether it can provide a data base that can be used to assess the risks associated with shipping and handling. Comment on whether this test also gives valid results on:

- the amount of respirable fines produced by real, potential impacts during shipping and handling.
- the increase in surface area that will possibly degrade chemical durability.

5. Provide comments and recommendations on MCC-10 and impact testing of waste forms.

The participants were given opportunities, both during the course of the presentation and during the discussion, to make comments and recommendations on test methods relevant to mechanical testing and fines analysis.

2.2 ORGANIZATION OF THE MEETING

The meeting was organized according to the agenda, Appendix C. The presentations were intended to provide a common basis for discussion and participants were given opportunities to comment during the course of each presentation. The MCC recorded significant contributions and prepared a summary statement at the end of the first day. This summary statement provided the basis for discussions during the second day.

After the meeting the MCC prepared a second summary of the meeting with the recommendations of the participants. This summary was mailed to the participants for further comments. The recommendations are given in Section 4. Additional comments are recorded verbatim in Section 5.

3.0 PRESENTATIONS BY INVESTIGATORS

To establish common ground for discussion of impact testing of waste forms, approximately one-half of the first day was a review of impact test methods, data obtained and limitations of the data. The visual aids used by each speaker and the notes taken by an MCC recorder are used to summarize the presentations given at the meeting. The significant issues are summarized and are not intended to represent in detail the full presentation given by each speaker.

3.1 SUMMARY OF PRIOR IMPACT TESTING OF WASTE FORMS

M. D. Merz, MCC, presented a summary of the impact test data reported in the MCC publication, State-of-the-Art Review of Materials Properties of Nuclear Waste Forms, PNL-3802, April 1981. Methods and data are summarized in Figures 1, 2 and 3. The variety of methods made it difficult to compare the wt% fines less than 10 μm for the various waste forms. Lack of standardization of testing was a limitation of currently available data. No systematic study has been made to determine the effect of test parameters such as energy input or impact velocity for crushing type tests such as done by Bunnell, Wallace and Kelley, Ramm and Ferenczy, and Jardine and Mecham. The lack of a standardized test method makes comparison of waste forms difficult. The applicability of the data to accidents has not been established.

L. Jardine, ANL, summarized studies on impact testing of glass and the methods used for fines analysis. He presented typical results, summarized known problem areas and proposed some recommendations for the MCC-10 Impact Test Method. Problems with drop weight tests that were mentioned were: energy losses are unknown, loss of fragments is suspected and characterization of fragments is difficult for the small particle sizes. The objective of ANL studies has been to obtain the fraction of fragment sizes in the respirable, or airborne, size range and to measure the increase in surface area. Jardine indicated that independent test variables were impact energy, specimen size and shape, specimen orientation, impact device, velocity and mass of impacting device and specimen composition. The ANL apparatus consisted of a falling

weight that fractured the specimen in a closed chamber, as shown in Figure 4. The tests used a diametrally loaded specimen, chosen for the convenience of effecting fracture with a relatively small falling weight (10 kg).

The method for particle analysis involved wet sieving followed by the combination of a Coulter counter and microscopic examination (Figure 5). Data plotted on lognormal probability paper typically gave a linear relation (Figure 6). A summary of results for three glass types was presented (Figure 7).

Jardine pointed out that surface area measurement is difficult, considering material differences and collection problems. Methods mentioned were BET and microscopic examination. He indicated that the literature supports the idea that surface area increase is directly proportional to impact energy absorbed by the specimen.

In summary, Jardine recommended that the MCC-10 Impact Test Method include:

- specification of available impact energy/specimen volume ($\sim$10 j/cm^3)
- diametrally loaded impact specimen
- toleration of modest specimen size variations (as a practical matter)
- lognormal size analyses.

3.2 PROPOSED MCC-10 IMPACT TEST METHOD

G. Dudder, PNL, presented a summary of the proposed MCC-10 Impact Test Method (Appendix A). The MCC had done only limited verification testing with the test fixture through Effects Technology Incorporated and had not selected any final test parameters such as velocity, mass and particle analysis method(s). However, the draft contained significant departures from past tests in the following areas:

- A cylinder will be impacted axially rather than diametrally.
- A relatively large mass, 200 to 1000 kg rather than 10 to 20 kg will be used to fracture the specimen.
- A rigid specimen support will be specified if necessary.
- Preferably, particle analysis will be done by direct airborne sampling at the time of impact. However, subsequent determination of

respirable fraction from the collected, total specimen mass by sampling a generated aerosol may be necessary. Sieving analysis will be an essential part of test development and will probably be part of the test method.

The MCC, in selecting such test conditions, contended that a uniformly stressed specimen was more meaningful than a diametrally loaded specimen since the fines produced are referenced to specimen volume, or mass, under stress during impact. A large falling mass was preferred to provide a reproducible, essentially constant, high loading rate during the impact. The intent of the proposed MCC test is to subject the waste form to a reproducible impact condition. Obtaining the respirable fraction from an airborne sample was selected because of uncertainties in obtaining aerodynamic diameter from sieving and Coulter counter methods. Physical size data cannot be related reliably to aerodynamic diameter without density and shape factor data for particles generated.

3.3 DYNAMIC TEST FIXTURE DESIGN AND RESULTS

W. Adler of Effects Technology Inc. (ETI) presented the results of fixture design and verification testing done for the MCC by ETI. The test data, with no fines analysis, consisted of load-time records taken from impacts performed on pyrex, alumina and glass-bonded mica. The object was to demonstrate reproducibility of loading to verify stressing of the specimen volume to a stress measurable by an external load cell, and to verify capability of the fixture to withstand anticipated loads. The load cell may not be an essential element in the MCC-10 Impact Test Method, but was an essential test-development, diagnostic aid. Typical load-time traces are shown in Figures 8 and 9. A schematic of the test fixture is shown in Figure 10. The fixture performed adequately and load-time records provided information on energy absorbed by the specimen. The absorbed energy varied from 0.4 to 5% of the available energy and was different for each material tested. Maximum loads were reproducible to within approximately 5 to 10% for alumina and pyrex.

Considerable discussion ensued as a result of W. Adler's presentation on use of diametrally loaded specimens versus axially loaded cylinders. The principal argument, though as yet unsubtantiated, was that the diametrally loaded specimen will provide more reproducible results than the axially loaded specimen; the basis for selection of axial loading was uniformity of stressing with resultant wt% fines normalized with respect to a specimen volume under relatively uniform stress rather than the highly nonuniformly stressed volume in the diametrally loaded specimen. There were no confirming experimental data for selection of either the diametrally loaded specimen or the axially loaded specimen with respect to reproducibility of fines generation in lab-scale experiments.

3.4 RESPIRABLE FINES AND SURFACE ANALYSIS TECHNIQUES

J. Briant and O. Moss, PNL, outlined the MCC plan for characterizing the particles produced by impact fracture. Data will be obtained on both wt% respirable fines and increase in surface area. A basic assumption was that several kinds of waste forms, such as glass, ceramic, concrete, and glass-ceramic, must be compared and that two basic questions must be answered:

1. What is the mass fraction of respirable fines produced?
2. What is the specific surface?

Two general approaches were proposed for characterizing the particles produced by impact fracture, analysis of a slurry and analysis of an aerosol. For each approach, size and surface area could be measured. The overall scheme envisioned for particle analysis is shown in Figure 11. Considerable background information was presented on the determination of respirable fraction by cyclone separation. This technique can be used directly on the airborne particles produced by impact and on particles collected and made airborne in a fluidized bed aerosol generator. Consideration of the necessity for knowing aerodynamic diameter which depends on particle dimension, shape and density was the primary justification for recommending an aerodynamic size classifier (Figures 12 and 13) rather than sizing techniques such as sieving and a Coulter counter. The analysis of a hypothetical sample, based on size distributions typical of ANL data, was presented (Figure 14). Surface area determination

could be derived from sieving analysis combined with assumption of a lognormal distribution over the range of sizes that contribute significantly to surface area.

Professor V. Marple, University of Minnesota, presented a summary of commercially feasible methods for particle selection and analysis. His background in design of particle classifiers provided the participants with considerable evidence of the practicality of designing and using with confidence airborne particle classifiers for determining respirable fraction and for obtaining data that will be accepted as relevant to aerodynamic diameter and inhalation hazards.

- **DROPPING OF GLASS-FILLED CANISTERS (T.A. SMITH & W. ROSS) 1/2 SCALE (15cm DIA. × 150cm, 150 kg)**
- **ROTATING ARM IMPACT OF GLASS-FILLED CANISTERS (T.A. SMITH & W. ROSS) 1/6 SCALE (5cm DIA. × 10cm, TO 35 m/s)**
- **DROPPING OF FULL SCALE GLASS-FILLED CANISTERS (R. BUNNELL) (40cm DIA. × 270cm, 7.6m DROP)**

FIGURE 1. Large Scale Impact Tests on Waste Forms (M. D. Merz, MCC)

- **SINGLE & MULTIPLE IMPACTS ON ENDS OF CYLINDERS (1cm×3cm)**

BUNNELL - PNL	**BOROSILICATE GLASS WITH CALCINE**
WALLACE & KELLEY - SRL	**VARIOUS CONCRETES WITH SLUDGE**
	BOROSILICATE GLASS WITH SLUDGE
RAMM & FERENCZY - AUSTRALIA	**SYNROC CERAMIC WITH WASTE**

- **SINGLE & MULTIPLE IMPACTS ON DIAMETERS OF CYLINDERS (4cm×6cm)**

JARDINE & MECHAM - ANL	**PYREX, QUARTZ**
	VARIOUS WASTE FORMS IN PROGRESS

FIGURE 2. Small Scale Tests (M. D. Merz, MCC)

