PNNL-10944
UC-602

K-Basin Spent Nuclear Fuel Characterization Data Report II

J. Abrefah
W. J. Gray
G. L. Ketner
S. C. Marschman
T. D. Pyecha
T. A. Thornton

March 1996

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

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute

Battelle

PNNL-10944

Use this Legal Notice for an 1830 Report Available to the Public

DISCLAIMER

PACIFIC NORTHWEST NATIONAL LABORATORY
operated by
BATTELLE MEMORIAL INSTITUTE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-AC06-76RLO 1830

Printed in the United States of America

Available to DOE and DOE contractors from the Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831; prices available from (615) 576-8401.

Available to the public from the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Rd., Springfield, VA 22161

This document was printed on recycled paper.

PNNL-10944
UC-602

K-Basin Spent Nuclear Fuel Characterization Data Report II

J. Abrefah
W. J. Gray
G. L. Ketner
S. C. Marschman
T. D. Pyecha
T. A. Thornton

March 1996

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

Pacific Northwest National Laboratory
Richland, Washington 99352

Summary

An Integrated Process Strategy has been developed to package, condition, transport, and store in an interim storage facility the spent nuclear fuel (SNF) currently residing in the K-Basins at Hanford. Information required to support the development of the conditioning process and to support the safety analyses must be obtained from characterization testing activities conducted on fuel samples from the Basins. Some of the information obtained in the testing was reported in PNL-10778, *K-Basin Spent Nuclear Fuel Characterization Data Report* (Abrefah et al. 1995). That report focused on the physical, dimensional, metallographic examinations of the first K-West (KW) Basin SNF element to be examined in the Postirradiation Testing Laboratory (PTL) hot cells; it also described some of the initial SNF conditioning tests.

This second of the series of data reports covers the subsequent series of SNF tests on the first fuel element. These tests included optical microscopy analyses, conditioning (drying and oxidation) tests, ignition tests, and hydrogen content tests.

The results of the conditioning, hydrogen content, and ignition testing of the element sections may be summarized as follows:

- An SNF specimen with added water was cold vacuum dried (CVD) at 60 Torr and 323 K, hot vacuum dried (HVD) at 60 Torr and 573 K, and then conditioned at 473 K in 2% oxygen/98% argon mixture. The added water came off during the first 3 hours of the CVD cycle. Relatively little moisture evolved during the HVD cycle. A small oxygen pickup was noted during passivation.
- Two SNF specimens with free water added were CVD at 60 Torr and 323 K, HVD at 60 Torr and 573 K, and then conditioned at 523 K in 2% oxygen/98% argon mixture at 1 atm. Most of the added water came off during the CVD cycle. Relatively little moisture was observed during the HVD cycle. A total of 27 mg of oxygen was picked up during passivation. The two specimens lost a total of 505.6 mg of weight as a result of oxide spalling. Analysis of the spalled residue indicated that U_4O_9 and U_3O_8 were the predominant chemical phases. -The moisture content in the off-gas stream during the passivation cycle in this run was higher than in tests when water was not added to the specimens
- Hydrogen release was measured from a decladded fuel specimen at 573 K for about 24 hours and at 623 K for 96 hours in an argon gas flow of 100 cc/min. System pressure was about one atmosphere. The concentration of hydrogen gas peaked during the temperature ramp. The total hydrogen measured was 0.12 mg. An appreciable amount of moisture was also detected in the gas stream.
- Significant amounts of both moisture and hydrogen were released from cladding material peeled off the damaged end of the element. These values peaked sharply (about 260 ppm moisture and 75 ppm hydrogen in the gas stream) during the temperature ramp but, remained fairly high throughout the run.

- Hydrogen release was measured from a decladded fuel specimen sectioned from the uncorroded mid-length of N-reactor fuel element. A total of about 1.7 mg of hydrogen was released from the sample between 573 K and 748 K. The sample was not depleted of hydrogen at the end of the test.
- Hydrogen release was measured from cladding material at 573 K. The initial release of hydrogen occurred during the temperature ramp. The release peaked at about 13 ppm (volume fraction in the gas stream) and decreased quickly to below detection after the first 4 hours. The total hydrogen release was about 10 micrograms
- In ignition tests on two unconditioned fuel specimens in dry air flowing at 500 cc/min and a temperature ramp rate of about 15 K/min, ignition occurredat about 913 K. Hydrogen released during the oxidation reaction was small, but when the oxidation reaction was terminated by switching the gas supply from dry air to pure argon, hydrogen content in the offgas increased as the the specimen cooled. Post-test examinations of the specimens revealed extensive surface oxidation and a powdered residue in the specimen boat.
- Ignition of conditioned fuel specimens in dry air flowing at 500 cc/min and a temperature ramp rate of 15 K/min could not be discerned from the specimen temperature plot. Again, little hydrogen release occurred during the oxidation reaction, but when the oxidation reaction was terminated by switching the gas supply from dry air to pure argon, the hydrogen content in the off-gas stream increased as the speciment cooled. Post-test examinations of the specimens revealed extensive surface oxidation and a uranium oxide residue in the specimen boat.
- Ignition was again indiscernible in an ignition test on another conditioned fuel specimen in dry air at 500 cc/min and a temperature ramp rate of 15 K/min again, even though the maximum set temperature for the furnace was increased from 973 K (for the previous tests) to 1073 K. The pattern of hydrogen release was the same as in previous ignition tests.
- In two ignition tests conducted on samples that had been cut from the damaged end of an N-Reactor outer fuel element stored in the K-West basin canister 4378 in dry air, ignition occurred at about 551 K. On post-test examination, the sample surfaces were oxidized and powdered residue (likely oxides of uranium) was present in the sample boat. The hydrogen and moisture content measured in the off gas stream during the temperature ramp were much higher than in tests on undamaged samples from the mid-section of the same element.

Most of the oxides formed on the cut uranium surfaces were nonadherent and spalled to generate oxide residue. Analysis of the residue by X-ray diffractometer (XRD) identified the chemical phase to be predominantly UO_{2+x} (U_4O_9 and U_3O_7) and a minor component of U_3O_8. Analysis of particulates from the damaged end of SNF element SFEC5,4378 by XRD identified a hydrated oxide phase, $UO_3.2H_2O$, in addition to the higher oxide phases.

Acknowledgments

We extend our appreciation to the Postirradiation Test Laboratory staff for their relentless efforts in providing the test specimens. Our thanks go to Ray Bell for his assistance in performing the furnace tests, to George Salazar for providing the optical photomicrographs, and to Craig Buchanan, Evan Jenson, and JimYoung for providing the scanning electron microscope, X-ray diffractometer, and Energy Dispersive Analysis for X-Ray results.

Acronyms and Initialisms

CVD	Cold Vacuum Drying
DQO	Data Quality Objective
GC	Gas Chromatograph
HVD	Hot Vacuum Drying
ICDD	International Centre for Diffraction Data
IPS	Integrated Process Strategy
ITA	Independent Technical Assessment Group
LRB	Laboratory Record Book
MCO	Multi-Canister Overpack
PNNL	Pacific Northwest National Laboratory
PTL	Postirradiation Testing Laboratory
ppm	parts per million by weight
XRD	X-Ray Diffractometer
SFEC	Single Fuel Element Canister
SNF	Spent Nuclear Fuel
TI	Test Instruction
WHC	Westinghouse Hanford Company

Contents

Figures

Section 2

Section 3

Appendix B

Tables

1.0 Introduction

The Spent Nuclear Fuel (SNF) project characterization activities provide technical data on SNF stored at the K Basins that will support the disposal pathway of SNF in interim storage. The first characterization data report (Abrefah et al. 1995) on this effort provided the results of the visual inspection of fuel samples, physical characterization (weight and immersion density measurements), metallographic examinations, and controlled atmosphere furnace testing of three fuel samples shipped from the KW Basin to the Post-Irradiation Testing Laboratory (PTL). This report continues the data reporting activity providing the results of further metallographic examinations, conditioning process testing, and ignition testing in support of the pathway.

The test data about the response to the conditioning process supports decisions related to conditioning process design and safety analyses. The testing generally consisted of conditioning sibling SNF specimens, metallographic analyses on unconditioned and conditioned SNF specimens, and ignition testing on unconditioned and conditioned specimens from the middle (undamaged) area and the damaged end of SNF element SFEC5, 4738, transported to the PTL in the first shipment.

The first set of data quality objectives (DQOs) for the first shipment of KW-Basin SNF samples to be characterized were developed in *Data Quality Objectives for the Initial K West Fuel Examinations* (Lawrence et al. Feb. 1995). Those DQOs focused on placing the SNF in multi-canister overpacks (MCOs), transporting the MCOs to the interim storage facility, and short-term storage.

Additional DQOs, related to the Path Forward/IPS SNF conditioning requirements at the interim storage facility, are provided in *Data Quality Objectives for the Initial Fuel Conditioning Examinations* (Lawrence 1995). This Test Report describes conditioning and ignition test data and supporting analyses on the first KW-Basin SNF samples to be used in support of this conditioning DQO. The testing consisted of total hydrogen content analyses, conditioning tests, metallographic examinations of unconditioned (as-cut) and conditioned specimens, and ignition tests on unconditioned and conditioned specimens sectioned from SNF element SFEC5,4378 -The tests provided data required by the conditioning DQO to satisfy two generic information needs: Drying-Oxidation-Dissolution Kinetics and Pyrophoricity-Combustibility.

Optical Microscopy Analyses: These studies provide information concerning the type and quantity of corrosion products that have formed on the SNF during storage in the KW Basin, the effect of the corrosion on the mechanical integrity of the SNF assemblies, the approximate relative amounts of metal, oxide, and hydride to be loaded into MCOs in the basins, and the integrity of the oxide layer formed on the metal surfaces by the conditioning process.

Conditioning (Drying and Oxidation) Tests: These tests provide water-removal and oxygen-uptake data that will support decisions concerning the conditioning required for long term dry

storage. The furnace data concerning the dewatering, drying, hydrogen emission, and passivation kinetics of the as-received K Basin SNF specimens facilitate evaluation of 1) dewatering and/or drying as a step in the conditioning of canister sludge for interim storage, and 2) oxidation as a step in forming passivated fuel surfaces and/or chemically stable oxides. The data will be used to establish the fuel performance parameters for the pathway conditioning process, that is, the proposed Integrated Process Strategy (IPS 1995) process and/or the modified version that includes cold vacuum drying (CVD).

Ignition Tests: Ignition tests on the unconditioned and conditioned SNF samples support decisions related to the stability of the SNF before conditioning (for transportation and canister staging) and after conditioning (for the design and safety evaluation of the dry storage facility). The tests provide a basis for comparing the pyrophoricity of unconditioned and conditioned KW-Basin SNF from providing ignition curves for the conditioned samples and visual and/or metallographical determinations of the amount of potentially pyrophoric metal (U, Zr) or metal hydrides remaining.

Hydrogen Content Tests: These tests provided data to support analyses related to the pressurization of the MCO, the efficacy of the IPS conditioning process, and the potential for pyrophoric behavior of the fuel during design basis accidents. The test data provide a basis for estimating the total available hydrogen in the spent fuel in an MCO and in the off-gas stream during the conditioning process.

This report provides results from metallographic examinations and the controlled atmosphere furnace testing of specimens from SNF element SFEC5,4378.

2.0 Tests Performed

The series of examinations described herein addressed the hydrogen content, conditioning process response, and ignition characteristics of specimens taken from the undamaged middle and damaged end of a corroded KW-Basin fuel element (SFEC5,4378) from the first shipment of SNF to the PTL. The examinations included optical microscopy of IPS conditioned specimens. A comprehensive, up-to-date listing of the specimens taken is attached as Appendix C . Appendix C contains information about all the SNF samples generated to date and the test in which each was used. The sectioning of the element is shown schematically in Figure 2.1, and the details of the sectioning methodology are given in Test Instructions(TI) (Appendix D); 1. SNF-CT-003, 2. SNF-CT-006, 3. SNF-CT-008, and 4. SNF-CT-010.

2.1 IPS Drying And Conditioning Test

The hot-cell furnace test activities were performed in the PTL G-cell. A schematic of the furnace system used for the conditioning tests, its inlet gas control capabilities, and its off-gas analyses capabilities is shown in Figure 2.2. Specimen preparation and testing followed requirements in the TI listed in Table 2.1.

Table 2.1. Applicable Test Instructions for the Furnace Tests

Furnace Run #	Applicable TI
5 through 8	SNF-CT-007
9	SNF-CT-013
10	SNF-CT-014
11	SNF-CT-012
12	SNF-CT-015
13	SNF-CT-016
14	SNF-CT-018
15	SNF-CT-019
16	SNF-CT-025
17	SNF-CT-021
18	SNF-CT-026
19	SNF-CT-029

The SNF specimens used in furnace tests are listed in column 3 of Table 3.1 Each specimen was weighed and measured before the test and weighed after the test (Tables A.1 through A.15 in Appendix A). The dimensions of the specimens were determined from photographs. Figures B.1 through B.37 (Appendix B) show photographs of the following specimens SFEC5,4378-S2-F1 (B.1 - B.5), SFEC5,4378-S2-E2 (B.6 - B.11), SFEC5,4378-S2-E1-CD1 (B.12 - B. 15), SFEC5,4378-S2-E3A (B.16 - B.21), SFEC5,4378-S2-H (B.22 - B.27), SFEC5,4378-S1A-D (B.28 - B.32), and SFEC5,4378-S1A-H (B.33 - B.37).

Before being loaded in the furnace for the tests, the specimens were stored in a container filled with ultrahigh-purity argon. They were transferred from their temporary storage in E-cell to G-cell, the location of the furnace apparatus, just before loading. The TIs (Appendix D) describe the configuration of the test specimens in the furnace and detail the test parameters.

The basic conditioning cycle proposed in the IPS consisted of two steps:

- *Fuel drying*. The SNF was dewatered (free water removed) at 373K under vacuum for approximately 10 hours, dried at 573K under vacuum for approximately 24 hours (to remove water of hydration and partially decompose any uranium hydride present), and cooled to ambient cell temperature.

- *Fuel conditioning*. The fuel was exposed to a 98% helium-2% oxygen atmosphere at temperatures ranging from 423K to 523K for about 10 hours, to create a passive oxide film on any exposed uranium-metal surfaces. In the testing runs for this report, this step was conducted at near atmospheric pressure.

The off-gas from the tests was monitored only for moisture content during the drying steps, when the gas chromatograph (GC) could not be used to monitor the other components of the gas stream. Off-gas moisture, hydrogen, and oxygen were monitored during the conditioning step.

For each conditioning test, a specimen was fitted with a Type-K thermocouple. The furnace was purged at ambient temperature with ultrahigh-purity argon and dried to a moisture level of about 1 ppm, after which the time-temperature-atmosphere conditioning schedule instructed in the TIs was imposed. The moisture trap shown in Figure 2.2 was configured to have only one of the two parallel traps valved-in during the drying part of the conditioning cycle. At the end of the drying step, this trap was valved-off and the other trap valved-in. The first trap was weighed for integral analysis of the water removed from the specimens during the drying operation. Likewise, at the end of the conditioning part of the cycle the second trap was valved-off, removed for analysis, and replaced.

A continuous measurement of water emission from the specimens was provided by a moisture monitor placed in the off-gas train of the furnace, between the furnace and the moisture trap. This instrument measured the water in the off-gas by measuring the dew-point of the gas down to -

383K, corresponding to a detection limit of 1-10 ppm.

On completion of the conditioning cycle, the specimen(s) was removed from the furnace for visual examination and then stored in high-purity argon for later analysis.

After furnace run 15, the furnace system was reconfigured by connecting a bellows pump in parallel to the rotary pump (Figure 2.3) so that the GC could monitor the off-gas stream during the vacuum drying portion of the test. Run 16 was the first IPS drying test after this modification. Specimen SFEC5,4378-S2-F4 from the undamaged portion of the element was used.

2.2 Hydrogen Release Test

Hydrogen release tests were performed on decladded fuel specimens SFEC5,4378-S2-E1 (Run #7) and SFEC5,4378-S2-E2 (Run #9), cladding specimen 4378-S1-CLAD1 from the damaged end of the element (Furnace Run #8), and a defueled cladding specimen (SFEC5,4378-S2-CD1) taken from specimen E1 (Run #10).

The furnace system was dried by flowing high purity argon through it for about 24 hours while the lines were being heated to about 373K. Each specimen was mounted in the sample holder so that the specimen was in the center of the heated zone and in contact with the thermocouple. The sample holder was then loaded in the furnace tube and the rest of the system assembled. The furnace system was again purged with ultrahigh- purity argon flowing at 100 cc/min for at least 30 minutes. The furnace was then turned on and programed to heat at 10-15 K per minute to a stipulated temperature, where it was held constant for several hours until the next segment of heating cycle. The furnace was then allowed to cool, and the sample was recovered for weighing and further examination. The off-gas stream was analyzed for hydrogen by GC.

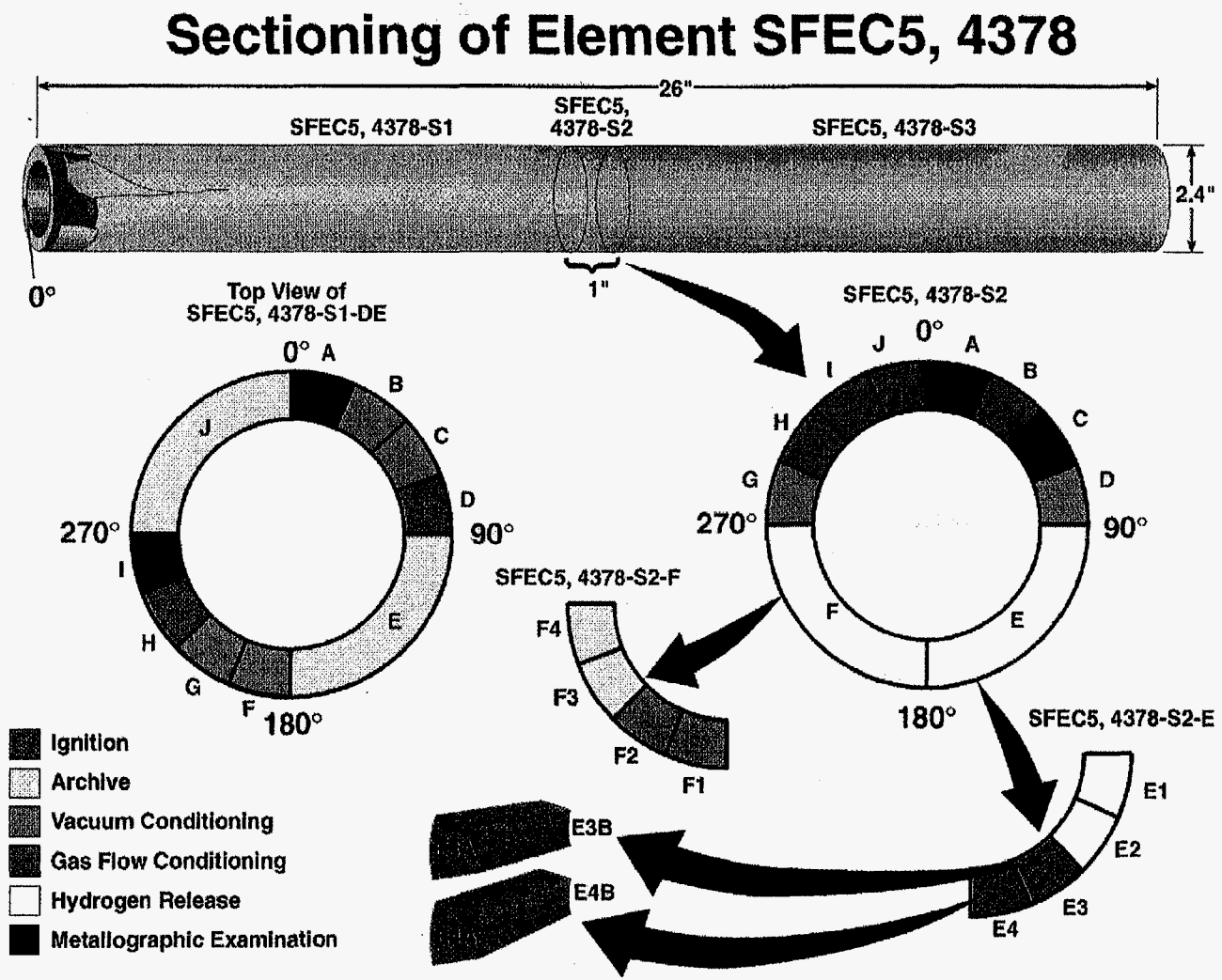


Figure 2.1. Sectioning diagram of SNF element SFEC5,4378, showing location of the test specimens.

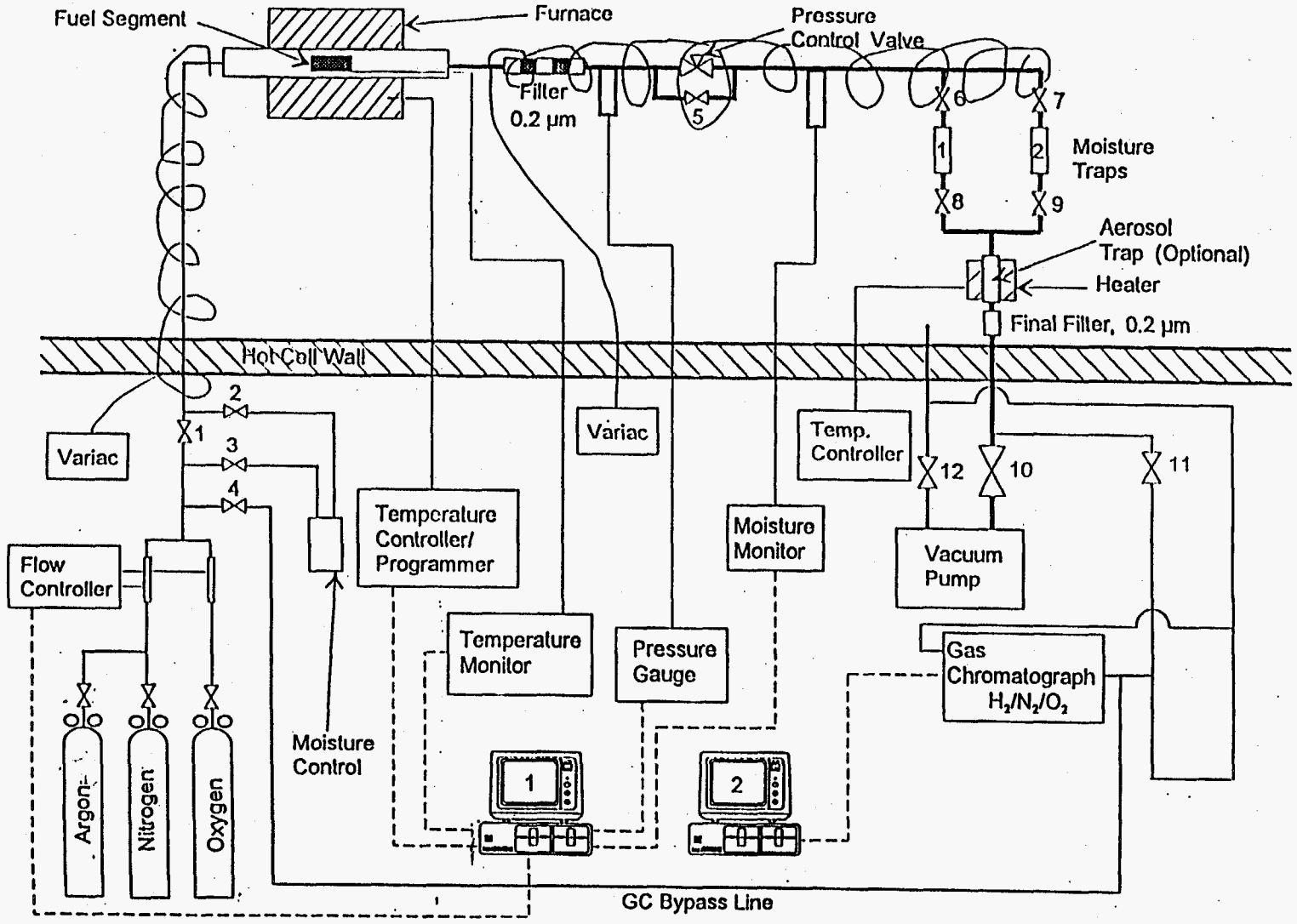


Figure 2.2: Schematic of the Furnace Testing System.

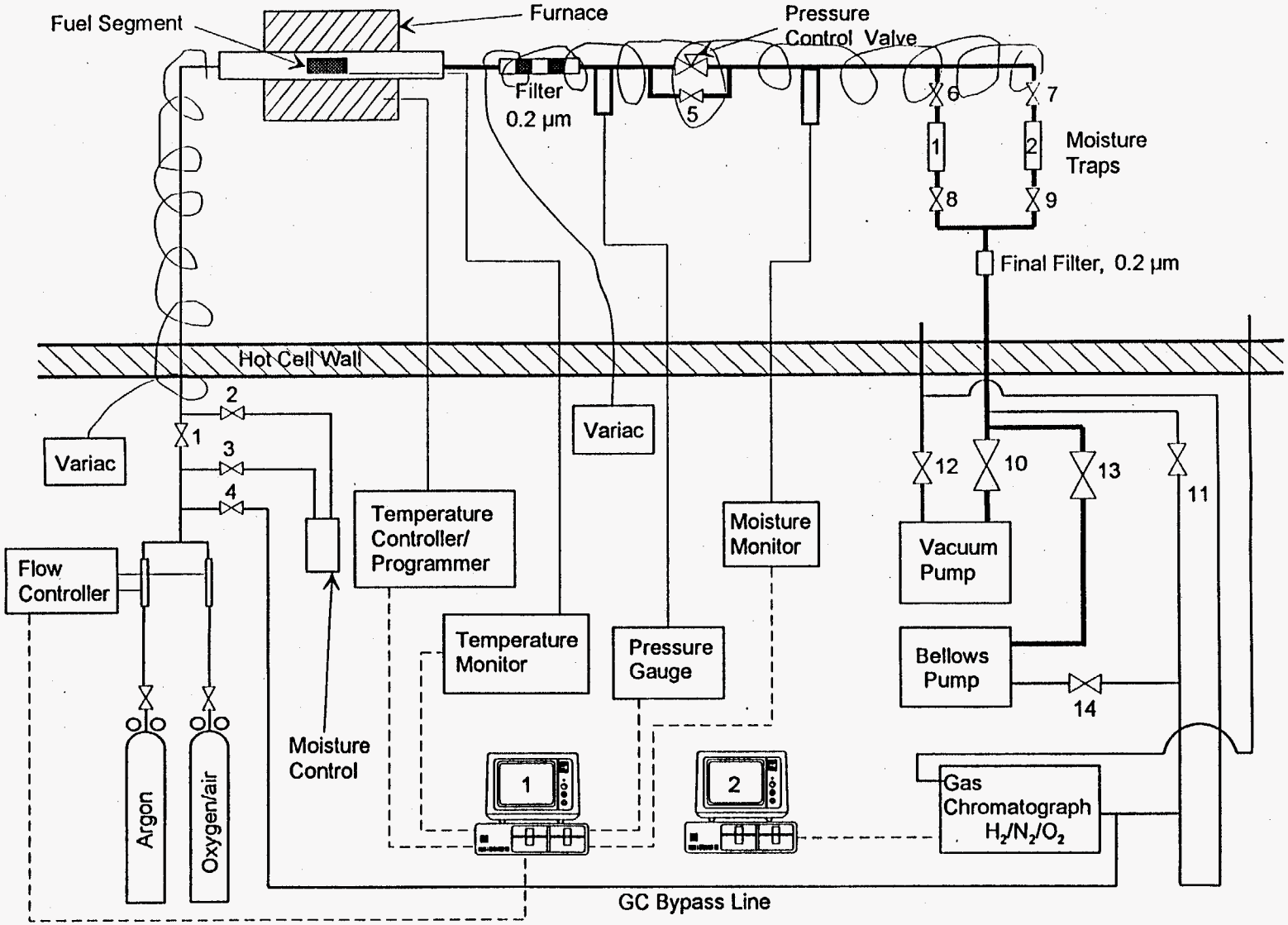


Figure 2.3: Schematic of the Furnace Testing System After the Installation of a Bellows Pump..

2.3 Ignition Test

Ignition testing was performed in an atmosphere of dry bottled air at the approximate schedule exemplified in Figure 3.6. The geometry/configuration in the furnace during ignition testing is described in the applicable TI (Table 2.1).

The specimens were placed in the hot-cell furnace. The atmosphere of the furnace was dried by pumping it to a low pressure and then purging with ultra high purity argon before the test. To prepare the as-cut test specimens, the annular fuel element was sectioned under an inert gas purge to minimize oxidation of the cut surfaces.

The ignition tests were done in a small (approximately 2.5 cm diameter) alumina-tube furnace. The specimens were tested with the cladding intact, and they were accurately weighed and measured prior to testing. A continuous measurement of water emission from the specimens during the ignition test was provided by a moisture monitor placed in the off-gas train of the furnace, and the hydrogen in the off-gas was monitored by GC.

To maintain dry-air atmosphere, bottled dry air was passed through the furnace at 500 cc/min during the tests. The furnace was heated at 15 K/min from the hot-cell ambient temperature to 973K during the tests. When each test was terminated, the furnace apparatus was flooded with dry argon to ensure any oxidation reactions which were occurring were stopped. The apparatus was then allowed to cool to the ambient hot-cell temperature and then opened. When the furnace was opened, the samples were visually examined, photographed, carefully removed, transferred to a weighing boat, and weighed.

The ignition temperature of the SNF specimen was determined graphically from the temperature history of the specimen as the point of intersection between the extension of the pre-ignition heating rate and the post-ignition self-heating rate.

2.4 Optical Microscopy

Metallography was performed on specimens to obtain information concerning microstructure, oxide thickness and hydride inclusions. The optical micrographs shown in this report are of conditioned specimens SFEC5,4738-S2-B and SFEC5,4378-S2-J. These specimens from the undamaged middle element SFEC5,4378 were put through the ITA conditioning process. Specimen 4378-S2-J was further subsectioned into specimens SFEC5,4378-S2-J1 and SFEC5,4378-S2-J2.

Cutting of the specimens was done in oil-based Hyprez™ coolant to reduce the likelihood that the fuel element would react with air. The sectioning followed Test Instruction SNF-CT-009 and was done in the E-cell air environment with an Isomet™ slow-speed saw. The approximate dimensions and configuration of the specimens are shown on the cutting diagram in the TI.

The approximately 22.5°-sector specimens were mounted for metallographic examination; 4378-S2-B and -J2 longitudinally, and -J1 transversely.

The samples were polished according to the procedure of Gardner and Riches (1956). The specimens were ground progressively through 180-, 240- and 600-grit silicon-carbide (SiC) abrasive paper and then with 800- to 1200-grit SiC abrasive paper lubricated with Hyprez OS Type IV (paraffin in kerosene). The specimens were then polished with a "gold-polishing" cloth (e.g., Glennel Corp. Gold-Label™ polishing cloth) in the following sequence:

- 4-8 micron diamond paste suspended in Hyprez OS Type IV lubricant
- 1-2 micron diamond paste suspended in Hyprez OS Type IV lubricant
- 0.05-micron Buehler Micropolish II™ or equivalent on a polishing cloth saturated with a 2% solution of chromic acid or silica slurry and use of the rotor- or the Vibra-polisher™ for 30 seconds to 5 minutes.

The polished surfaces were then partially oxidized under a heat lamp to highlight uranium hydride 'halos' and examined with the metallograph for the following series of photomicrographs:

- Fuel-surface structure at about 50 to 750X
- Fuel/cladding interface at about 50 to 750X
- Fuel edge at about 50 to 750X.

2.5 XRD Examination

Samples SFEC-5-4378-S2-FR2, 5-S1-PM1 and 5-S2-FR3 were examined by X-ray diffraction to identify the crystalline phases. Table 2.2 identifies and describes the samples.

The samples were prepared in the 325 Building Shielded Analytical Facility. Samples were prepared by pulverizing the material in a boron carbide mortar and pestle, adding collodion/amyl acetate to make a slurry, and a placing a few drops of the resulting slurry on a glass slide as described in Technical Procedure PNL-ALO-268. Only a few milligrams of sample were analyzed. The XRD running parameters were 0.02 degrees 2-theta step size, a count time (or dwell time) of 25 seconds/step (15 seconds for FR3), and a range of 5 to 65 degrees on the Scintag™ X-ray diffractometer. X-ray parameters were set at 45 KV and 40 ma with a copper anode. Slits were 4, 2, 0.5 and 0.3 mm. The instrument was within calibration limits. (Note - The inaccurate 1-second dwell time shown on the plots is due to a bug in the software).

Table 2.2. XRD Specimen Identification And Description

Sample	Description
FR2	SFEC5,4378-S2-FR2, Residue from Furnace Run 6 (SNF Specimens SFEC5,4378-S2-F1 & -F2 were used)
PM1	SFEC5,4378-S1-PM1, Particulate material from the damaged end of SFEC5,4378-S1 during removal of cladding piece.
PM1-DUP	Duplicate sample of PM1
FR3	SFEC5,4378-S2-FR3, Debris collected from first furnace tube after tube replaced (after Furnace Run 8).
FR3-DUP	Duplicate sample of FR3

Data reduction and search/match operations were performed with JADE 3+ software from Materials Data Inc. The ICDD data base through set 45 (December 1995) was used for search/match operations.

