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Dr. Martin J. Steindler Materials Review Board Argonne National Laboratory 9700 South Cass Avenue Argonne, Illinois 60439

Dear Dr. Steindler:

In response to your request dated January 7, 1985, we have reviewed the MCC-7 Test Procedure entitled, "Method for Preparation of Isothermally Heat-Treated Waste Forms (MRB-0391)." Based on our review we are submitting a NEGATIVE ballot for full approval of this procedure.

The MCC-7 procedure provides only general guidance for heat treating specimens for evaluating time-temperature-transformations in glass and ceramic waste forms. The procedure fails to contain sufficient specificity to allow others to duplicate the laboratory conditions for tests used to construct isothermal transformation plots. We assume that there are few specific recommendations in the procedure in order to allow workers the flexibility to establish desired laboratory conditions. If this much flexibility is needed in establishing laboratory conditions for these tests, we question the usefulness of the proposed procedure as a method to define a standard practice.

We have also enclosed several detailed comments which need to be resolved before we could approve this procedure.

If you have any questions, please contact me at FTS-427-4200.

Sincerely,

Michael J. Bell, Deputy Director Division of Waste Management Office of Nuclear Material Safety and Safeguards WM Project WM Record File Enclosures: Docket No. 109.6 As stated FD1 LPUR_____ Distribution: CZ (Roturn to WM 623-SS) : : 8502280465 850205 PDR WASTE . WM-1 PDR DATE :85/02/05 : : : •

BALLOT FOR MATERIALS REVIEW BOARD

BALLOT DEADLINE: FEBRMARY 4, 1985

BALLOT LEVEL: X Procedures Panel; Data Panel; Full Board

TITLE: MCC-7, Method for Preparation of Isothermally Heat-Treated Waste Forms (MRB-0391)

This ballot is for FULL APPROVAL

CHECK ONE:

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YES (without comments--return this ballot).

YES (with comments--letter attached).

XX NO (with REQUIRED comments--letter attached).

Members who are unable to complete this request for ballot by the deadline are requested to contact W. Seefeldt (FTS 972-4390) ASAP. Depending on circumstances, members may be given a grace period to complete this ballot. The vote of a member who has not returned this ballot or has not contacted this office by the deadline will be recorded as an abstention.

5 FEB 85 (DATE)

Michael Bill (SEGNATURE)

Enclosure 1

Specific Comments on MCC-7

1. Page 7-4, Section 3.0, USES AND LIMITATIONS

The third paragraph states that "current Waste Form Acceptance Specifications requires an estimate of the extent of devitrification in a glass waste form or require that crystalline or metallic inclusions in glass waste forms do not exceed a certain value (5 vol %)." The current waste form acceptance specifications should be referenced or appended.

2. Page 7-5, Section 5.1, Equipment

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In the first paragraph, the third and fourth sentences state "when radioactive specimens are used, determine that self-heating does not interfere with maintaining the desired specimen temperature or produce an unsatisfactory temperature gradient. Report the results of such an evaluation."

The temperature of the specimen and/or the temperature gradient within the specimen may be an important variable(s) in preparing isothermally heat treated specimens from borosilicate glass containing waste. Therefore, MCC-7 should state how the temperature of the radioactive specimen and the temperature gradient within the specimen should be measured or measured and calculated.

3. Page 7.6, Section 6.2. Test Specimens

The first sentence says "the test specimens must be representative of the bulk waste form." A description of the procedures to be followed to assure that the test specimens are representative of the bulk waste form should be included.

4. Page 7-7, Section 7.1, Isothermal Heat Treatments

Under Item 3 it is stated that, "it may be necessary to preheat test specimens." If preheating may be used by laboratory workers, the test procedure should provide specific guidance on how this should be performed.

5. Page 7.7, Section 7.1., Isothermal Heat Treatments

In the first paragraph under item 3, the last sentence says, "an alternative approach, particularly for glass waste forms, is to remelt each test specimen and insert it into the furnace directly from the melt temperature."

Section 6.1 requires that "before heat treatment, fully characterize and report the microstructure of the test material according to appropriate MCC or other standard methods. Use these methods to document microcracking, porosity, identification of all phases, shapes of grains or crystals, concentration of each phase, and extent of homogeneity."

If the test specimen is remelted the rate of cooling between the melt temperature and the furnace temperature will be greater for the specimen than for the bulk material from which the specimen was taken. The remelting and faster cooling from melt temperature to furnace temperature may change some of the physical properties described in section 6.1, e.g., porosity and microcracking. Specific conditions should be defined which control the remelt procedure to ensure that physical property changes will be minimized.

6. Page 7.7, Section 7.1, Isothermal Heat Treatment

Under Item 3, the procedure states, "the effect of any preheating can be assessed after the isothermal transformation behavior is determined. If an effect is suspected, then the experimenter should report this and attempt to reconduct the heat treatment without heating."

Specific guidance should be provided on how these performance assessments should be performed to ensure that all pertinent information is provided and reported in a standard format.

7. Page 7-7, Section 7.1, Isothermal Heat Treatments

Under Item 5, the procedure states that the test specimens should be cooled "as rapidly as possible."

The procedure should provide guidance regarding the conditions under which cooling should be performed.

8. Page 7-8 to 7-10, Section 7.2, Test Matrices

The term "Temperature interval" should be defined.

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9. General

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The Jantzen paper, enclosed with the MCC-7 review package, indicates the importance of the environmental conditions when constructing time-temperature-transformation plots. Because these conditions are important, the procedure should provide guidanceon defining and controlling appropriate testing conditions.

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