WASTE FORM	IMPACT ENERGY/VOLUME	IMPACT VELOCITY (m/s)	WT% FINES < 10μm	SURFACE AREA ENERGY	SURFACE AREA INCREASE (RATIO NEW/OLD)
BOROSILICATE GLASS +304 CANISTER	3200J/2.5×10⁴cm³ (10 ft DROP)	7.6 36	0.001 2	— —	1.3 TO 3 10 TO 100
BOROSILICATE GLASS IN FULL SCALE CANISTER	—	12.2	0.13	—	24.3cm²/g
BOROSILICATE GLASS WITH PW-7-2 CALCINE	217J/1.14cm³	4.88	2.4	16cm²/J	574
BOROSILICATE GLASS + SLUDGE	78J/3cm³	4.0	0.0*	9.5cm²/J	58
SYNROC CERAMIC SINTERED	217J/1.53cm³	(~5)	2	—	—
SYNROC CERAMIC HOT PRESSED	217J/1.53cm³	(~5)	1	—	—
HAC CONCRETE +40% SLUDGE	94J/5.68cm³	4.0	0.1	28.9	154
IP CONCRETE +40% SLUDGE	94J/5.65cm³	4.0	0.4	19.4	103
TYPE III CONCRETE +40% SLUDGE	94J/5.80cm³	4.0	0.63	25.3	136

FIGURE 3. Impact Data on Waste Forms (M. D. Merz, MCC)

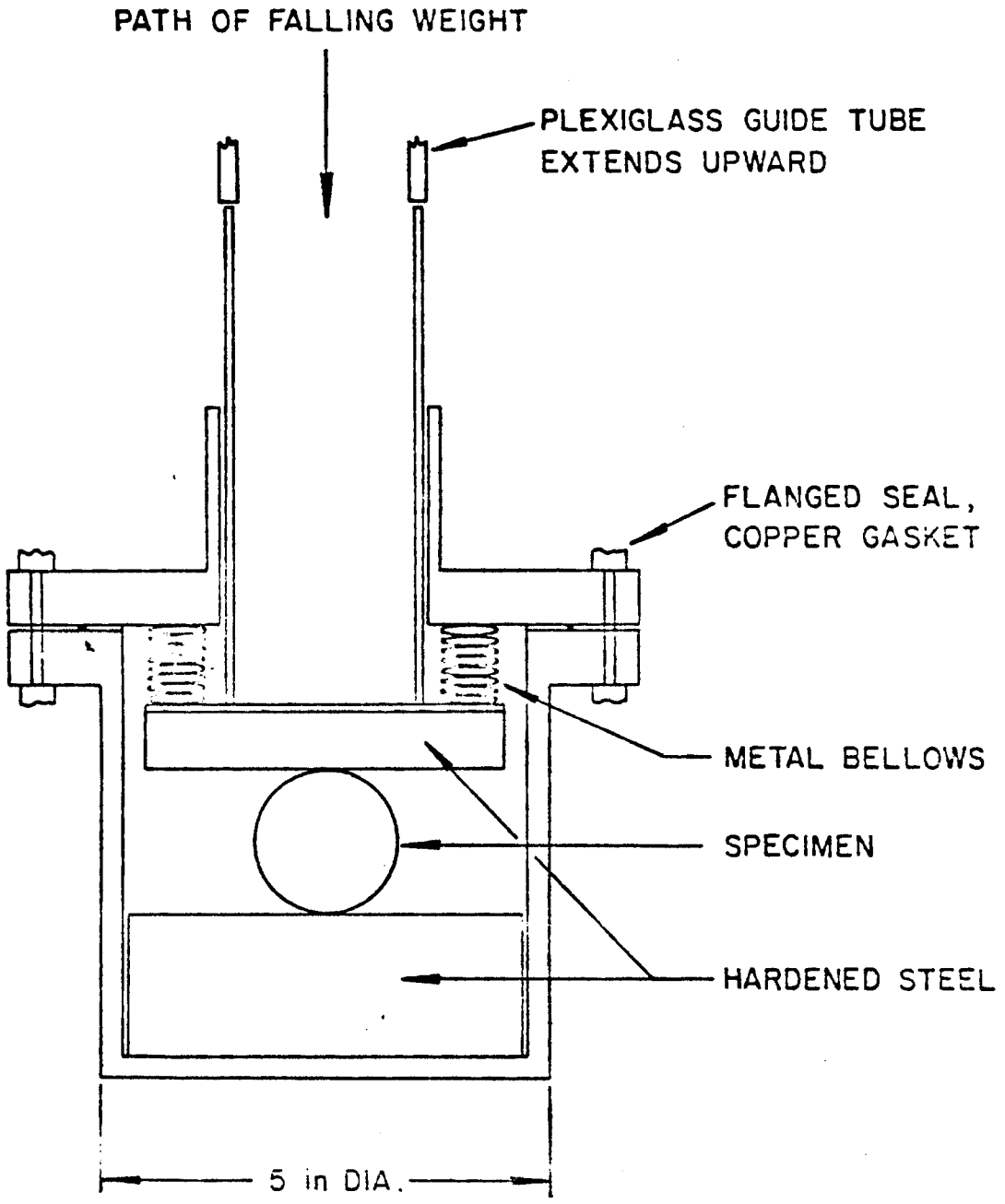

FIGURE 4. Falling Weight in a Closed Chamber (L. Jardine, ANL)

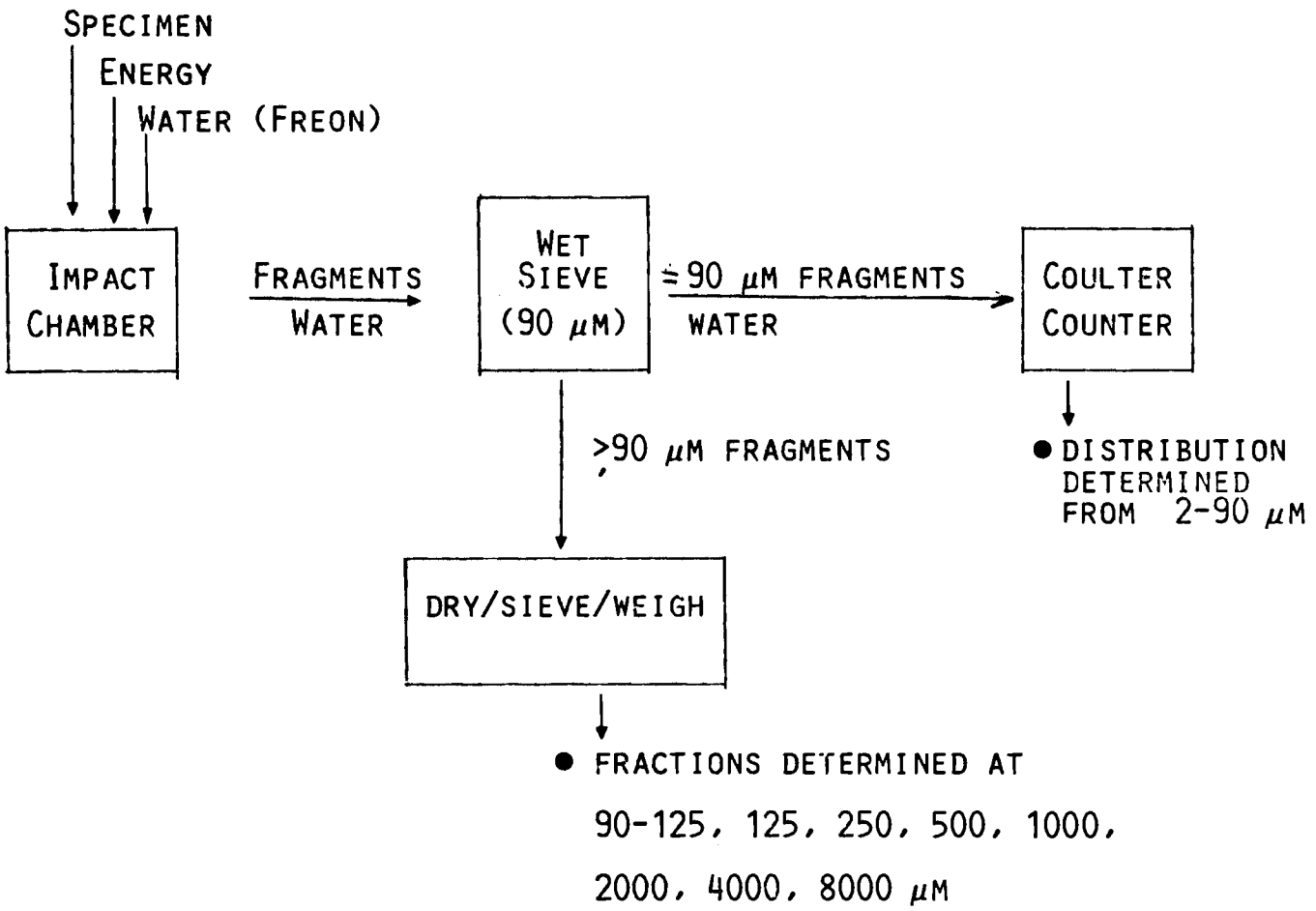

FIGURE 5. Typical Procedure for Size Analysis (L. Jardine, ANL)