3.0 Data And Discussions

3.1 IPS Process Drying And Conditioning

3.1.1 Test Data

The results of the cold-vacuum drying (CVD) followed by a hot-vacuum drying (HVD) on SNF specimen SFEC5,4378-S2-F1 and -F2 (furnace run 6) are plotted in Figures 3.1a and 3.1b (Plots for furnace run 5, specimen SFEC5,4378-S2-G are shown in Figures B.38 and B.39 in Appendix B). The time course of the moisture concentration measured by the moisture monitor (Figure 3.1a) shows that most of the added water came off during CVD and the relatively small amount left desorbed during HVD. Figure 3.1b also depicts the conditioning step of the test. The isothermal oxidation step was conducted at 523K. The plot shows depletion in the oxygen concentration in the off-gas stream, that is, oxygen pickup by the specimens. Integration of the oxygen depletion yields a total of about 27 mg of oxygen. Before and after-test weighings indicate that the specimens lost a total of about 506 mg of material due to oxide spalling. Analysis of the residue by XRD (Section 3.5) identified U_4O_9 as the main chemical phase and U_3O_8 as a minor component. The moisture content in the off-gas stream (Figure 3.1b) during the conditioning step of this test was greater than in the tests without added water. During the conditioning step of run # 5 (Figure B.29) oxygen pickup by the specimen was less than the detection limit of the GC, and at 473K no hydrogen gas evolved from the specimen even though the moisture level in the off-gas reached about 5 ppm.

Figures 3.2a & 3.2b show the results of CVD, HVD, and conditioning (run 16) when the furnace system (Figure 2.3) was configured to monitor the gas stream during the fuel drying step. Hydrogen release during HVD reached a maximum of about 37 ppm in the gas stream during the temperature ramp and decreased to about 2 ppm at the end of that step, for a total of 0.031 mg of hydrogen as measured by GC. At 473K, the oxygen pickup by the specimen was undetectable by KGC, and no hydrogen evolved from the specimen (Figure 3.2b).

3.1.2 Discussion

The results of the vacuum drying indicate that the CVD might be adequate to remove most of the free water in the system. However, the specimen picked up additional moisture, which came off during HVD. Hydrogen gas was not detected during the drying steps (CVD and HVD) of runs 5 and 6 because the furnace system was not configured to monitor the off-gas stream under vacuum. In run 16 the system was set up to detect hydrogen, but hydrogen was measured only

during HVD. The lack of detctable hydrogen during CVD suggests that the specimen reacted minimally with free water as follows:

$$U + (2 + x)H_2O \rightarrow UO_{2+x} + (2 + x)H_2$$

and such reactivity might not contribute significant hydrogen to the gas stream. The hydrogen evolved during the HVD and conditioning portions of the test might be from two main sources: (a) reaction of uranium with the moisture in the fuel and/or gas stream, and (b) decomposition and/or reaction of uranium hydride with moisture and/or oxygen.

The consistency of probable oxide spalling (weight loss) from the specimens suggests that 10 hours probably is too long for oxidation at 523K probably is too long, if the objective is to form a passivated layer. The spalling of most of oxide layer formed by the oxidation, however, does not preclude a thin layer of oxide being left on the exposed uranium surfaces. The XRD analyses of the residue show that the higher uranium oxides (U_4O_9 and U_3O_8) phases were formed rather than the anticipated UO_{2+x}. These higher oxides may have formed non-adherent oxides, resulting in the weight loss.

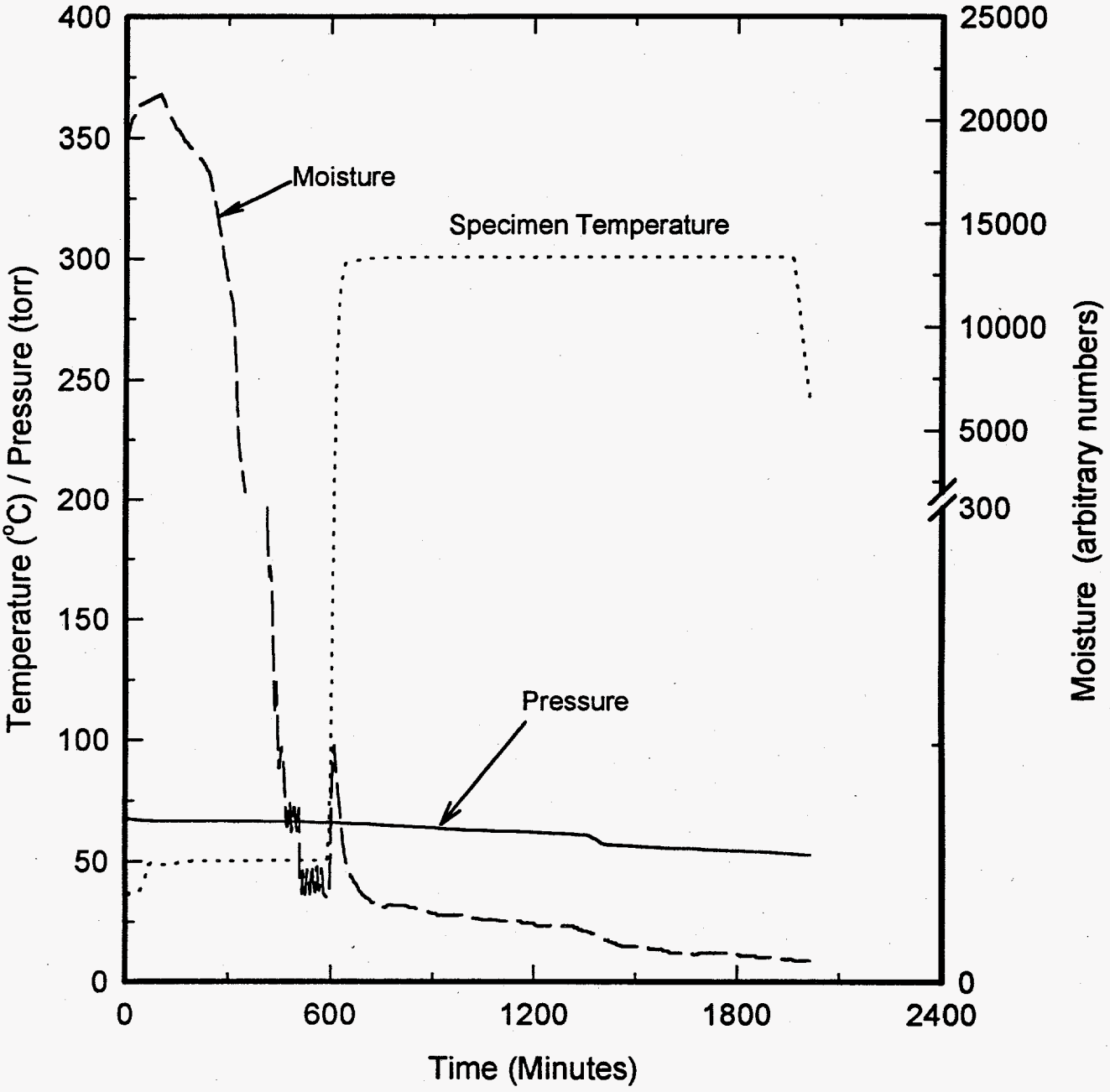


Figure 3.1a: Plots Of moisture Content of the Off Gas Stream, Furnace Pressure and Specimen Temperature during the CVD and HVD Steps of Furnace Run #6.

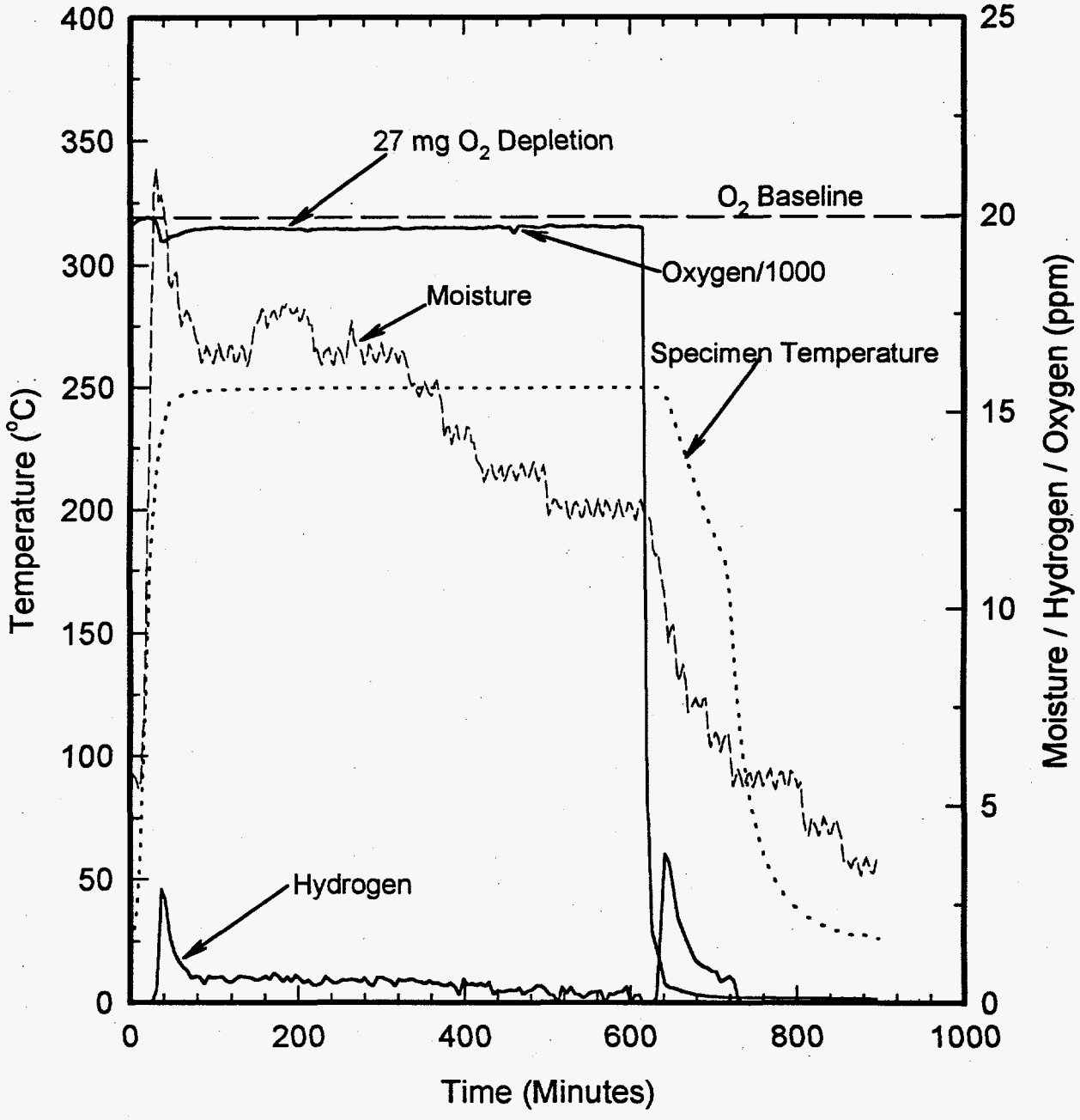


Figure 3.1b: Plots Of Moisture and Oxygen Contents in the Off Gas Stream, and Specimen Temperature during the Conditioning Step of Furnace Run #6. The moisture axis numbers are arbitrary numbers.

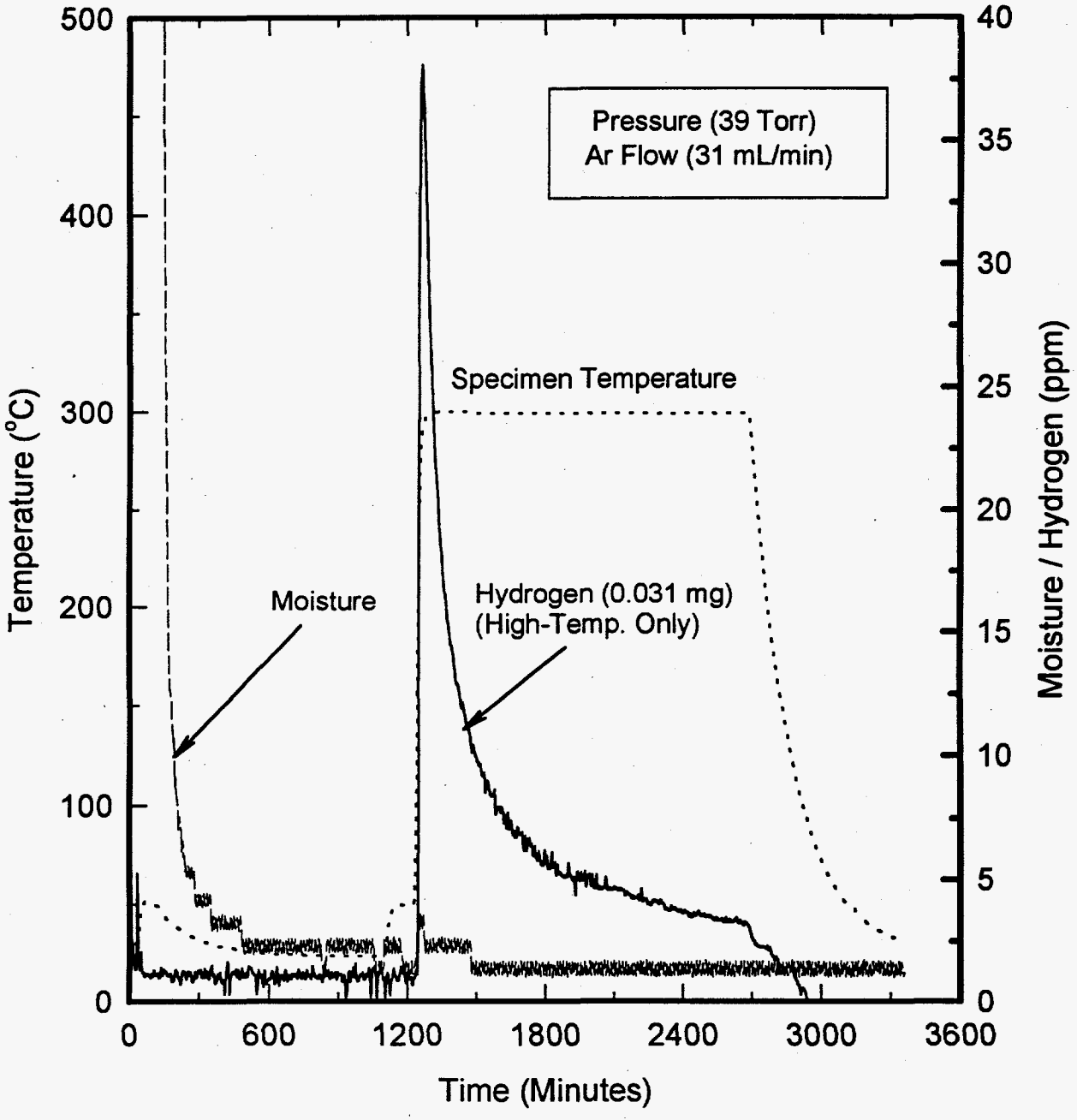


Figure 3.2a: Plots Of Moisture Content of the Off Gas Stream, Furnace Pressure and Specimen Temperature during the CVD and HVD Steps of Furnace Run #16. The moisture axis numbers are arbitrary numbers

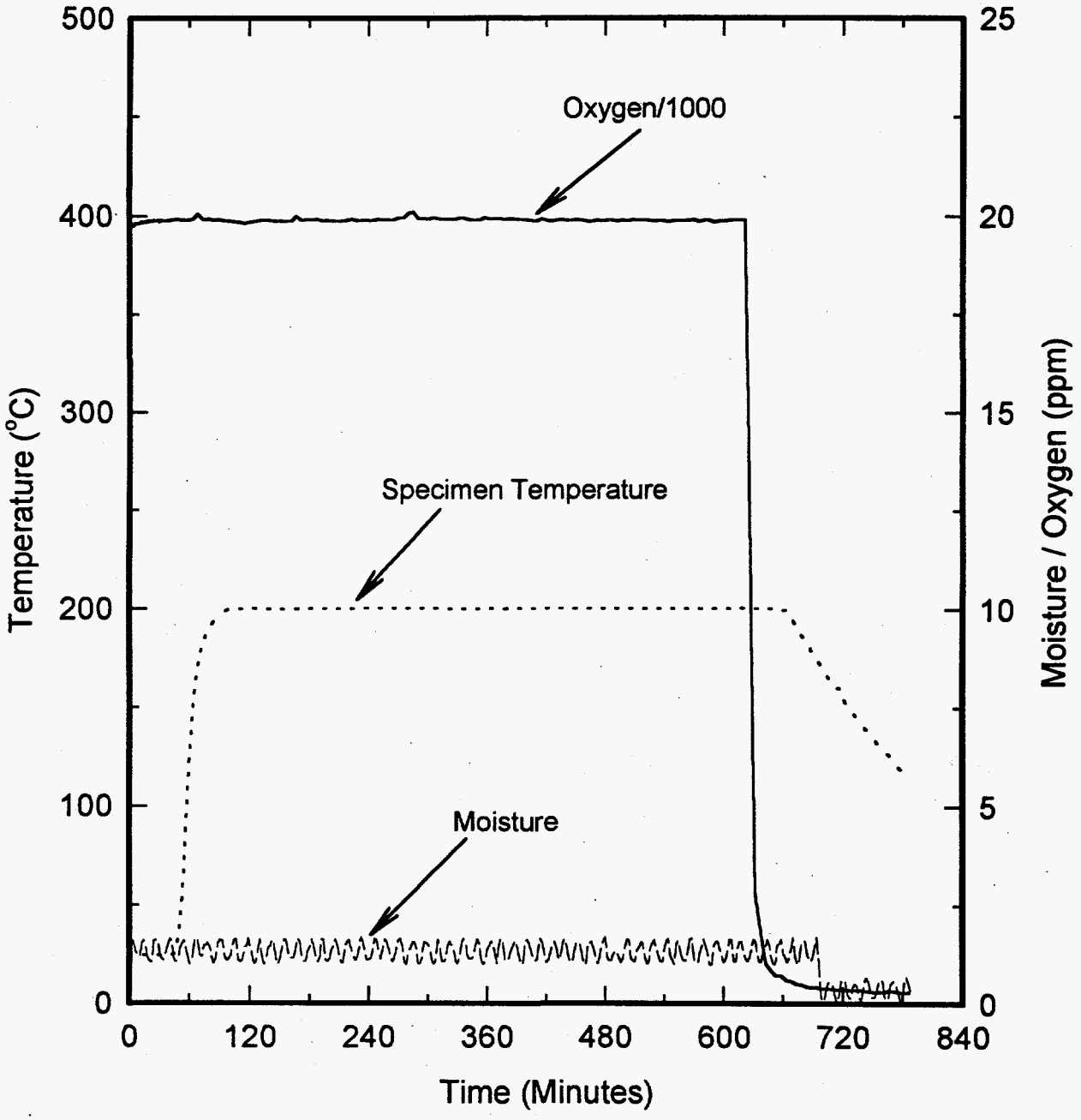


Figure 3.2b: Plots Of Moisture and Oxygen Contents in the Off Gas Stream, and Speciemen Temperature during the Conditioning Step of Furnace Run # 16.

3.2 Hydrogen Release

3.2.1 Test Data

This report contains the hydrogen release data for decladded fuel specimens SFEC5,4378-S2-E1 (Furnace Run #7) and SFEC5,4378-S2-E2 (Furnace Run #9), cladding specimen SFEC5,4378-S1-CLAD1 from the damaged end of the element (Furnace Run #8), and a defueled cladding specimen SFEC5,4378-S2-CD1 taken from specimen SFEC5,4378-S2-E1 (Furnace Run #10).

The results of hydrogen release from the decladded fuel specimen SFEC5,4378-S2-E2 are shown in Figure 3.3. The test was performed at 6 isothermal segments: 1). 523K 2). 648K, 3). ambient temperature, 4). 648K, 5). 698K, and 6). 748K. The furnace temperature ramp rate to each set specimen temperature was 10°C/min, and cooling to ambient temperature was by natural convection. Integration of the hydrogen curve (Figure 3.3) yielded 1.7 mg of hydrogen released from the specimen, which weighed 20.95 g before the test. At the end of the test, about 37 ppm of hydrogen was detected in the off-gas stream. After segment 3, the furnace was turned off and the specimen temperature fell to ambient, where GC detected no hydrogen in the off-gas. The hydrogen released from the specimen peaked during the temperature ramp except during segment 5 (the ramp to 698K) when the hydrogen level in the off gas had increased only slightly at the set temperature.

Figure 3.4 shows the results of a blank test (no specimen in the furnace) which established the baseline hydrogen and moisture content in the off-gas when the system was at high temperature. The negligible amount of detectable hydrogen is ascribed to residual hydrogen from run 9; the moisture level was only 1 ppm.

The release of hydrogen from a defueled cladding specimen SFEC5,4378-S2-CD1 is plotted in Figure 3.5. The initial release of hydrogen from the specimen during the temperature ramp peaked at about 13 ppm (volume fraction), and a total of about 10 micrograms was detected by GC during the first 4 hours of the test. Hydrogen release for furnace runs 7 and 8 is shown in Figures B.30 and B.31, respectively.

3.2.2 Discussion

The hydrogen released from the fuel specimen came from two main sources; (a) decomposition of uranium hydride inclusions in the fuel matrix, and (b) hydrogen in solid solution. The contribution from the latter should be small, given the low (about 1.8 ppm at 473K) solubility limit of hydrogen in uranium metal (Wilkinson 1962). Hydrogen generation by reaction of moisture from the gas stream with the specimen is also insignificant. The initial hydrogen release from the cladding is likely from three sources: reaction of the absorbed water with a) the Zircaloy cladding ($Zr + 2H_2O \rightarrow ZrO_2 + 2H_2$), b) the surface adsorbed/absorbed hydrogen, and c) hydrogen contained in solid solution. After that initial transient, no significant hydrogen release can be

attributed to the specimen. The total amount of hydrogen released from the cladding (0.0099 mg) is small relative to that measured during the conditioning tests. Thus, the contribution of hydrogen from the cladding was insignificant.

3.3 Ignition Runs

3.3.1 Test Data

Results of seven ignition tests are presented in this report. Five tests were performed on samples from the undamaged middle of the element: as-cut (unconditioned) samples SFEC5,4378-S2-E3A and SFEC5,4378-S2-E4A, and conditioned fuel specimens SFEC5,4378-S2-H, SFEC5,4378-S2-D and SFEC5,4378-S2-I. Two were performed on as-cut samples from the damaged end: SFEC5,4378-S1A-D and SFEC5,4378-S1A-H.

Data on the mid-length specimens are summarized in Table 3-1 (Furnace Runs 11 - 15) and plotted in Figures 3.6 through 3.9 and B.42 through B.47 (Appendix B). Figures 3.6 and 3.7 show the ignition test results for the unconditioned specimens, and Figures 3.8 and 3.9 show results from tests on IPS conditioned specimens. Figures 3.6 and 3.8 show the hydrogen detected by the GC, and the moisture content measured by the moisture monitor in the off-gas, and the specimen temperature history. Two significant observations are

1. Very little hydrogen was released when the sample was oxidizing, but hydrogen release became significant when the atmosphere was switched to pure argon.

2. Debonding of strongly bound water (water of hydration) occurred at about 673K. The peak moisture content in the off-gas from the conditioned specimen (Figure 3.8) is significantly lower than that from the unconditioned specimen (Figure 3.6).

Figures 3.7 and 3.9 are expanded plots of the specimen and furnace temperatures to graphically estimate the ignition point. In both, an abrupt change in the rate of specimen temperature increase as the furnace temperature reaches about 923K indicates that ignition has been initiated. Furthermore, the specimen temperatures did not continue to rise, but leveled off at a maximum. The major factors influencing that maximum temperature are the constraints of the tests, for example the maximum furnace temperature of 973K, specimen size, changes in the gas atmosphere (switching from dry air to pure argon), and intentionally turning off the furnace to quench the ignition. Phase transition in metallic uranium may also be a factor because the maximum temperature reached by the specimens is the beta-to-gamma phase transition temperature. The post-test examination of the specimen (Figure 3.12) showed the specimen to be highly oxidized and a powdered residue was observed in the specimen boat.

Figures 3.10 and 3.11, and B.48 and B.49 show the ignition testing results for specimens from the damaged end of SNF element SFEC5,4378. Figures 3.11 and B.49 indicate that the rate of change of specimen temperature increased at about 573K, at about this temperature, the sample

temperature increases at a faster rate indicating the onset of rapid oxidation (ignition). The post-test examinations showed the sample surfaces to be oxidized and powdered residue (likely oxides of uranium) was observed in the sample boat.

The intersection point of straight lines drawn through the lower-slope and higher-slope portions of the of the curve represents the ignition temperature. The ignition temperature for these specimens was about 553K. After ignition, the sample temperature increases much more rapidly than the furnace temperature until the uranium alpha-to-beta phase transformation begins at about 923K. Because the samples are small the phase transformation consumes enough energy to slow the temperature increase. At about 973K, onset of the uranium beta-to-gamma phase transformation produces another step in the sample temperature. The furnace temperature was not allowed to rise above 973K, and the temperature of these small samples does not increase much above 923K due to insufficient mass.

Figures 3.10 and B.48 show the hydrogen and moisture content of the gas stream flowing through the furnace apparatus during the ignition tests. In both cases, the hydrogen and moisture content are much higher than observed in the tests conducted on undamaged samples of fuel taken from the mid-section of the same element (Figures 3.6 through 3.9). This indicates that the corroded fuel contains, singly or in combination, 1) a higher concentration of uranium hydrides, 2) a higher concentration of free hydrogen, 3) hydrated species that decompose or oxidize during the tests, releasing free hydrogen or water.

3.3.2 Discussion

These results suggest that the ignition temperature of these damaged, corroded fuel samples is significantly lower than that of samples from the undamaged midsection of the same element. The lower ignition temperature is more likely due to reactions associated with relatively higher concentrations of uranium hydride inclusions in these specimens; that is, hydrogen release in these tests was greater than in the tests on undamaged mid-section samples. This effect of uranium hydride on the ignition temperature is due to following: a) direct oxidation of exposed hydrides, which have larger effective surface area (smaller particle size), b) decomposition of the uranium hydride inclusions to small metallic uranium particles, which will oxidize at a higher rate due to increased surface area, and c) decomposition of uranium hydride inclusions in the matrix and consequent cracking in the surrounding uranium matrix, due to the large volume changes associated with the transformation of hydride particles to the metallic particles. The increased reaction rate caused by these factors will release sufficient heat to initiate ignition at a lower furnace temperature.

Cracks on the exposed uranium surfaces (Figures B.28 through B.37) of the corroded samples used in the tests also increase their effective surface area, increasing the reaction rate and further lowering the ignition temperature.

The ignition temperature of specimens from the undamaged portion of the outer N-Reactor fuel

element is comparable to the value for bulk metallic uranium reported by Schnizlein and colleagues (Schnizlein et al. 1959). Therefore, the typical specimens used in these tests and the use of the burning-curve technique (Musgrave 1972) to determine ignition characteristics of the SNF material are adequate.

Table 3-1. Summary of the Furnace Testing

Run #	Test	Specimen(s)	Status		Observations
			Start Date/Time	End Date/Time	
5	CVD at 60 Torr and 323 K followed by HVD at 60 Torr and 573 K. The specimen was then conditioned at 473 K in 2% oxygen/98% argon mixture.	SFEC5,4378-S2-G plus added water	07-24-95 / 2130	07-27-95 / 1300	Added water came off during the first 3 hours of the CVD cycle. Relatively small amount of moisture evolved during the HVD cycle. Small amount of oxygen pickup during passivation step.
6	CVD at 60 Torr and 323 K followed by HVD at 60 Torr and 573 K. The specimen was then conditioned at 523 K in 2% oxygen/98% argon mixture. Pressure was one atm during conditioning.	SFEC5,4378-S2-F1 & F2 with added free water	08-07-95 / 2200	08-10-95 / 1330	Most of the added water came off during the CVD cycle. Relatively, small amount of moisture was observed during the HVD cycle. A total of 27 mg of oxygen was picked up during the passivation step. The two specimens lost a total of 505.6 mg of weight due to oxide spalling. Analysis of the spall off residue indicates U_4O_9 and U_3O_8 as the predominant chemical phases. Compared to the tests in which water was not added to the specimens, the moisture content in the off-gas stream during the passivation cycle in this run is relatively high.
7	Hydrogen release from decladded fuel specimen at 573 K for about 24 hours and at 623 K for 96 hours in an argon gas flow of 100 cc/min. Pressure of the system was about one atmosphere.	SFEC5,4378-S2-E1 (Decladded)	08-23-95 / 1300	08-28-95 / 1300	Hydrogen gas was measured in the gas stream and the hydrogen concentration peaked during the temperature ramp. The total hydrogen measured was 0.12 mg. An appreciable amount of moisture was also detected in the gas stream.
8	Hydrogen Release from Cladding Material	Peeled Off Cladding from the damaged end of SFEC5,4378	08-30-95 / 0930	09-05-95 / 1100	Significant amounts of both moisture and hydrogen were measured. These peaked sharply (about 260 ppm moisture and 75 ppm hydrogen in the gas stream) during the temperature ramp, but remained fairly high throughout the run

Run #	Test	Specimen(s)	Status		Observations
			Start Date/Time	End Date/Time	
9	Hydrogen release from decladded fuel specimen.	SFEC5,4378-S2-E2 (Decladded)	9-28-95 /0830	10-6-95 /1415	Compared to previous runs, a significant amount of hydrogen was released from this specimen.
Blank	Furnace was heated at 748 K.	N/A	10-6-95 /1450	10-7-95 /2350	No hydrogen was observed and the moisture leveled off at about 1ppm in the gas stream.
10	Hydrogen Release from Cladding Material at 573 K.	Cladding specimen SFEC5,4378-S2-E1-CD1	10-10-95 /1400	10-11-95 /1330	The results show initial release of hydrogen during the temperature ramp. The release peaked at about 13 ppm (volume fraction) and decreased quickly to below detection after the first 4 hours. The total amount of hydrogen released is about 10 micrograms
11	Ignition of unconditioned fuel specimen in dry air. The Air flow rate was 500 cc/min and the temperature ramp rate was about 15 K/min	SFEC5,4378-S2-E3A	10-25-95 /1030	10-25-95 / 2400	The specimen ignited at about 913 K. Hydrogen released during the oxidation reaction was small but when the oxidation reaction was terminated by switching the gas supply from dry air to pure argon there was an increase in the hydrogen content in the off gas stream during the specimen cooling down.
12	Ignition of unconditioned fuel specimen in dry air. The Air flow rate was 500 cc/min and the temperature ramp rate was about 15 K/min	SFEC5,4378-S2-E4A	11-01-95 /0850	11-01-95 /1506	The results of this run was very similar to what was observed in Run # 11. The ignition temperature was about 913 K.
13	Ignition of conditioned fuel specimen in dry air. The Air flow rate was 500 cc/min and the temperature ramp rate was about 15 K/min	SFEC5,4378-S2-H	11-03-95	11-03-95	The ignition of this specimen was not very discernible from the specimen temperature plot. Hydrogen released during the oxidation reaction was again small but when the oxidation reaction was terminated by switching the gas supply from dry air to pure argon there was an increase in the hydrogen content in the off gas stream during the specimen cooling down.

Run #	Test	Specimen(s)	Status		Observations
			Start Date/Time	End Date/Time	
14	Ignition of conditioned fuel specimen in dry air. The Air flow rate was 500 cc/min and the temperature ramp rate was about 15 K/min	SFEC5,4378-S2-D	11-10-95	11-10-95	The results of this run show ignition at about 903 K. The hydrogen release mechanism was similar to that of Run # 13.
15	Ignition of conditioned fuel specimen in dry air. The Air flow rate was 500 cc/min and the temperature ramp rate was about 15 K/min	SFEC5,4378-S2-I	11-17-95	11-17-95	The ignition of this specimen was not very discernible from the specimen temperature plot even though the maximum set temperature for the furnace was increased from 973 K (for the previous tests) to 1073 K. Hydrogen released during the oxidation reaction was again small but when the oxidation reaction was terminated by switching the gas supply from dry air to pure argon there was an increase in the hydrogen content in the off gas stream during the specimen cooling down.
16	CVD at 39 Torr and 323 K followed by HVD at 39 Torr and 573 K. The atmosphere during drying was maintained inert by flowing high purity argon at 31 cc/min. Hydrogen in the off gas stream was monitored by a GC. The specimen was then conditioned at 473 K in 2% oxygen/98% argon mixture. Pressure was one atm during conditioning.	SFEC5,4378-S2-F4 with added free water	12-20-95 / 1545	12-29-95 / 2100	Most of the added water came off during the CVD cycle. Relatively, small amount of moisture was observed during the HVD cycle. Hydrogen evolution during the CVD cycle was below the detection limit of the GC but at HVD the content of hydrogen in the off gas stream was comparable to that measured in the gas flow drying test at the same temperature (Runs 1 & 2). However, hydrogen evolution during the HVD shows a decay with time. At 473 K the oxygen pickup by the specimen during the conditioning cycle was below the detection limit of the GC.
17	Ignition of as cut (i.e., unconditioned) fuel specimen from a damaged region of a SNF element in dry air. The Air flow rate was 500 cc/min and the temperature ramp rate was about 15 K/min	SFEC5,4378-S1A-D	01-09-96	01-09-96	The specimen ignited at a lower temperature compared to specimens from the undamaged portion of the SNF element. The ignition temperature is about 551 K. Higher hydrogen and moisture evolved from the specimen during the temperature ramp. The moisture curve shows double peaks indicating two distinct bound water in the specimen.

