# ACTIVITY PLAN

# PARAMETRIC STUDIES OF METAL DEGRADATION AND MICROSTRUCTURE: Measurement of Threshold Stress Intensity for Stress Corrosion Cracking.

Sub-Activity E-20-18d of Activity E-20-18 of the scientific investigation "Metal Barrier Selection and Testing"

WBS # 1.2.2.3.2

Harkirat S. Ahluwalia

Revision 0

Aug 1, 1989



J. Jardine, YMP Project Leader Date

D. W. Short, YMP OA Manager

0/11/89 Date

W. L. Clarke, Technical Area Leader

Data

R. D. McCright, Task Leader

Date

#### 1.0 INTRODUCTION

# 1 Identification of activity

This activity plan for sub-activity E-20-18d and is written pursuant to quality procedure 033-YMP-QP 3.0, [1]. Sub-activity E-20-18d is part of activity E-20-18 which is known as "Parametric studies of metal degradation and microstructure". Activity E-20-18 is a part of the scientific investigation known as "Metal Barrier Selection and Testing" which is identified with WBS # 1.2.2.3.2. and published in UCID-21262.

# 1.2 Quality Assurance Level Assignment

A quality assurance level of QA-II has been assigned to this activity (E-20-18).

# 1.3 Responsibilities

Harkirat S. Ahluwalia, John Estill, Greg E. Gdowski and Joseph C. Farmer are the Principal Investigators for this activity and are responsible for the conduct of this work. R. Daniel McCright is the Task Leader for the Metal Barrier Selection and Testing Investigation. Willis L. Clarke is the Technical Area Leader for Container Materials, Modeling and Testing.

# 2.0 PURPOSE AND OBJECTIVES

The objective of this sub-activity is to provide specific stress corrosion cracking data needed for material selection and model development.

#### 3.0 ACTIVITY DESCRIPTION

This activity will be used to access the threshold stress intensity for stress corrosion cracking,  $(K_{ISCC})$  of the candidate materials. Slow stress corrosion crack growth does not occur at all values of stress intensity and the minimum initial value at which environmental sensitive crack growth occurs is designated  $K_{ISCC}$ . The value of  $K_{ISCC}$  determined by this activity and the value for  $K_{IC}$  (plane-strain fracture toughness) determined by activity E-20-18c will be used to rank the candidate alloys in terms of the embrittlement index,  $K_{IC}/K_{ISCC}$ . This activity will also be used to monitor stress corrosion crack propagation rates using the reversing D.C. instrument (Crack monitoring system).

One of two testing techniques may be used for the determination of  $K_{\text{ISCC}}$ ; the Rising Load testing method or/and Wedge-Opening-Loading technique.

Rising load Kisco testing method. The testing technique used to determine Kisco is essentially identical to the procedure used for Kic fracture testing (ASTM E399-83) [2] except that a slower rate of loading is involved normally, and the specimen is exposed to the environment while ing loaded. The slower rate of loading is essentially to allow for vironmentally induced crack initiation and to cause time-dependent sub-critical propagation. This technique is often referred to as the Rising load Kisco testing method [3]. In this technique stress corrosion characteristics are measured in terms of crack growth rate. The specimen

is fixed in a holding device and the environment applied to the tip of the hined notch. The test environment should be brought into contact with specimen before it is stressed. This enhances access of the corrodent To the crack tip to promote earlier initiation of stress corrosion cracking and to decrease the variability of the test method. If the specimen has been pre-cracked, it is deflected, in the presence of the corrodent, to a predetermined RI value. Crack length using the reversing DC potential drop method is measured and the crack opening displacement, is also measured along the line of load application when the load is maintained at the same level for the duration of the test. Once the environment is applied to the specimen, the crack length is monitored as a function of time elapsed from deflection. The overall result of this procedure is to cause the stress intensity factor to decrease as the crack extends under the influence of the corrodent. The slope of the crack length versus time curve at any crack length provides crack growth rate. From the K-calibration the stress intensity level is determined.

The data are plotted as logarithms crack growth rate or crack velocity versus stress intensity factor. Generally, three stages of crack growth rate may be identified in stress-corrosion results presented in this manner. Stage I occurs at low stress intensities where crack growth rate is strongly stress-intensity dependent and the crack may eventually arrest, thus indicating K<sub>ISCC</sub>. Stage II occurs at intermediate stress intensities where crack growth rate is independent of stress intensity. Stage III occurs at stress intensities close to K<sub>IC</sub> where crack growth rate again becomes dependent upon stress intensity. The reversing P C.potential drop method will also be used to monitor crack propagation i.es.

Wedge-Opening-Loaded Technique. Figure 1 shows 8 schematic illustration of the loading technique and instrumentation involved. The bolt loaded specimen is loaded initially to relatively stress-intensity levels  $(K_{I\,\,I})$ , exposed to the environment of interest for a predetermined length of time, and unloaded. Since the test is conducted under constant displacement conditions, the load on the specimen and, consequently , the nominal stress intensity factor decreases as the leading to crack arrest as the decreasing K<sub>1</sub> approaches the threshold for cracking,  $K_{\text{Iscc}}$ . From knowledge of the initial loading conditions and the final crack length at the end of the test, the stress intensity level associated with crack arrest can be computed [4].

#### 3.1 Technical Reviews

- A formal surveillance will be held before any experimental work begins. This review will insure that:
- 1. Measurement and test equipment (M&TE) are properly calibrated as specified in quality procedure 033-YMP-QP-12.0.
- 2. Test samples are procured as specified in quality procedure 3-YMP-QP-4.0 and controlled as specified in quality procedure 3-YMP-QP-8.0.

- 3. Collected data will be controlled as specified in quality ocedure 033-YMP-8.0.
- 4. Laboratory notebooks are being maintained as specified in quality procedure 033-YMP-QP-3.4.

After completion of an experiment or a series of experiments, a UCID report will be written. The UCID report will undergo review as specified in quality procedure 033-YMP-QP-3.3.

#### 3.2 Hold Points

There are no formal hold points associated with this activity, but the results will be evaluated on a continuous basis by the Principal Investigators to insure that work is proceeding according to plan. If significant unanticipated problems arise, the Principal Investigators will inform the Task Leader. A joint decision will be made about corrective actions.

Progress will be reported to the Task Leader in monthly report. If changes in project scope require that experimental work change direction, it is the responsibility of the Task Leader to communicate this to the Principal Investigator in writing.

# 3.3 Equipment

Required M&TE include: Constant extension rate testing machine (CERT), specifically Cortest Series 34000 Floor Model; Load cell, Sensotec model D/3971-01, identification no: 5015939; Controller, Cortest model SC12, identification no: 4403461. These instruments are found on the list of calibrated equipment (see Appendix I). The Reversing d.c. potential drop instrument and the Instron testing machine Model 8500 are on order. Displacement gages and Caliper or micrometers are also required and are in the process of being acquired. The identification numbers and calibration records of all M&TE used will be identified in the scientific notebook.