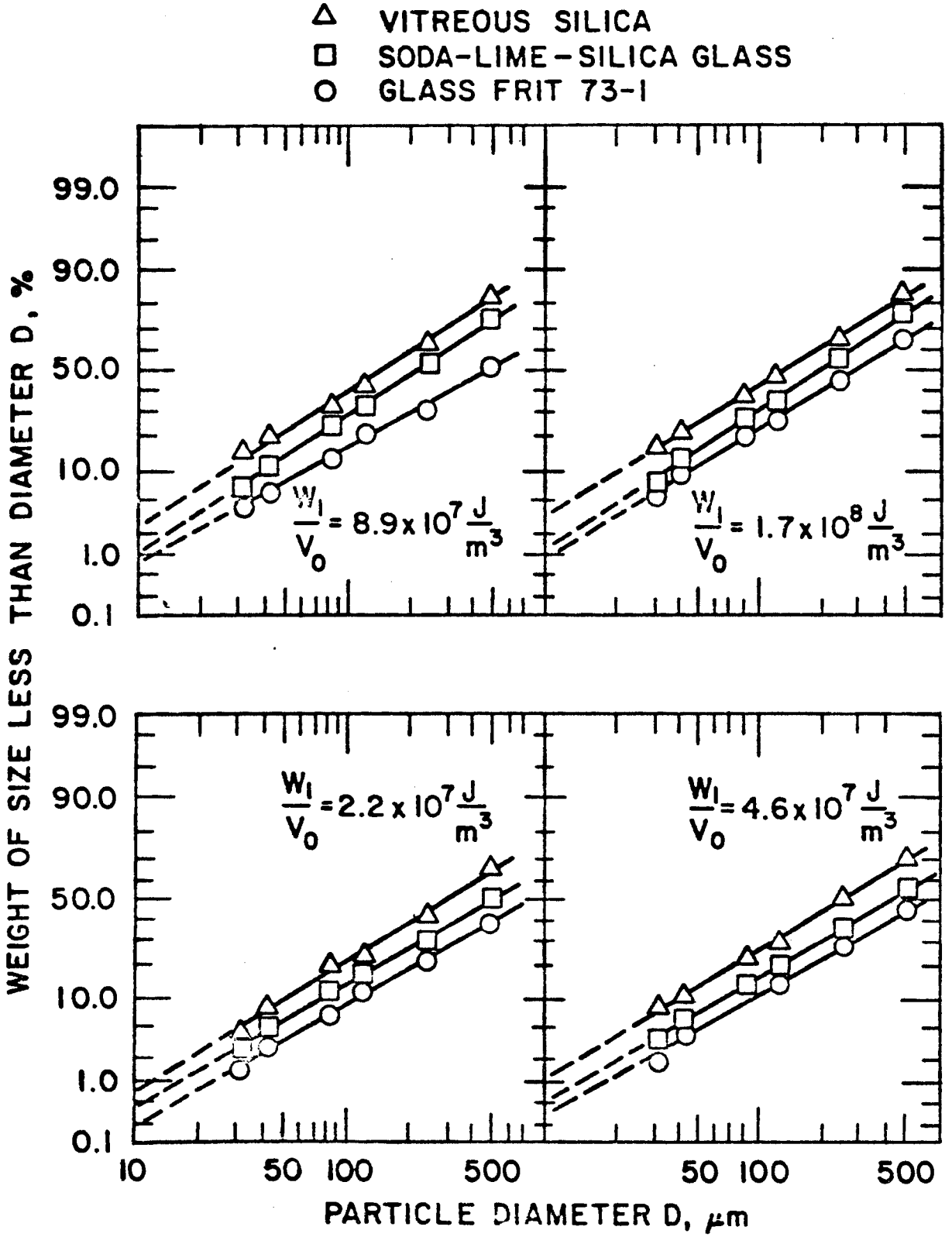

FIGURE 6. Lognormal Size Distribution for Three Glasses (L. Jardine, ANL)

Glass Type	RESULTS			
	$W/V_0 \times 10^6$ (J/m^3)	Wt % ≤ 10 μm	Dg (μm)	σg
Vitreous Silica	170	3.2	135	4.1
	89	2.8	150	4.0
	46	1.4	220	4.0
	22	0.6	330	4.0
Soda-Lime Silica Glass	170	1.2	192	3.7
	89	0.8	220	3.7
	46	0.6	370	4.1
	22	0.4	490	4.4
Glass Frit (73-1)	170	1.0	290	4.3
	89	0.4	490	4.4
	46	0.4	530	4.4
	22	0.2	720	4.2

FIGURE 7. Summary of Fines Produced by Impact for Three Glasses (L. Jardine, ANL)

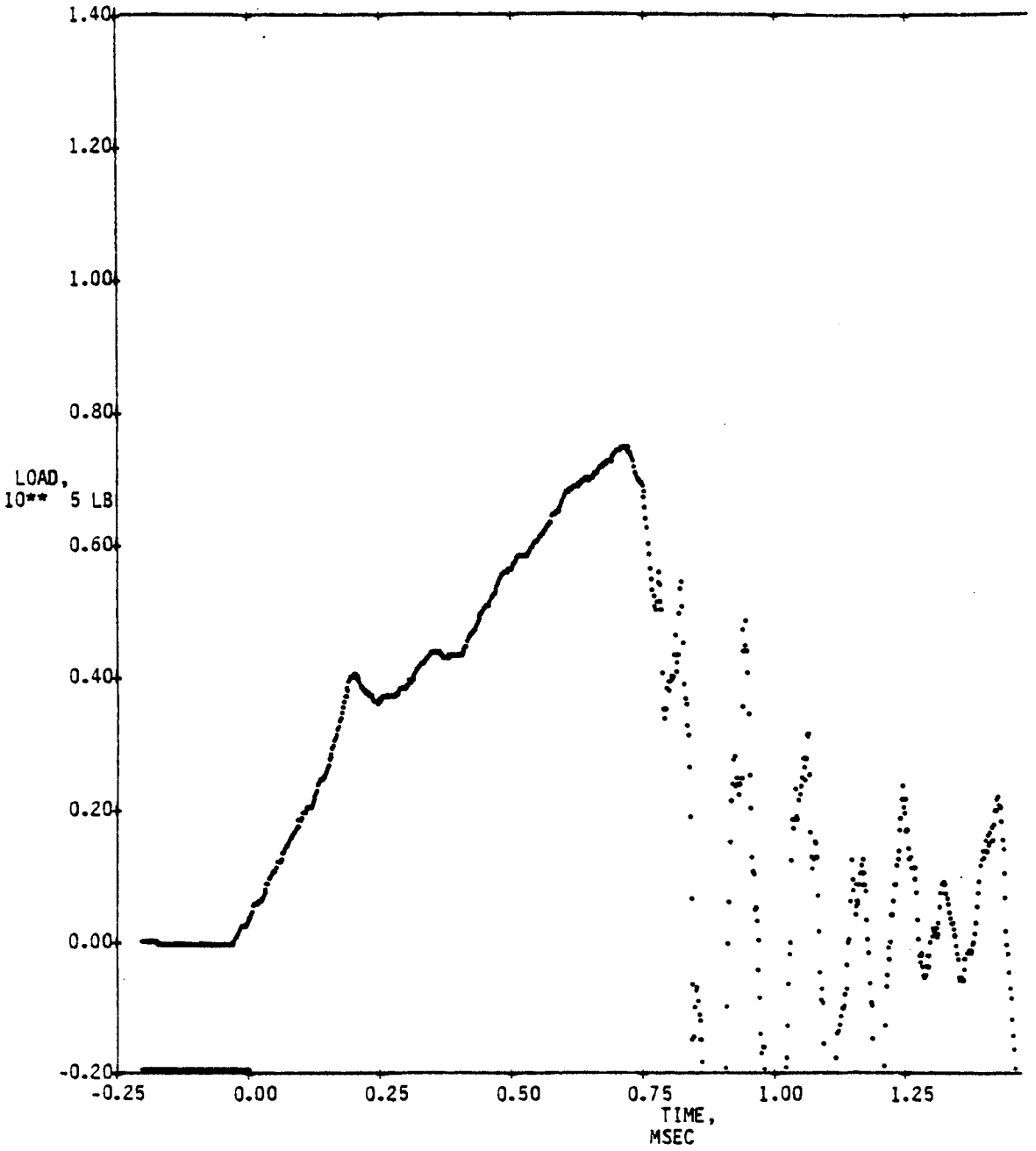

FIGURE 8. Load-Time Trace for Pyrex B (W. Adler, ETI)

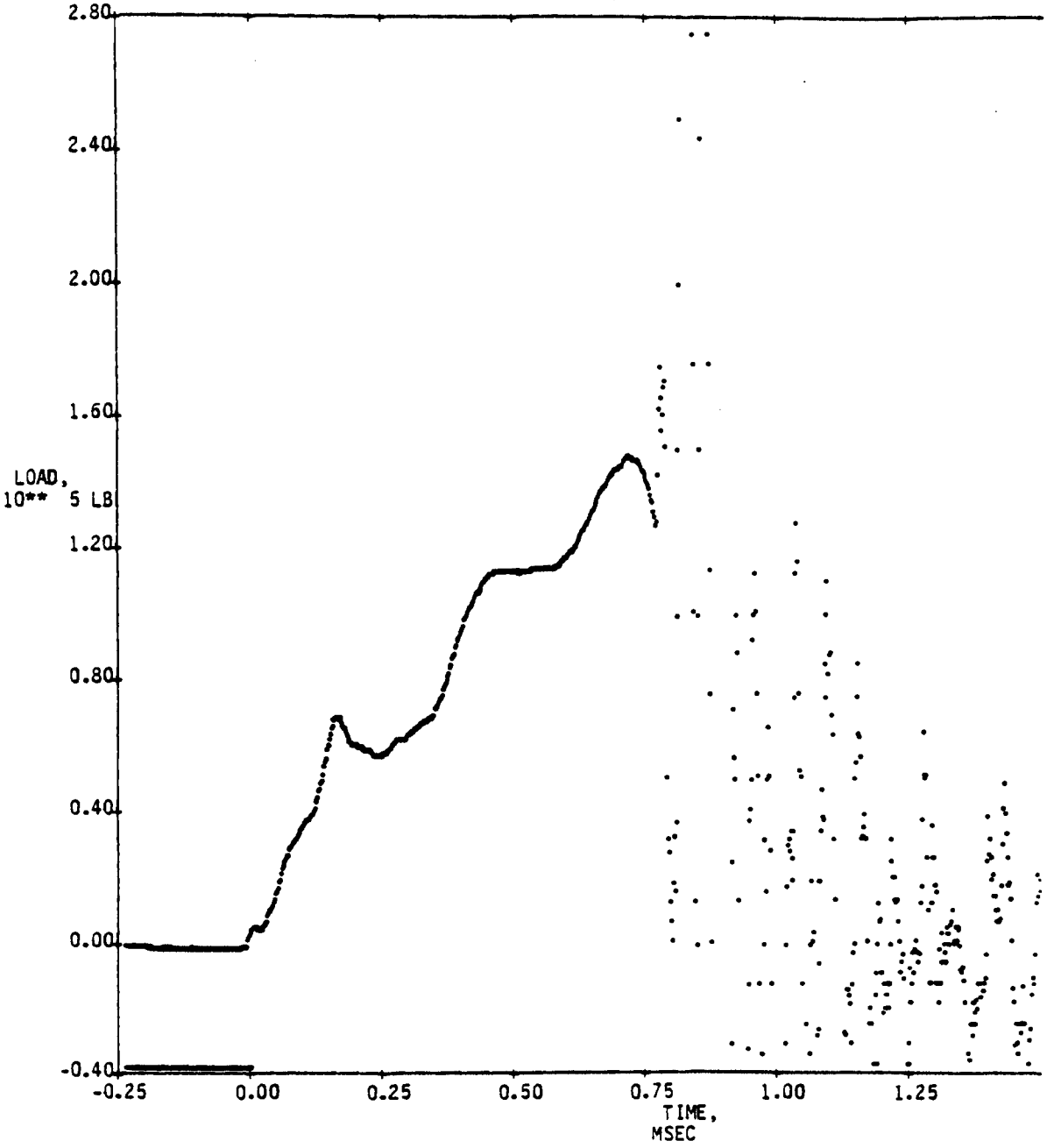

FIGURE 9. Load-Time Trace for Alumina B (W. Adler, ETI)