Run #	Test	Specimen(s)	Status		Observations
			Start Date/Time	End Date/Time	
18	CVD at 39 Torr and 323 K followed by HVD at 39 Torr and 573 K. The atmosphere during drying cycles was air. Hydrogen in the off gas stream was monitored by a GC. Specimen was not conditioned	SFEC5,4378-S1A-C with added free water	01-15-96 / 2200	01-17-96 / 1330	Most of the added water came off during the CVD cycle. But the amount of moisture observed in the off gas stream during the HVD cycle was still very high. Hydrogen evolution during the CVD cycle was below the detection limit of the GC but at HVD hydrogen in the off gas stream was measured.
19	Ignition of as cut (i.e., unconditioned) fuel specimen from a damaged region of a SNF element in dry air. The Air flow rate was 500 cc/min and the temperature ramp rate was about 15 K/min	SFEC5,4378-S1A-H	01-22-96	01-22-96	The specimen ignited at a lower temperature compared to specimens from the undamaged portion of the SNF element. The ignition temperature is about 550 K. Higher hydrogen and moisture evolved from the specimen during the temperature ramp. The moisture curve shows double peaks indicating two distinct bound water in the specimen. The hydrogen curve also shows a structure (i.e., double peak) that indicates at least two forms of hydrogen with different bond strength evolved from the specimen.

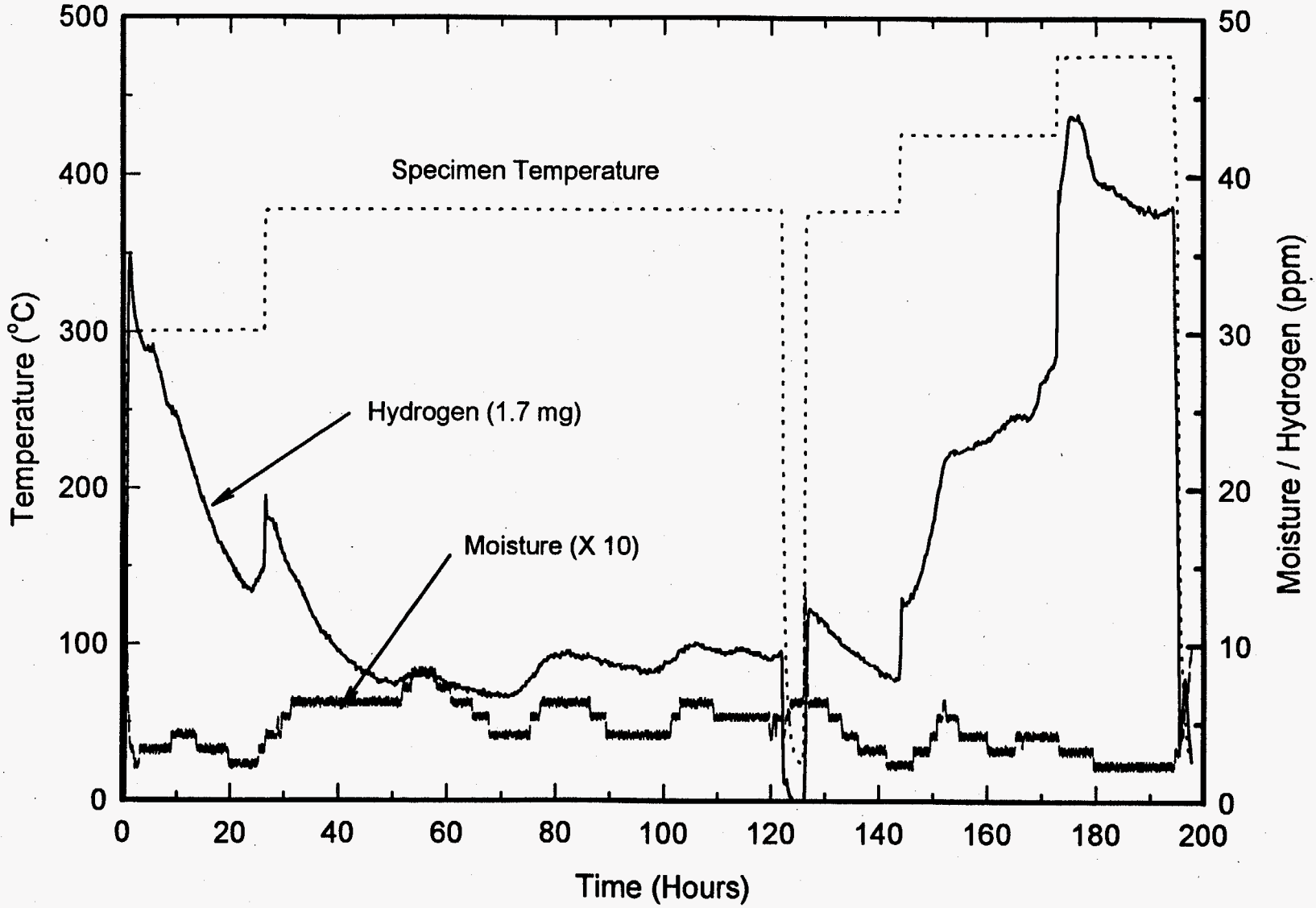


Figure 3.3: Results of Hydrogen Release from a Decladded Fuel Specimen SFEC5,4378-S2-E2 Showing the History of Moisture and Hydrogen In the Off Gas Stream at Different Specimen Temperatures. Arbitrary numbers for the Moisture axis

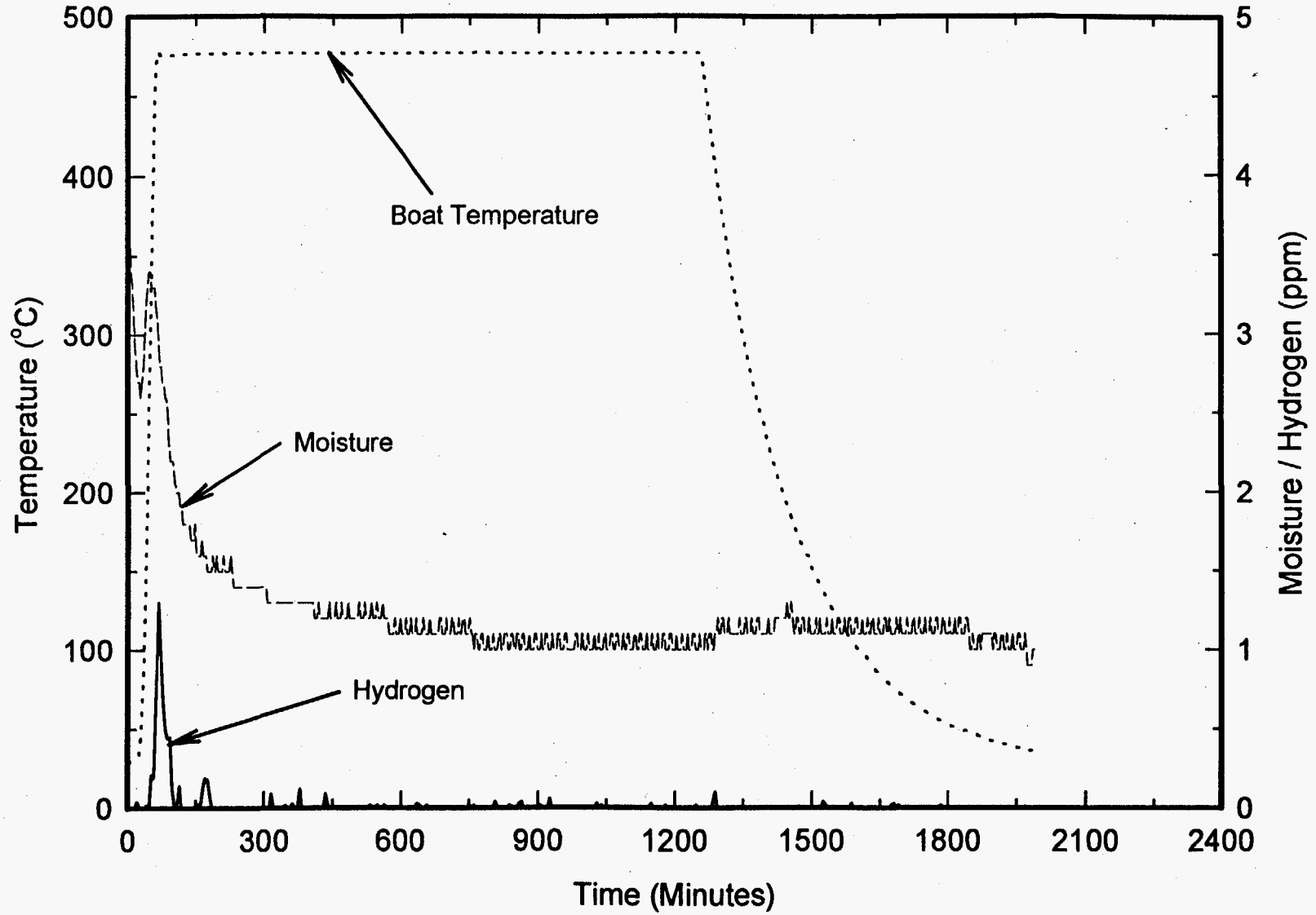


Figure 3.4: Results of Hydrogen Release Blank Test Showing the History of Moisture and Hydrogen In the Off Gas Stream From the Furnace. The numbers of the moisture axis are arbitrary numbers.

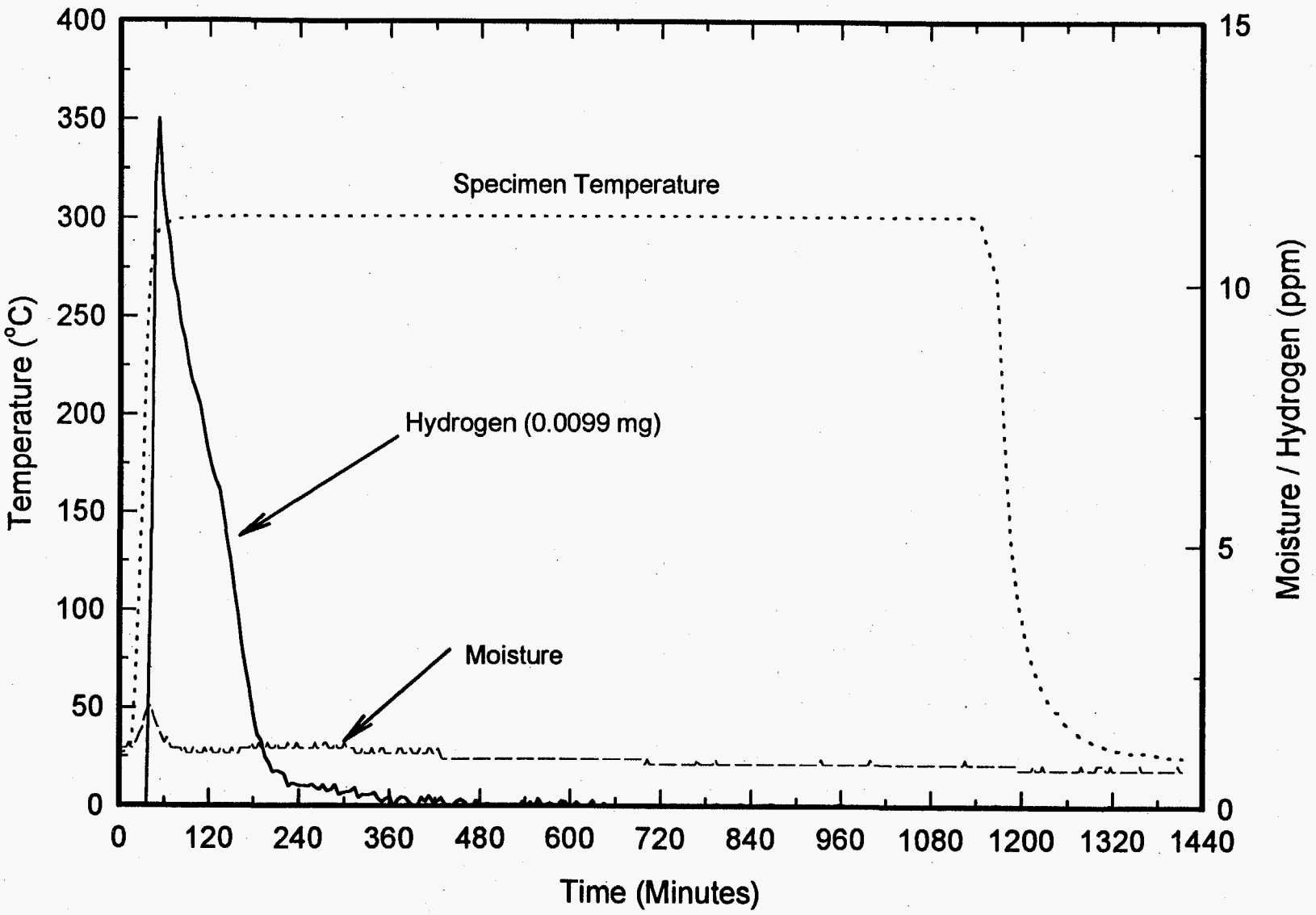


Figure 3.5: Results of Hydrogen Release from a Defueled Cladding Specimen SFEC5,4378-S2-E1-CD1 Showing the History of Moisture and Hydrogen In the Off Gas Stream. The moisture axis numbers are arbitrary numbers.

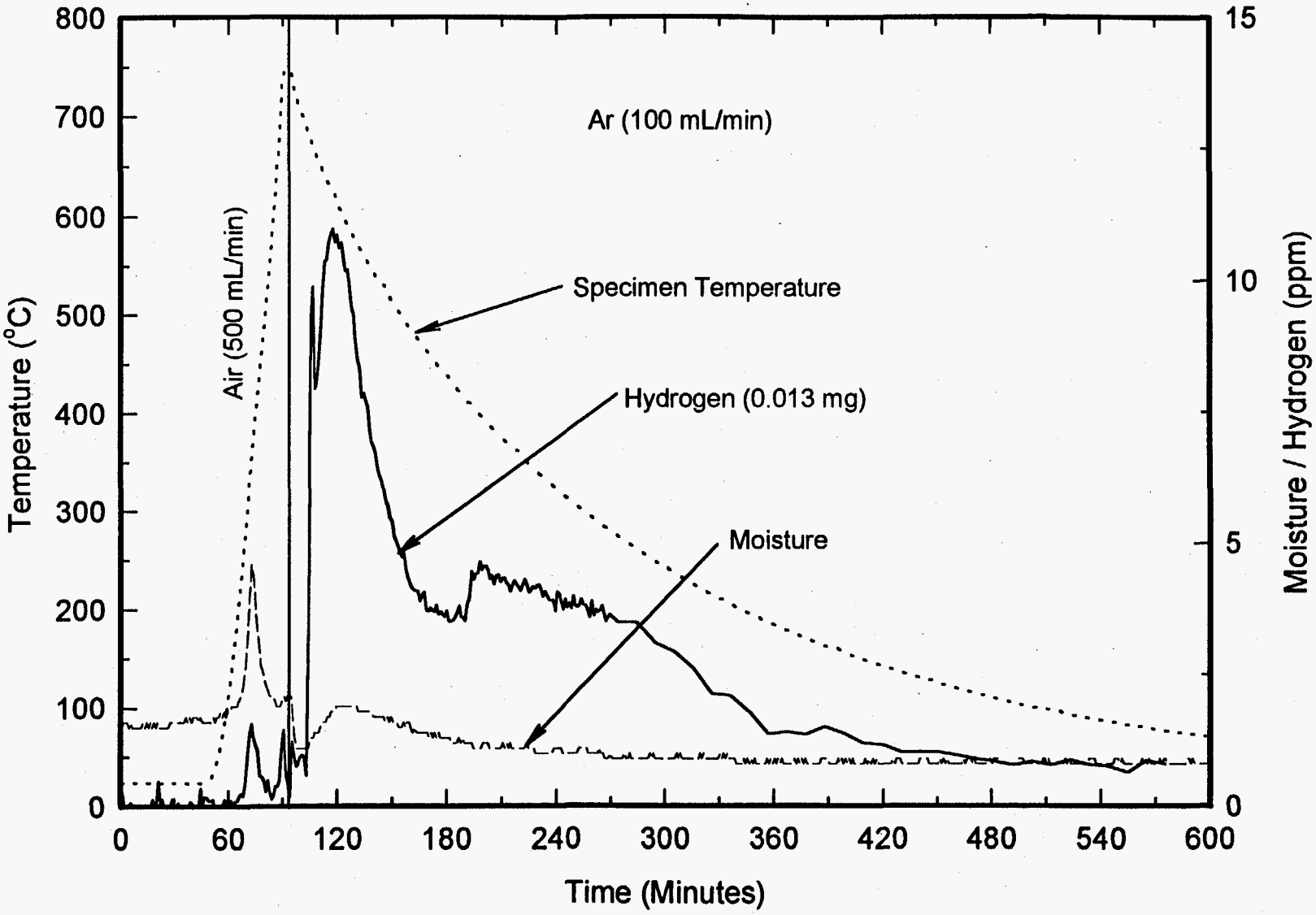


Figure 3.6: Ignition Test Results of An As-Cut Specimen SFEC5,4378-S2-E3A. The Vertical Line Indicates the Time When the Furnace Power Was Turned Off. The moisture axis numbers are arbitrary numbers.

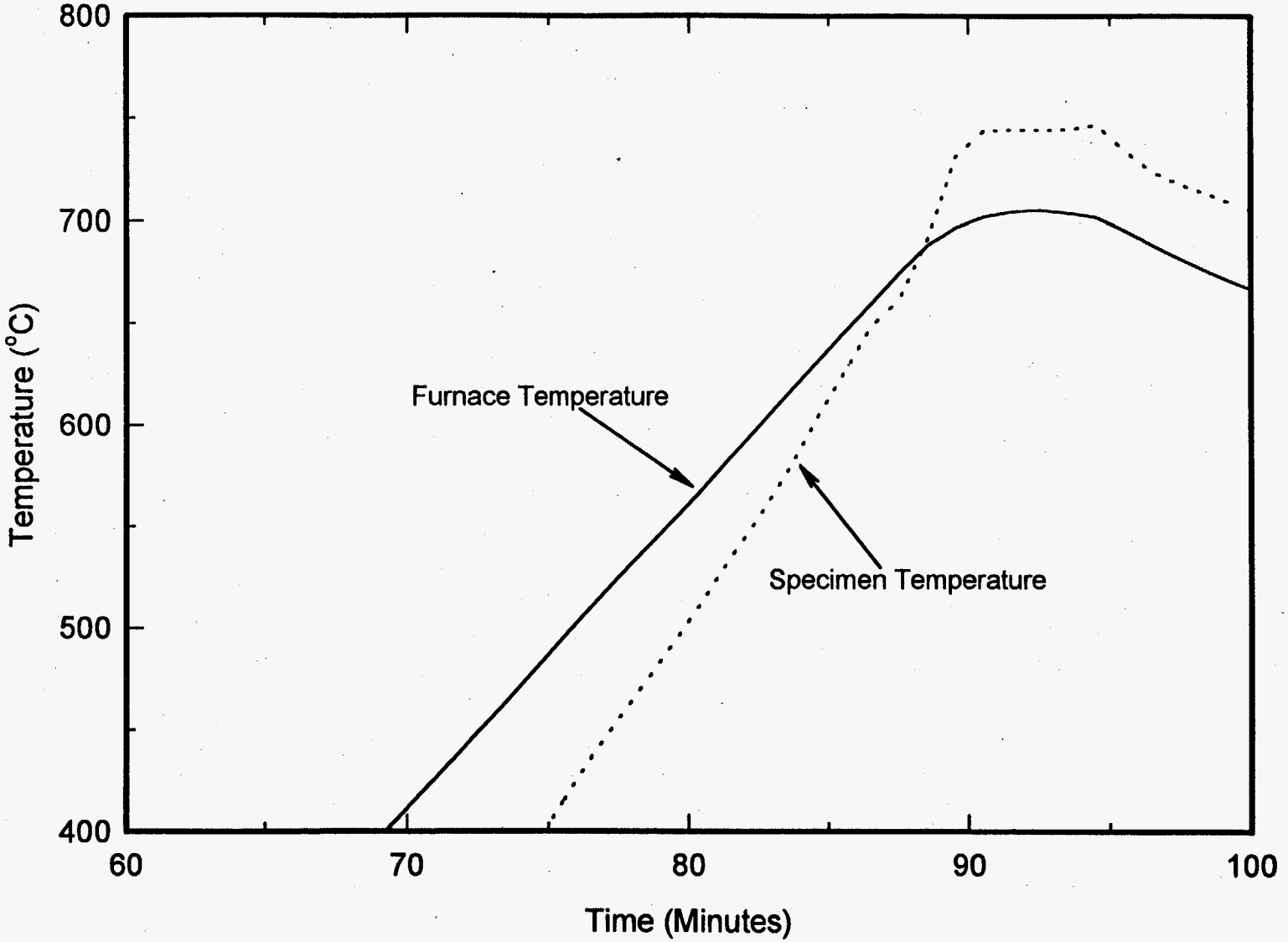


Figure 3.7: An Expanded Plots of Specimen and the Furnace Temperatures In the Region Where Ignition Of Figure 3.6 is Expected to Occur.

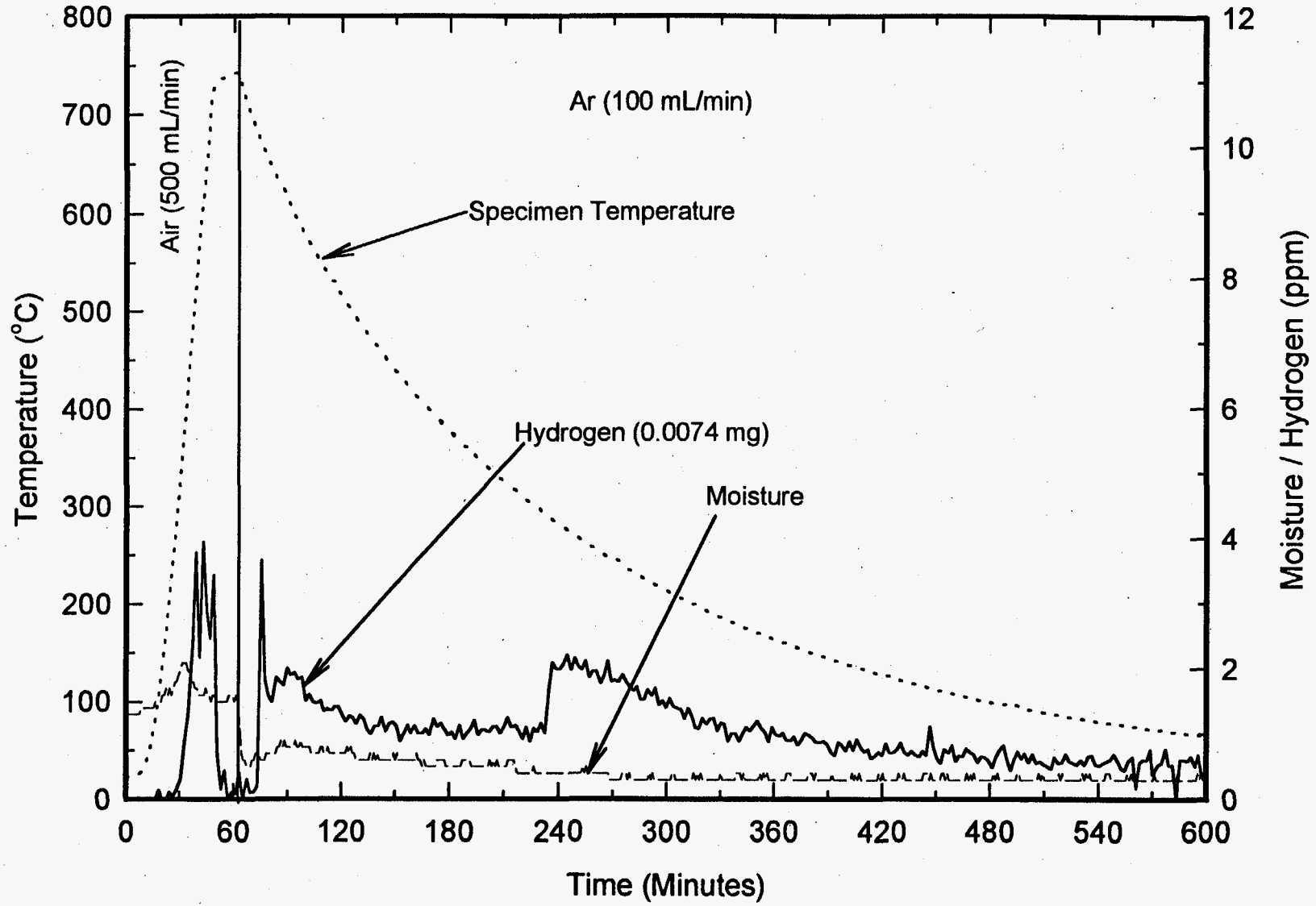


Figure 3.8: Ignition Test Results of An IPS Conditioned Specimen SFEC5,4378-S2-D. The Vertical Line Indicates the Time When the Furnace Power Was Turned Off. The moisture axis numbers are arbitrary numbers.

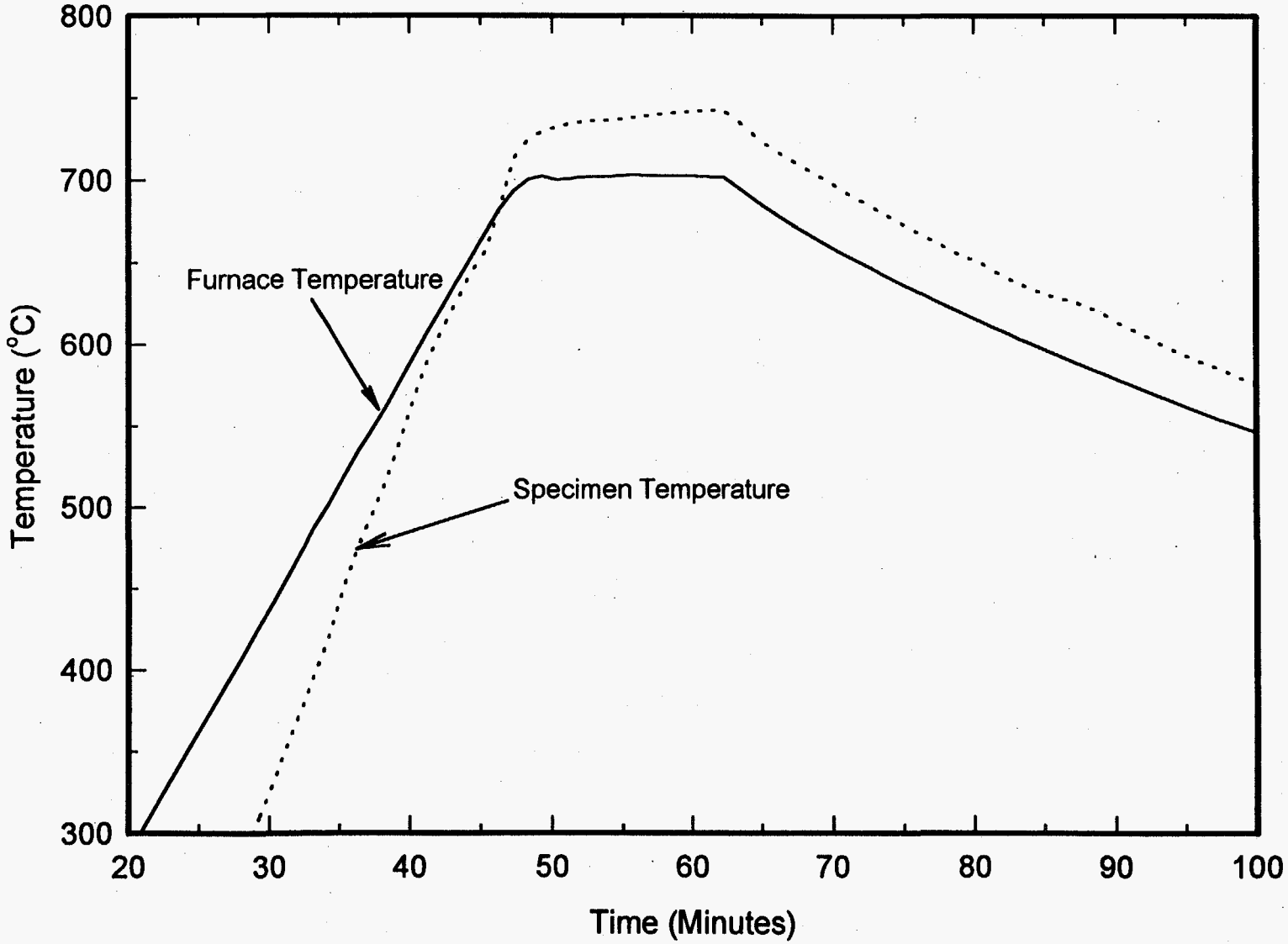


Figure 3.9: An Expanded Plots of the Specimen and the Furnace Temperatures In the Region Where Ignition Of Figure 3.8 is Expected to Occur.

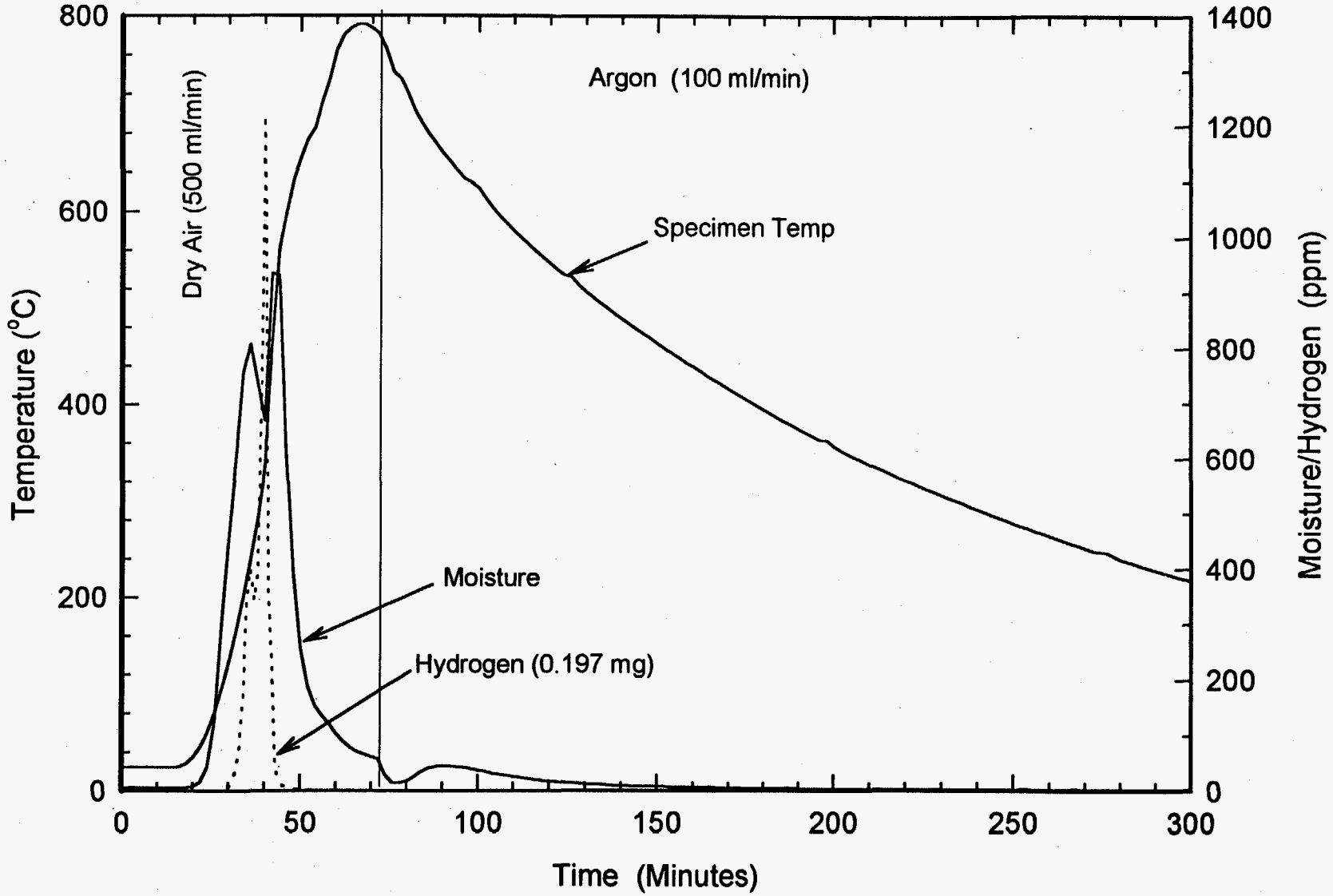


Figure 3.10: Ignition Test Results of As-Cut SNF Specimen SFEC5,4378-S1A-H. The Vertical Line Indicates the Time When the Furnace Power Was Turned Off. The moisture axis numbers are arbitrary numbers.