# 3.4 Materials

All samples tested will be procured as specified in quality procedure 033-YMP-QP-4.0. and controlled as specified in quality procedure 033-YMP-QP-8.0.

# 3.5 Special Environmental Conditions

Electrolytes used for testing will be prepared so as to maintain the same relative concentrations of ions as found in water from well J-13, if possible. Absolute concentrations may be greater or less than those found in water from J-13 (reference condition). Measurements in other aqueous environments (NaCl solutions, etc) will be made if necessary. Tests may also be conducted in a vapor-phase environment containing  $NO_x$  species; the environmental variables will include temperature, partial pressure of water and partial pressure of  $NO_x$  species. Solutions may be refreshed when necessary.

# 3.6 Special Training/Qualification Requirements

No special training/qualification are required.

# 3.7 Activity Closeout

The final product of this sub-activity will be a UCID report documenting all results. Supporting documentation such as laboratory notebooks and technical review comments will be retained by the responsible individual until the document package is transferred to the local records center at the conclusion of the sub-activity.

#### ) PRECISION AND ACCURACY

The precision of a Kisco determination is a function of the accuracy and bias of the various measurements of linear dimensions of the specimen and testing fixtures, the precision of the displacement measurements, and the bias of the load measurement as well as the bias of the recording devices used to produce the load displacement record and the precision of the constructions made on this record. The accuracy of the various measurements will be recorded in the scientific notebook.

## 4.1 Calibration requirements

All M&TE must be calibrated as specified in quality procedure 033-YMP-QP-12.0. Identification numbers of equipment used for this sub-activity will be found on the approved list of M&TE for the Yucca Mountain Program.

# 4.2 Conditions Which May Adversely Affect Results

In order for a result for  $K_{IC}$  to be considered valid it is required that both the specimen thickness, B, and the crack length, a, exceed  $2.5(K_{Q}/\sigma_{VS})^{2}$ , where  $\sigma_{VS}$  is the 0.2% offset yield strength of the material for the temperature and loading rate of the test and  $K_{Q}$  is the conditional result used to establish if a valid  $K_{IC}$  has been measured. However, it is not clear whether the same criteria should be applied during the designed of pre-cracked specimens for stress-corrosion testing. It is recommended that the dimensions of the plastic zone be kept at a minimum compared with the thickness dimension of the specimen and the relationship for the validity of  $K_{IC}$  be used as a guide to test the validity of  $K_{ISCC}$ .

#### 5.0 IN-PROCESS DOCUMENTATION

In process documentation will include stress-strain curves, optical and electron micrographs. Such records will be kept in a controlled laboratory notebook identified as Metal Barrier Selection and Testing Task Controlled Notebook No.00079. Copies of all in-process documents will be kept by all the principal investigators identified in Section 1.3. Results will be periodically transmitted to the Task Leader in the monthly report and the Task Leader is responsible for transferring the document package to the local records center at the conclusion of this sub-activity.

# 1 Data Recording and Data Reduction

All relevant data for the determination of Kisco shall be kept in a bound, scientific notebook, as well as an appropriate data base. The data from the x-y recorder and any construction on that record will be pasted in the scientific notebook. Data collected from computers will be stored on magnetic media and a hard copy will also be presented in the scientific notebook.

# 5.2 Analysis

The interpretation of test records and calculation of  $K_{ISCC}$  shall be conducted according to the Technical Implementing Procedures [5,6].

# 6.0 INTERFACES

This sub-activity can proceed independent of any other activity, however activity E-20-19 ("Metal Barrier Selection") cannot proceed without this activity.

#### 7.0 SCHEDULE

The readiness review for this sub-activity was scheduled for the July 1, 1989. The final UCID report will summarize all of the data and a draft copy will be completed prior to the first week in April of 1990.

#### 8.0 TECHNICAL IMPLEMENTING PROCEDURES

A TIP for the determination of plane-strain fracture toughness ( $K_{IC}$ ) and the threshold stress intensity for stress corrosion cracking ( $K_{ISCC}$ ) will be prepared in accordance with Quality Procedure No. 033-YMP-QP 5.0, "Technical Implementing Procedures". [5,6]

# 9.0 SPECIAL CASES (PROCUREMENT)

Technical services provided by Hira Ahluwalia are provided by contract and meet the requirements of 033-YMP-QP 4.0 "Procurement Control and Documentation"[1]. All such services will be performed under the LLNL YMP Quality Assurance Plan.

# 9.1 QA Requirements Specification

Not applicable

#### 9.2 Statement of work

The statement of work for technical support for this activity is to provide technical support for electrochemical corrosion experiments. An example of service contract statement of work is provided in Appendix II.

# 9.3 Subcontractor Interface Control

The technical contacts at LLNL for the contracts discussed in Section 9.0 are Joseph C. Farmer, William Halsey, R.Daniel McCright and Willis Clarke.

# 9.4 Materials/Equipment Provided

Access to laboratory space is provided so that work on this sub-activity can be accomplished.

## 9.5 Deliverables

Deliverables for the technical support contractor will include a UCID report documenting the results and scientific notebooks and data accumulated on magnetic media.

# 10.0 REFERENCES

- [1] Yucca Mountain Project, Quality Procedures Manual.
- [2] Standard test method for plane-strain fracture toughness of Metallic Materials: E399-83, ASTM Philadelphia, p680-701. 1983.
- [3] W.G.Clark Jr. and J.D.Landes. 'An evaluation of Rising Load Testing' Stress corrosion- New Approaches, ASTM STP 610, p108-127, 1976.
- [4] S.R.Novak and S.T.Rolfe, Journal of Materials, JMLSA, Vol. 4, No. 3, Sept. 1969, pp701-728.
- [5] Technical Implementing Procedure for Kic and Kisco. (TIP-CM-1)
- [6] Technical Implementing Procedure for Kisco determination using WOL specimens (TIP-CM-5).

# 11.0 APPENDIXES

- [I] Current list of calibrated equipment p2.
- [II] Example statement of work.

APPENDIX I

Current list of calibrated equipment.