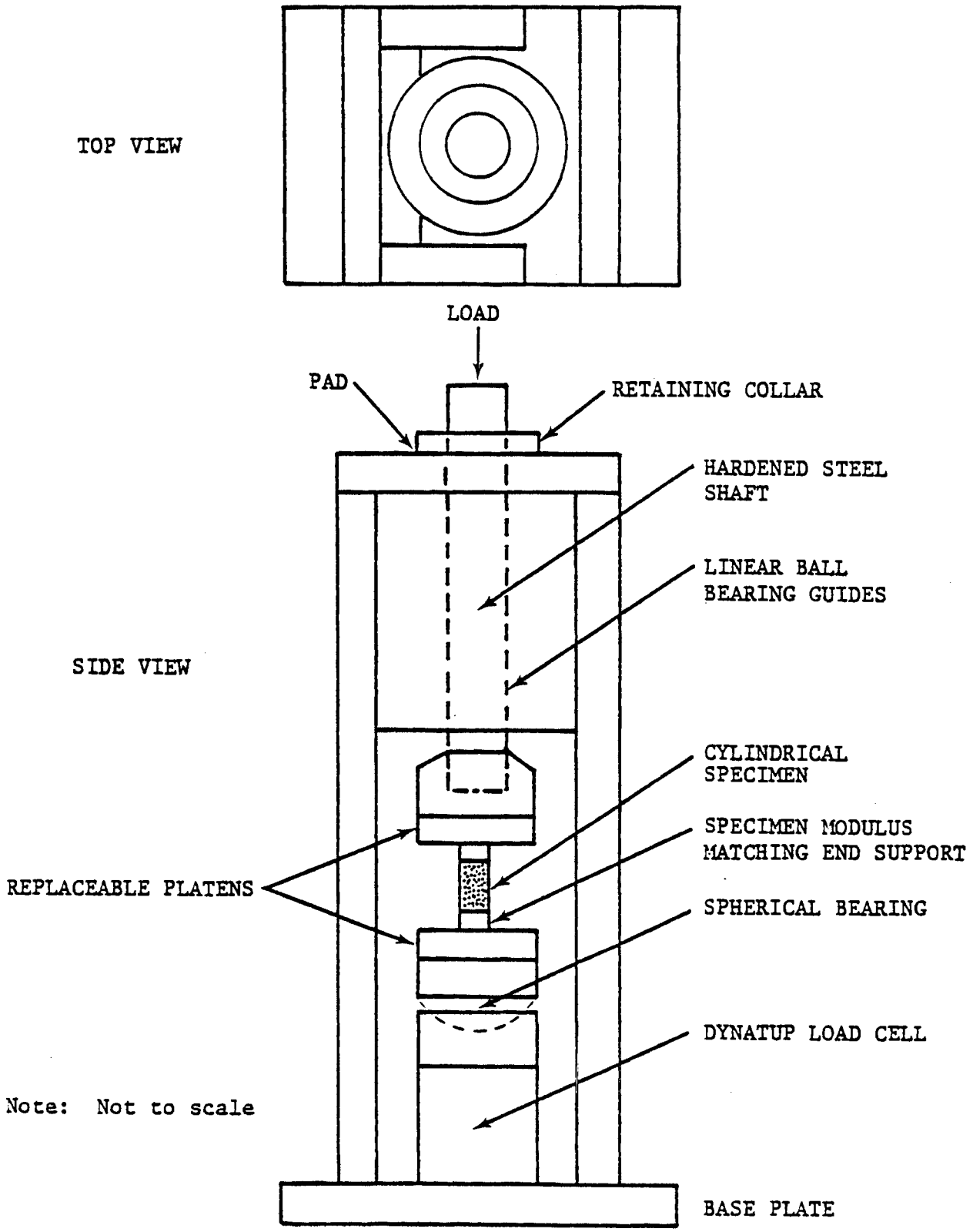

FIGURE 10. Schematic of the Compression Test Fixture (W. Adler, ETI)

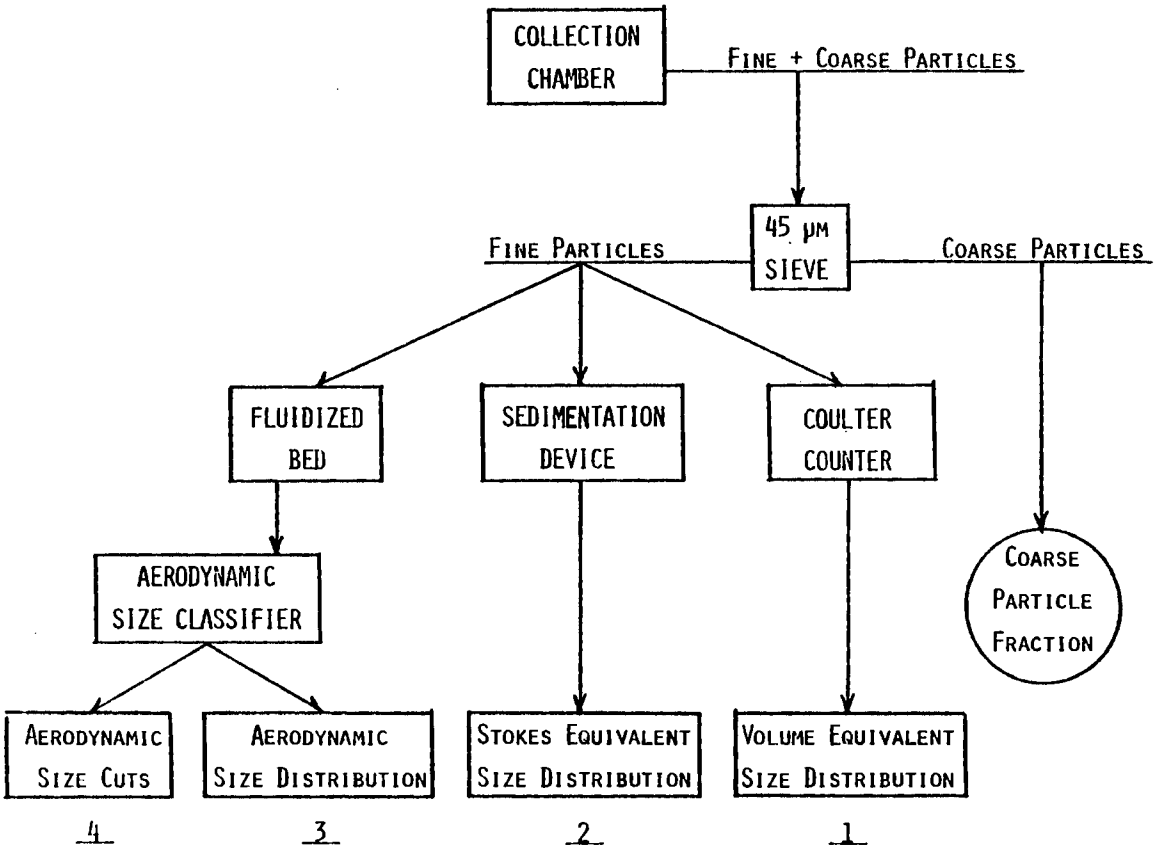

FIGURE 11. Scheme for Particle Analysis (J. Briant and O. Moss, PNL)

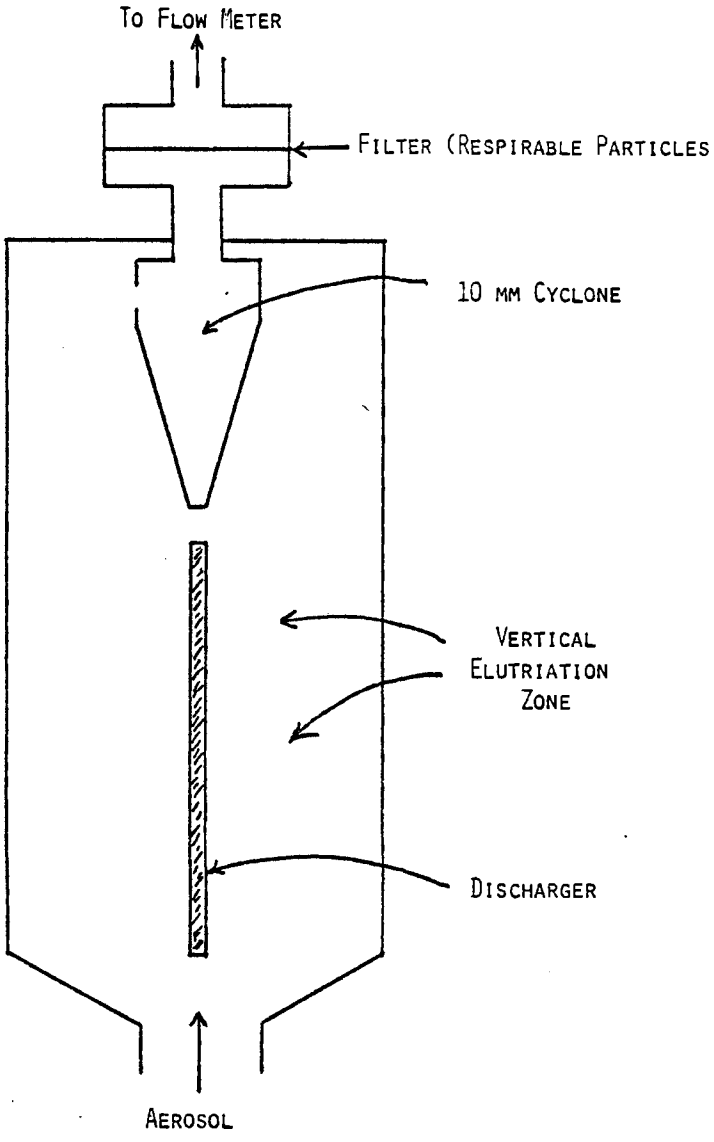

FIGURE 12. Aerodynamic Size Classifier (J. Briant and O. Moss, PNL)