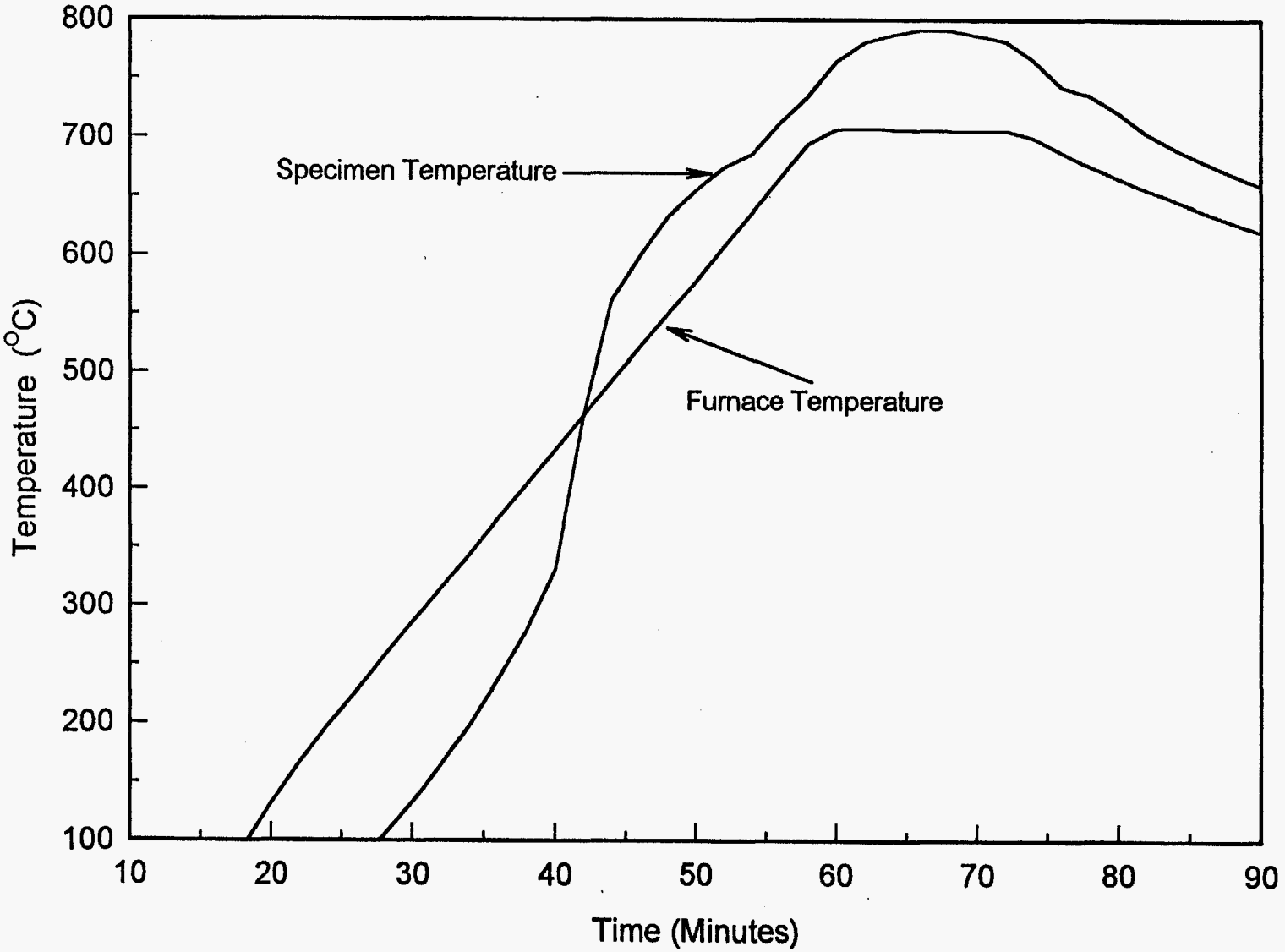


Figure 3.11: An Expanded Plots of Specimen and the Furnace Temperatures In the Region Where Ignition Of Figure 3.10 is Expected to Occur.

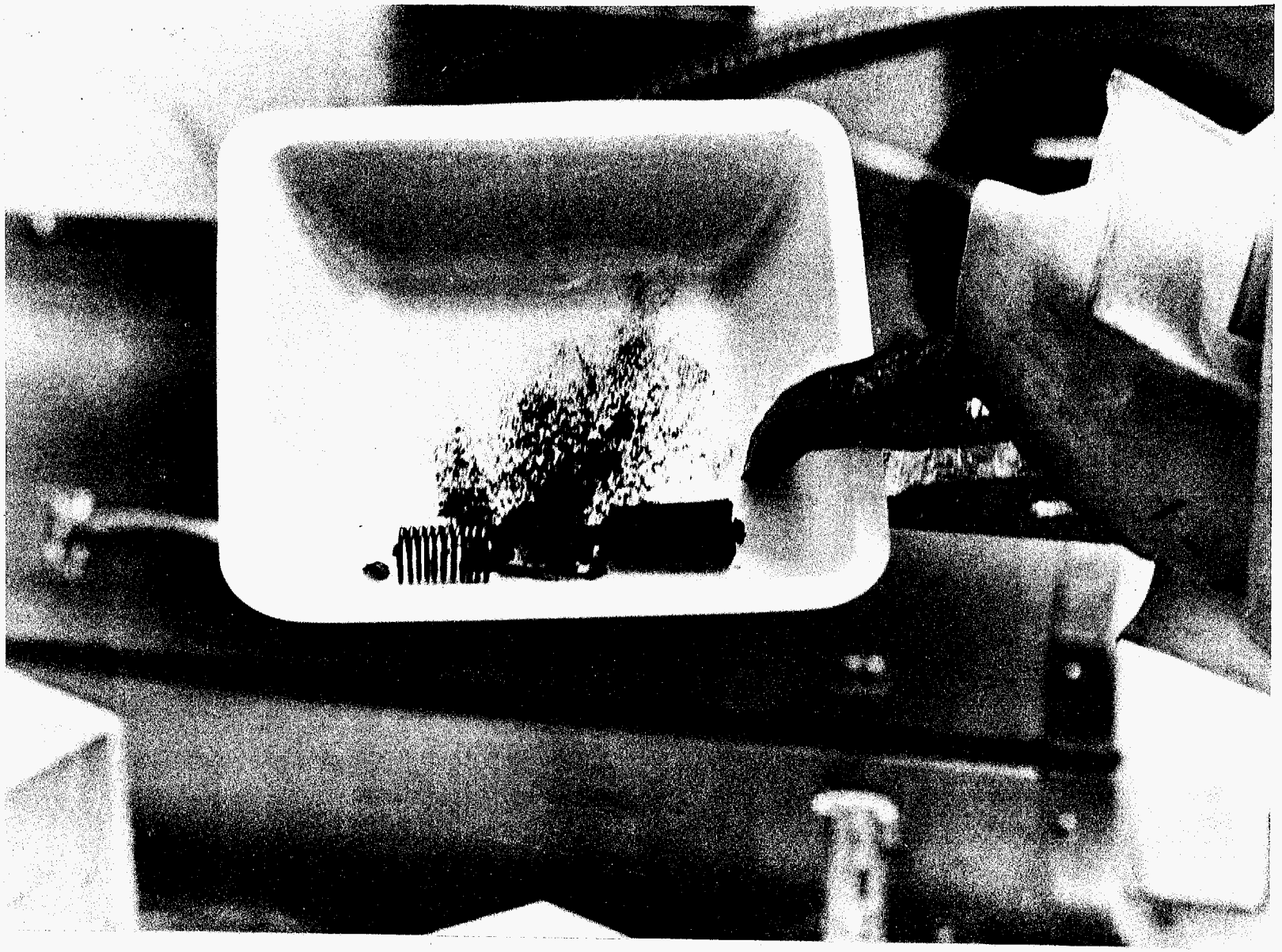


Figure 3.12: Photograph of Specimen SFEC5,4378-S2-D Showing Highly Oxidized Specimen, the Residue from the Spalled Uranium Oxide and Spring Used to Keep Specimen In Contact With the K-Type Thermocouple Throughout the Test.

3.4 Optical Microscopy Results

Metallography was performed on specimens to obtain information concerning microstructure and hydride inclusions in the fuel matrix, morphology of the oxide film formed during the conditioning test, and thickness and adherence of the oxide layer. Optical micrographs shown in this report are for specimens SFEC5,4738-S2-B and SFEC5,4378-S2-J, which were dried and conditioned by the proposed ITA process in Furnace Runs 1 and 2 (Abrefah et al. 1995).

Figures 3.13 through 3.17, and B.49 through B.64 are photomicrographs of the polished surfaces of specimens SFEC5,4378-S2-J1 & -J2 mounted in the transverse and longitudinal planes, respectively, and SFEC5,4378-S2-B in the longitudinal plane. All of the photomicrographs show inclusions in the uranium matrix, which appear as bright, regular geometric shapes or as dark spots away from and around the bright inclusions.

The boundary between the cladding and uranium fuel is shown in Figures 3.15 B.51, and B.64). These photomicrographs show no separation of the cladding from the fuel material and no visible material degradation in the region around the boundary. Thus, the drying and conditioning process did not degrade the bonding of cladding to fuel. There is no indication of porosity at the fuel/cladding boundary.

Figures 3.13, 3.14, B.54, and B.56 through B.59 show the microstructure of the oxide film formed by the oxidation in 2% oxygen/98% argon gas mixture at a temperature of 523K. Rather than a well defined oxide layer adhering to the fuel substrate, these photomicrographs show a loosely spalling oxide layer on the cut surfaces of the fuel. The thickness of the oxide layer is non-uniform (Figures B.58 and B.59), and the maximum thickness estimated for the separated oxide film is about 16 microns (Figure 3.14).

The result of the heat tinting used to identify probable hydride phase inclusions as 'halo' in the matrix is shown in Figures 3.16 and 3.17. The identified hydride inclusions are randomly distributed and could not be distinctly associated with grain boundaries or microcracks.

3.4.1 Discussion

Two distinct phase structures appear in the photomicrographs of the as-polished uranium surface: uranium-carbide precipitates and what are probably uranium-hydride inclusions. The carbides are the regularly structured bright-phase inclusions, and the uranium hydride phase are most likely around the carbide precipitates, grain boundaries, and micro-cracks. Some of the dark structures could be voids created by oxidized phases, fission gases, or both. The grain boundaries and the micro-cracks are not visible on the polished surfaces. Possible sources of hydrides in the fuel matrix include a) residual "tramp" hydrogen from the fuel fabrication process, b) hydrogen produced as the fuel corroded during storage in the K Basin, and c) hydrogen generated by corrosion of the cladding during N reactor operation. Thermodynamically, all of the hydrogen generated by corrosion of the Zircaloy should reside in the cladding. Thus, items (a) and (b) are

the most probable sources of hydride in the fuel matrix.

The spalling of the oxide film formed during oxidation in 2% oxygen/98% argon gas mixture at 250°C specimen temperature confirms the weight loss from the specimens and the residue observed in the specimen boat. Most of the oxide film formed at the test conditions are non-protective and nonadherent. The results agree with the report (Wilkinson 1962) that above 75°C oxidation of uranium metal is markedly accelerated to form nonadherent oxide film.

The presence of uranium hydride phase in the conditioned samples explains the leveling off of the hydrogen content in the off-gas stream during the conditioning testing (Furnace Runs 1 and 2, reported in Abrefah 1995) as an indicator that the drying process did not remove all the bulk hydride phase in the uranium matrix.

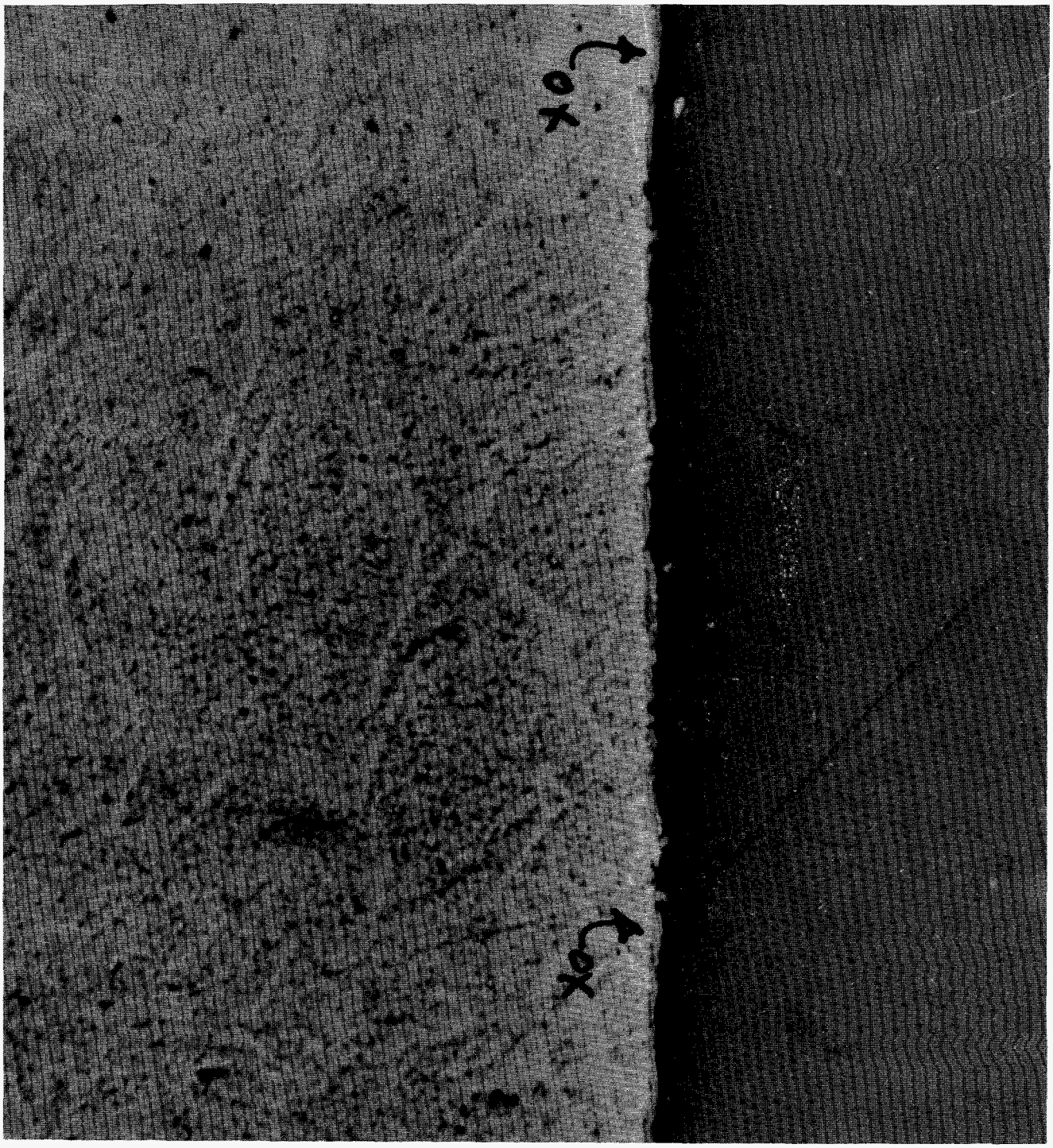


Figure 3.13: Photomicrograph, 750X, of the Mid-Region of As-Polished Conditioned Specimen SFEC5,4378-S2-J1 in the Transverse Plane. The photo shows a thin loosely adhered oxide layer (film between the black mounting material and the fuel) on the uranium cut surface. PTL Log Q-1473, Photo # 240.

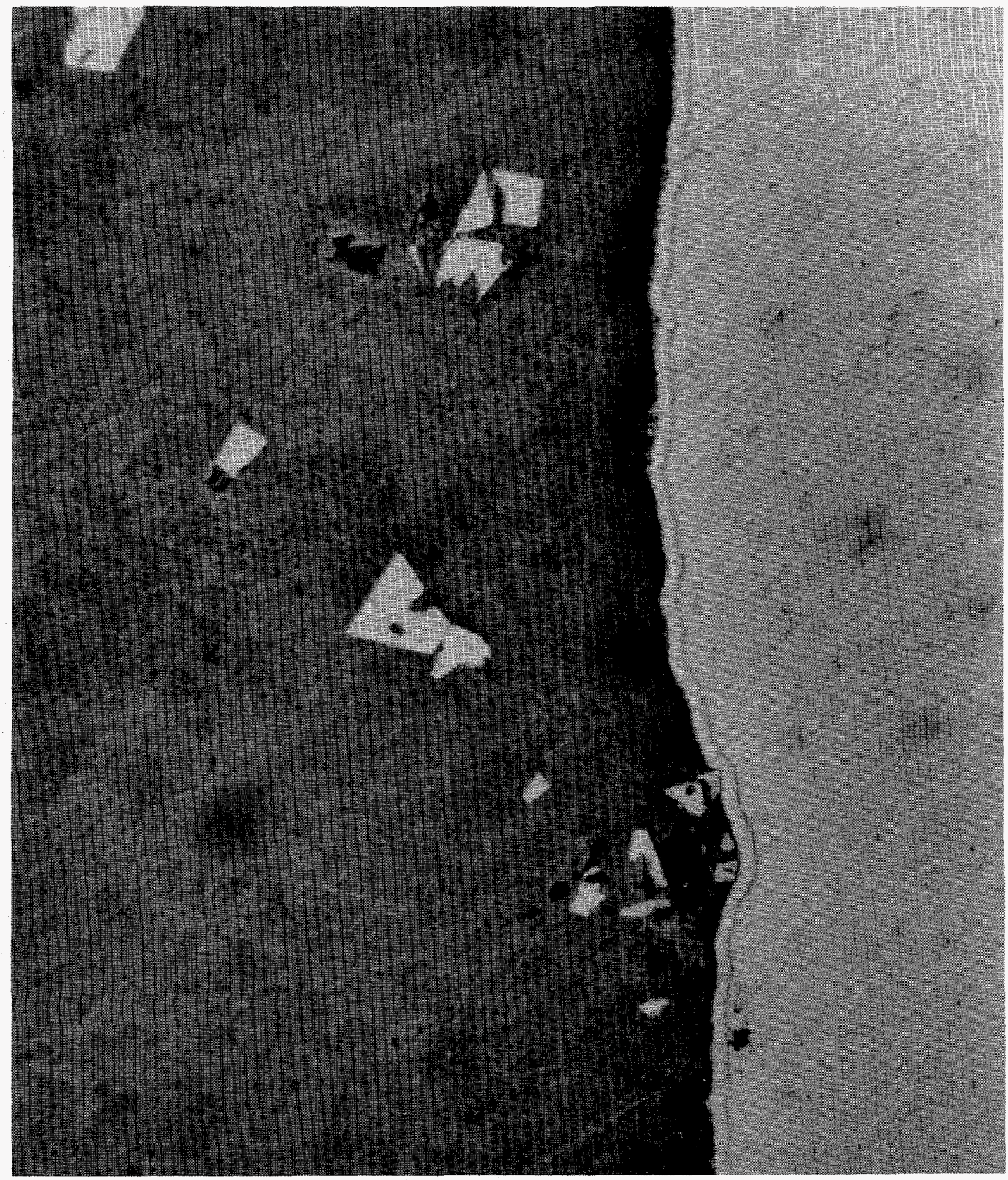


Figure 3.14: Photomicrograph, 250X, of As-Polished Conditioned Specimen SFEC5,4378-S2-J1 in the Transverse Plane. The photo shows the boundary region of the fuel (dark) and cladding (bright), and precipitates of uranium carbide. PTL Log Q-1461, Photo # 280.

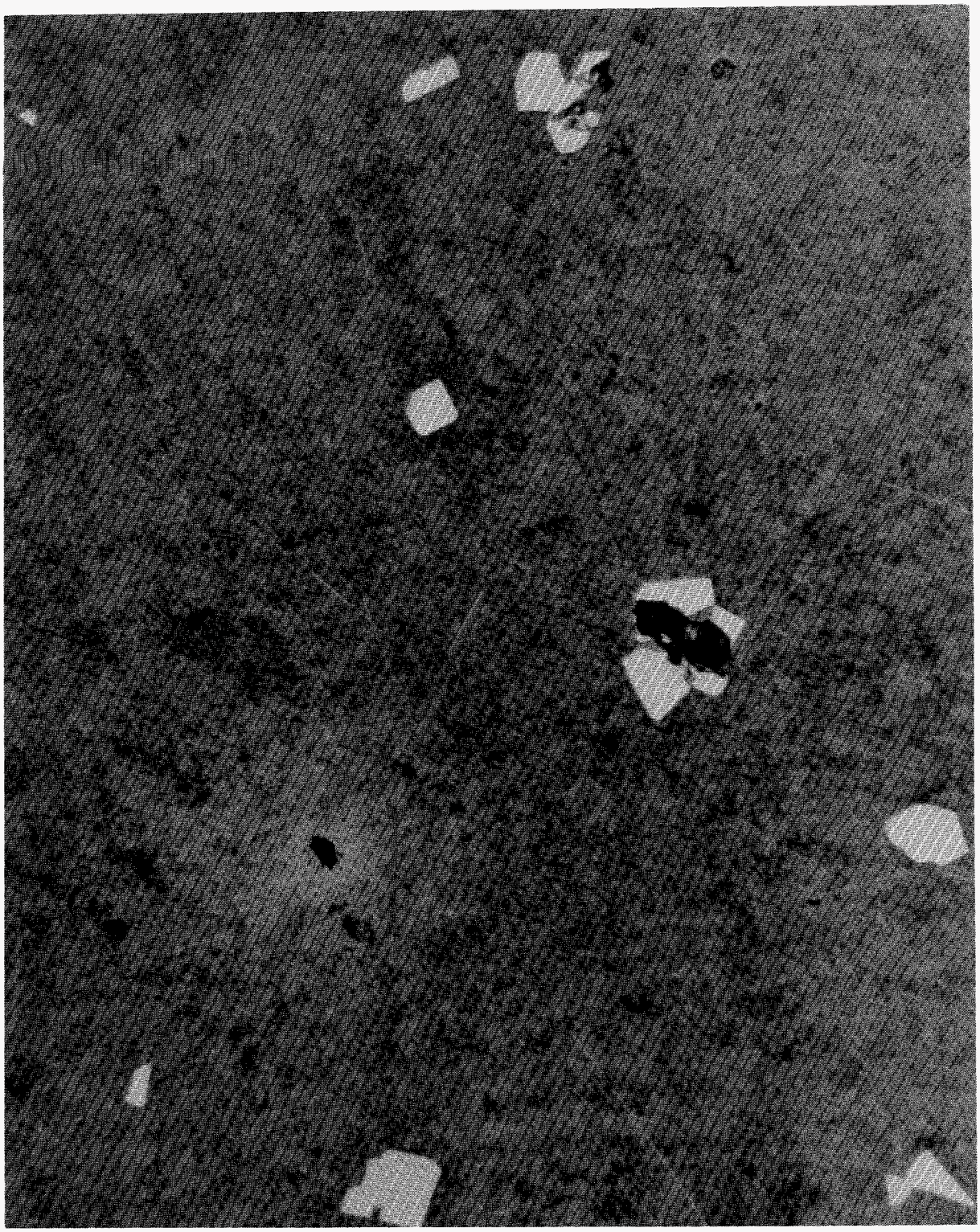


Figure 3.15: Photomicrograph, 250X, of a Location Close to the Inner Diameter Surface of Conditioned Specimen SFEC5,4378-S2-J1 in theTransverse Plane. The photo shows probable hydride phase and uranium carbide precipitates.
PTL Log Q-1455, Photo # 274.

Figure 3.16: Photomicrograph, 250X, of the As-Polished Conditioned Specimen SFEC5,4378-S2-B in the Longitudinal Plane Illustrating Spalling Off of the Oxide Layer. PTL Log Q-1508, Photo # 326.

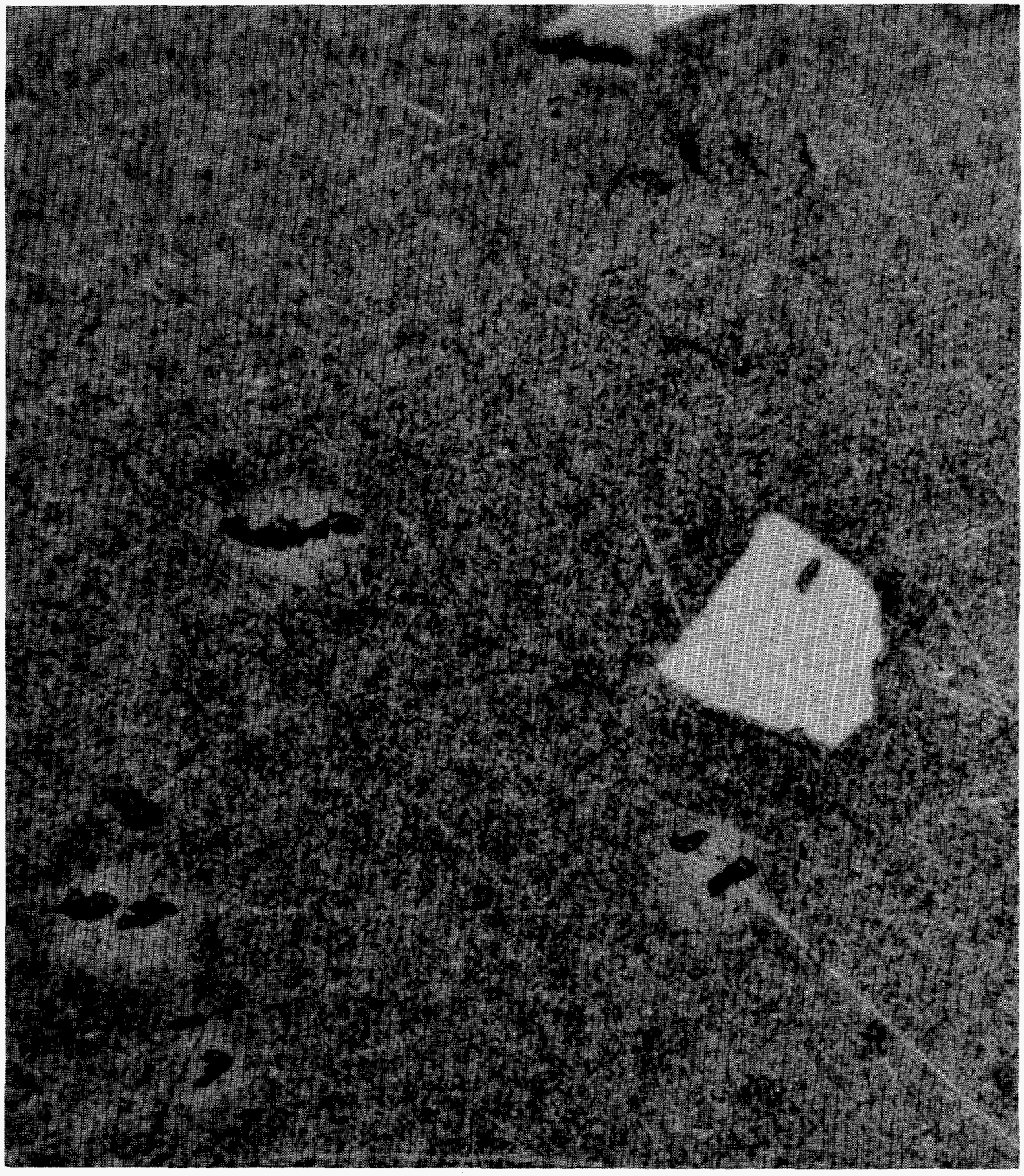


Figure 3.17: Photomicrograph, 750X, of As-Polished Conditioned Specimen SFEC5,4378-S2-B in the Longitudinal Plane. The heat tinted polished surface shows hydride inclusions as 'halo'. PTL Log Q-1500, Photo # 318.

3.5 XRD Examination

3.5.1 Test Data

The raw data diffractograms are shown in Figures 3.18 through 3.22. Background subtracted data with "stick figure" patterns of identified phases are shown in Figures 3.23 through 3.27. These samples are all characterized by a rather high background count (as high as 16,000) and a series of superimposed crystalline peaks. Some of the peaks are rather sharp, others are quite broad (> 0.5° 2-theta). The broad peaks are indicative of very small crystallites, on the order of 10 microns; the sharp peaks represent crystallites on the order of 100 microns or larger.

PM1-DUP and FR3-DUP are duplicate runs of the earlier PM1 and FR3 samples. These samples were removed from the same sample containers as the earlier runs (as delivered to the Shielded Analytical Laboratory, SAL).

3.5.2 Discussion

Broad peaks present in all samples are associated with UO_2, U_4O_9, and U_3O_7. The XRD patterns for these phases are nearly identical; however, the pattern of U_3O_7 has some double peaks and can sometimes be inferred from the presence of split peaks or shoulders. These broad peaks are herein referred to as UO_{2+x}, recognizing that x may have intermediate values from the presence of additional oxygen in the lattice. The combination of similar spectra and broad peaks makes identification based on XRD patterns only very difficult.

A similar situation exists with U_3O_8 and UO_3. Diffraction patterns are nearly identical except for small differences in intensities. With thin samples, intensity differences may be brought on by preferred orientation, and identification based only on the x-ray pattern is difficult. These patterns are referred to as U_3O_{8+x}, and again the oxygen content may be greater than that indicated by U_3O_8. The similarities in the UO_{2+x} and U_3O_{8+x} systems are apparent in the "stick figures" of Figure 3.26.

The broad peaks in sample FR2 can be ascribed to the UO_{2+x} system; the U_4O_9 pattern has a slightly better fit to the data (0.15 degree difference at 28.4 degrees). These peaks are quite broad, and the data fit profiles from both phases about equally well, hence the uncertainty as to which is present. The peaks at 33 and 56 degrees have pronounced shoulders on the low 2-theta sides, which suggest the presence of U_3O_7, but certainly not as a single phase. All three phases could be present. In addition, U_3O_{8+x} is definitely present, although in small quantity. Figure 3.23 shows the background subtracted plot and the "stick figure" displays of each phase.

Samples PM1 and PM1-DUP show the broad peaks associated with UO_{2+x}. Shoulders are not distinguishable in the 33 and 56 degree peaks, so that U_3O_7 is likely not present. U_4O_9 is a slightly better fit to the data than UO_2 in both samples. Schoepite, $UO_3 \cdot 2H_2O$, is present in both samples in moderate amounts (high peak intensity but low area, so probably less than UO_{2+x}).

Metaschoepite is present at low levels in PM1-DUP, but was not detectable in sample PM1. Figures 3.24 and 3.25 shows the background subtracted data and the "stick figures" for the identified phases of these samples.

Samples FR3 and FR3-DUP also show the broad peaks for the UO_{2+x} systems. Some shoulders are apparent in the 33 and 56 degree peaks; therefore some U_3O_7 may be present. U_3O_{8+x} is present in a moderate amount. U_4O_9 is a slightly better fit to the data than UO_2 in both samples. Schoepite, (and /or metaschoepite), $UO_3 \cdot 2H_2O$, is a minor presence. These samples are essentially the same within experimental error.

The request was made to look specifically for the phase $UO_2 \cdot 2H_2O$. This phase is not in the International Centre for Diffraction Data (ICDD) set 45 published in September 1995.