	INSTRUMENT	HODEL	HFG	IDENT NO	LAST CAL	CAL EXP	RECALL	ACTIVITY	BLDG	ROOM	TAL	TL	
	THERMOCOUP	TYPE K	Mer.	7/	••••••	*******	••••••	•••••		•••••		•••••••	
	THE OUP	TYPE K	CHEGA	34	8/09/88	8/09/89	6/28/89	8-20-4	281	1160	WILDER	GLASSLEY	
	STR. GAU	5550	OHEGA	17	8/09/88	8/09/89	6/28/89	8-20-4	281	1160	WILDER	GLASSLEY	
	STRAIN GAU	5550	PRECISE SE	23479	8/09/88	8/09/89	6/28/89	8-20-4	281	1160	WILDER	GLASSLEY	1
	STRAIN GAU	5550 VS	PRECISE SE	24380	8/09/88	8/09/89	6/28/89	8-20-4	281	1160	WILDER	GLASSLEY	
	STRAIN GAU	5550 V6	PRECISE SE	23342	8/09/88	8/09/89	6/28/89	B-20-4	281	1160	WILDER	GLASSLEY	
	STRAIN GAU	2105	TABER	23341 721223	8/09/88	8/09/89	6/28/89	8-20-4	281	1160	WILDER	GLASSLEY	
	WEIGHT SET	NONE	TROEMNER		8/09/88	8/09/89	6/28/89	8-20-4	281	1160	WILDER	GLASSLEY	
	WEIGHT SET	6137/1540	TROEMNER	4935825	9/14/88	9/15/89		0-20-31	243	2026	SHAW	RYERSON	
	CONTROLLER	SC12	CORTEST	4935832 4403461	9/15/88	9/15/89		0-20-31	243	2026	WAKE	RYERSON	
	STRIP CHAR	585/11/13	CHEGA	4347352	CAL LAB		INITIAL	E-20-23	241	1878	CLARKE	McCRIGHT	
	STRIP CHAR	585/11/13	CHEGA	4414849	CAL LAB		INITIAL	E-20-23	241	1877	CLARKE	McCRIGHT	ŀ
	STRIP CHAR	585/11/13	CHEGA	4076603	CAL LAB		INITIAL	E-20-23	241	1877	CLARKE	McCRIGHT	ļ
	STRIP CHAR	585/11/13	CHEGA	4350130	CAL LAB		INITIAL	E-20-23	241	1877	CLARKE	McCRIGHT	1
	POTENTIOST	173	PAR		CAL LAB		INITIAL	E-20-23	261	1877	CLARKE	McCRIGHT	
	POTENTIOST	173	PAR	4015329	4/28/89	10/28/89		E-20-23	241	1878	CLARKE	McCRIGHT	
	CONTROLLER	175	PAR	4369149	CAL LAB		INITIAL	E-20-23	241	1877	CLARKE	McCRIGHT	1
	POTENTIOST	273	PAR	3676767	4/28/89	10/28/89		E-20-23	241	1878	CLARKE	McCRIGHT	
	TROITKETC	273	PAR	4053581	4/21/89	10/20/89		E-20-23	241	1877	CLARKE	McCRIGHT	
	TEOTHETC	362	PAR	4066826	CAL LAB		JAITIKI	E-20-23	241	1878	CLARKE	McCRIGHT	
	POTENTIOST	363	PAR	4272227	3/13/89	9/13/89		E-20-23	241	1878	CLARKE	MCCRIGHT	
	POTENTIOST	363	PAR	4345822	1/09/89	7/09/89	6/5/89	E-20-23	241	1878	CLARKE	MCCRIGHT	
	POTENTIOST	363	PAR	4277185 3328949	3/07/89	9/07/89		E-20-23	241	1878	CLARKE	McCRIGHT	
	POTENTIOST	363	PAR	3711963	3/07/89	9/07/89		E-20-23	241	1878	CLARKE	McCRIGHT	
	POTENTIOST	363	PAR	_	3/06/89	9/06/89		E-20-23	241	1878	CLARKE	McCRIGHT	
	CT TER	376	PAR	3329106	CAL LAB			E-20-23	241	1878	CLARKE	McCRIGHT	
	LON CELL	0/3971-01	SENSOTEC	4764128	4/29/89	10/29/89		E-20-23	241	1878	CLARKE	McCRIGHT	ı
	LVDT	351-000	TRANS-TEX	5015939 4764111	4/28/89	10/28/89		E-20-23	241	1878	CLARKE	HECRIGHT	1
	WEIGHT SET	NONE	TROEMNER		CAL LAB	<b></b>	INITIAL	E-20-23	241	1878	CLARKE	McCR1GHT	
•	PROFILEOME	DEKTAK IIA	SLOAN	4935818	9/15/88	9/15/89		E-20-23	241	1883	CLARKE	HECRIGHT	-
	OSCILLOSCO	2465	TEXTRONIX	88503	1/12/89	1/12/90		G-20-3.1	151	1034A	SHAW	RYERSON	
		\$73-25-230	AHE	4160616	9/01/88	9/10/89		H-20-6	327		CLARKE	RUSSELL	
	WATT TRANS		AME	4763411 4763428	10/07/88	10/07/90		s-20-1	G	THL	WILDER	RAMIREZ	
	DEPTH PROB	501DR	CPN .		10/07/88	10/07/90		s-20-1	G	THE	WILDER	RAMIREZ	
	DA CONTROL	3497A	НР	4737900	7/28/88	7/28/89	6/5/89	s-20-1	G	THL	WILDER	RAMIREZ	
	PRES TRAN	2279-1	ASHCROFT	3854172	9/25/88	9/25/89		S-20-1	G	THL	WILDER	RAMIREZ	
	PRES TRAN	2279-2	ASHCROFT	5038761	4/12/89	4/12/90		S-20-1	G	THE	WILDER	RAMIREZ	
	PRES TRAN	2279-3	ASHCROFT	5038785	4/12/89	4/12/90		S-20-1	G	THL	WILDER	RAHIREZ	
	PRES TRAN	2279-4	ASHCROFT	5038792 5038761	4/12/89	4/12/90		s-20-1	G	TNL	WILDER	RAMIREZ	
	BALANCE	2404	SARTORIUS	3792559	4/12/89	4/12/90		s-20-1	G	THL	WILDER	RAMIREZ	
	BALANCE	MGSD	HETTLER		CAL LAB		INITIAL	J-20-8	281		AINES	SILVA	
	OSCILLOSCO	11402	TEXTRONIX	3559879	CAL LAB		INITIAL	J-20-8	281		AINES	SILVA	
	OSCILLOSCO	2335	TEXTRONIX	4928490	CAL LAB		INITIAL	J-20-8	281		AINES	SILVA	
	PULSE GEN	DG535		4728496	CAL LAB		INITIAL	J-20-8	281		AINES	SILVA	
	RX THERM	2804A	SRS	4778699	CAL LAB		INITIAL	J-20-8	281		ATHES	SILVA	
	VERT AMP	11A34	HP	5038655	CAL LAB		INITIAL	J-20-8	281		AINES	SILVA	
	THERHOCOUP	TYPE K	TEXTRONIX	4921767	CAL LAB		INITIAL	1-20-8	281		AINES	SILVA	
	THERMOCOUP	TYPE K	ONEGA	3977512	3/15/89	3/15/90		B-20-4	281		WILDER	GLASSLEY	
	THENHOCOUP	TYPE K	CHECA	3977529	3/15/89	3/15/90		B-20-4	281		VILDER	GLASSLEY	
	OCCUP	TYPE K	OHEGA	3977536	3/15/89	3/15/90		3-20-4	281		WILDER	GLASSLEY	
		· · · · · · · · · · · · · · · · · · ·	OMEGA	3977543	3/15/89	3/15/90		8-20-4	281		WILDER		4

APPENDIX II

Example statement of work.