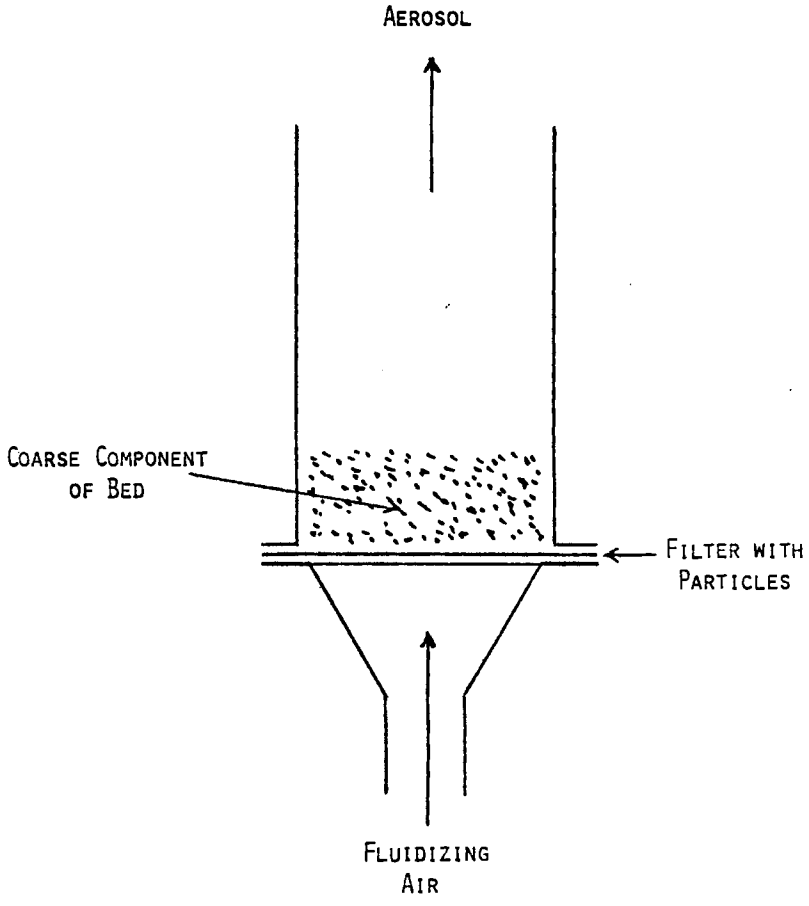

FIGURE 13. Fluidized Bed Aerosol Generator (J. Briant and O. Moss, PNL)

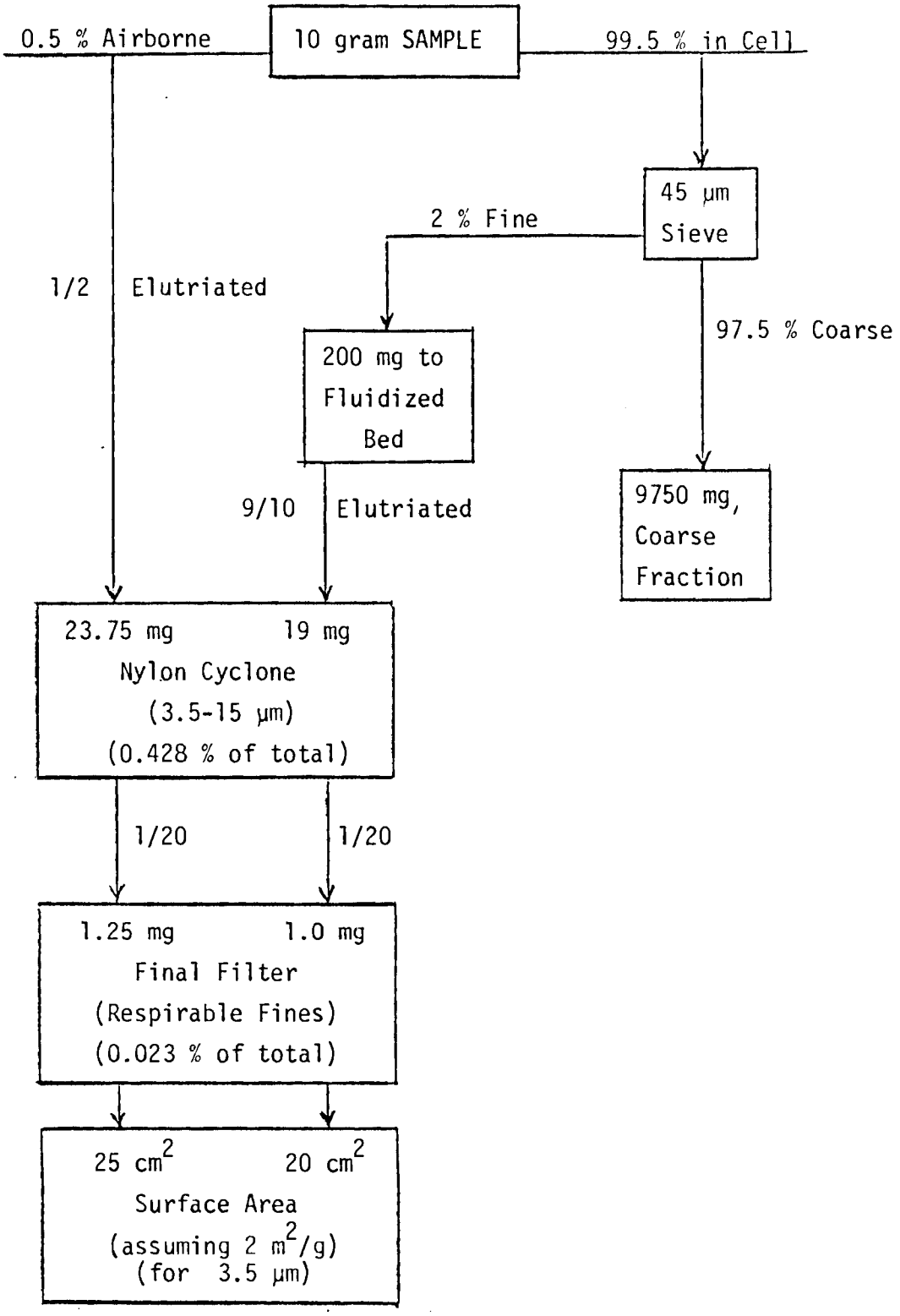

FIGURE 14. Hypothetical Mass Balance of Fines Generated by Impact (J. Briant and O. Moss, PNL)

4.0 RECOMMENDATIONS OF PARTICIPANTS

To gain some degree of consensus from the participants, a summary statement was prepared and discussed on the second day. As a result of these discussions a second summary statement was prepared and mailed to the participants subsequent to the meeting for further comment. Two participants provided additional comments. These comments are included verbatim in Section 5.0. This section presents the recommendations taken as the consensus of the group with the exceptions noted in Section 5.

4.1 IMPACT METHOD

The attached draft of MCC-10, Appendix A, describes the method and equipment proposed for MCC-10. The test fixture is essential to proper loading. The method of delivering the specified impact load to the fixture can vary from laboratory to laboratory. Certain minimum foundation requirements will be specified to ensure reproducibility.

The following summary statements were generally accepted in the discussions on March 26.

- Every brittle waste form tested shall fracture. The primary purpose of MCC-10 is a test for brittle waste forms. The test could be applied to "ductile" waste forms such as metal matrix waste forms but fines are not likely to be generated in such a test. The weight and height (energy input) will be specified in MCC-10 to ensure that fracture occurs for conceivable brittle waste forms. Waste forms will be evaluated by MCC-10 on the basis of fines analysis, not load-to-fracture or energy absorbed or any other mechanical property. More elaborate testing of scaled canister and waste form will be necessary to relate intrinsic waste form properties such as fracture toughness and strength to full scale impact behavior. Such tests are beyond the scope of MCC-10.
- MCC-10 will specify a certain energy (height and weight) for the impact. The basis for selection of the energy will be that the "strongest" waste form fractures in the test.

- The energy selected for the test cannot, at this time, be selected on the basis of simulating real accidents other than providing sufficient energy to fracture the specimen.
- One specimen size is preferred for MCC-10. However, this restriction may be relaxed if verification tests can justify some variation in specimen size to avoid unnecessary costs in specimen preparation.
- Selection of the specimen size will be based on achieving reasonable loads during impact and having a specimen for which each dimension is at least ten times the dimension of the largest microstructural inhomogeneity of the waste form.
- There is currently no method or data to predict the results of large scale accidents from a small scale test, though scaled tests of canisters and waste form are possible. On a ranking basis, the results of MCC-10 can be expected to give the correct relative performance of waste forms in a full scale accident. The only reservation expressed was that the effects of triaxial stress state, which are likely to be present in real waste forms in canisters, are not included in MCC-10. Tests with a confining cylinder of canister material around the waste form can assess whether this is an important factor, but such testing is beyond the scope of MCC-10.
- The selection of an axially loaded cylinder (as specified in the draft MCC-10 Impact Test Method) rather than a diametrally loaded cylinder was debated. Reproducibility of testing was of concern and may be a deciding factor in selection. However, the axially loaded cylinder is superior in concept and is preferred because the specimen volume is more uniformly stressed and because the amount of respirable fines and increase in surface area are referenced to the original specimen weight (i.e., volume). The axially loaded cylinder will be used if adequate reproducibility is demonstrated in verification testing.