Table 3.2 identifies the peak area for each phase in each of the above samples (units are total counts in major peak/ratio of counts for that phase to total of all three phases):

Table 3.2: Peak Area for Major Peak, and Ratio of Area to Phases Found

Sample ID	Phase					
	UO_{2+x}		U_3O_{8+x}		$UO_3.2H_2O$	
	Major Peak Area	Phases Area Ratio	Major Peak Area	Phases Area Ratio	Major Peak Area	Phases Area Ratio
FR2	1155	0.97	35	0.03	----	-----
PM1	429	0.72	27	0.04	142	0.24
PM1-DUP	25550	0.68	----	-----	11895	0.32
FR3	6769	0.63	3866	0.36	162	0.01
FR3-DUP	12896	0.48	13771	0.50	410	0.02

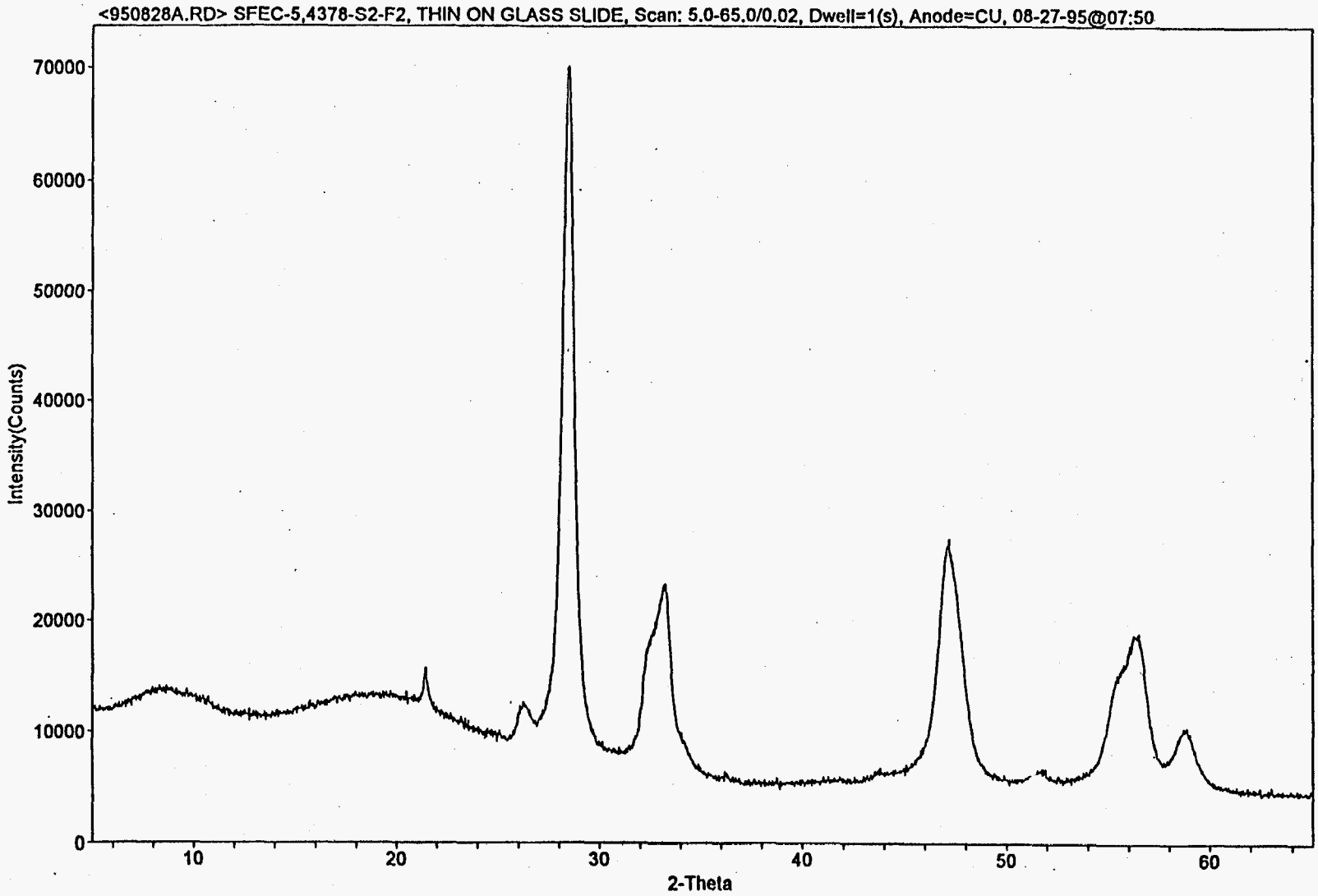


Figure 3.18: Raw Data Diffractogram of Specimen SFEC5,4378-S2-FR2

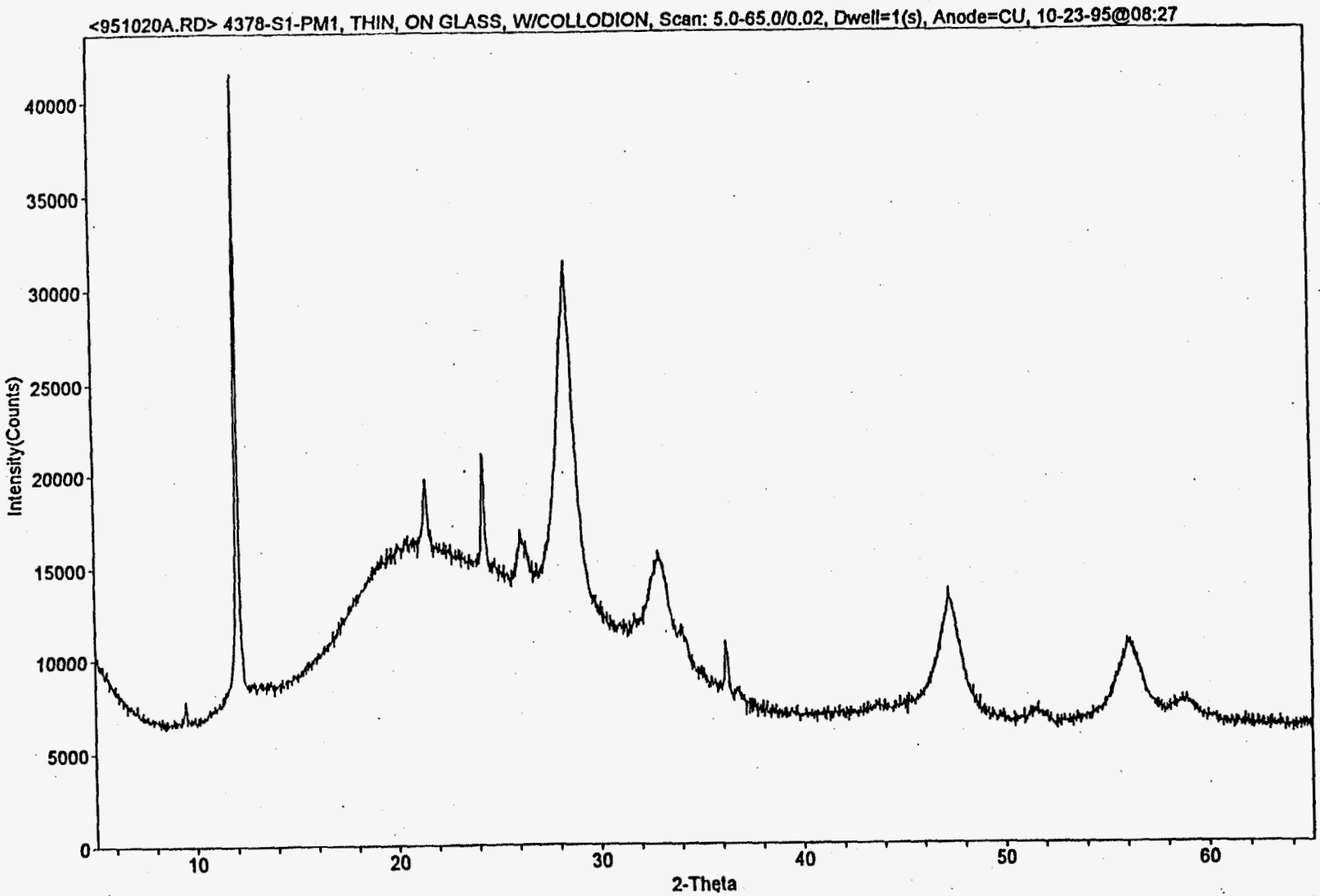


Figure 3.19: Raw Data Diffractogram of Specimen SFEC5,4378-S1-PM1

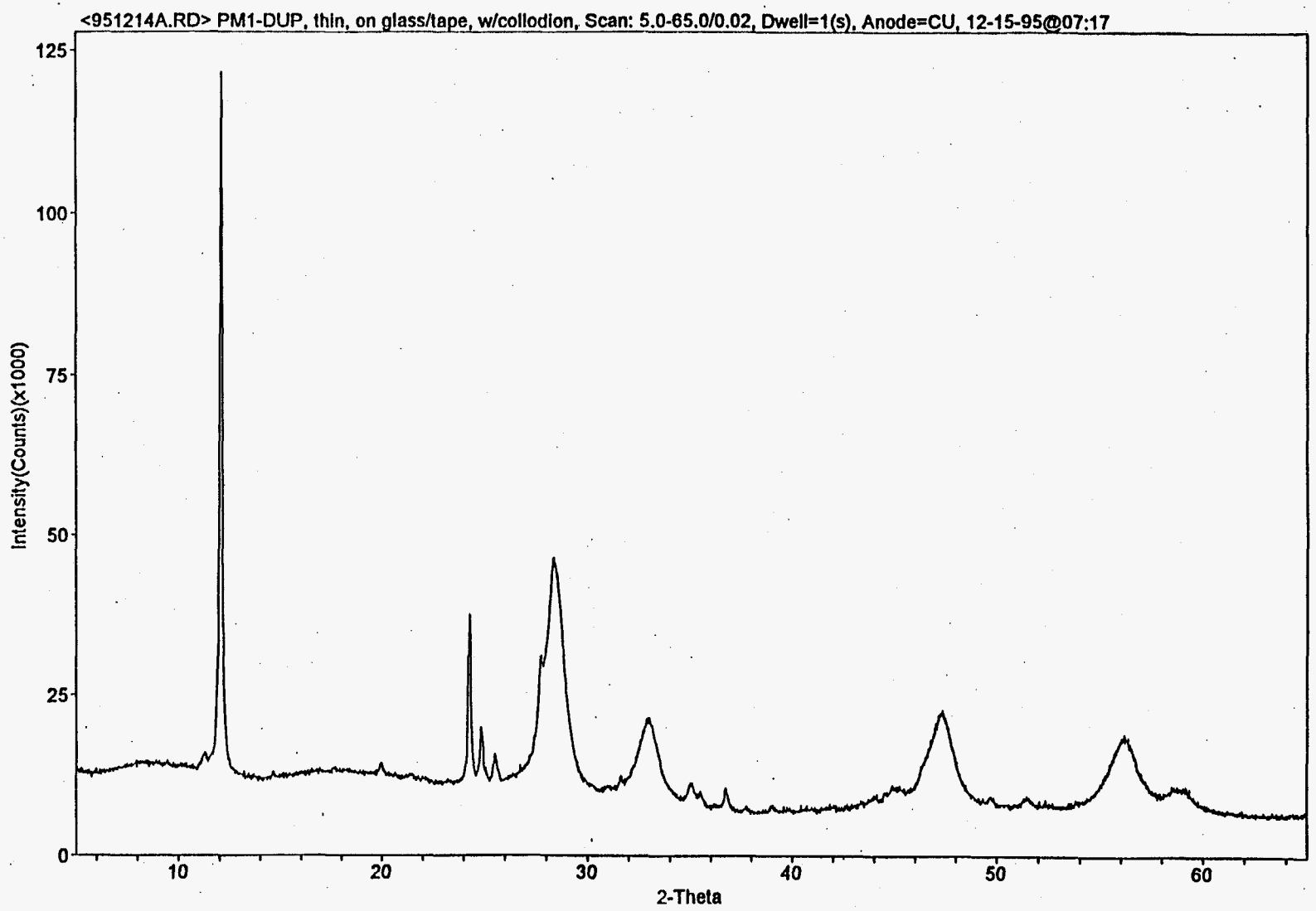


Figure 3.20: Raw Data Diffractogram of Specimen SFEC5,4378-S1-PM1-DUP

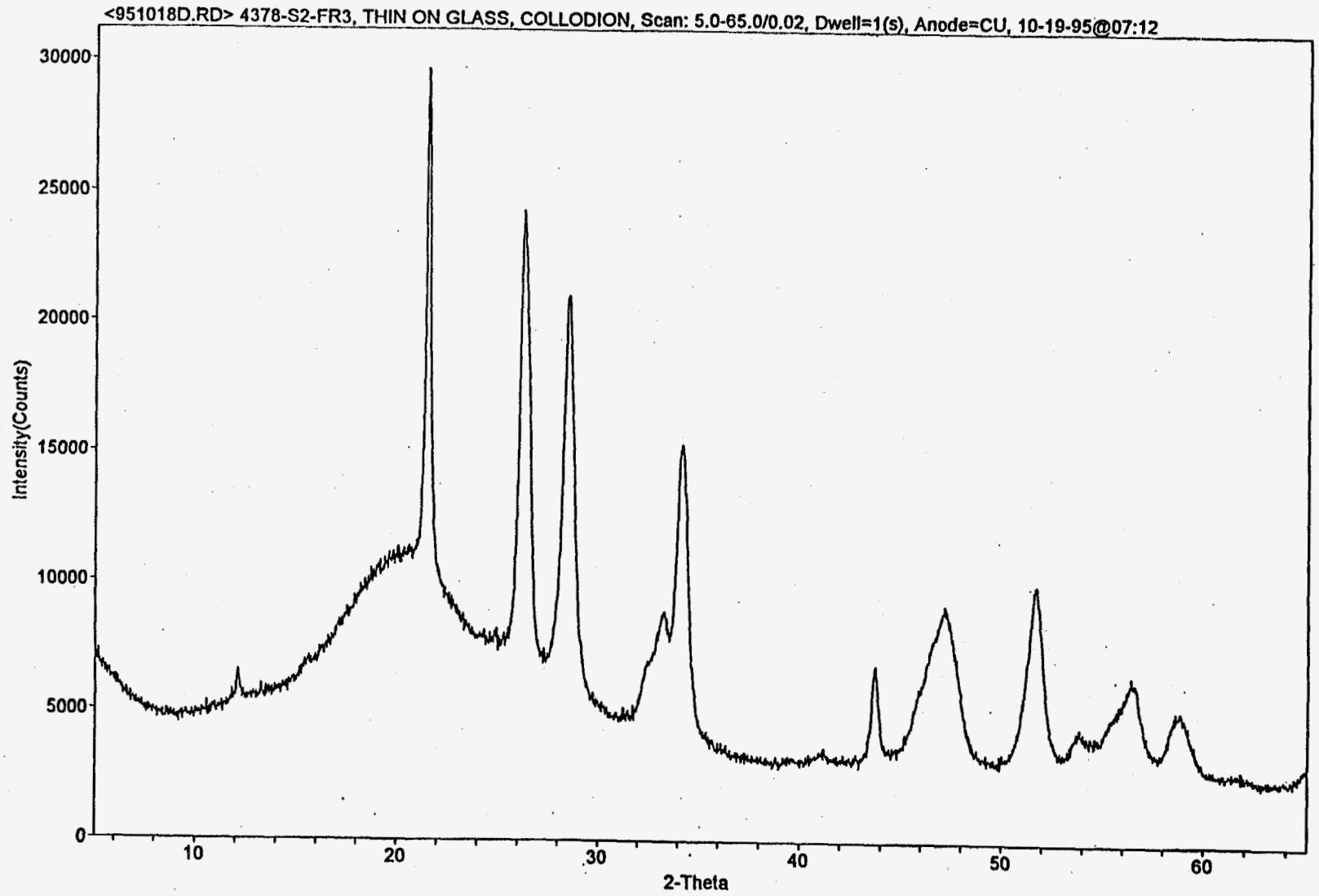


Figure 3.21: Raw Data Diffractogram of Specimen SFEC5,4378-S2-FR3

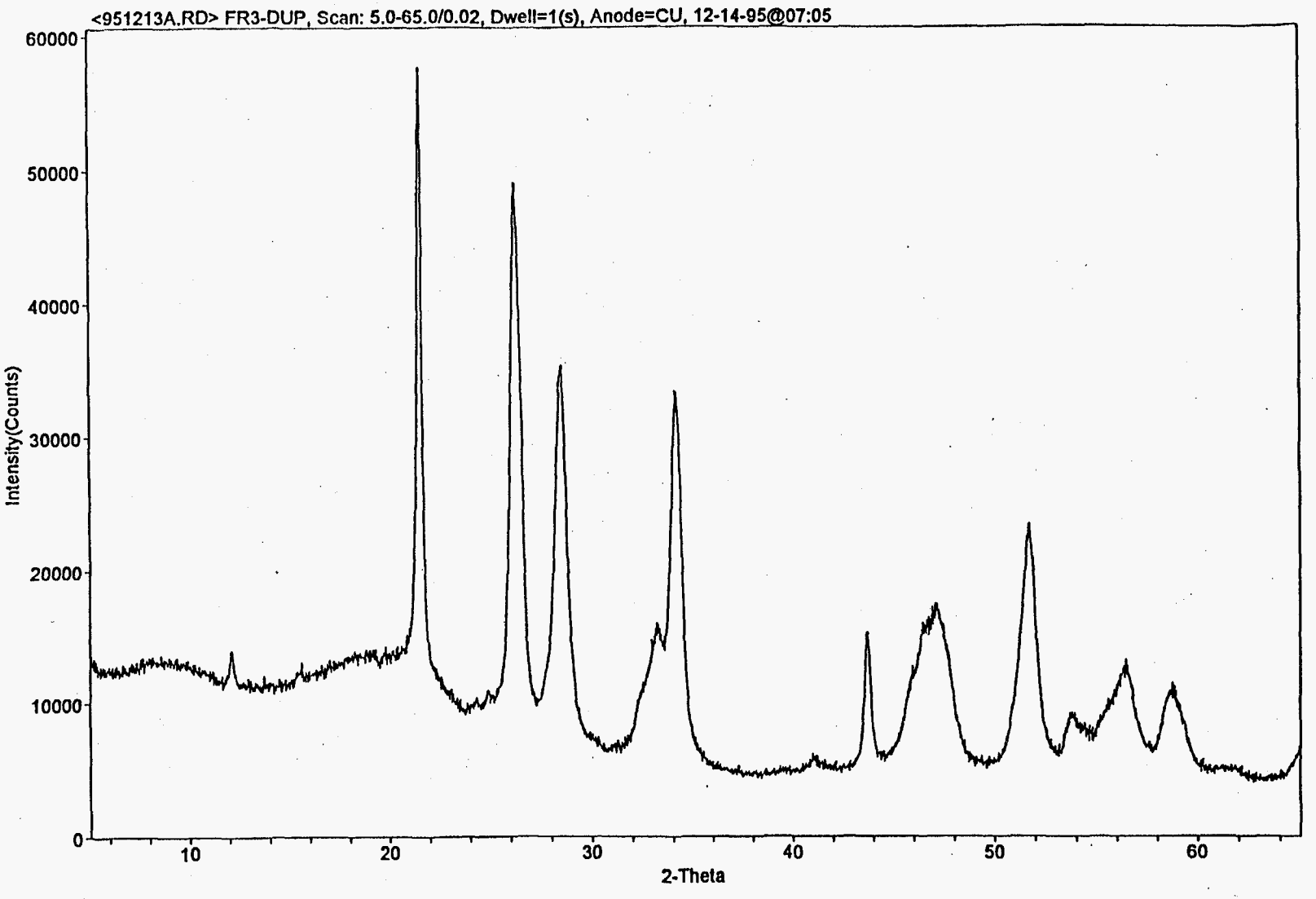


Figure 3.22: Raw Data Diffractogram of Specimen SFEC5,4378-S2-FR3-DUP

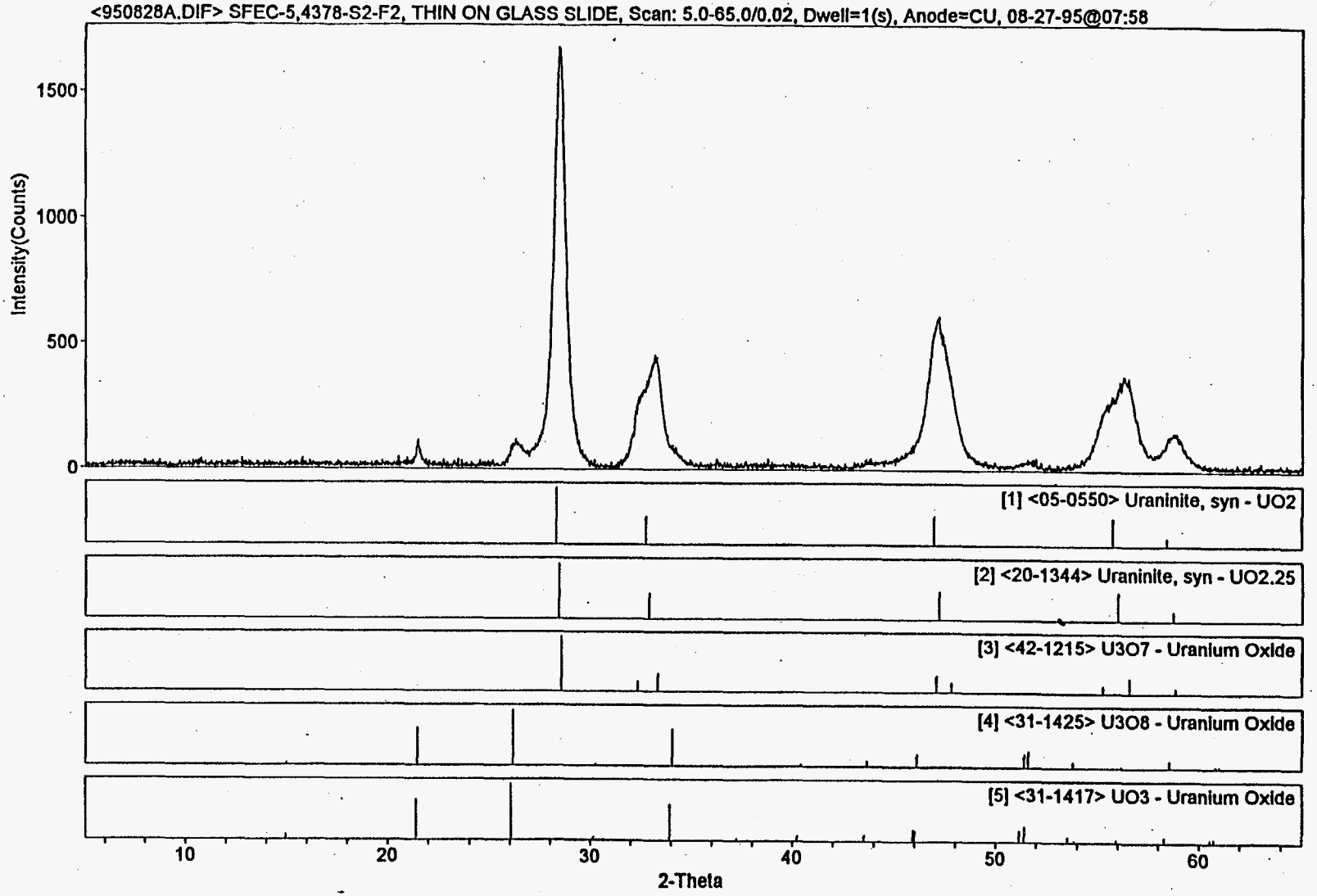


Figure 3.23: Background Subtracted Diffractogram of Specimen SFEC5,4378-S2-FR2. "Stick Figure" Pattern of Phases Identified also Shown.

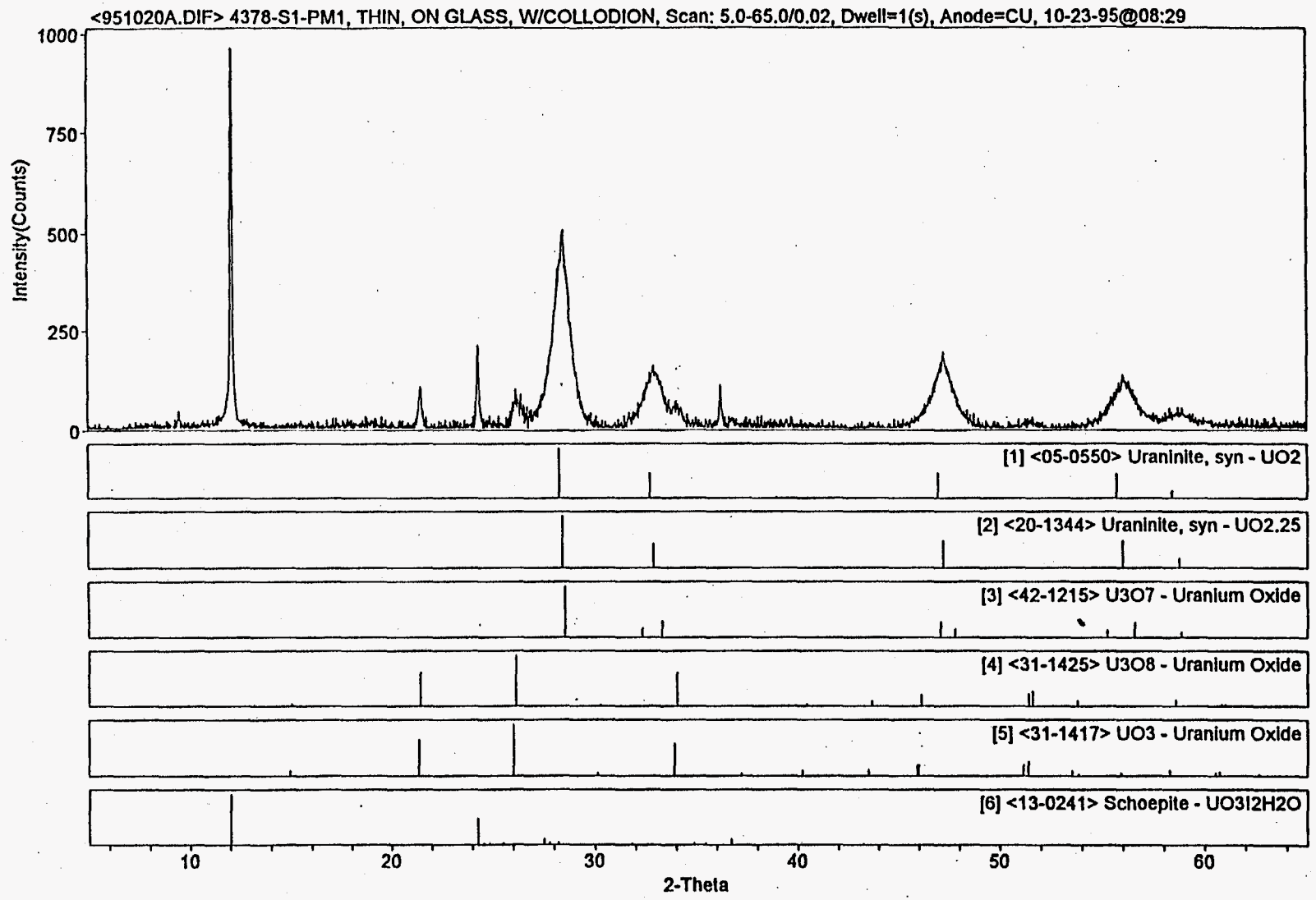


Figure 3.24: Background Subtracted Diffractogram of Specimen SFEC5,4378-S1-PM1. "Stick Figure" Pattern of Phases Identified also Shown.

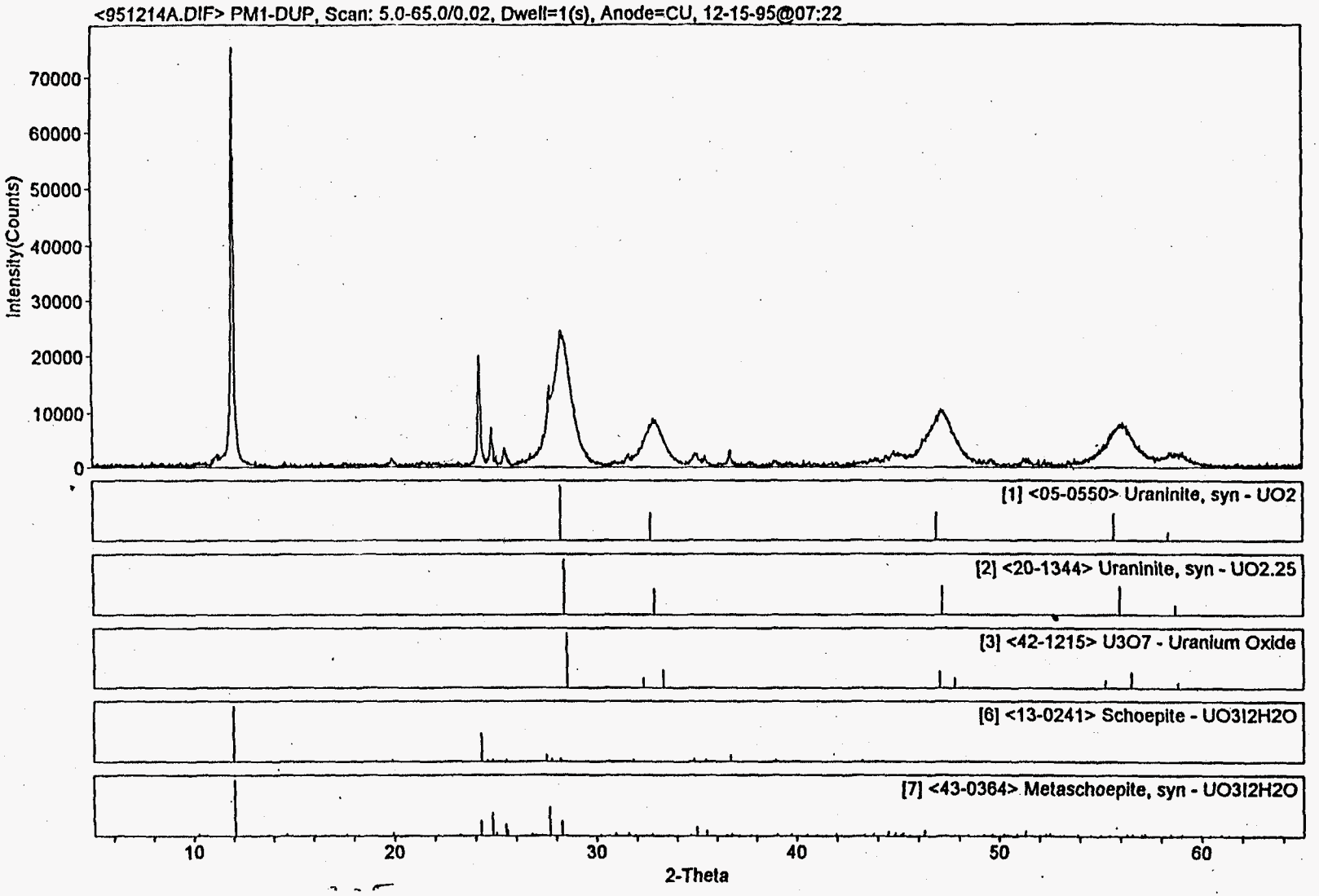


Figure 3.25: Background Subtracted Diffractogram of Specimen SFEC5,4378-S1-PM1-DUP and "Stick Figure" Patterns of Phases Identified

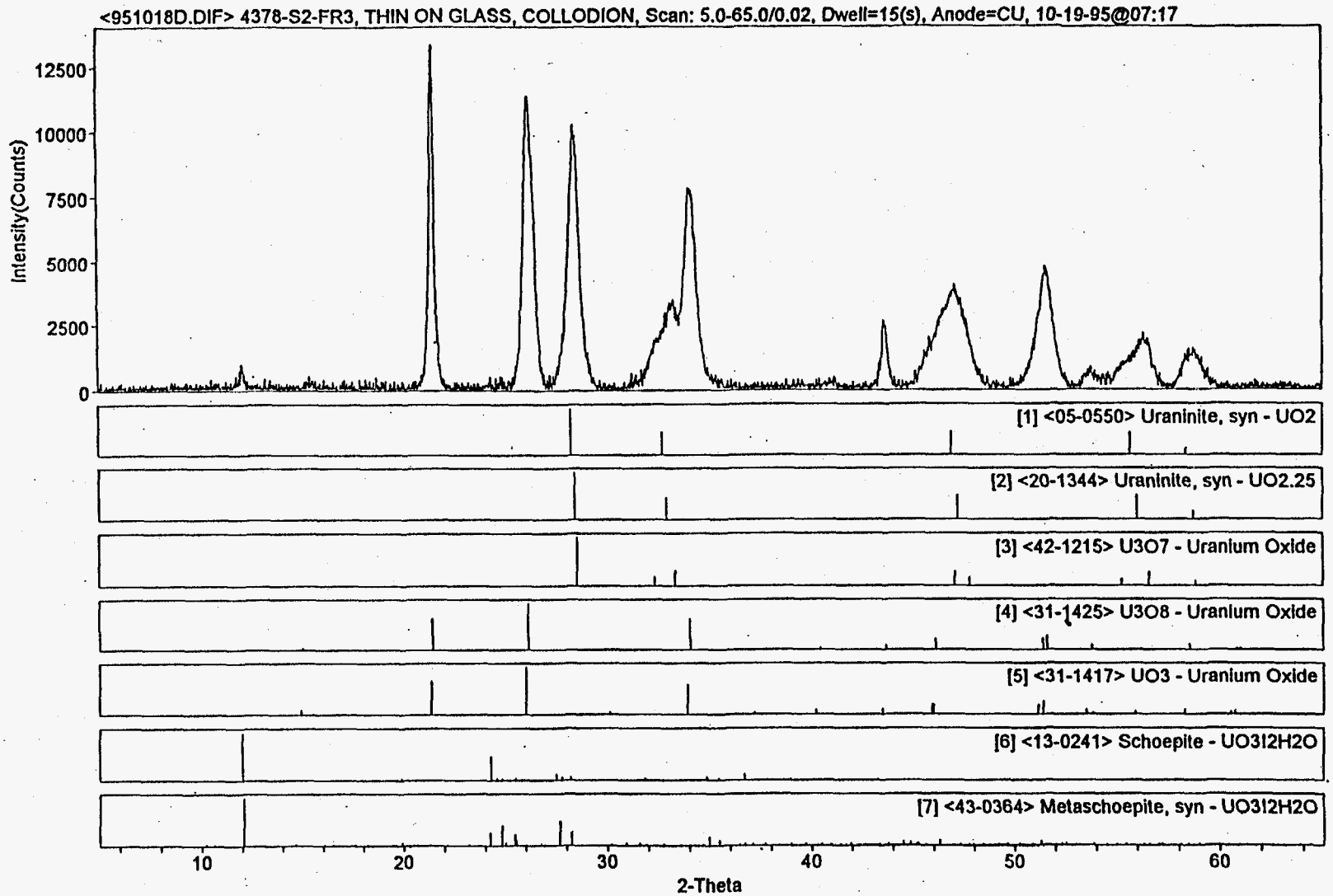


Figure 3.26: Background Subtracted Diffractogram of Specimen SFEC5,4378-S2-FR3 and "Stick Figure" Patterns of Phases Identified.