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# MATERIAL SCIENCE SUPPORT FOR THE YUCCA MOUNTAIN PROJECT.

# STATEMENT OF WORK PROPOSAL.

# Introduction

The Metal Barrier Selection and Testing (MBST) Task of the Yucca Mountain Project at Lawrence Livermore National Laboratory (LLNL) is responsible for the selection of the metal barrier material for application in the high-level nuclear waste repository being designed for the Yucca Mountain Site in Nevada. The Scientific Investigation Plan (SIP) for the MBST task includes: (i) development of models for degradation modes, mechanical properties and microstructure (E-20-16); (ii) experimental technique development (E-20-17); (iii) parametric studies of degradation and microstructure (E-20-18); (iv) degradation mode surveys (E-20-13).

It is proposed that <u>Science and Engineering Associates</u>, <u>Inc.</u> continue to provide significant scientific and engineering support to: (i) evaluate existing mechanistically based models of stress corrosion cracking and crevice corrosion in alloy 825 and CDA 715 under repository conditions; i) perform constant extension rate testing with simultaneous measurement acoustic emissions and electrochemical noise; (iii) provide technica support for electrochemical corrosion experiments including accessment of plane-strain fracture toughness (K<sub>1</sub>c) and threshold stress intensity for stress corrosion cracking, (K<sub>1</sub>scc) of the candidate materials; (iv) complete an evaluation of the suitability of titanium, zirconium and monel as corrosion resistant materials for high-level Nuclear Waste Containers for emplacement at the Yucca Mountain repository.

All of these tasks, which are described in detail below, will be completed in accordance with the Quality Assurance Program Plan for YMP (033-YMP) at the Quality Assurance level assigned in the Scientific Investigation Plan.

This procurement action deals with the acquisition of support personnel only. The scientist, who is required due to his technical expertise, will support the activities of the Yucca Mountain Project. The scientific personnel will work under the direct supervision of LLNL-YMP staff. This work will be completed in accordance with approved QA procedures as defined in the YMP Quality Assurance Program Plan. The qualifications of personnel assigned to work on this subcontract has been documented and submitted to the YMP QA staff.

# TECHNICAL SUPPORT OF WORK.

Jeller will provide a Ph.D. Scientist to support the Nuclear Waste Management Program at Lawrence Livermore National Laboratory.

Task 1 Complete an evaluation of the availability and applicability of existing mechanistically based models of stress corrosion cracking and crevice corrosion, adapt these models or develop new models to help predict the effects of stress corrosion cracking and crevice corrosion in alloy 825 and CDA 715 under repository conditions. This task includes completion of a survey of the technical literature to identify mechanistically based models of localized corrosion, crevice corrosion, and stress corrosion cracking and modifying these models to aid in the prediction of the effects of crevice corrosion and stress corrosion cracking on alloy 825 and CDA 715 in a repository environment. This task is an important element of activity E-20-16, building upon already existing data and information previously identified in E-20-13. This task, which will provide significant input to activity E-20-19 (metal barrier material selection), will be performed at a quality assurance level QA II.

Task 2 Conduct constant extension rate testing with simultaneous measurement of acoustic emissions and electrochemical noise. Current transients correlated with acoustic emissions can be used to determine repassivation rates at crack tips. Repassivation rates may also be determined with the strained electrode technique. Additionally provide technical assistance with other experimental technique development ctivities. This task is an element of activity E-20-17, "Experimental chnique development" and will be performed at a quality assurance level-OA III.

Task 3 Provide technical support for electrochemical corrosion experiments including accessment of plane-strain fracture toughness  $(K_{1:c})$  and threshold stress intensity for stress corrosion cracking,  $(K_{1:s:c:c})$  of the candidate materials. The values of  $K_{1:c:c}$  and  $K_{1:s:c:c}$  will be used to rank the candidate alloys in terms of the embrittlement index,  $K_{1:c:c}/K_{1:s:c:c}$ . This task is an element of activity E-20-18, "Parametric studies of degradation and microstructure" and will be performed at a quality assurance level of QA I.

Task 4 Complete an evaluation of the suitability of titanium, zirconium and monel as corrosion resistant materials for high-level Nuclear Waste Containers for emplacement at the Yucca Mountain Repository. The overall project schedule is such that the container material must be chosen before the environmental conditions at the site are fully characterized by tests conducted in exploratory shafts. There is, therefore, some potential that the actual site conditions may prove to be too aggressive for successful employment of the alloys currently being evaluated as metal container materials. There is also some potential that performance assessment models will predict metal container degradation rates that are not consistent with meeting the goal of "substantially complete containment" included in the NRC regulations for the repository.

While both of these potentials are small, it is prudent to consider other loys as a backup to the alloys currently being considered. This tas' ll be performed at a quality assurance level of QA III.

Reports The seller will submit monthly progress reports.

# TABLE OF CONTENTS

									]	Page
1.0	INTR	RODUCTION			•	•	•		٠	1
	1.1	Activity Identity		•			f .		•	1
	1.2	Quality Assurance Level Assignment			•	•		•		1
	1.3	Responsibilities	•	•	•	•	•	•	•	1
2.0	PURI	POSE AND OBJECTIVES	• •	•	•	•	•	, •	•	1
3.0	ACT	TIVITY DESCRIPTION	• •	•	•	•	•	•	•	2
	3.1	Technical Reviews				•		•	•	3
	3.2	Hold Points		•	•			•	•	3
	3.3	Equipment		•	•			•		3
	3.4	Materials		•	•			•	•	3
-	3.5	Special Environmental Conditions		•	.:	•		•		4
	3.6	Special Training/Qualification Requirements		•	•	•			•	4
	3.7	Activity Closeout	• • •	•	•	•	•	•	. •	4
4.0	PREC	CISION AND ACCURACY	• •	•	•	•	•	•	•	4
	4.1	Calibration Requirements	• . •	•	•	•	•	•	•	4
	4.2	Conditions That May Adversely Affect Results .		•	• `		•	•	•	5
	4.3	Sources of Uncertainty and Error to be Controlled	and	Mea	sure	d	•	•	•	5
5.0	IN-P	PROCESS DOCUMENTATION		•	•	•	•	•	. •	5
	5.1	Data Recording and Data Reduction		•.	•	•	•	•		6
	5.2	Analysis		•	•	•	•	•	•	6
6.0	INTE	ERFACES		•	•		•	•		6