4.2 FINES ANALYSIS METHOD

The methods for measurement of respirable fraction were ranked as indicated:

1. Direct sampling with cyclone
2. Direct sampling with impactor
3. Washing of fines + sieving + cyclone
4. Washing of fines + sieving + impactor
5. Washing + sieving + sedimentation
6. Washing + sieving + Coulter counter
7. Washing + sieving + extrapolation of lognormal distribution

Methods 5 and 6 may be reversed if the amount of respirable fines is too small for sedimentation analysis. The Coulter counter requires only a small volume of material for analysis and this is an advantage that could override other considerations. Overall materials balance is of great importance in fines analysis and enhances greatly the credibility of particle analysis.

The methods for surface measurement were ranked as indicated:

1. No sieving, gas adsorption
2. Lognormal analysis of seiving data plus data on respirable fines
3. Lognormal analysis of sieving data and Coulter counter data

Method 1 gives specific surface area, and though it is the most reproducible and is a standard method, it gives the highest value for surface area and includes area that may not be accessible to leachants. Particle size can be determined in a relatively straightforward manner to sizes as small as 100 m by sieving; determination of smaller sizes requires either data from a Coulter counter or extrapolation of sieving data according to an assumed particle size distribution, e.g., lognormal, data from the airborne sampling or data obtained from sedimentation analysis. Complete particle size characterization data over the entire size range may be necessary to assure material balance. Sieving analysis and Coulter counter analysis have the disadvantages of possible, unknown effects of the solutions on the waste form and the necessity of assuming a shape factor to arrive at surface area. Method 2 was considered most appropriate for MCC-10.

5.0 COMMENTS FROM PARTICIPANTS SUBSEQUENT TO MEETING

Additional comments were received from E. Wilmot, Sandia National Laboratory and Bom Soon Lee, Brookhaven National Laboratories after the meeting. These comments are presented verbatim in this section.

5.1 E. WILMOT, SANDIA NATIONAL LABORATORY

Specific Requirements for Risk Assessment

At the Transportation Technology Center (TTC), we are particularly concerned about the characteristics of materials that may well end up as contents in our casks and packagings. The concern focuses on two requirements: for licensing of packagings and for risk assessments that are included in environmental impact statements. Fortunately, the data needs for each of the components are essentially the same: particle size distribution, mass fraction of material that is less than 10 microns mean aerodynamic diameter, and increased surface area. The first two types of data are by far the most important to us, at least for HLW forms, because submersion scenarios with ingress of water are not considered in our transportation risk assessments and because the casks used to transport HLW are not cooled by water. I think it is appropriate to mention that we are interested in measuring all particles that are less than 10 microns (AMAD) even though they may not become airborne during the impact test itself. In a discussion with Owen Moss, the term "potentially airborne" particulates less than 10 microns (AMAD) often surfaced. Knowing only the quantity airborne during the MCC-10 test is not the measurement needed by us. What is needed is the potentially airborne mass fraction. This bit of information may help you to decide between a Coulter counter or an aerodynamic size classifier. Again, as mentioned earlier, increase in surface area is not important because the transportation environment should not credibly include leaching.

In our analyses, energy inputs to the contents of a cask during a credible accident (even the incredible accident environments of our crash tests) would not be estimated to exceed 0.5 joule/cm^3, nominally. Unfortunately, the TTC

did not measure the energy inputs to contents during the crash tests so we can only state an upper bound with much confidence. The correlation to the high energy densities your test proposes is nebulous at best, but despite this fact, we at the TTC agree that testing to a failure threshold of the material is very important. Clearly a range of energy inputs plotted against respirable material generated is what our needs will ultimately be. And clearly, the need for a load cell is justified at least in the long term. Another approach might be to test materials until an arbitrary threshold of fines generated, say 10 percent, was reached; in other words, testing to a failure threshold.

Furthermore, the crushing effect of multiple impacts may more closely resemble real accident environments because the waste form will be containerized so that waste will be held in a confined area during impact and because multiple impacts with crushing may occur.

Dual Purpose of MCC-10

The MCC-10 test is to satisfy two objectives, as I understand it: ranking of waste forms and providing data for estimating risks of full-scale accidents. I think that the test may be useful for both even though the data may not be directly correlated to full-scale results at least without a full-scale testing program. Since ranking waste forms is an immediate need for waste form selection and efforts to support this must obviously be expedited, I feel that MCC-10 should be written to accommodate a phased approach. Initially, MCC-10 should be written to incorporate the simplest apparatus and procedures possible to allow ranking. Caveats about the extrapolation to full scale should be included in the procedure to avoid problems of people using data for risk analysis. The first phase of MCC-10 should not include load cells and axial loading. The simplicity of apparatus and procedures expedites testing by minimizing sample preparation time and reduces cost to all waste form developers. A simple test will allow all waste form developers to purchase the apparatus and to perform testing.

Then, after the elimination of the majority of the waste forms has been accomplished using the simple test, a more complex apparatus and procedure can be established to include load cells and axial loading. I suspect, however, that despite elaborate testing procedures, scaling MCC-10 data to expected

full-scale data may not be possible. If correlations are possible between MCC-10 test data and full-scale test data, they will be possible only after full-scale tests have been performed. Correlations can be attempted initially using pyrex.

Other Concerns

At this juncture, I would like to introduce two of my personal biases that you might consider: 1) MCC-10 should be applicable to all candidate HLW forms, including metal matrix, and 2) material balance problems may negate the usefulness of any of the test results.

My knowledge of the preliminary HLW selection criteria allows me to state that, if a test is not applicable to all candidate waste forms, then it cannot be used to discriminate among waste forms, period. The other problem, material balance, is not administrative, but rather, experimental. The weight percent of material lost during the test is so significant in all cases that the values for respirable fines can be completely distorted by the way in which the particle distribution curves are adjusted. Unless material loss can be minimized to a less than significant (?) level, the comparison of data from MCC-10 may not be truly valid. As a minimum, MCC-10 should specify a minimum acceptable material balance necessary for acceptable test results.

MCC-10 Apparatus and Procedure

A number of specific comments about MCC-10 are listed below. The comments are generally suggestions and concerns about the test as now proposed.

1. Compliance pieces required to reduce end effects will probably have to be tailored to each material type or waste form tested.
2. Aerodynamic size classification of fines will result in considerable mass loss which makes accountability nearly impossible.
3. If a Coulter counter is used, will particle characteristics be altered by the fluid selected for use in it?
4. The test should be simple and apparatus not complex in order to enhance the reproducibility of testing. Problems may occur in preparing axial testing specimens.

5. Cost must be considered as a major factor in apparatus specification.

6. Procedures for sample preparation must be stated explicitly and carefully so as not to favor one waste form over another.

7. Reproducibility of results must be ensured.

8. Because there may be some multiphase waste forms that partition radionuclides to non-respirable size particles, all particle size classes must be analyzed for radionuclide content. Maybe the results should be given in terms of weight percent of major radionuclides in fines less than 10 microns (AMAD).

5.2 BOM SOON LEE, BROOKHAVEN NATIONAL LABORATORIES

For the surface area increase caused by the impact from an accident, a realistic condition with a container will give a fracture pattern which is probably different from the one generated by this test procedure. Thus, even if you can calculate the increase in surface area accurately using this procedure, it might be difficult to predict the surface area increase for the real accident. I believe that the whole package, or at least waste form in a container, should be used for impact testing with a realistic impact energy.

APPENDIX A

PROPOSED MCC-10 IMPACT TEST METHOD - DRAFT

This draft of MCC-10 was the most up-to-date version at the time of the meeting on Impact Testing in March 1981 and it is included here to document the development of the test method. It is not complete and cannot be used to obtain data for publication in the Nuclear Waste Materials Handbook.

CONTENTS

MCC-10

BRITTLE-MATERIALS IMPACT TEST

1.0 SCOPE

The MCC-10 Brittle-Materials Impact Test specifies standard conditions for impact loading of the waste forms and subsequent analysis of the post-test fragments. Impact loading has two detrimental effects on waste forms--production of respirable fines and an increase in surface area. Thus, the purpose of this procedure is to obtain the fragmentation characteristics of brittle nuclear waste forms as a function of impact energy absorbed by the specimen.

2.0 SUMMARY

A right-circular cylinder of brittle waste is impact-compression loaded between two hardened, parallel steel platens, one of which is moved relative to the other. The displacement of the moving platen is limited to less than the height of the test specimen to ensure total fracture of the specimen while avoiding further comminution of the fragments by the contacting end pieces. The specimen is axially loaded so that the flat surfaces of the specimen are normal to the applied load (Figure A.1). The impact loads are supplied to the dynamic test fixture by a falling weight. The test fixture, in turn, transfers the impact load to the cylindrical specimen. The resultant fragments are captured for subsequent particle analysis in order to determine the weight percent respirable fines and the surface area.

3.0 LIMITATIONS

This test can be used only on brittle waste materials that exhibit minimal plastic deformation prior to fracture, such as ceramics, glasses, and concretes. The test cannot be used for metal matrix, bituminous, or plastic waste forms because of the limited displacement feature of the platens.