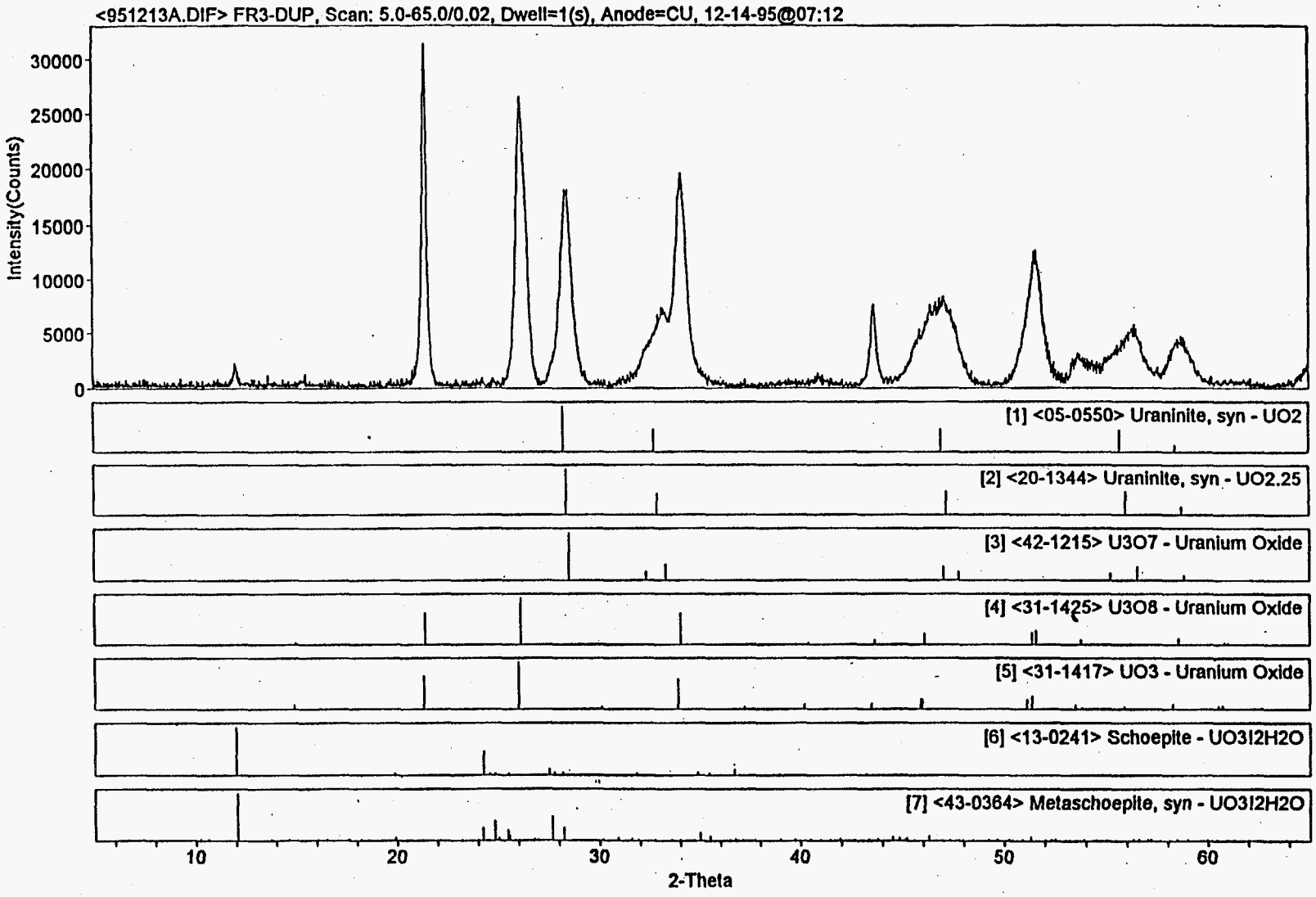


Figure 3.27: Background Subtracted Diffractogram of Specimen SFEC5,4378-S2-FR3-DUP and "Stick Figure" Patterns of Phases Identified.

4.0 References

Abrefah, J, WJ Gray, GL Ketner, TD Pyecha, TA Thornton, and SC Marschman, 1994, *K-Basin Spent Nuclear Fuel Characterization Data Report*, PNL-10778, Battelle Pacific Northwest National Laboratory, Richland, Washington.

Gardner, HR, and JW Riches, 1956, *The Effect of Cooling Rate on the Nucleation and Growth of Beta-Uranium Hydride in Metallic Uranium,* HW-43428, Hanford Atomic Products Operation, Richland, Washington.

Independent Technical Assessment (ITA), 1994, *Dry Storage of N Reactor Fuel,* Volume 1, U.S. Department of Energy Richland Operation Office, Richland, Washington.

Integrated Process Strategy for K Basins Spent Nuclear Fuel. Volume 1: Strategy and Recommendations, WHC-SD-SNF-SP-005, Vol. 1, Rev 0, (July 1995), Westinghouse Hanford Company, Richland, Washington.

Lawrence, LA, TA Thornton and J Abrefah, *Data Quality Objectives for the Initial K. West Fuel Examinations* WHC-SD-SNF-DQO-001, Rev. 0, (Feb. 1995), Westinghouse Hanford Company, Richland, Washington.

Lawrence, LA, *Data Quality Objectives for the Initial Conditioning Examinations* WHC-SD-SNF-DQO-004, Rev. 0, (May 1995), Westinghouse Hanford Company, Richland, Washington.

Musgrave, LE, *Theory of Burning Curve Ignition of Nuclear Metals*, J. Of Nuclear Materials, 43 (1972) 155.

Wilkinson, WD, *Uranium Metallurgy, Volume II: Uranium Corrosion and Alloys*, Interscience Pub. (1962).

Schnizlein, JG, PJ Pizzalato, HA Porte, JD Bingle, DF Fischer, LW Mishler, and RC Vogel, "*Ignition Behavior and Kinetics of Oxidation of the Reactor Metals, Uranium, Zirconium, Plutonium and Thorium, and Binary Alloys of Each*", ANL-5974, ANL, Lemont, Illinois (1959).

Appendix A

Tables

Table A.1. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S2-G

Test Instruction Data Sheet	Title: Vacuum Drying and Conditioning Testing of Initially Wet SNF Element SFEC5,4378 Specimens	Rev. No.: 0	Page 9 of 10

Operator: W J Gray Date: 7/24/95 Cognizant Scientist: [signature] Date: 7/25/95

EQUIPMENT DESCRIPTION

1 - Sample Balance Description: Mettler, AT-200	2 - Sample Balance Calibration Number: 370-06-01-007	3 - Calibration Expiration Date: 01-06-96
4 - Moisture Trap Balance Description: Mettler PM600	5 - Moisture Trap Balance Calibration Number: 354-01-01-009	6 - Calibration Expiration Date: 5/2/96
	7 - Ruler Calibration Number: SL524; CoRe 366-66-02-001	8 - Ruler Calibration Date: 7-13-95

SAMPLE MEASUREMENTS

9 - Specimen Identification Code: SFEC5, 4378-S2-G

Measurement Number	Weight Before Test (grams) Avg. of 3 Wgts.	Weight After Test (grams) Avg. of 3 Wgts.	Sample Dimensions (mm) Approx. Range of Values; Dimensions include Cladding: Length Z_a	Length Z_b	Thickness	Inner Chord X_{1a}	Inner Chord X_{1b}	Outer Chord X_{2a}	Outer Chord X_{2b}
1	10 35.6939	11 35.6977	12 24.0	13 24.1	14 9.6	15 7.5	16 7.5	17 10.6	18 10.7
2	19 ± 0.0001	20 ± 0.0000	21 24.0	22 24.0	23 9.8	24 7.5	25 7.6	26 10.5	27 10.6

28 - Specimen Identification Code: N/A

Measurement Number	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) Length Z_a	Length Z_b	Thickness	Inner Chord X_{1a}	Inner Chord X_{1b}	Outer Chord X_{2a}	Outer Chord X_{2b}
1	29	30	31	32	33	34	35	36	37
2	38	39	40	41	42	43	44	45	46

Measurement Number	Moisture Trap #1 Weight Before Test (g)	Moisture Trap #1 Weight After Test (g)	Moisture Trap #2 Weight Before Test (g)	Moisture Trap #2 Weight After Test (g)
1	47	48	49 433.30	50 433.12
2	51	52	53 433.29	54 433.12

Moisture Trap #1 not weighed nor used.

SYSTEM STATUS

OK 55 - Time/Date 7/24/95 @ 2133	OK 56 - Time/Date 7/26/95 @ 2050	OK 57 - Time/Date 7/26/95 @ 2121	OK 58 - Time/Date 7/27/95 @ 0902
62 J.A	63 J.A	64 J.A	65 J.A

Table A.2. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S2-F1& -F2

Test Instruction Data Sheet	Title: Vacuum Drying and Conditioning Testing of Initially Wet SNF Element SFEC5,4378 Specimens	Rev. No.: 0	Page 9 of 10

Operator: W.F. Gray Date: 8/7/95 Cognizant Scientist: [signature] Date: 8/8/95

EQUIPMENT DESCRIPTION

1 - Sample Balance Description: Mettler, AT-200	2 - Sample Balance Calibration Number: 370-06-01-007	3 - Calibration Expiration Date: 01-06-96
4 - Moisture Trap Balance Description: Mettler PM600	5 - Moisture Trap Balance Calibration Number: 354-06-01-009	6 - Calibration Expiration Date: 5/2/96
	7 - Ruler Calibration Number: SL524; Cadd 670-66-02-001	8 - Ruler Calibration Date: 7-13-95

SAMPLE MEASUREMENTS

9 - Specimen Identification Code: SFEC5, 4378-S2-F1

Measurement Number	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) ~ Range of Measured Values; Dimensions include Cladding						
			Length		Thickness	Inner Chord		Outer Chord	
	Avg. 3 Weighings	Avg. 3 Weighings	Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	10 39.6110	11 39.3493	12 23.6	13 23.7	14 9.6	15 8.7	16 8.4	17 11.2	18 11.7
2	19 ±0.0001	20 ±0.0000	21 23.7	22 23.9	23 9.9	24 8.8	25 8.7	26 11.5	27 12.0

28 - Specimen Identification Code: SFEC5, 4378-S2-F2

Measurement Number	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) ~ Range of Measured Values						
			Length		Thickness	Inner Chord		Outer Chord	
	Avg. 3 Weighings	Avg. 3 Weighings	Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	29 36.7232	30 36.4793	31 23.6	32 23.6	33 9.5	34 7.9	35 8.0	36 11.3	37 10.4
2	38 ±0.0001	39 ±0.0001	40 23.9	41 23.7	42 9.9	43 8.0	44 8.1	45 11.5	46 10.9

Measurement Number	Moisture Trap #1		Moisture Trap #2	
	Weight Before Test (g)	Weight After Test (g)	Weight Before Test (g)	Weight After Test (g)
1	47	48	49 442.08	50 442.00
2	51	52	53 442.08	54 442.00

Trap #1 not weighed nor used.

SYSTEM STATUS

62 - Time/Date 8/7/95 @ 2222	63 - Time/Date 8/9/95 @ 2150	64 - Time/Date 8/9/95 @ 2218	65 - Time/Date 8/10/95 @ 0855
62 J.A	63 J.A	64 J.A.	65 J.A.

Table A.3. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S2-E1

Test Instruction Data Sheet	Title: Furnace Testing of SNF Element SFEC5,4378	Rev. No.: 0	Page 8 of 9

Operator: W. J. Gray Date: 8/23/95 Cognizant Scientist: [signature] Date: 8/24/95

EQUIPMENT DESCRIPTION

1 - Sample Balance Description: Mettler, AT-200	2 - Sample Balance Calibration Number: 370-06-01-007	3 - Calibration Expiration Date: 01/06/96
4 - Moisture Trap Balance Description: N/A	5 - Moisture Trap Balance Calibration Number: N/A	6 - Calibration Expiration Date: N/A
	7 - Ruler Calibration Number: SL524; Cadd #670-66-02-001	8 - Ruler Calibration Date: 7-13-95

SPECIMEN MEASUREMENTS

9 - Specimen Identification Code: SFEC5, 4378-S2-E1 orientation lost During De-cladding ~Range of measured values after cladding removal

Measurement Number	Weight Before Test (grams) Avg of 3 weighings (Including Debris)	Weight After Test (grams)	Sample Dimensions (mm) Length Z_a	Length Z_b	Thickness	Inner Chord X_{1a}	Inner Chord X_{1b}	Outer Chord X_{2a}	Outer Chord X_{2b}
1	10 32.2051	11 32.2043	12 25.2	13 25.3	14 5.4	15 10.3	16 9.3	17 10.5	18 9.7
2	19 32.002	20 32.0041	21 25.3	22 25.5	23 7.1	24 10.5	25 9.4	26 11.0	27 9.8

28 - Specimen Identification Code: N/A

Measurement Number	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) Length Z_a	Length Z_b	Thickness	Inner Chord X_{1a}	Inner Chord X_{1b}	Outer Chord X_{2a}	Outer Chord X_{2b}
1	29	30	31	32	33	34	35	36	37
2	38	39	40	41	42	43	44	45	46

Measurement Number	Moisture Trap #1 Weight Before Test (g)	Moisture Trap #1 Weight After Test (g)	Moisture Trap #2 Weight Before Test (g)	Moisture Trap #2 Weight After Test (g)
1	47	48	49	50
2	51	52	53	54

Moisture traps were not weighed because previous tests where no water was added to specimen test showed insignificant moisture lost. This was verified by the moisture monitor in this case with an indicated loss of 0.82 mg.

SYSTEM STATUS

55 - Time/Date 8/23/95 @1300	56 - Time/Date N/A	57 - Time/Date N/A	58 - Time/Date N/A

Table A.4. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S1-CLAD-1

Test Instruction Data Sheet	Title: Furnace Testing of SNF Element SFEC5,4378	Rev. No.: 0	Page 8 of 9

Operator: W.F. Gray Date: 8/31/95 Cognizant Scientist: [signature] Date: 9/1/95

EQUIPMENT DESCRIPTION

1 - Sample Balance Description: Mettler AT-200	2 - Sample Balance Calibration Number: 370-06-01-007	3 - Calibration Expiration Date: 01-06-96
4 - Moisture Trap Balance Description: N/A	5 - Moisture Trap Balance Calibration Number: N/A	6 - Calibration Expiration Date: N/A
	7 - Ruler Calibration Number: SL524; Calc 670-66-02-001	8 - Ruler Calibration Date: 7-13-95

SPECIMEN MEASUREMENTS

9 - Specimen Identification Code: SFEC5, 4378-S1-CLAD-1 - Irregular Shape, measurements not taken per

Measurement Number	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) Length Z_a	Length Z_b	Thickness	Inner Chord X_{1a}	Inner Chord X_{1b}	Outer Chord X_{2a}	Outer Chord X_{2b}
1	① 10 1.8564	① 11 1.8262	12	13	14	15	16	17	18
2	19 ±0.0000	20 ±0.0001	21	22	23	24	25	26	27

① Average of 3 weighings.

28 - Specimen Identification Code: N/A

Measurement Number	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) Length Z_a	Length Z_b	Thickness	Inner Chord X_{1a}	Inner Chord X_{1b}	Outer Chord X_{2a}	Outer Chord X_{2b}
1	29	30	31	32	33	34	35	36	37
2	38	39	40	41	42	43	44	45	46

Measurement Number	Moisture Trap #1 Weight Before Test (g)	Moisture Trap #1 Weight After Test (g)	Moisture Trap #2 Weight Before Test (g)	Moisture Trap #2 Weight After Test (g)
1	47	48	49	50
2	51	52	53	54

Moisture trap not weighed due to anticipated low moisture total. This was verified by the low total moisture (2.4 mg) measured by the moisture monitor.

SYSTEM STATUS

55 - Time/Date: 8/31/95 @ 0918 W.F. Gray	56 - Time/Date: N/A	57 - Time/Date: N/A	58 - Time/Date: N/A

Table A.5. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S2-E2

Test Instruction Data Sheet	Title: Hydrogen Release Test of Specimen SFEC5,4378-S2-E2	Rev. No.: 0	Page 7 of 8

Operator: W K Hoag Date: 9/28/95 Cognizant Scientist: [signature] Date: 9/29/95

EQUIPMENT DESCRIPTION

1 - Sample Balance Description: Mettler, AT-200	2 - Sample Balance Calibration Number: 370-06-01-007	3 - Calibration Expiration Date: 01-06-96
4 - Moisture Trap Balance Description: N/A	5 - Moisture Trap Balance Calibration Number: N/A	6 - Calibration Expiration Date: N/A
	7 - Ruler Calibration Number: 52524; Code H670-66-02-001	8 - Ruler Calibration Date: 7-13-95

SPECIMEN MEASUREMENTS

9 - Specimen Identification Code: SFEC5,4378-S2-E2 Orientation lost during de-cladding ~Range~ % measured values after cladding removal

Measurement Number	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) Length Z_a	Length Z_b	Thickness	Inner Chord X_{1a}	Inner Chord X_{1b}	Outer Chord X_{2a}	Outer Chord X_{2b}
1	① 10 21.1015	① 11 20.9465	12 23.6	13 24.6	14 6.0	15 5.8	16 5.7	17 8.9	18 9.0
2	19 ±0.0001	20 ±0.0001	21 23.7	22 24.7	23 6.6	24 5.9	25 5.8	26 9.0	27 9.0

① Average of 3 weighings; Residue not included.

SYSTEM STATUS

28 - Time/Date: 9/28/95 @ 0840 W K Hoag

Table A.6. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S2-CD1

Test Instruction Data Sheet	Title: Hydrogen Release Test of Cladding Specimen SFEC5,4378-S2-E1-CD1	Rev. No.: 0	TI No.: SNF-CT-014	Page 7 of 8

Attachment 2

Operator: W J Gray Date: 10/11/95 Cognizant Scientist: [signature] Date: 10/12/95

EQUIPMENT DESCRIPTION

1 - Sample Balance Description Mettler, AT-200	2 - Sample Balance Calibration Number 370-06-01-007	3 - Calibration Expiration Date 01-06-96
	4 - Ruler Calibration Number SL524; Code #670-66-02-001	5 - Ruler Calibration Date 7-13-95

SPECIMEN MEASUREMENTS

Measurement Number	6 - Specimen Identification Code SFEC5,4378-S2-E1-CD1				
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) Range & measured Values		
			Length, Z	Thickness, t	Width, X
1	① 7 1.1337	① 8 1.1337	9 25.2	10 0.7	11 9.9
2	12 ±0.0001	13 ±0.0001	14 25.5	15 0.8	16 10.5

① Averaged 3 weighings

SYSTEM STATUS

17 - Time/Date 10/11/95 @ 1407 W J Gray

Table A.7. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S2-E3A

Test Instruction Data Sheet	Title: Ignition testing of Specimen SFEC5,4378-S2-E3A	Rev. No.: 0	TI No.: SNF-CT-012	Page 7 of 8

Attachment 2

Operator: W. F. Gray Date: 10/25/95 Cognizant Scientist: [signature] Date: 10/26/95

EQUIPMENT DESCRIPTION

1 - Sample Balance Description Mettler, AT-200	2 - Sample Balance Calibration Number 370-06-01-007	3 - Calibration Expiration Date 01-06-96
4 - Moisture Trap Balance Description Mettler PM600	5 - Moisture Trap Balance Calibration Number 354-06-1-09	6 - Calibration Expiration Date 5/2/96
	7 - Ruler Calibration Number SL524; Calg 670-06-02-001	8 - Ruler Calibration Date 7-13-95

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S2-E3A Dimensions include cladding								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) Range of measured values:						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_o	Z_b		X_{ia}	X_{ib}	X_{oa}	X_{ob}
1	① 10 14.7046	① 11 14.1771	12 11.3	13 10.8	14 9.4	15 7.8	16 6.8	17 9.2	18 8.9
2	19 ± 0.0001	20 ± 0.0000	21 12.1	22 12.1	23 9.6	24 7.9	25 7.3	26 9.3	27 9.0

① Average of 3 weighings; Residue not included.

Measurement Number	Moisture Trap #1	
	Weight Before Test (g)	Weight After Test (g)
1	28 302.97	29
2	30 302.97	31

Not weighed after test because moisture indicated by the moisture monitor was insignificant

SIGNATURE/VERIFICATION BLOCKS

32 10/25/95 @ 1114 W. F. Gray

Table A.8. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S2-E4A

Test Instruction Data Sheet	Title: Ignition testing of Specimen SFEC5,4378-S2-E4A	Rev. No.: 0	TI No.: SNF-CT-015	Page 7 of 8

Attachment 2

Operator: W J Gray Date: 11/1/95 Cognizant Scientist: [signature] Date: 11/2/95

EQUIPMENT DESCRIPTION

1 - Sample Balance Description Mettler, AT-200	2 - Sample Balance Calibration Number 370-06-01-007	3 - Calibration Expiration Date 01-06-96
4 - Moisture Trap Balance Description N/A	5 - Moisture Trap Balance Calibration Number N/A	6 - Calibration Expiration Date N/A
	7 - Ruler Calibration Number SL524; Code 670-66-02-001	8 - Ruler Calibration Date 7-13-95

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S2-E4A								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) ~ Range of measured values; Dimensions include cladding						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	① 10 9.4117	① 11 8.8302	12 10.6	13 10.4	14 9.4	15 4.1	16 3.8	17 7.1	18 6.9
2	19 ±0.0001	20 ±0.0001	21 10.8	22 11.0	23 9.6	24 4.5	25 3.9	26 7.1	27 7.1

① Average of 3 weighings; Residue not included.

Measurement Number	Moisture Trap #1	
	Weight Before Test (g)	Weight After Test (g)
1	28	29
2	30	31

Moisture trap not weighed because of insignificant moisture lost from duplicate specimen E3A.

SIGNATURE/VERIFICATION BLOCKS

32 11/1/95 @ 0900 W J Gray

Table A.9. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S2-H

Test Instruction Data Sheet	Title: Ignition testing of Specimen SFEC5,4378-S2-H	Rev. No.: 0	TI No.: SNF-CT-016	Page 7 of 8

Attachment 2

Operator: W J Gray Date: 11/3/95 Cognizant Scientist: [signature] Date: 11/6/95

EQUIPMENT DESCRIPTION

1 - Sample Balance Description Mettler, AT-200	2 - Sample Balance Calibration Number 370-06-01-007	3 - Calibration Expiration Date 01-06-96
4 - Moisture Trap Balance Description N/A	5 - Moisture Trap Balance Calibration Number N/A	6 - Calibration Expiration Date N/A
	7 - Ruler Calibration Number 51524; Cat 670-66-02-001	8 - Ruler Calibration Date 7-13-95

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S2-H								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) ~ Range of measured values; values include cladding						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_1	Z_2		X_{1i}	X_{2i}	X_{1o}	X_{2o}
1	ⓘ 10 28.5468	11 27.4339	12 24.1	13 24.1	14 9.4	15 5.5	16 6.0	17 8.7	18 8.8
2	19 ±0.0001	20 ±0.0001	21 24.2	22 24.2	23 9.6	24 5.5	25 6.1	26 8.8	27 8.9

ⓘ Average of 3 weighings; residue not included.

Measurement Number	Moisture Trap #1	
	Weight Before Test (g)	Weight After Test (g)
1	28	29
2	30	31

Moisture trap not weighed because previous tests with specimens E3A & E4A showed there was insignificant loss of moisture.

SIGNATURE/VERIFICATION BLOCKS

32 11/3/95 @ 1107 W J Gray

Table A.10. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S2-D

Test Instruction Data Sheet	Title: Ignition testing of Specimen SFEC5,4378-S2-D	Rev. No.: 0	TI No.: SNF-CT-018	Page 7 of 8

Attachment 2

Operator: W J Gray Date: 12/6/95 Cognizant Scientist: [redacted] Date: 11-28-95

EQUIPMENT DESCRIPTION

1 - Sample Balance Description METTLER, AT-200	2 - Sample Balance Calibration Number 370-06-01-007	3 - Calibration Expiration Date 01-06-96
[redacted]	4 - Ruler Calibration Number 670-66-02-001	5 - Ruler Calibration Date 07-13-95

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S2-D Dimensions include cladding								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			[redacted]	[redacted]		[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	6 28.7914	7 27.2973	8 25.2	9 25.3	10 9.2	11 4.9	12 6.4	13 8.0	14 8.9
[redacted]	15 ±0.0002	16 ±0.0001	17 25.3	18 25.2	19 9.4	20 5.6	21 6.7	22 8.1	23 9.0

SIGNATURE/VERIFICATION BLOCK

24 1431 h 11/10/95 WJ Gray

1) Weights are the average of three weighings.
2) "Weight After Test" does not include the weight of the material collected as particulate residue in the furnace boat (About 1.5540 ± 0.0001 g).

Table A.11. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S2-I

Test Instruction Data Sheet	Title: Ignition testing of Specimen SFEC5,4378-S2-I	Rev. No.: 0	TI No.: SNF-CT-019	Page 7 of 8

Attachment 2

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: 3/11/96

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
METTLER AT-200 (1)	370-06-01-007	01-06-96
	4 - Ruler Calibration Number: SL 524 670-66-02-001 ~~670-02-0001~~ PR 2/8/96	5 - Ruler Calibration Date: 7-13-95 (EXPIRES 7-12-96)

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S2-I								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) Plates H 34.9 Through H 35.4						
			Length (3)		Thickness (4)	Inner Chord (4)		Outer Chord (4)	
			Z_1	Z_2		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	6 37.2672 (2)	7 36.8158 (2),(5)	8 24.6	9 24.8	10 9.3	11 7.3	12 7.8	13 11.3	14 11.1
2	15 ±0.0001	16 ±0.0001	17 24.4	18 25.0	19 9.6	20 7.5	21 8.0	22 11.5	23 11.3

SIGNATURE/VERIFICATION BLOCK

24

Notes:
(1) Balance certified accurate to ± 0.0016 g.
(2) Average of three weighings, ± 1 standard deviation on the mean.
(3) Fuel length.
(4) Measurements include cladding.
(5) Does not include the weight of particulate residue collected from the furnace boat. (Residue sample, 5-52-FR9, weighs 0.1155 g ± 0.0001 g.)

2/8/96

Table A.12. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S2-F4

Test Instruction Data Sheet	Title: Vacuum Drying and Conditioning Testing of Wet SNF Specimen SFEC5,4378-S2-F4	TI No.: SNF-CT-025 Rev. 00	Page 8 of 9

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: 3/11/96

EQUIPMENT DESCRIPTION

1 - Sample Balance Description: Mettler AT-200 ①	2 - Sample Balance Calibration Number: 370-06-01-007	3 - Calibration Expiration Date: 01-06-96
4 - Moisture Trap Balance Description	5 - Moisture Trap Balance Calibration Number	6 - Calibration Expiration Date
	7 - Ruler Calibration Number: # SL524 670-66-02-001	8 - Ruler Calibration Date: 7-13-95 (Expires 7-12-96)

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code SFEC5,4378-S2-F4								
	Weight Before Test (grams) ②	Weight After Test (grams) ②	Sample Dimensions (mm) See Photos # 172 Through # 176 Length ③ Z_a	Z_b	Thickness ④	Inner Chord ④ X_{1a}	X_{1b}	Outer Chord ④ X_{2a}	X_{2b}
1	10 28.8024	11 28.8100	12 23.6	13 24.0	14 9.7	15 5.8	16 5.1	17 9.6	18 9.0
2	19 ±0.0001	20 ±0.0001	21 24.0	22 24.2	23 10.1	24 5.9	25 5.3	26 9.7	27 9.2

Measurement Number	Moisture Trap #1		Moisture Trap #2	
	Weight Before Test (g)	Weight After Test (g)	Weight Before Test (g)	Weight After Test (g)
1	28	29	30	31
2	32	33	34	35

NOTES:
① Balance certified accurate to ± 0.00016 g.
② Average of 3 weighings, ± 1 standard deviation on the mean.
③ ~~Full~~ Fuel length only
④ Measurements include cladding. VAByrd 2/9/96

SYSTEM STATUS

36 - Time/Date	37 - Time/Date	38 - Time/Date	39 - Time/Date	40 - Time/Date

Table A.13. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S1A-D

Test Instruction Data Sheet	Title: Ignition testing of Specimen SFEC5,4378-S1A-D1 DP 2-7-96	Rev. No.: 0	TI No.: SNF-CT-021	Page 7 of 8

Attachment 2

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: 3/11/96

EQUIPMENT DESCRIPTION

1 - Sample Balance Description: Mettler AT-200 (1)	2 - Sample Balance Calibration Number: 370-06-01-007	3 - Calibration Expiration Date: 01-08-97
	4 - Ruler Calibration Number: SL524 670-66-02-001	5 - Ruler Calibration Date: 7-13-95 (EXPIRES 7-13-96)

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S1A-D1 DP 2-7-96 Refer to Photos # 422 Through # 427									
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)							
			Length		Thickness	Inner Chord		Outer Chord	
			Z_1 (4)	Z_2 (4)	(3)	X_{i1} (5)	X_{i2} (5)	X_{o1} (5)	X_{o2} (3)
1	6 36.4920 (2)	7 36.9550 (2)	8 28.6	9 25.0	10 9.0	11 6.7	12 6.4	13 10.3	14 9.7
2	15 ± 0.0001	16 ± 0.0001	17 29.5	18 22.5	19 9.0	20 6.5	21 6.5	22 10.4	23 9.8

SIGNATURE/VERIFICATION BLOCK

24

NOTES:

(1) Balance is certified accurate to ± 0.0016 g.

(2) Average of three weighings ± 1 standard deviation on the mean.

(3) Includes cladding.

(4) Does not include cladding (Cladding length: I.D. Side ~ 33.5 ± 2 mm, O.D. Side ~ 50.0 ± 0.5 mm)

(5) As measured at the damaged edge of the remaining uranium fuel. Values include cladding

DP 2-7-96

Table A.14. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S1A-C

Test Instruction Data Sheet	Title: Vacuum Drying and Conditioning Testing of Wet SNF Specimen SFEC5,4378-S1A-C	TI No.: SNF-CT-026 Rev. 00	Page 8 of 9

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: 3/11/96

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
Mettler AT-200 (1)	370-06-01-007 #SL524	01-08-97
	4 - Ruler Calibration Number 670-66-02-001	5 - Ruler Calibration Date 07-13-95 (Expires 7-13-96)

SAMPLE MEASUREMENTS

Measurement Number	6 - Specimen Identification Code SFEC5,4378-S1A-C								
	Weight Before Test (grams) (2)	Weight After Test (grams) (2)	Sample Dimensions (mm) See Photos # 428, 429, and #431 - #433 Length FUEL ONLY (3) Z_a	Z_b	Thickness (4)	Inner Chord (4) X_{1a}	X_{1b}	Outer Chord (4) X_{2a}	X_{2b}
1	7 52.2238	8 51.4456	9 37.7	10 33.0	11 9.3	12 6.8	13 7.2	14 10.8	15 11.3
2	16 ±0.0001	17 ±0.0001	18 40.5	19 37.5	20 9.5	21 7.0	22 7.4	23 11.0	24 11.5

SYSTEM STATUS

25 - Time/Date	26 - Time/Date	27 - Time/Date	28 - Time/Date	29 - Time/Date

NOTES: (1) Balance certified accurate to ± 0.0016 g.
(2) Average of 3 weighings, ± 1 standard deviation on the mean.
(3) Damaged fuel end has very irregular surface geometry; values shown are for fuel only.
(4) Dimensions shown include cladding.
(5) Cladding length (irregular): O.D. side ~ 46.5 ± 2.0 mm
I.D. side ~ 40.0 ± 3.0 mm
TA Rych 2/7/96

Table A.15. Dimensions And Weight Measurements Of Specimen SFEC5,4378-S1A-H

Test Instruction Data Sheet	Title: Ignition testing of Specimen SFEC5,4378-S1A-H	Rev. No.: 0	TI No.: SNF-CT-029	Page 6 of 7

Attachment 2

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: 3/11/96

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
METTLER AT-200 (1)	370-06-01-007	01-08-97
	4 - Ruler Calibration Number: #SL 524, 670-66-02-001	5 - Ruler Calibration Date: 7-13-95 (EXPIRES 7-13-96)

SAMPLE MEASUREMENTS

9 - Specimen Identification Code: SFEC5,4378-S1A-H ~~(Refer to Photos #440 through #444)~~ VDB 2-7-96

Measurement Number	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm) Refer to Photos #440 Through #444						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_1 (5)	Z_2 (5)	(4)	X_{1a} (4)	X_{1b} (4)	X_{2a} (4)	X_{2b} (4)
1	6 48.6016 (2)	7 49.1828 (2),(3)	8 22.0	9 21.3	10 9.4	11 8.5	12 8.5	13 12.0	14 11.8
2	15 ± 0.0001	16 ± 0.0001	17 31.5	18 33.3	19 9.7	20 8.6	21 8.6	22 12.1	23 11.9

SIGNATURE/VERIFICATION BLOCK

24

(4) measurements taken at fuel boundaries; values shown include the cladding.

(5) Does not include cladding; Fuel length is irregular. Cladding length:
O.D Side ~ 21.5 ± 0.5 mm
I.D. Side ~ 51.5 ± 1.0 mm

VDBrack 2/6/96

NOTES:
(1) Balance is certified accurate to ± 0.0016 g
(2) Average of three weighings, ± 1 standard deviation of the mean.
(3) Sample and residue weighed together - dropped while unloading furnace, but swept up prior to weighing, with possible loss of ~10% of the residue (per log notes).