7.0	SCHE	DULE				•	•	•	•	•	•	•	•	٠	•	•	•	•	•	•	7
	7.1	Durat	ion			•			•	•		•	•	•	•	•				•	7
	7.2	Staffi	ng Re	quir	emer	nts	•	•	•	•	•	•	•	•	•	•	•	•	•	•	7
8.0	TECH	INICAI	_ IMI	PLEN	MEN	TIN	G P	RO	CED	UR	ES	•	•	•	•	•	•	•	•		7
9.0	SPEC	IAL CA	SES	(PR	OCL	JRE:	ME	NT)		•	•		•		•	•	•	•	•	•	7
	9.1	QA R	equir	emei	nt Sp	ecifi	catio	ons	•		•	•		. •	•	•	•	•		•	8
	9.2	Stater	nent o	of W	ork	•		•				•		•	•	•	•	•	•		8
	9.3	Subco	ntrac	tor I	nterf	ace	Con	trol	•			•	•			•	•	•	•		8
	9.4	Mater	ials/E	Quip	men	t Pro	ovide	ed						•		•	•	•		•	8
	9.5	Delive	erable	s		•	•	•	٠	•	•	•			•	•	•	٠	٠	•	8
10.0	REFE	ERENC	ES	•	• •	•	•	•	•	•	•	•	•		•	•	. •	•	•		9
11.0	APPE	ENDICE	is .	•	• •	•	•	•	•	•	•	•	•	•	•	•	•	•	•		A-1
	11.1	Appe Oxida		1	-	evel	Ass	ignr	nent	She	ets	for	Lov	v-Te	mpe	eratı	ıre (	Ove	a.		
	11.2	Appe. Spent					for I	Long	у-Те	rm,	Lov	w-T	emp	erai	ure	Oxi	datio	on c	f B	WR	•
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# 1.0 INTRODUCTION

The LLNL-YMP Scientific Investigation Plan for Spent Fuel Waste Form Testing YMP WBS Element 1.2.2.3.1.1 [1] identifies an activity for oxidation tests on spent fuel and UO<sub>2</sub> by measuring the weight gained during the oxidation process in a low-temperature oven over long time periods. This activity will be performed at the Pacific Northwest Laboratories (PNL) of Battelle in Richland, WA. This activity plan describes performance details for this activity according to the guidelines prescribed in LLNL-YMP Quality Procedure 033-YMP-QP 3.0.

# 1.1 Activity Identity

The activity number assigned in the SIP (Spent Fuel Waste Form Testing) [1] for the oxidation tests on spent fuel and UO<sub>2</sub> using a low-temperature oven method is D-20-45.

# 1.2 Quality Assurance Level Assignment

Activity D-20-45 is assigned as QA Level I (see Appendix A).

# 1.3 Responsibilities

Key personnel responsible for performing the work in this activity are identified below:

Technical Area Leader:

Dr. Henry F. Shaw (LLNL)

Task Leader:

Dr. Ray B. Stout (LLNL)

Principal Investigator:

Dr. Robert E. Einziger (PNL)

Dr. Einziger will be supported by his colleagues, H. C. Buchanan (PNL), who has co-authored several papers on oxidation testing apparatus and the oxidation data measured [3-8] and Dr. W. J. Gray (PNL).

# 2.0 PURPOSE AND OBJECTIVES

Low-temperature experimental data on UO<sub>2</sub> spent fuel oxidation kinetics are necessary to develop performance assessment models that describe the behavior of the spent fuel in a

repository. The objective of these spent fuel experimental tests will be to evaluate the effects of variables such as moisture, temperature, burnup, and various other spent fuel characteristics on oxidation rate and phase formation, to evaluate and identify the various operative oxidation mechanisms, and to confirm results of an alternative short-term thermogravimetric testing method [4,5]. Results from these experimental tests will be used to develop a mechanistic model for the oxidation of UO<sub>2</sub> activity D-20-50.1, "Generate Models for Release of Radionuclides from the Spent Fuel Waste Form."

# 3.0 ACTIVITY DESCRIPTION

This low-temperature oven oxidation testing activity will be performed under the current LLNL-YMP QA Program Plan (QAPP) 033-YMP-R [2]. Some initial oxidation testing and scoping studies of spent fuel was conducted under the previous LLNL Nuclear Waste Management Program QAPP. This experience has been incorporated into this activity plan.

The sequence of steps in the activity and the connections with model development and leaching activities in the spent fuel task WBS 1.2.2.3.1.1 are illustrated in Figure 1.1 (pp 1.2) of the test plan entitled, "Test Plan for Thermogravimetric Analyses of BWR Spent Fuel Oxidation" document no. PNL-6745 [5]. Defining a set of precise decision points that identify completion of the activity is difficult because the activity for obtaining experimental data of oxidation kinetics and the activity for model development of oxidation kinetics are complementary, iterative and continuously coupled. However, at this stage in activity planning for low-temperature oven oxidation tests, decision points occur when:

- i sufficient experimental data are provided to support or refute a proposed two stage mechanism of an initially rapid grain boundary oxidation process and then a slower grain volume oxidation process;
- ii the proposed two stage mechanism is refuted, therefore, consideration needs to be given in possibly developing an alternative model for spent fuel oxidation kinetics with the available experimental data;
- sufficient experimental data are provided to analytically represent, through a parallel model development activity, the temperature, moisture, and description parameters of spent fuel characteristics (burnup, grain size, fission gas content, etc) effects on spent fuel

oxidation kinetics over the expected range of environmental history conditions expected in a repository.

# 3.1 Technical Reviews

At this time, no technical reviews are planned for this activity. However, at least two meetings per year between PNL and LLNL personnel are planned to discuss and report the status and future plans for low-temperature oven oxidation tests and, at which time, the need for technical reviews will be re-examined. These meetings will be in addition to the anticipated formal reports and papers that will be written to document the results and to distribute spent fuel oxidation data amongst all the related activities in YMP and the scientific community for review and comment by peers. The timing of these meetings will be determined, in part, by the progress of the experimental work.

# 3.2 Hold Points

No hold points for directional changes in testing are currently identified. However, the need for establishing hold points will be considered during each meeting described in Section 3.1 of this activity plan.

# 3.3 Equipment

The experimental equipment required for this activity is identified and described in Section 2.0 of the test plan entitled, "Test Plan for Long-Term, Low-Temperature Oxidation of BWR Spent Fuel" document no. PNL-6427. This test plan is attached as Appendix B.