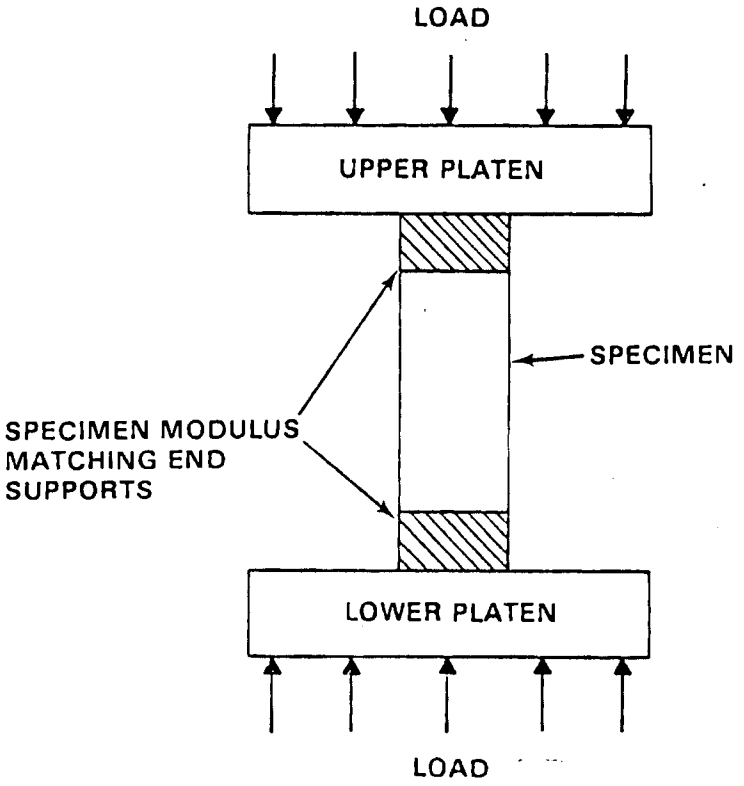

FIGURE A.1. Axially Loaded Specimen with Flat Surface of the Specimen Normal to the Applied Load

4.0 TESTING EQUIPMENT AND APPARATUS

The testing equipment and apparatus consist of a load-supplying system, a dynamic compression fixture, a particle-collection system, and particle-analysis equipment. The load-supplying system delivers the impact load to the dynamic-compression fixture, which in turn transfers the load to the specimen in a well controlled and repeatable manner. The specimen fragments are contained by the particle-collection system for subsequent analysis. The particle-analysis equipment consists of a system for measuring respirable fines and a system for measuring specific surface area.

4.1 Load-Supplying System

The load-supplying system is composed of a guided falling weight with a massive foundation. The falling weight should be adjustable with a maximum capacity of at least 750 kg and a minimum drop height of 50 cm above the top

of the dynamic-compression fixture. These limits provide the impact loads required (up to 1.0 MN) to fragment the higher strength materials. The compliance of the load-supplying system must be low in order for the high loads to be achieved. Thus, the weight of the foundation required for high rigidity is to be at least 10 times the maximum falling-weight capacity. A Dynatup® Model 8100 drop tower, such as shown in Figure A.2, which has a maximum drop weight of 1000 kg and a 10,000 kg foundation, is a suitable system.

The values of the falling weight and drop height are tentative and will be determined in the verification test program. Effects Technology, Inc. found 750 kg to be adequate for fracturing alumina as part of the load-capacity verification of the MCC test fixture. Since smaller weights reduce the cost of testing machines, the MCC will establish the validity of using a smaller weight in verification tests and will qualify machines of lower weight capacity for MCC-10.

4.2 Dynamic-Compression Fixture

The dynamic-compression fixture 1) assures proper alignment of test specimen with the impact-loading axis, 2) transfers the impact load from the load-supplying system to the specimen with minimal (less than 1%) decrease in load, 3) limits the displacement of the upper platen to 19.0 mm to avoid comminution of the particles generated during the fracture of the specimen, 4) has a sufficiently low compliance to ensure that the loads required to fracture the specimen can be obtained, and 5) is able to accommodate a particle-containment vessel to collect the fragmented specimen. A schematic of a compression-test fixture that meets these requirements is shown in Figure A.3 with an optional load cell. The test fixture is designed with a spherical bearing to accommodate any lack of parallelism between the ends of the cylindrical specimen and the load-transmitting platens. A linear ball-bearing alignment fixture is used to maintain axial loading throughout the test.

®Dynatup is a registered tradename of Effects Technology, Inc.

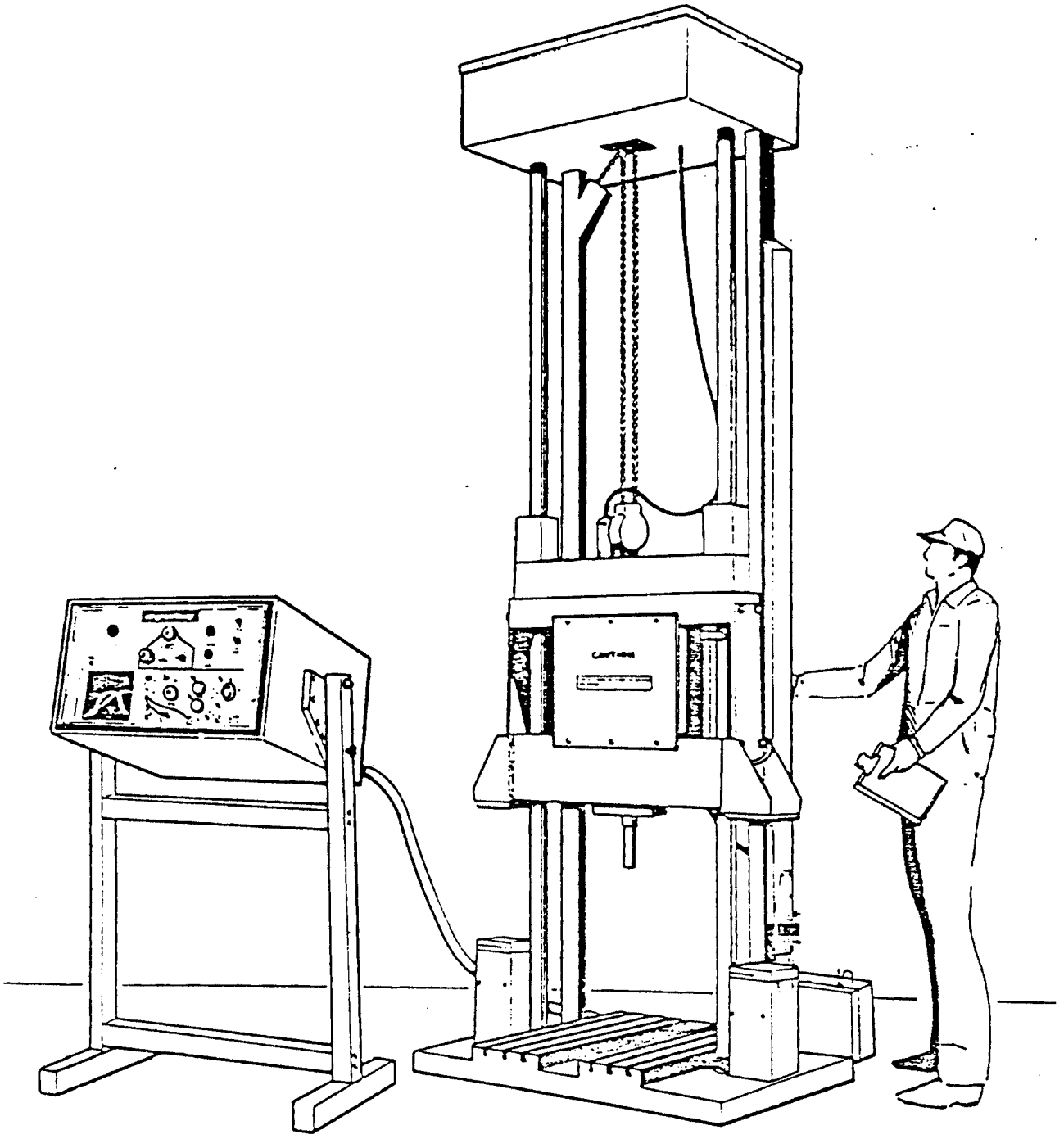

FIGURE A.2. Drop Tower Impact Machine

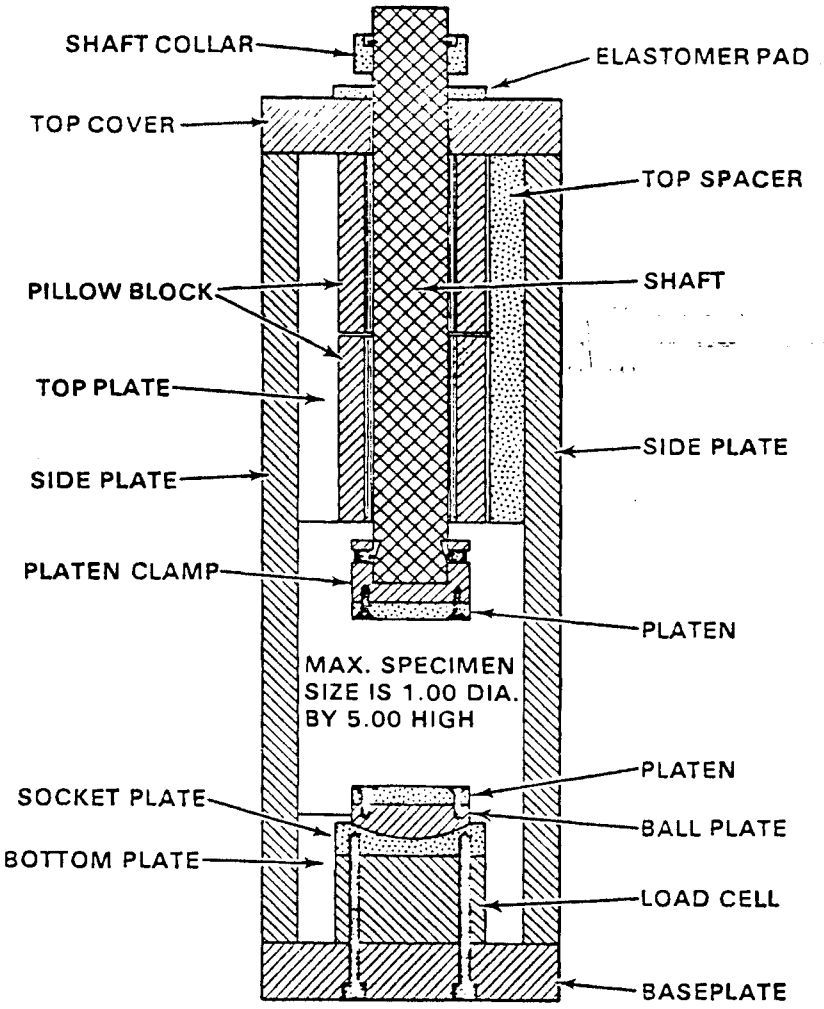

FIGURE A.3. Compression Test Fixture

A collar which impacts the top plate of the test fixture to stop its downward motion is placed on the loading shaft to restrict the displacement of the platens to less than 19 mm. It is necessary to confine the displacement of the falling weight to less than 13 mm, after it impacts the loading shaft, to prevent further loading of the shaft by the falling weight. Special stop blocks are located on each side of the dynamic-compression fixture to stop the falling weight within these tolerances.