Appendix B

Figures

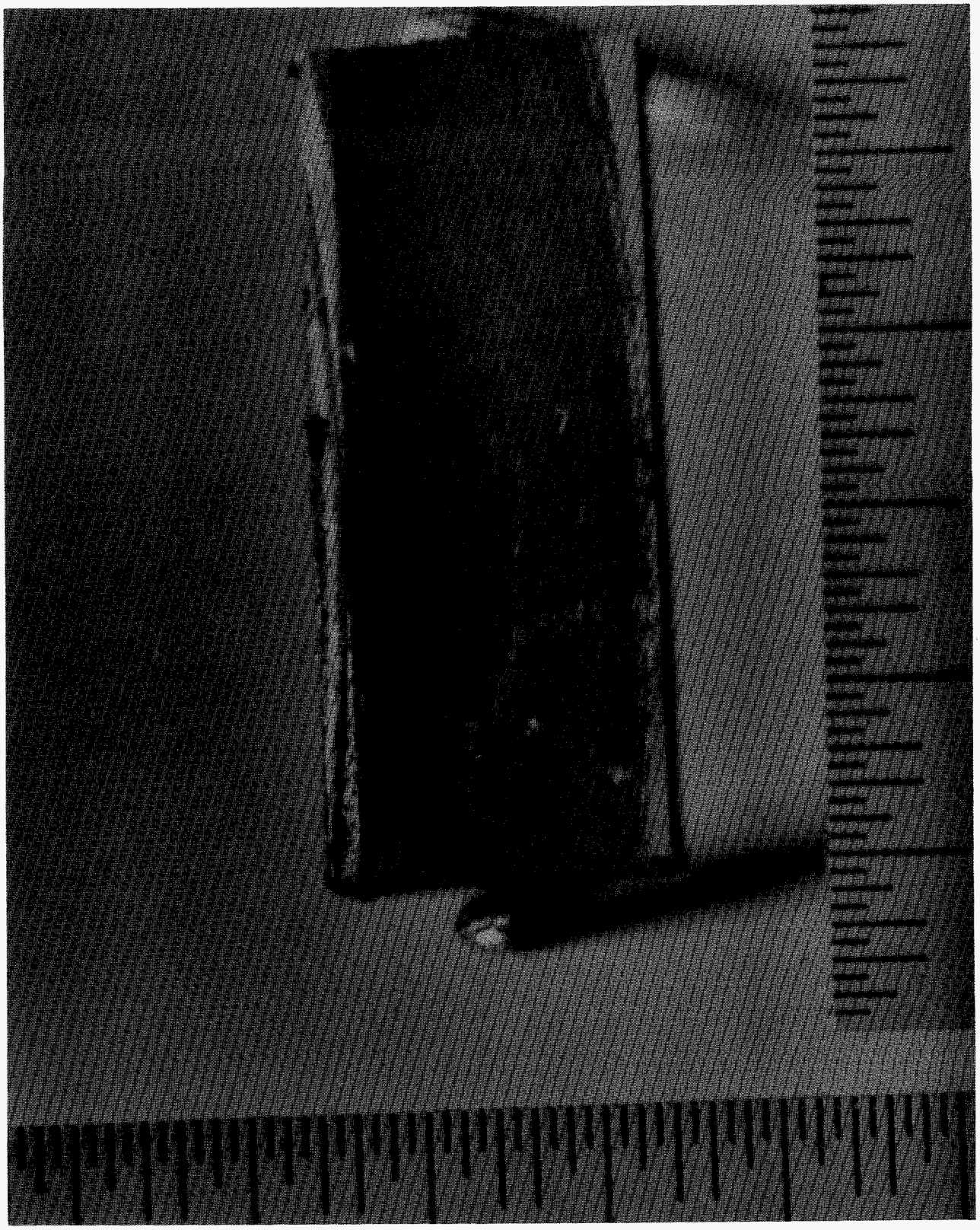


Figure B.1: Longitudinal Plane of Specimen SFEC5,4378-S2-F1. Each division of the attached scale is 0.5 mm. The PTL log is Q-1344, Photo # 156

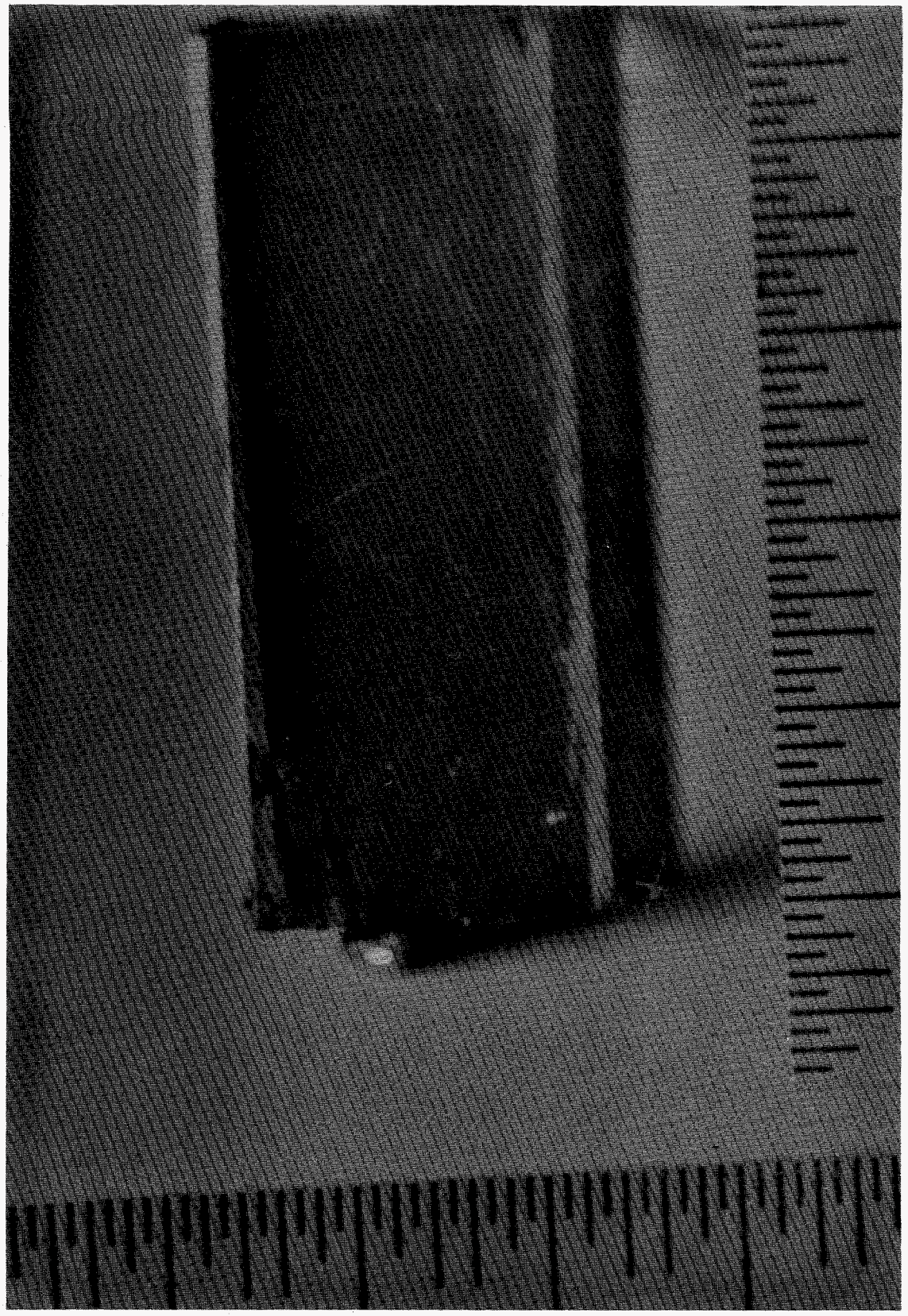


Figure B.2: Other Longitudinal Plane of Specimen SFEC5,4378-S2-F1. Each division of the attached scale is 0.5 mm. The PTL log is Q-1345, Photo # 157.

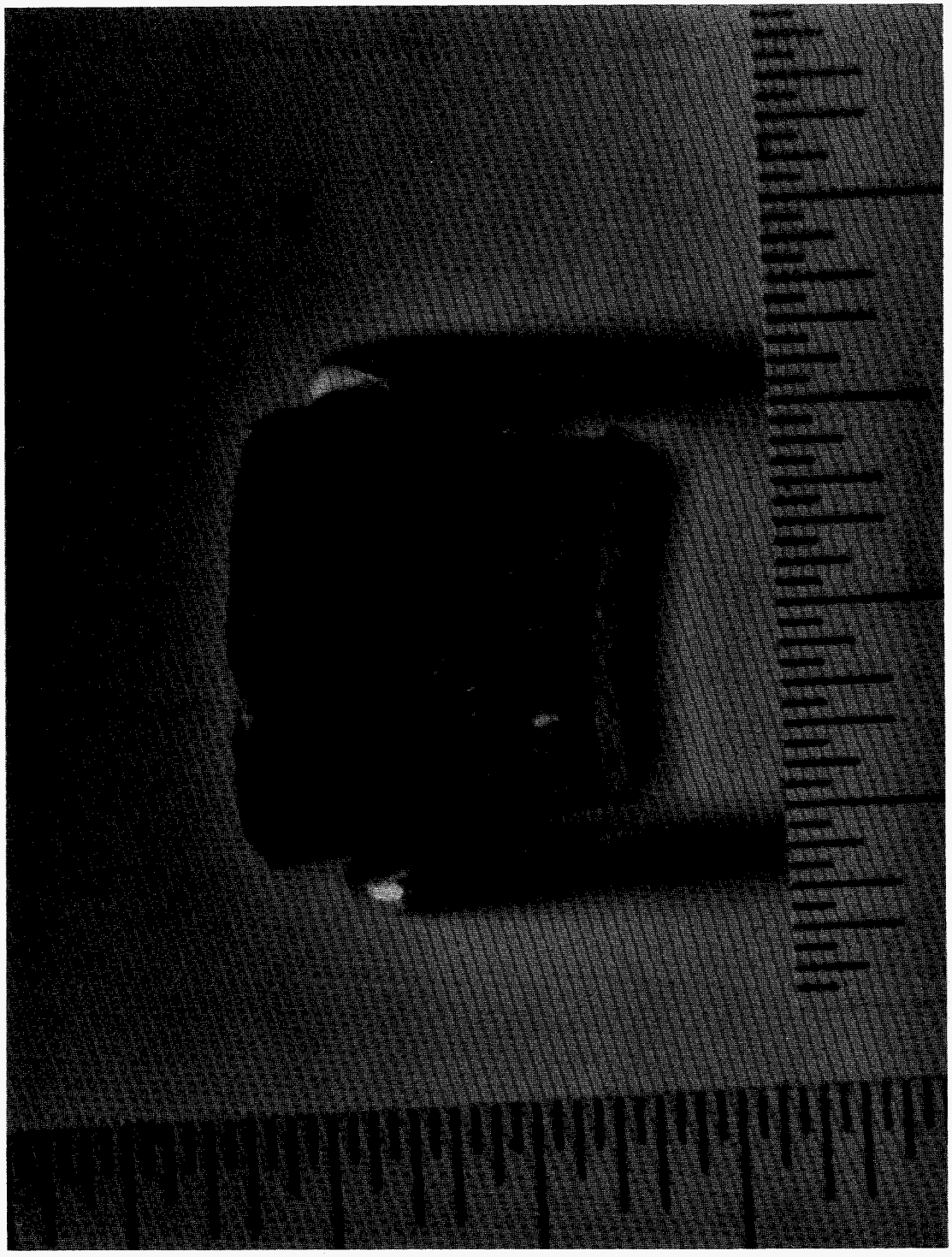


Figure B.3: Top Plane of Specimen SFEC5,4378-S2-F1. Each division of the attached scale is 0.5 mm. The PTL log is Q-1346, Photo # 158.

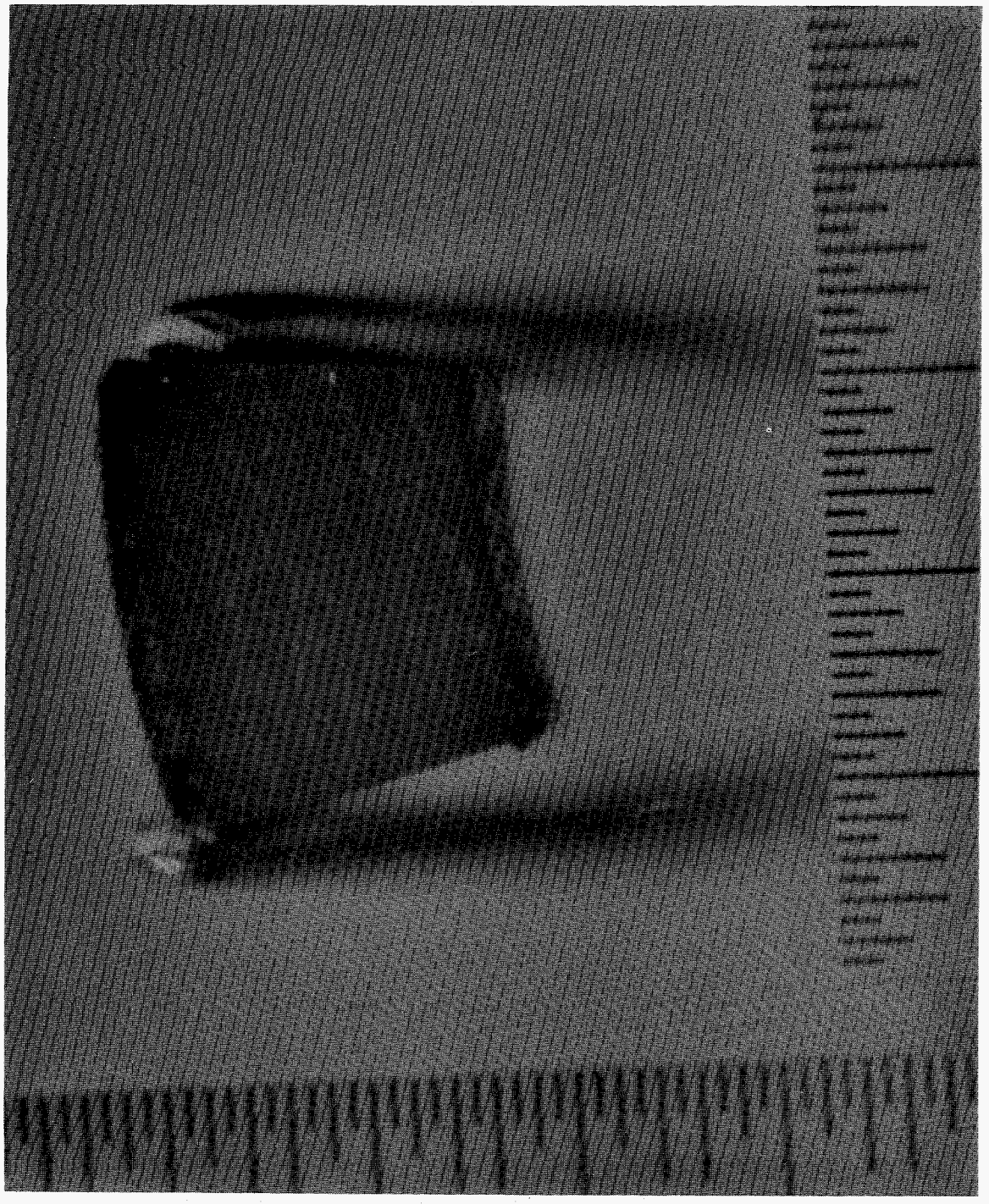


Figure B.4: Bottom Plane of Specimen SFEC5,4378-S2-F1. Each division of the attached scale is 0.5 mm. The PTL log is Q-1347, Photo # 159.

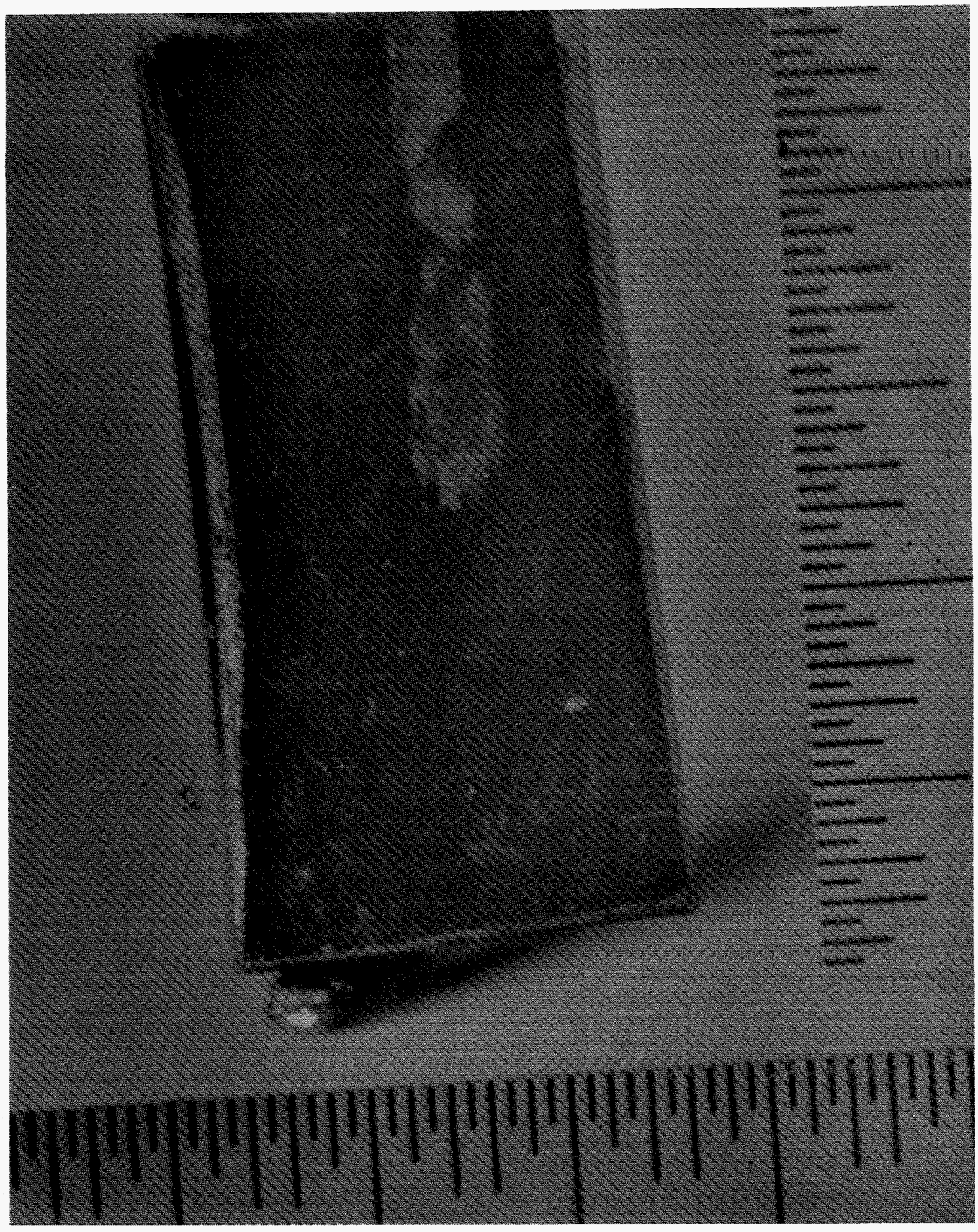


Figure B.5: Outside Cladding Plane of Specimen SFEC5,4378-S2-F1 Showing the Saw Mark. Each division of the attached scale is 0.5 mm. The PTL log is Q-1348, Photo #160.

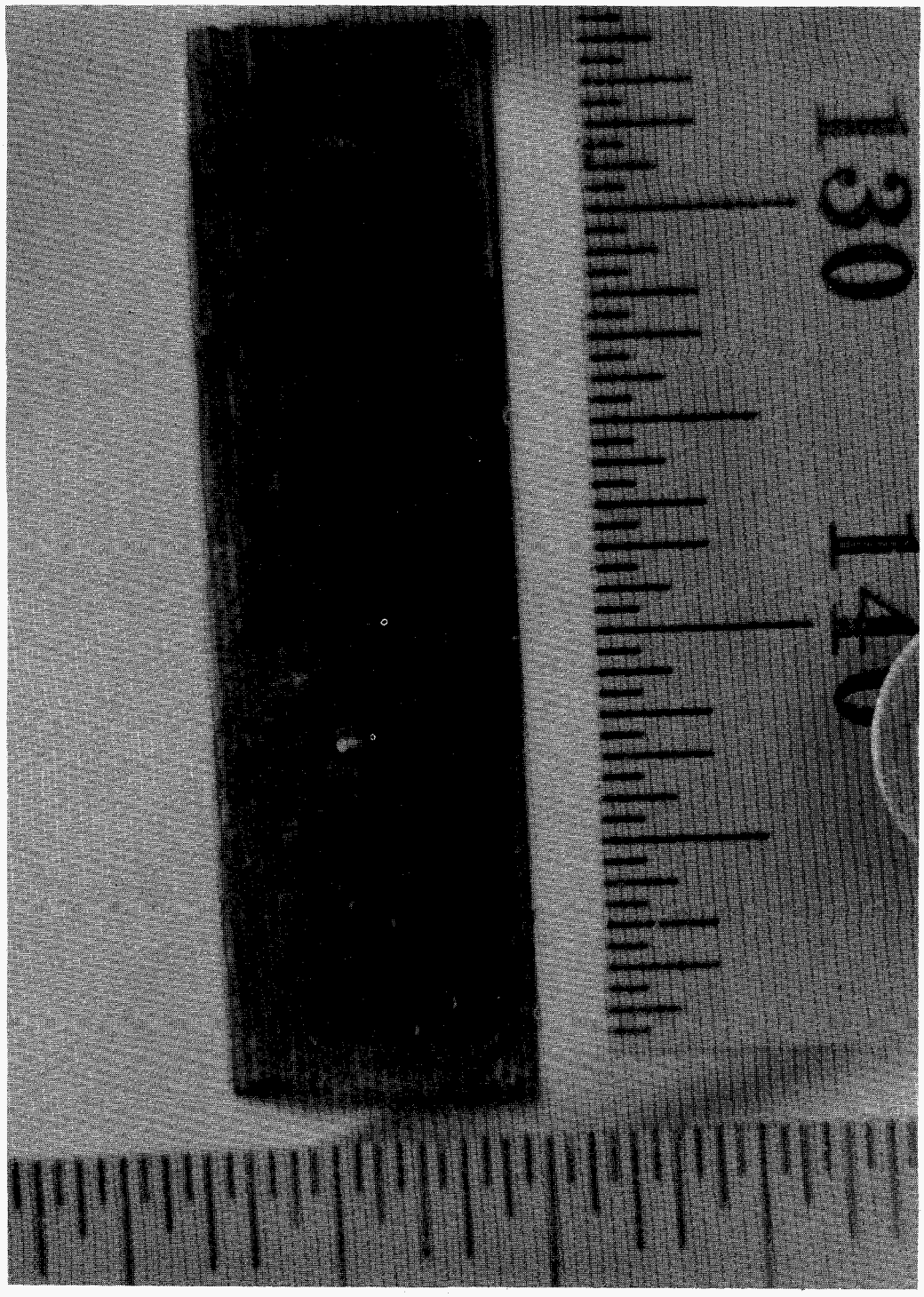


Figure B.6: Longitudinal Plane of Specimen SFEC5,4378-S2-E2. Each division of the attached scale is 0.5 mm. The PTL log is Q-1386, Photo # 196.

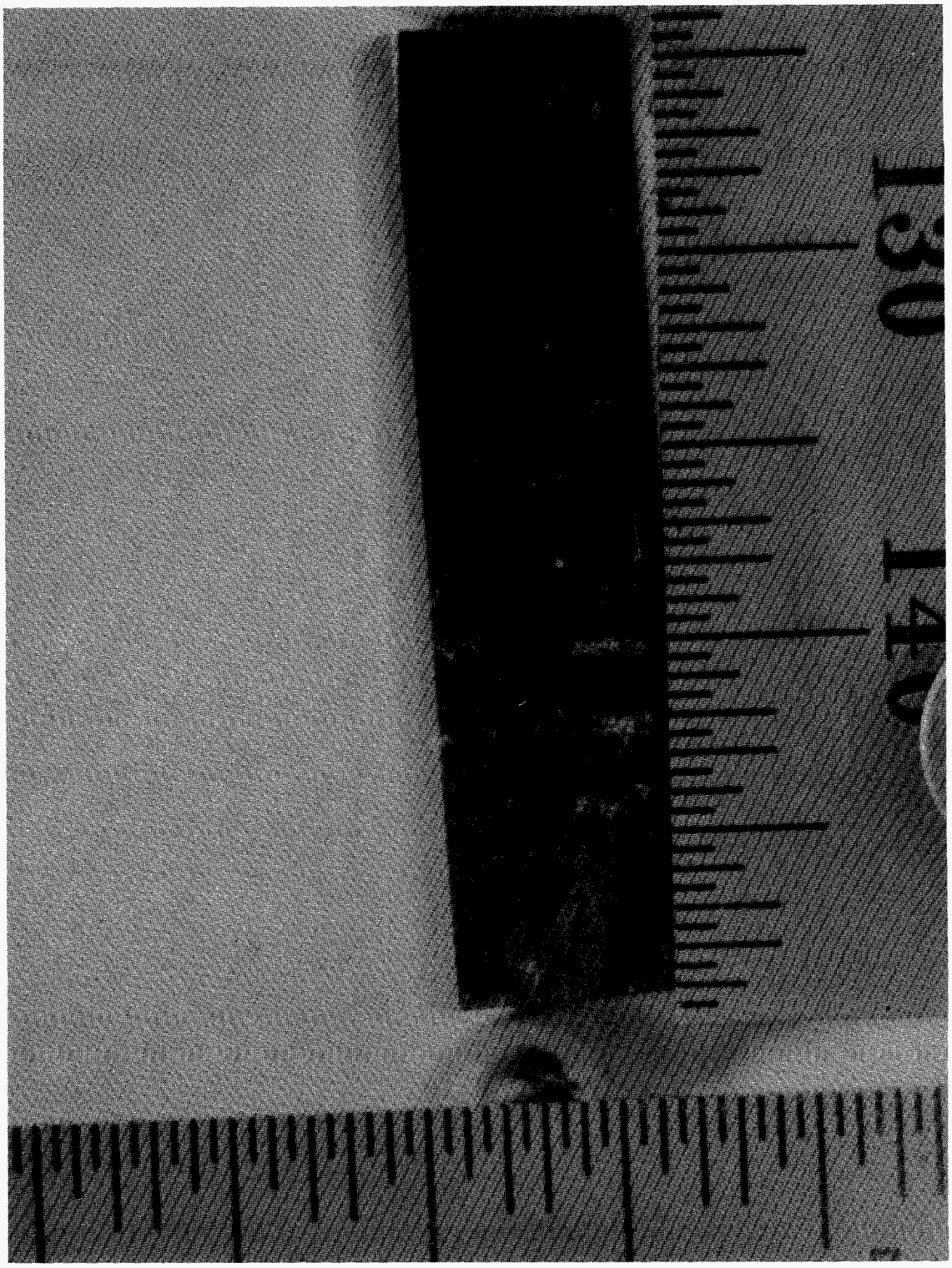


Figure B.7: Longitudinal Plane of Specimen SFEC5,4378-S2-E2. Each division of the attached scale is 0.5 mm. The PTL log is Q-1387, Photo # 197.

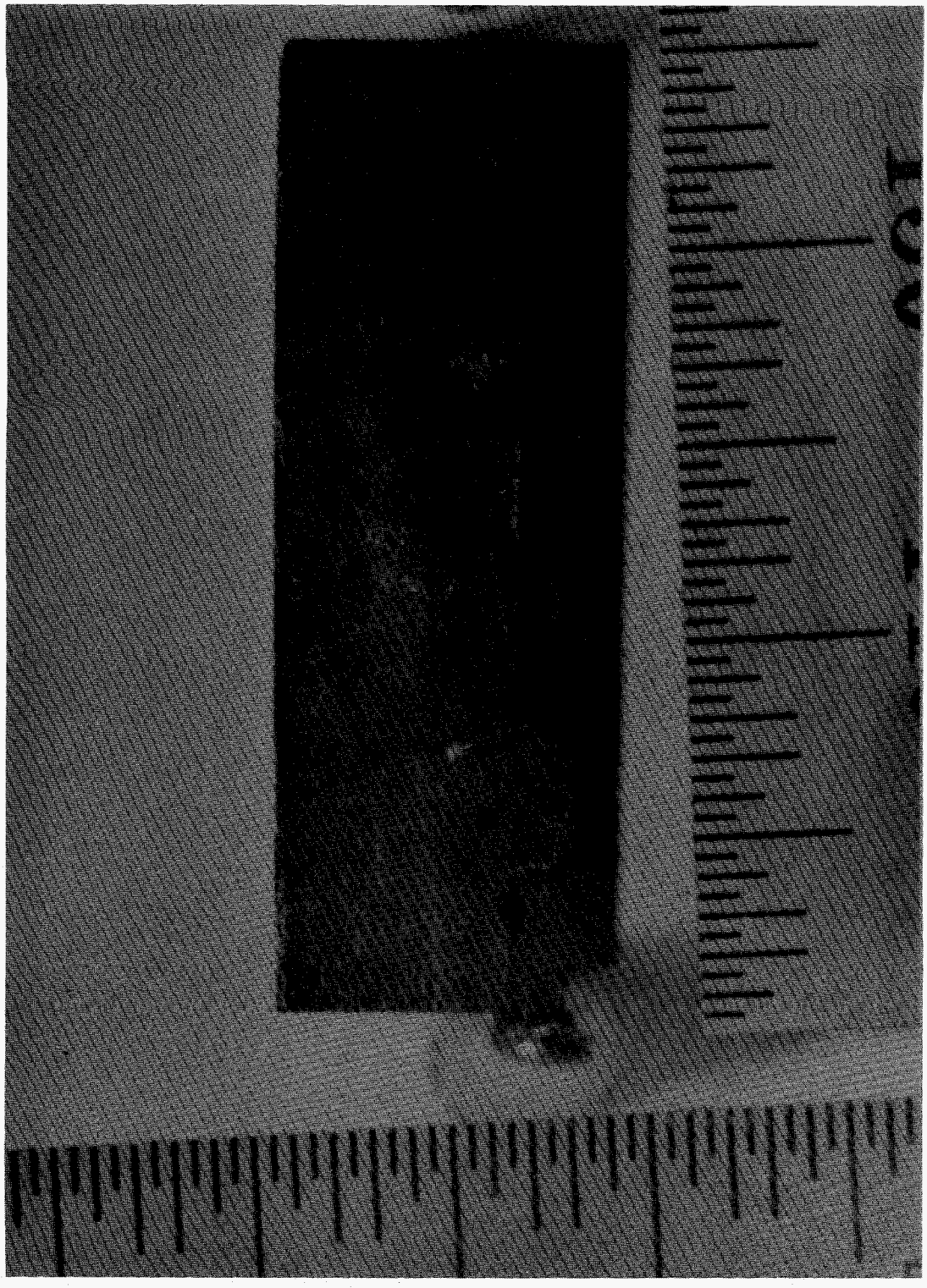


Figure B.8: Longitudinal Plane of Specimen SFEC5,4378-S2-E2. Each division of the attached scale is 0.5 mm. The PTL log is Q-1388, Photo # 200.