# 3.4 Materials

Initial oxidation tests have been conducted and are still continuing for PWR fuel samples (Series Test 1) and BWR fuel samples (Series Test 2) which are described in sections 1.2 and 2.0 of Appendix B. New samples of UO<sub>2</sub> spent fuel used in this activity are identified and described in the addendum to Test Plan for Long-Term, Low-Temperature Oxidation of High Burnup Spent Fuel. This addendum is attached as Appendix C. These samples are obtained from the Material Characterization Center (MCC) [9] which has been assigned the responsibility by DOE-HQ of providing QA Level I

specimens for spent fuel oxidation testing by YMP. Additional samples of different fuel types will be added to the test matrix as they become available. This will greatly increase the amount of data that can be acquired for use in oxidation modeling development activities and performance assessments.

# 3.5 Special Environmental Conditions

The test specimens are radioactive and the oxidation testing conditions require temperature and moisture control. The environmental testing conditions are described in Sections 2.1.3, 2.1.5, and 2.3 of Appendix B.

# 3.6 Special Training/Qualification Requirements

Training will be required for personnel performing work in this activity relative to the procedure for low-temperature oven oxidation tests, technical implementing procedures listed in Section 8.0 of this activity plan, and appropriate examination procedures discussed in Section 2.7 of Appendix B. Training will be accomplished through reading assignments and on-the-job supervision, as appropriate, to gain and demonstrate proficiency. Training documentation will be included in the Personnel Qualification Records at PNL.

# 3.7 Activity Closeout

At the completion of the low-temperature oven oxidation testing, any remaining QA records such as scientific notebooks and technical reports will be submitted to the LLNL-YMP Local Records Center.

# 4.0 PRECISION AND ACCURACY

The overall measurement error of the low-temperature oven oxidation testing is specified at less than 20% in Section 1.2 of Appendix B.

# 4.1 Calibration Requirements

All measurement instrumentation (i.e., balances, thermocouples, and data recorders) will be calibrated against National Institute of Standards and Technology (NIST) traceable reference standards. Calibration procedures and requirements are given in PNL

Technical Implementing Procedure (TIP) No. SFO-2-1. This section supersedes the fourth sentence in section 2.4.1 of Appendix B.

# 4.2 Conditions That May Adversely Affect Results

Progress and results may be adversely affected by understaffing and personnel changes during the course of an experiment in progress. Also, as discussed under model evaluation in section 2.F of the test plan entitled, "Test Plan for Series 2 Thermogravimetric Analyses of Spent Fuel Oxidation", HEDL-7556 [4], there exist uncertainties in the current understanding of low temperature UO<sub>2</sub> spent fuel oxidation kinetics which make these tests non-routine and developmental in nature. Thus, changes and updates in experimental procedures and directions should not come as future surprises, although none are currently anticipated. Uncertainties in mechanistic interpretations for oxidation kinetics have been discussed in the report entitled, "Technical Test Description of Activities to Determine the Potential for Spent Fuel Oxidation in a Tuff Repository", HEDL-7540 [3].

# 4.3 Sources of Uncertainty and Error to be Controlled and Measured.

Once the test specimens are provided, sources of experimental error are moisture measurements, temperature measurements, weight measurements, and elapsed time measurements. The temperature and weight measurements are the most critical for this test procedure. The temperature control limit of  $\pm$  3°C at temperatures up to 300°C and weight change limit of  $\pm$  0.01% are given as controllable measurements in the Section 2.3 of Appendix B.

## 5.0 IN-PROCESS DOCUMENTATION

Documentation to be generated during the conduct of this activity include: scientific notebooks; magnetic computer disks, and photographs. Scientific notebooks are controlled and maintained in accordance with PNL's Act Now Directive 89-1 entitled, "Use of Laboratory Record Books (MG 4.3, Research Records)." Records will be transferred to the PNL Records Center for storage and maintenance prior to turnover to LLNL-YMP on an annual basis.

# 5.1 Data Recording and Data Reduction

The data acquisition system and data reduction techniques are described in PNL TIP No. SFO-2-1.

# 5.2 Analysis

Section II.F of Reference 4 discusses existing references, phenomenological models, and correlation functions to obtain empirically fitted models. This approach will be augmented with the model development for the oxidation of UO<sub>2</sub> activity D-20-50.1 as described in the LLNL-YMP Scientific Investigation Plan for Spent Fuel Waste Form Testing YMP WBS Element 1.2.2.3.1.1 [1]. The experimental testing and the model development activities will be carried out in parallel with close and continuous technical interchanges to maintain consistent and contiguous data input quality and model prediction capability.

# 6.0 INTERFACES

Activity D-20-45 involves experimental tests for obtaining data on oxidation rates and the various oxidation states of UO<sub>2</sub>. These data will be used in activity D-20-50.1 which is the activity for developing a mechanistic model of oxidation kinetics that can be extrapolated to the time domain for repository environmenal conditions. Activity D-20-45 is planned to be conducted in parallel with activity D-20-50.1. This will allow information to be "continuously" interchanged between the two activities. The Technical Area Leader for both of these activities is Dr. Henry F. Shaw, of LLNL. The Task Leader for both of these activities is Dr. Ray B. Stout, of LLNL. Thus, two levels of activity managers have direct technical information and budget control over the coordination between these activities.

Within the Spent Fuel Waste Form Testing YMP WBS Element 1.2.2.3.1.1, data and specimens at various oxidized states will be provided to activities for dissolution/leach testing. These activities are D-20-42, D-20-43, and D-20-53; and the Task Leader is Dr. Herman Leider, of LLNL. Other information, in terms of both experimental data and models developed, are provided to activities under the control of the LLNL-YMP Waste Package Performance Assessment WBS 1.2.2.5.1.

# 7.0 SCHEDULE

# 7.1 Duration

The duration of this activity is governed by the model development activity D-20-50.1 since this activity is planned to be an ongoing activity where data collected will be continuously provided to update and improve this model and provide input in future performance assessments. The duration of activity D-20-50.1 is approximately 7 years where the final model development will be provided into the final Licensing Application Design Performance Assessment.

The estimated test durations for the currently planned Series 3 oven oxidation testing are listed in Table 2.2 of Appendix C.

# 7.2 Staffing Requirements

Estimated staffing requirements are shown in Appendix D. Staffing requirements are based on the currently planned Series 3 oven oxidation tests described in Appendix C. As more fuel samples become available and are added to the test matrix, these estimates will be revised accordingly.

# 8.0 TECHNICAL IMPLEMENTING PROCEDURES

Procedures for performing the tests are discussed in Section 2.7 of Appendix B. In addition, the following TIPs will also be used:

- PNL TIP No. SFO-2-1.

# 9.0 SPECIAL CASES (PROCUREMENT)

The experimental testing, data acquisition/storage and some preliminary data analyses are performed and managed by the Principal Investigator, Dr. Robert Einziger of Pacific Northwest Laboratories (PNL) in Richland, WA 99352 as described in Appendix B.

# 9.1 QA Requirements Specifications

Work to be performed under this activity plan will be in accordance with the latest revision of PNL-MA-70 QA Plan No. WTC-018, which is consistent with the requirements of LLNL-YMP QA Requirements Specification No. QARS-001C. This section supersedes the second sentence in section 2.9 of Appendix B.