4.3 Particle-Collection System

The particle-collection system is designed to confine and subsequently release for analysis the fragments generated during the impact event. Figure A.4 is a schematic drawing illustrating how the chamber is accommodated in the dynamic-compression fixture.

4.4 Particle-Analysis Equipment

Two particle characteristics are to be measured: weight percent respirable fines and surface area. It is necessary to classify the particles by aerodynamic diameter in order to measure the weight percent respirable fines. The respirable fines are collected from the chamber surrounding the impact test specimen by passing air through the chamber, thus forcing the particles through a standard cyclone fines classifier onto a filter. The filter paper shall be weighed to determine the weight percent of respirable fines. The remaining fragments are to be sieved using standard sieving techniques and the resultant size cuts weighed in order to develop a log-log plot of particle size versus weight percent. A standard geometric size factor will then be applied to the data to convert it to an estimate of total geometric surface area. Specific surface area will be measured with gas adsorption, if warranted.

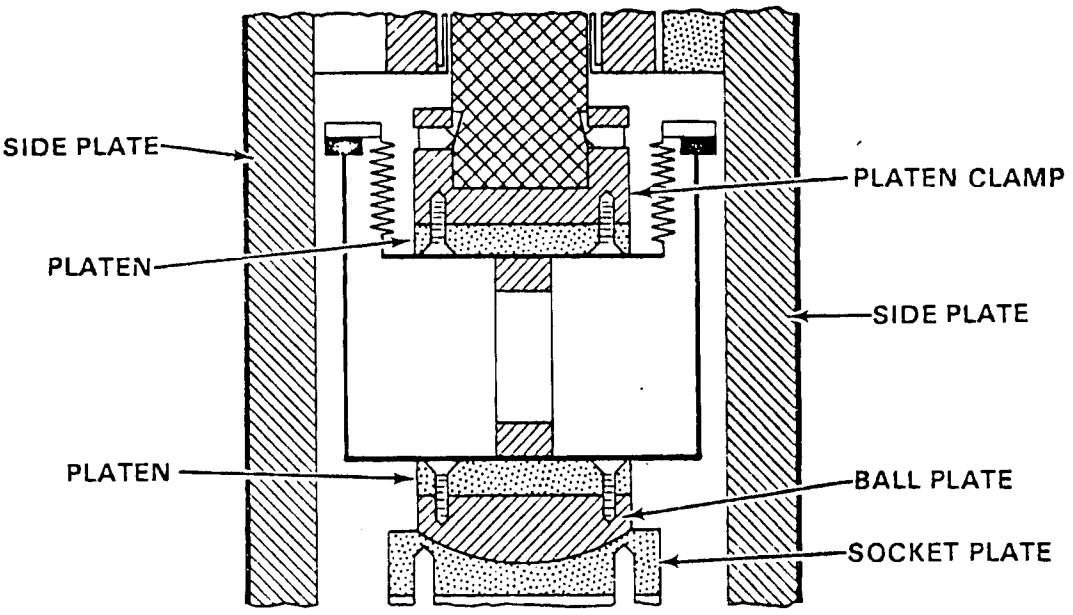

FIGURE A.4. Particle-Collection System

5.0 TEST SPECIMEN

5.1 Specimen Size

Use a specimen that is 19.05 ± 0.15 mm in diameter and 47.6 ± 0.15 mm long, round within a total tolerance zone of 0.025 mm, straight within a tolerance zone of 0.050 mm, and with the ends perpendicular to the cylinder axis within a tolerance zone of 0.050 mm, with ends of the specimen parallel within 0.050 mm.

These dimensions are tentative and will be determined in the verification test program. MCC prefers to use 13-mm-diameter x 25-mm-length specimen if reproducible loading can be achieved with this size. The larger size (19-mm diameter x 48-mm length) performed well in preliminary tests.

5.2 Specimen Preparation

Core drill specimens to obtain 19.05-mm-diameter rods from large pieces of waste form. Use centerless grinding with 200-grit SiC or a tool-post grinder on a lathe using a 60-grit Al_2O_3 wheel (32 Norton VBE or equivalent) to obtain an acceptable surface finish on the cylindrical surface. Then produce flat surfaces with a 200-mesh diamond cut-off saw. Use water as the coolant in the cutting or grinding operaions. Reject any specimen with chips or surface flaws greater than 0.1 mm.

5.3 Specimen End Pieces

Place the specimens on end pieces to reduce stress nonuniformities (Figure 1). The end pieces are to be the same diameter as the specimen and are necessary to reduce end constraint on the specimen. Hardened steel or alumina must be used.

One of these materials will be selected on the basis of verification test performed by the MCC.

6.0 PROCEDURES FOR TESTING

6.1 Record the specimen diameter and length to ±0.025 mm.

6.2 Weigh the specimen to ±1 mg.

6.3 Position the specimen between unused end pieces.

6.4 Seal the particle-collection chamber and start air flow for fines collection.

6.5 Load the drop tower with the specified weight.

6.6 Raise the weight to the specified height.

6.7 Record the weight and height above the dynamic-compression fixture.

6.8 Calculate the total available energy (height x weight).

6.9 Release the weight to fracture the specimen.

6.10 Collect the specimen fragments for subsequent particle analysis.

6.11 The particle analysis procedure for measuring weight percent respirable fine and specific surface are being developed.

7.0 NUMBER OF TESTS

The number of tests required will be determined in the verification test program.

8.0 QUALIFICATION OF TEST RESULTS

The load transfer shaft of the dynamic-compression fixture must be resting on its displacement-limiting collar after testing to ensure that there was sufficient energy available to fracture the specimen. In addition, the specimen end pieces must remain intact for the test to be valid.

9.0 REPORTING

Report the following:

9.1 Description of test systems used, both mechanical-test apparatus and fines-analysis equipment.

9.2 Description of the preparation and analyzed composition of the specimen.

9.3 Thermal history and significant processing variables that affect strength.

9.4 End-piece material and condition of end pieces after test.

9.5 Drop weight (kg), height (m), and available energy (J).

9.6 Specimen dimensions: length (L) and diameter (D).

9.7 Specimen mass (g).

9.8 Weight percent respirable fines; calculation based on original specimen weight and weight of particles captured on cyclone filter.

9.9 Geometric surface area and specific surface area, if warranted. Report as total surface area (m^2).

APPENDIX B

MEETING PARTICIPANTS

APPENDIX B

MEETING PARTICIPANTS
MATERIALS CHARACTERIZATION CENTER
IMPACT TESTING MEETING
MARCH 25-26, 1981

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APPENDIX C

MEETING AGENDA

APPENDIX C

MEETING AGENDA

NUCLEAR WASTE MATERIALS CHARACTERIZATION CENTER

IMPACT TESTING MEETING

MARCH 25-26, 1981

WEDNESDAY, MARCH 25 - BATTELLE AUDITORIUM LOBBY

8:00 am	Pick up visitors at Holiday Inn	
8:15	Badge in, Battelle ROB, Battelle Blvd.	
8:30	Coffee/rolls	
8:45	MCC Overview	Ron Nelson, PNL
9:00	Need for Impact Test Procedure Relative to Waste Management	Dan Merz, PNL
9:30	Review of Previous Impact Testing of Waste Forms	Dan Merz, PNL and Les Jardine, ANL
10:15	Break	
10:30	Proposed MCC-10 Impact Test Procedure	Gordon Dudder, PNL
12:00 noon	Lunch - Northwest Room	Visitors, and Atteridge, Dudder, Moss, Briant, Merz
1:15 p.m.	MCC-10 Dynamic Test Fixture Design and Preliminary Impact Data	Bill Adler, ETI
2:00	Respirable Fines and Surface Analysis Techniques	Jim Briant PNL, Owen Moss, PNL
2:30	Commmercially Feasible Methods for Particle Selection and Analysis	Virgil Marple University of Minn.
3:00	Discussion and coffee	Dan Merz, Moderator
3:45	Impact Testing Facilities Tour (Optional)	All
5:15	Return visitors to Holiday Inn	
6:30	Social Hour - Whitman Room Holiday Inn	All
7:00	Dinner - Whitman Room Holiday Inn	All
9:00	Preparation of Summary of Meeting	Dan Merz, Gordon Dudder, Dave Atteridge, Bille Neth

THRUSDAY, MARCH 26 - BATTELLE AUDITORIUM LOBBY

8:00 am	Pick up visitors at Holiday Inn	
8:15	Coffee/rolls	
8:30-11:30	Discussion of Meeting Summary and Further Recommendations	Dan Merz, Moderator
12:00	Return visitors to Holiday Inn	

APPENDIX D

QUESTIONS POSED BY MCC TO PARTICIPANTS

APPENDIX D

QUESTIONS POSED BY MCC TO PARTICIPANTS

1. For impact testing to rank waste forms, what criteria should be used for selection of the impact loading (mass, velocity, energy)?
2. What criteria should be used for selection of specimen size? Is it reasonable to have this a fixed, single size for all waste forms and to base ranking on use of a single impact condition?
3. Can a ranking test give meaningful quantitative data on respirable fines and surface area increases that can be translated to real accidents? Is it reasonable to expect that one test condition, e.g., a single impact energy or velocity, can provide sufficient data?
4. Can the respirable fraction be adequately determined by particle sizing such as with a Coulter counter, with sedimentation analysis, with cyclone and with sieving? A ranking of these methods (or combinations thereof) might be appropriate. Are there serious drawbacks to use of any of these?
5. For surface area determination, what ranking of the above techniques, plus a gas adsorption technique, would you suggest?

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