# 9.2 Statement of Work

The description of the work to be performed by the Principal Investigator, Dr. Robert Einziger of PNL, is provided in Appendix B.

# 9.3 Subcontractor Interface Control

The technical contacts and interfaces between LLNL-YMP and PNL are described in the Special Client Requirements Section, Part B16.0 of the PNL-MA-70 QA Plan No. WTC-018. This section also describes the documents/reports (i.e., Technical Procedures, Reports and Test Plans) to be submitted by PNL to LLNL-YMP for review and approval. Informal memo and telephone exchanges will be documented in LLNL-YMP controlled scientific notebooks assigned to this activity.

# 9.4 Materials/Equipment Provided

At present, no materials and equipment are expected to be provided directly from LLNL-YMP to PNL for TGA oxidation testing under activity D-20-45. The UO<sub>2</sub> spent fuel specimens will be obtained from MCC [9] as previously discussed in Section 3.4 of this activity plan. All other testing equipment is currently available at PNL.

# 9.5 Deliverables

As described in Section 2.8 of Appendix B, periodic progress reports, formal reports, and journal papers will be provided as warranted. Currently, monthly progress reports, test plans, formal reports, and papers for journal publication are submitted by PNL to LLNL-YMP for review and approval.

# 10. REFERENCES

- 1. LLNL-YMP Scientific Investigation Plan for Spent Fuel Waste Form Testing Rev. 1 (1989), Lawrence Livermore National Laboratory, Livermore, CA.
- 2. LLNL-YMP QA Program Plan (QAPP) 033-YMP-R (1989), Lawrence Livermore National Laboratory, Livermore, CA.
- 3. Einziger, R. E., (1985), Technical Test Description of Activities to Determine the Potential for Spent Fuel Oxidation in a Tuff Repository, HEDL-7540, Westinghouse Hanford Co., Richland, WA.
- 4. Einziger, R. E., (1986), Test Plan for Series 2 Thermogravimetric Analyses of Spent Fuel Oxidation, HEDL-7556, Westinghouse Hanford Co., Richland, WA.
- 5. Einziger, R. E., (1988), Test Plan for Thermogravimetric Analyses of BWR Spent Fuel Oxidation, PNL-6745, Pacific Northwest Laboratory, Richland, WA.
- 6. Einziger, R. E., and Woodley, R. E., (1985), Evaluation for the Potential for Spent Fuel Oxidation Under Tuff Repository Conditions, HEDL-7452, Westinghouse Hanford Co., Richland, WA.
- 7. Einziger, R. E., and Buchanan, H. C. (1988), Long-Term, Low-Temperature Oxidation of PWR Spent Fuel: Interim Transition Report, WHC-EP-0070, Westinghouse Hanford Co., Richland, WA.
- 8. Thomas, L. E., Einziger, R. E., and Woodley, R. E., (1989), Microstructural Examination of Oxidized PWR Fuel by Transmission Electron Microscopy, J. Nuclear Materials (in press).
- 9. MCC Characterization Plan for MCC Approved Testing Materials Draft Copy, (1989), Pacific Northwest Laboratory, Richland, WA.

# 11. APPENDICES

- 11.1 Appendix A QA Level Assignment Sheets for Low-Temperature Oven Oxidation Tests.
- 11.2 Appendix B Test Plan for Long-Term, Low-Temperature Oxidation of BWR Spent Fuel, PNL-6427 by Robert E. Einziger, December 1988.
- 11.3 Appendix C Addendum to Test Plan for Long-Term, Low-Temperature
  Oxidation of High Burnup Spent Fuel by Robert E. Einziger, August 4, 1989.
- 11.4 Appendix D Estimated Staffing Requirements for Series 3 Oven Oxidation Tests.

# Appendix A - QA Level Assignment Sheets for Low-Temperature Oven Oxidation Tests

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		SUMMAR	RY SHEET		
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S.I.P. Identification: WASTE FORM	- SPEN	T FUEL			
Activity Description: OVEN OXIDA	ATION TE	STS, SPENT	FUEL	AND UO2	
Quality Assurance Level: I			QALA	Meeting Date:	<u>4/26/89</u>
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Meeting Attendees:					
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	DECISION CRITERIA RECORD					
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WBS No	1.2.2.3.1.1 Activity No. D-20-45					
S.I.P. Identil	lication: WASTE FORM - SPENT FUEL					
Activity Des	cription: OVEN OXIDATION TESTS, SPENT FUEL AND UO2					
Decision Criteria	Applicability					
I	NO HARDWARE (ENGINEERED ITEMS)					
A.I1	INDETERMINATE					
11.8	NO SOFTWARE DESIGN					
II.C.1	INDETERMINATE					
II.C.2	APPLICABLE					
11.0.1	APPLICABLE					
II.E	NOT APPLICABLE - NO HISTORICAL REPORTS					
II.F	NOT APPLICABLE - NO ENVIRONMENTAL/SOCIOECONOMIC STUDIES					
II.G.1	APPLICABLE					
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# **GRADED QA CONTROL SPECIFICATION RECORD**

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WBS No. 1, 2, 2	3.1.1	Activity No	D-20-45
S.I.P. Identification:	WASTE FORM - SPENT I	FUEL	·
Activity Description:	OVEN OXIDATION TEST	S, SPENT	FUEL AND UO2
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YES YES	Section I - ORGANIZATION Section II - QA PROGRAM Section III - SCIENTIFIC INVES	STIGATION	033-YMP-QP 1.0 033-YMP-QP 2.series
YES YES	& DESIGN CO	ONTROL	
YES_ YES_			
<u> YES </u> <u>YES </u>	1.4 Scientific Investigation Data In and Analysis     1.5 Use of Computer Programs	terpretation	
YES_	1.6 The Use of Scientific Notebook     Use of Technical Implementing     1.7 Change Control		
YES YES	1.8 Interface Control 1.9 Verification of Scientific Invest		
YES YES	1.10 Surveillance of Scientific Inve- and Experiments 1.11 Reports, Conclusions and Re		s
YES_	1.12 Close-Out Verification 2.0 Design Control - NO EN	IG I NEERED	ITEMS INVOLVED
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YES	Section IV - PROCUREMENT DOCUMENT CONTROL	
YES_YES_	1.0 Requirements     1.1 Measures to Assure Adequate Qualit     2.0 Additional Requirements for Level I Activiti	
YES_	2.1 Content of Procurement Documents 2.2 Procurement Document Review	
YES YES	2.3 Procurement Document Changes 2.4 Distribution of Procurement Docume	
	Section V - INSTRUCTIONS, PROCEDURE PLANS, AND DRAWINGS	033-YMP-QP 5.0
<u> </u>	Section VI - DOCUMENT CONTROL Section VII - CONTROL OF PURCHASED	033-VMP-QP 5.0
YES	ITEMS AND SERVICES 1.0 General Requirements	
YES	1.1 Procurement Planning	•
YES YES	1.2 Source Evaluation and Selection 1.3 Bid Evaluation	·
YES	1.4 Supplier Performance Evaluation 1.5 Control of Documents Generated by Suppliers	$\sim$
YES	1.6 Acceptance of Item or Service 1.7 Acceptance of Services Only	
YES	1.8 Control of Supplier Nonconformance	ez
YES YES	2.0 Commercial-Grade tems 2.1 Alternatives	
YES	Section VIII - IDENTIFICATION AND CONTROL OF ITEMS, SAMPLES AND DATA	
NO	Part A - Identification and Control of Items -	- NO ENGINEERED (ITEMS INVOLVE
NO	1.0 Identification 1.1 General	
YES NO.	2.0 Control Part B - Identification and Control of Sample	25
YES	1.0 Identification 1.1 General	
YES	Part C - Identification and Control of Data 1.0 Identification	
NO_YES_	1.1 General Section IX - CONTROL OF PROCESSES	S - NO SPECIAL PROCESSES
<u>NO</u>	1.0 General Requirements 2.0 Process Control	
NO	2.1 Method 2.2 Identification of Special Processes	
NO	2.3 Qualification of Special Process	
NO	Procedures 2.4 Qualification of Personnel	
NO	Performing Special Processes  2.5 Special Process Equipment	<b>V</b>
NO	2.6 Special Process Records	~

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	NO NO	Section X - INSPECTION 1.0 General Requirements 2.0 Personnel	NO ENGINEERED ITEMS INVOLVED
	NO NO	2.1 Reporting Independence of Person 2.2 Qualification	nei
	_ <u></u>	3.0 Inspection Hold Points 4.0 Inspection Planning	
	-04-	4.1 Sampling 5.0 In-Process Inspection	•
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	<u> </u>	6.0 Final Inspection 6.1 Inspection Requirements 6.2 Acceptance	
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	NO NO	7.1 Methods 8.0 Qualifications Requirements	
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	NO	<b>Drawings</b>	
	_NC	3.3 Review of Procedures	
i	-MC	3.5 Alternatives	
	NO NO	4.0 Test Results 5.0 Records	
	YES	Section XII - CONTROL OF MEASURING AND TEST EQUIPMENT 1.0 General	G
	YES.	1.1 Maintaining Accuracy of Equipme	nt
		1.3 Description of Responsibilities 2.0 Purpose of Equipment	
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Page	
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APPLICABLE (YES/NO)	DESCRIPTION	JUSTIFICATION OR CONTROL PROCEDURE(S)
YES	Section XIII - HANDLING, SHIPPING AND STORAGE	
YES YES YES YES YES	1.0 General 1.1 Special Equipment and Protective Environments 1.2 Specific Procedures 1.3 Inspection and Testing of Special Tools 1.4 Operators of Special Equipment 1.5 Marking and Labeling	
<u>NO</u>	Section XIV - INSPECTION, TEST AND OPERATION STATUS	NO ENGINEERED ITEMS
NO NO NO	1.0 Indication of Status     2.0 Methods of Indicating Status     3.0 Application and Removal of Status Indicators	
YES	Section XV - CONTROL OF NONCONFORMING ITEMS	033-YMP-QP 15.0
YES YES	Section XVI - CORRECTIVE ACTION Section XVII - QUALITY ASSURANCE RECORDS	033-YMP-QP 16.00 033-YMP-QP 17.00
YES	Section XVIII - AUDITS	033-YMP-QP 18.00

Supplemental Controls Required:					
NONE					
Justification:		•			

Remarks:

NONE

Appendix B - Test Plan for Long-Term, Low-Temperature Oxidation of BWR Spent Fuel

# Test Plan for Long-Term, Low-Temperature Oxidation of BWR Spent Fuel

R. E. Einziger

December 1988

Prepared for Lawrence Livermore National Laboratory under a Related Services Agreement with the U.S. Department of Energy Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
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001-025	N	A02
026-050		A03
<b>0</b> 51-07 <b>5</b>		A04
076-100		A05
101-125		A06
126-150		A07
151-175		80A
176-200		A09
201-225		A10
226-250		A11
251-275		A12
276-300		A13
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TEST PLAN FOR LONG-TERM, LOW-TEMPERATURE OXIDATION OF BWR SPENT FUEL

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# SUMMARY

Preliminary studies indicated the need for more spent fuel oxidation data in order to determine the probable behavior of spent fuel in a tuff repository. Long-term, low-temperature testing was recommended in a comprehensive technical approach to 1) confirm the findings of the short-term thermogravimetric analysis tests; 2) evaluate the effects of variables such as burnup, atmospheric moisture, and fuel type on the oxidation rate; and 3) extend the oxidation data base to representative repository temperatures and better define the temperature dependence of the operative oxidation mechanisms.

This document presents the test plan to study the effects of atmospheric moisture and temperature on oxidation rate and phase formation using a large number of boiling-water reactor fuel samples. Tests will run for up to two years, use characterized fragmented and pulverized fuel samples, cover a temperature range of 110°C to 175°C, and be conducted with an atmospheric moisture content ranging from <-55°C to ~80°C dew point. After testing, the samples will be examined and made available for leaching testing.

# CONTENTS

SUMM	ARY .	••••••••••••••••••••••	iii	
1.0	INTR	ODUCTION 1	1.1	
	1.1	OBJECTIVE OF DRY BATH TESTING	1.1	
	1.2	BACKGROUND	1.2	
2.0	TEST	DESCRIPTION	2.1	
	2.1	TEST MATRIX	2.1	
		2.1.1 Test Temperature	2.1	
		2.1.2 Fuel Particle Size	2.2	
		2.1.3 Moisture in the Atmosphere	2.4	
		2.1.4 Number of Samples and Test Duration	2.4	
		2.1.5 Radiation Fields	2.5	
	2.2	SAMPLE SELECTION, PREPARATION, AND IDENTIFICATION	2.6	
	2.3	EQUIPMENT DESCRIPTION	2.9	
	2.4	.4 TEST OPERATION		
		2.4.1 Initial Startup	2.10	
		2.4.2 Interim Examination	2.10	
		2.4.3 Final Examination	2.11	
2.5		POST-TEST SAMPLE EVALUATION	2.11	
		2.5.1 Ceramography	2.11	
		2.5.2 X-Ray Diffraction	2.12	
		2.5.3 Scanning Electron Microscopy	2.12	
		2.5.4 Transmission Electron Microscopy and Electron Diffraction	2.12	
	2.6	ANALYSIS OF DATA	2.14	
	2.7	DDUCEUIDES	2 1/	