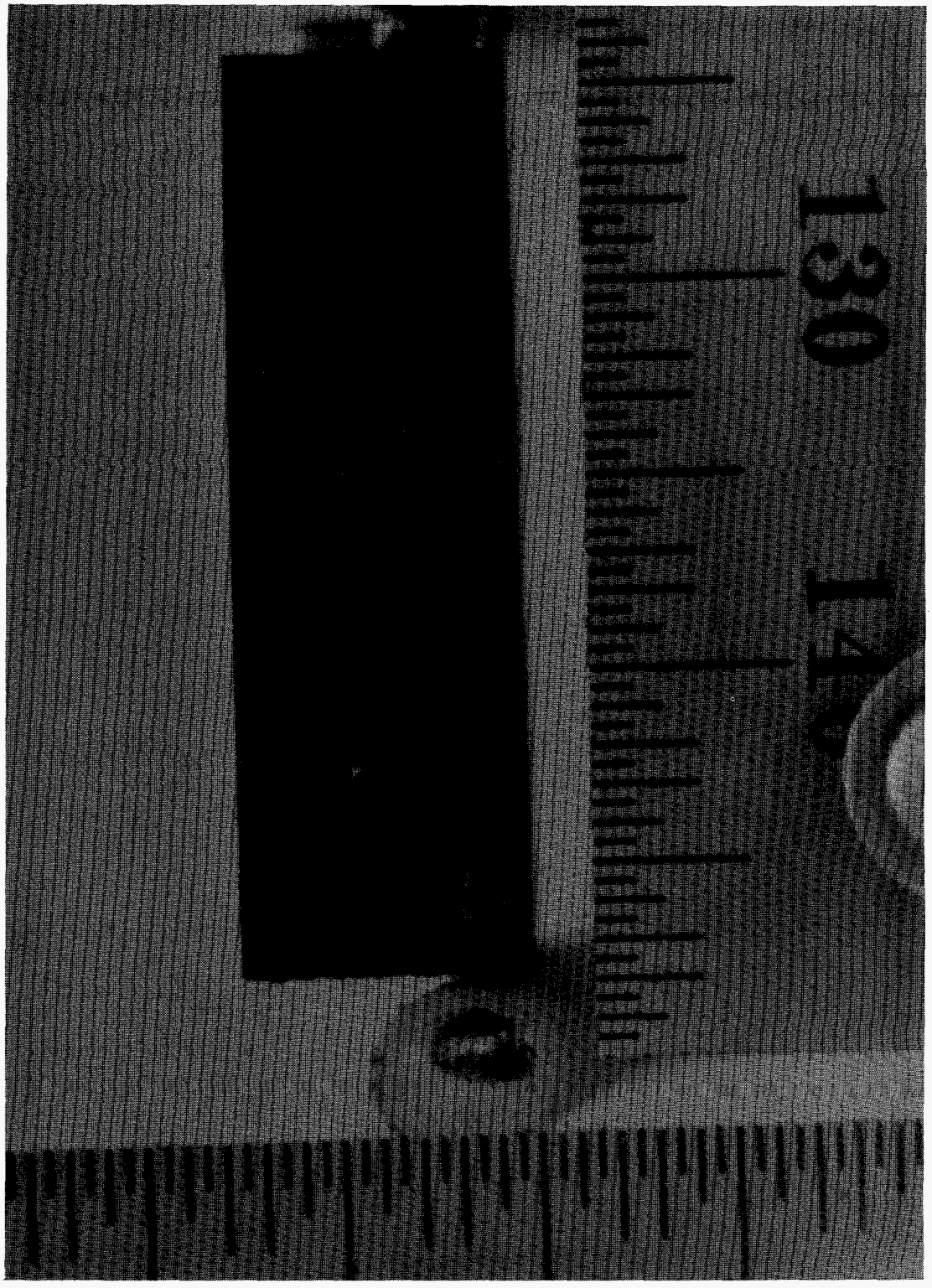


Figure B.9: Longitudinal Plane of Specimen SFEC5,4378-S2-E2. Each division of the attached scale is 0.5 mm. The PTL log is Q-1389, Photo # 201.

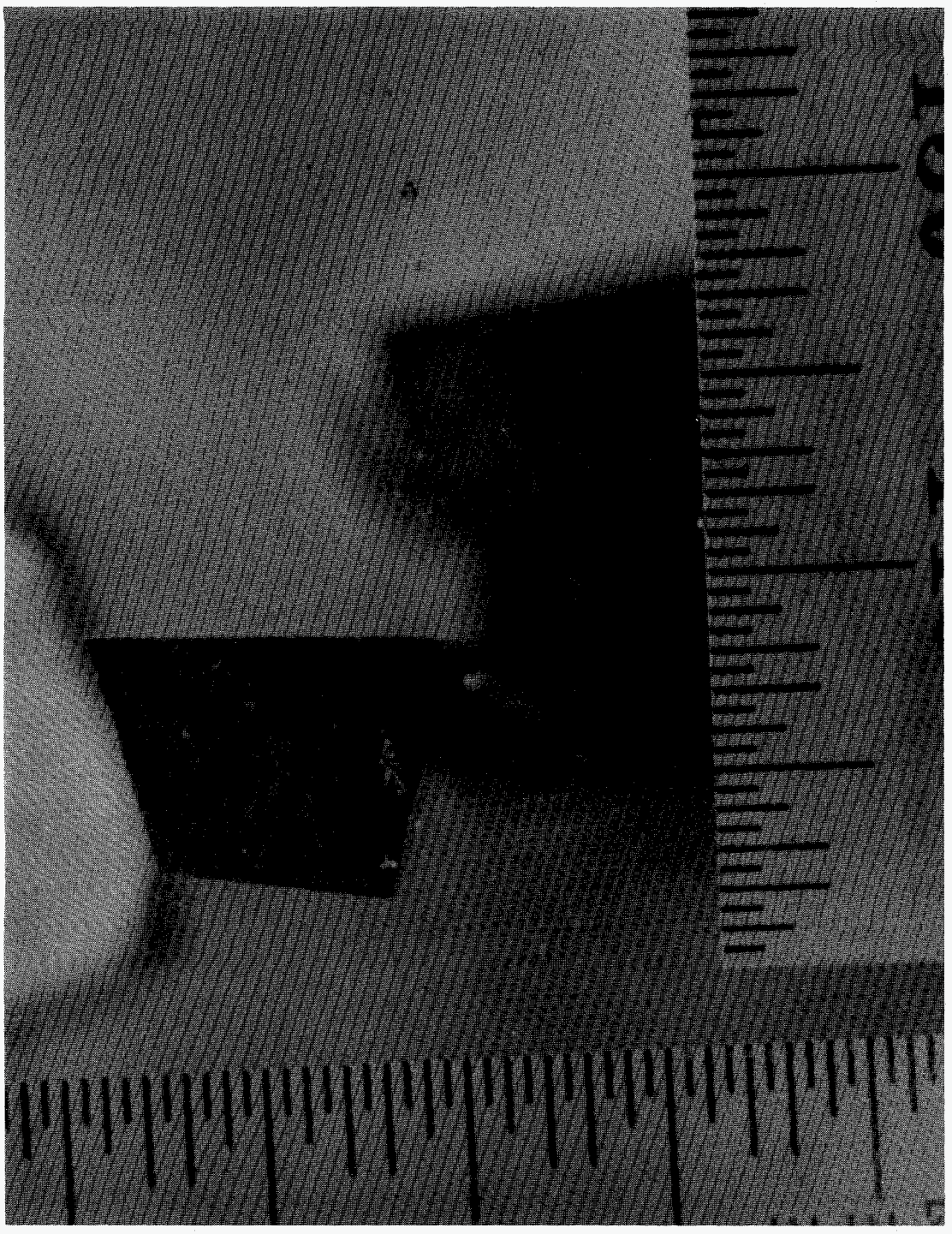


Figure B.10: End View of Specimen SFEC5,4378-S2-E2. Each division of the attached scale is 0.5 mm. The PTL log is Q-1390, Photo # 202.

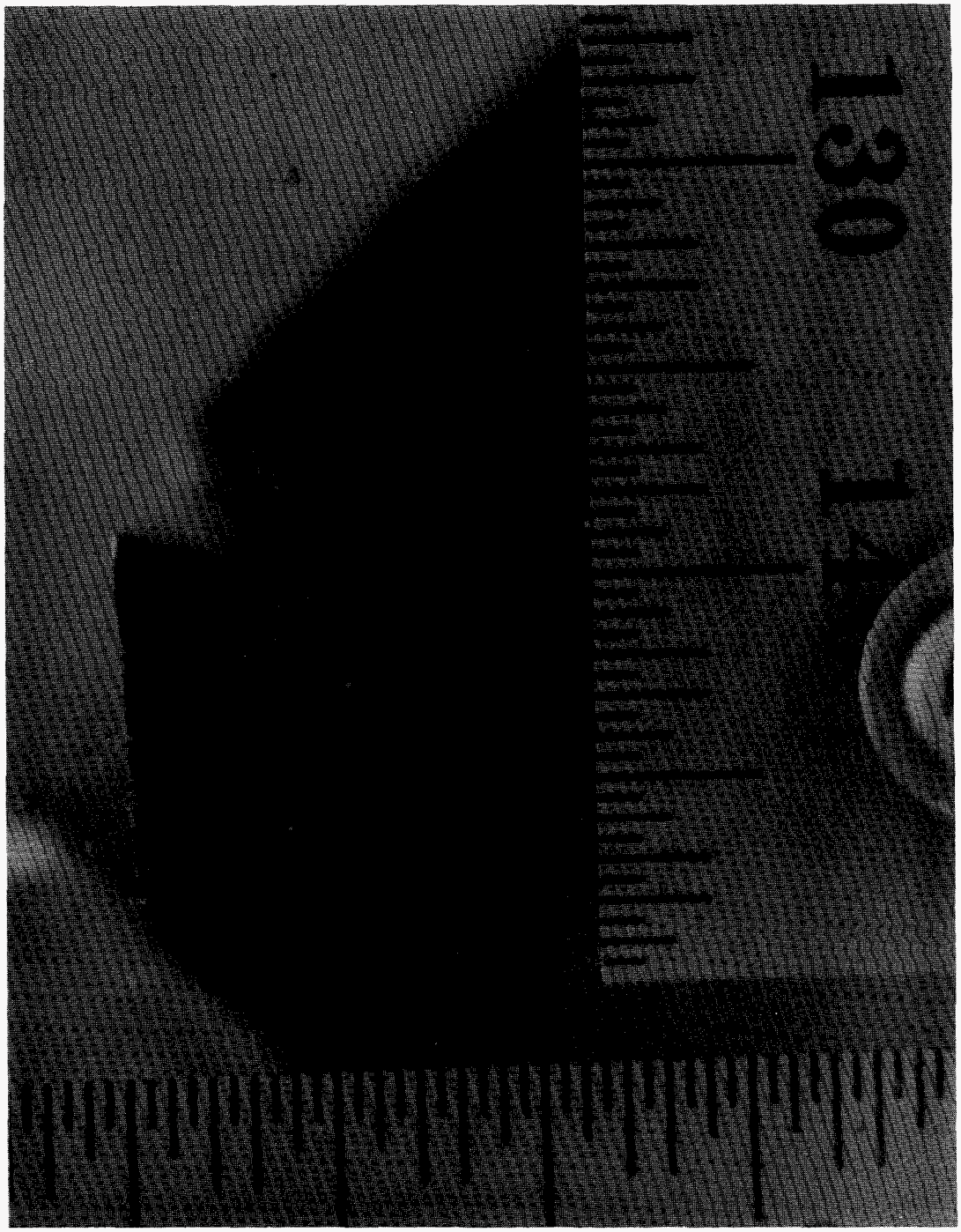


Figure B.11: Other End View of Specimen SFEC5,4378-S2-E2. Each division of the attached scale is 0.5 mm. The PTL log is Q-1391, Photo # 203.

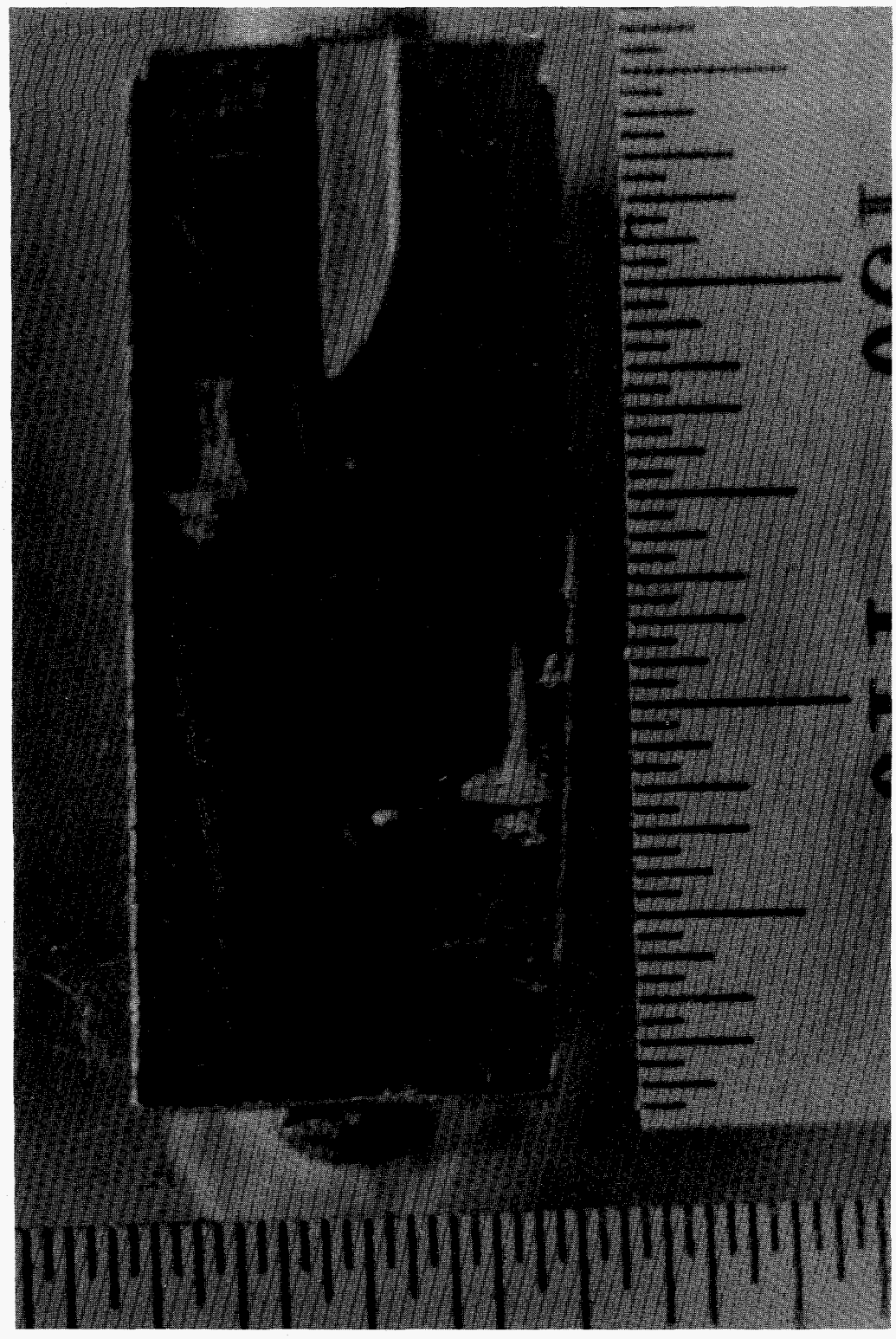


Figure B.12: Outer Surface View of Specimen SFEC5,4378-E1-CD1. Each division of the attached scale is 0.5 mm. The PTL log is Q-1475, Photo # 292.

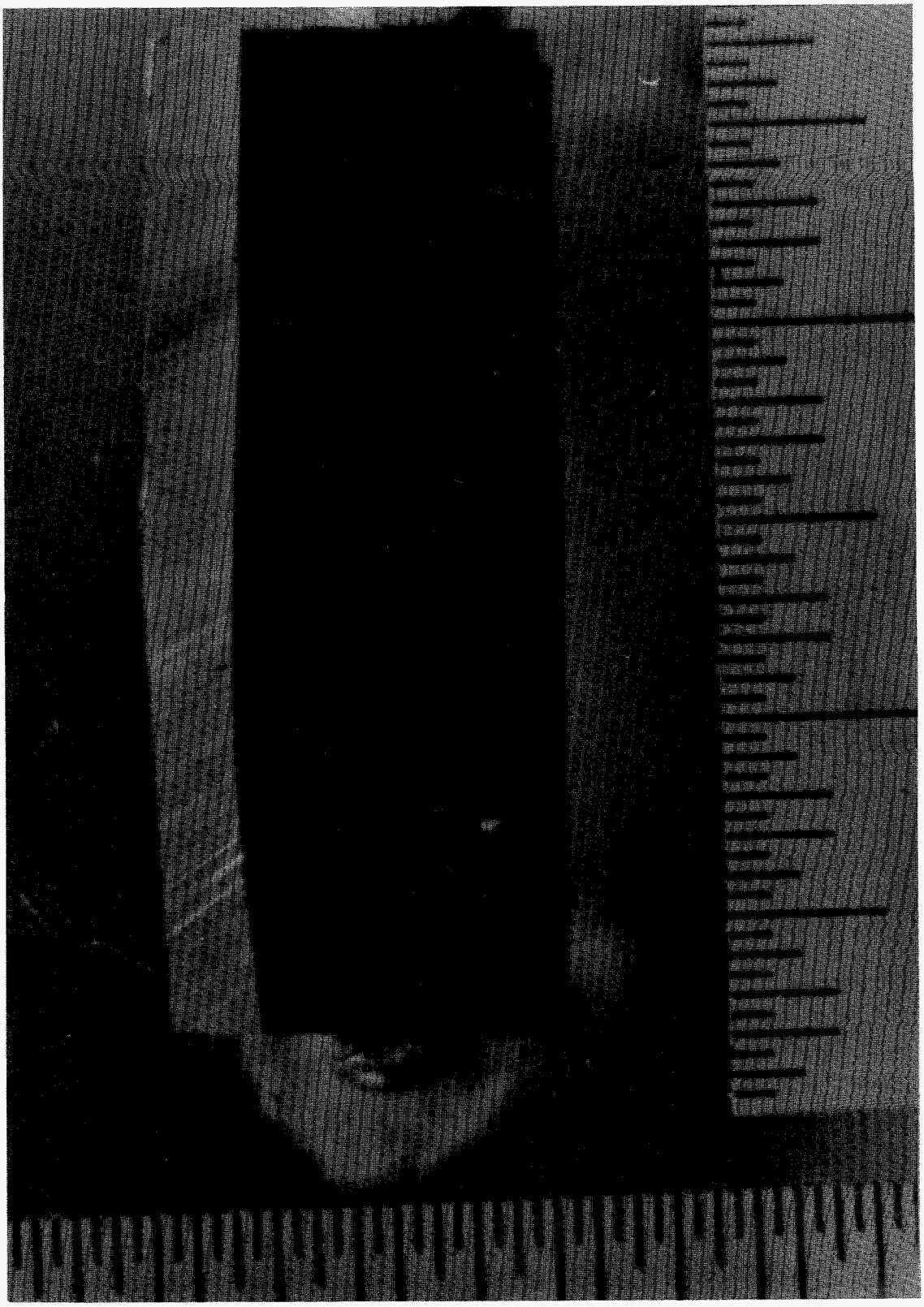


Figure B.13: Inner Surface View of Specimen SFEC5,4378-S2- E1-CD1. Each division of the attached scale is 0.5 mm. The PTL log is Q-1476, Photo # 293.

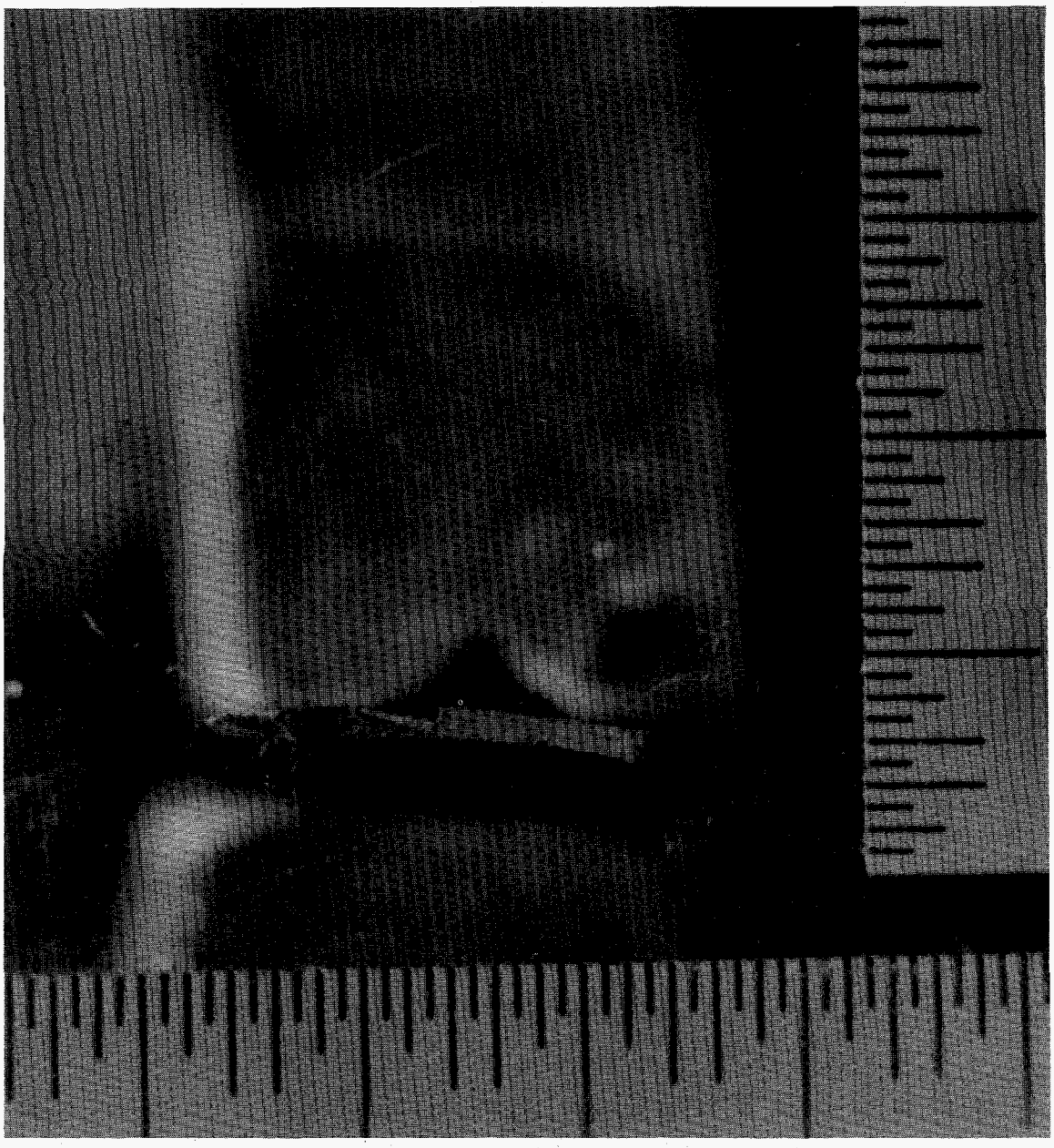


Figure B.14: Top End View of Specimen SFEC5,4378-S2-E1-CD1. Each division of the attached scale is 0.5 mm. The PTL log is Q-1477, Photo # 294.

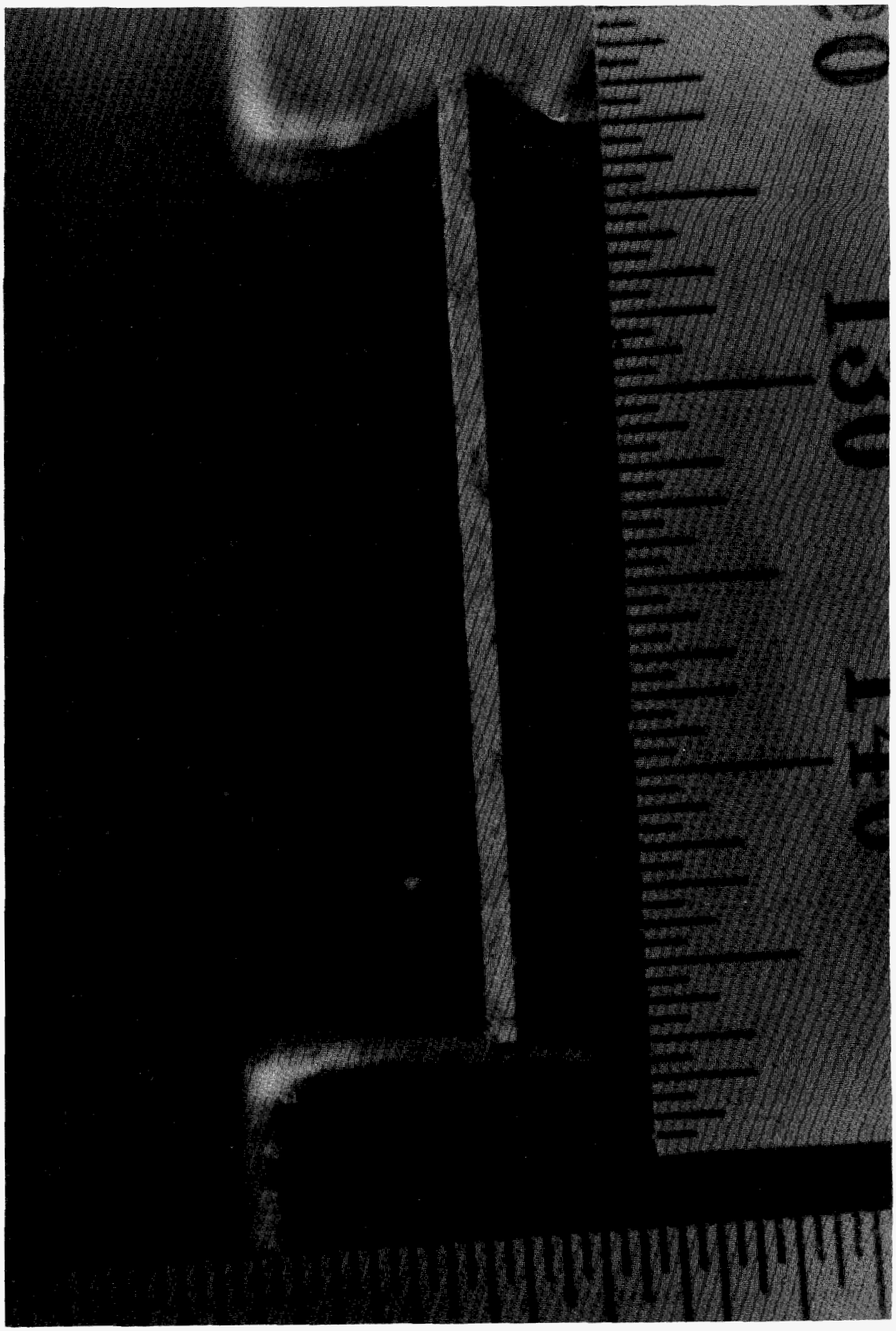


Figure B.15: Side View of Specimen SFEC5,4378-S2-E1-CD1. Each division of the attached scale is 0.5 mm. The PTL log is Q-1478, Photo # 295.

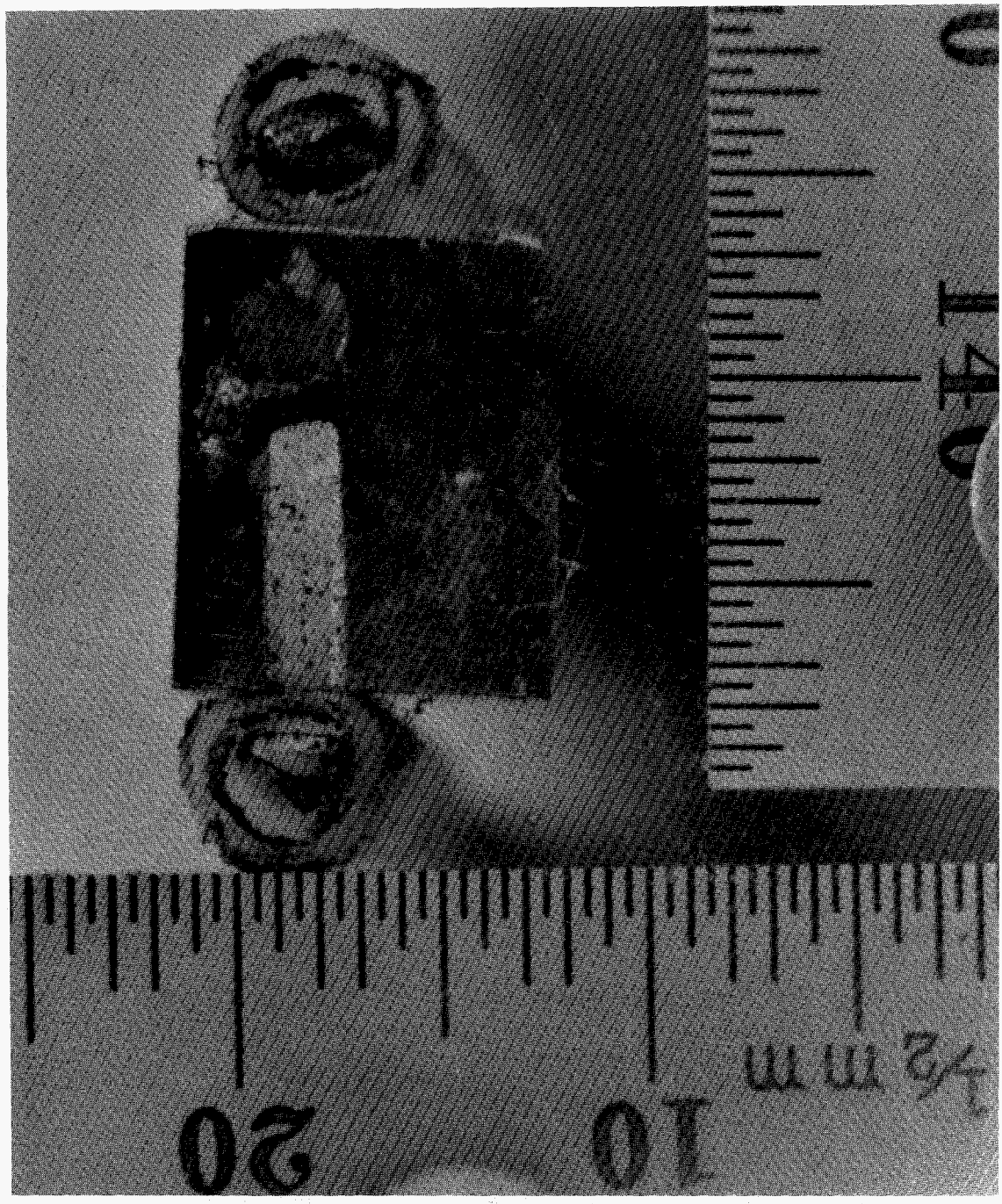


Figure B.16: Outer Cladding Surface of Specimen SFEC5,4378-S2-E3A. Each division of the attached scale is 0.5 mm. The PTL log is Q-1399, Photo # 216.

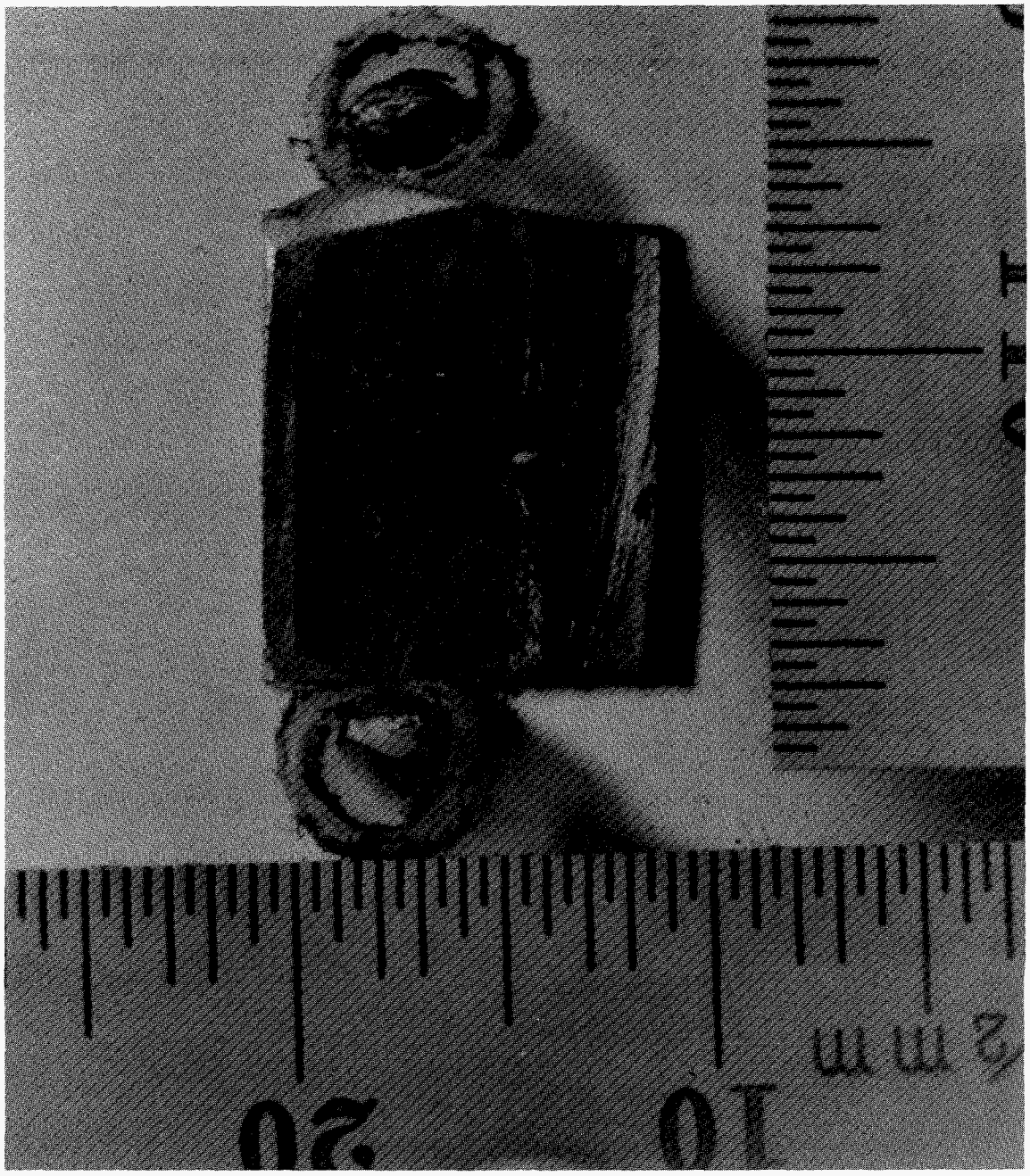


Figure B.17: Longitudinal Surface of Specimen SFEC5,4378-S2-E3A. Each division of the attached scale is 0.5 mm. The PTL log is Q-1400, Photo # 217.

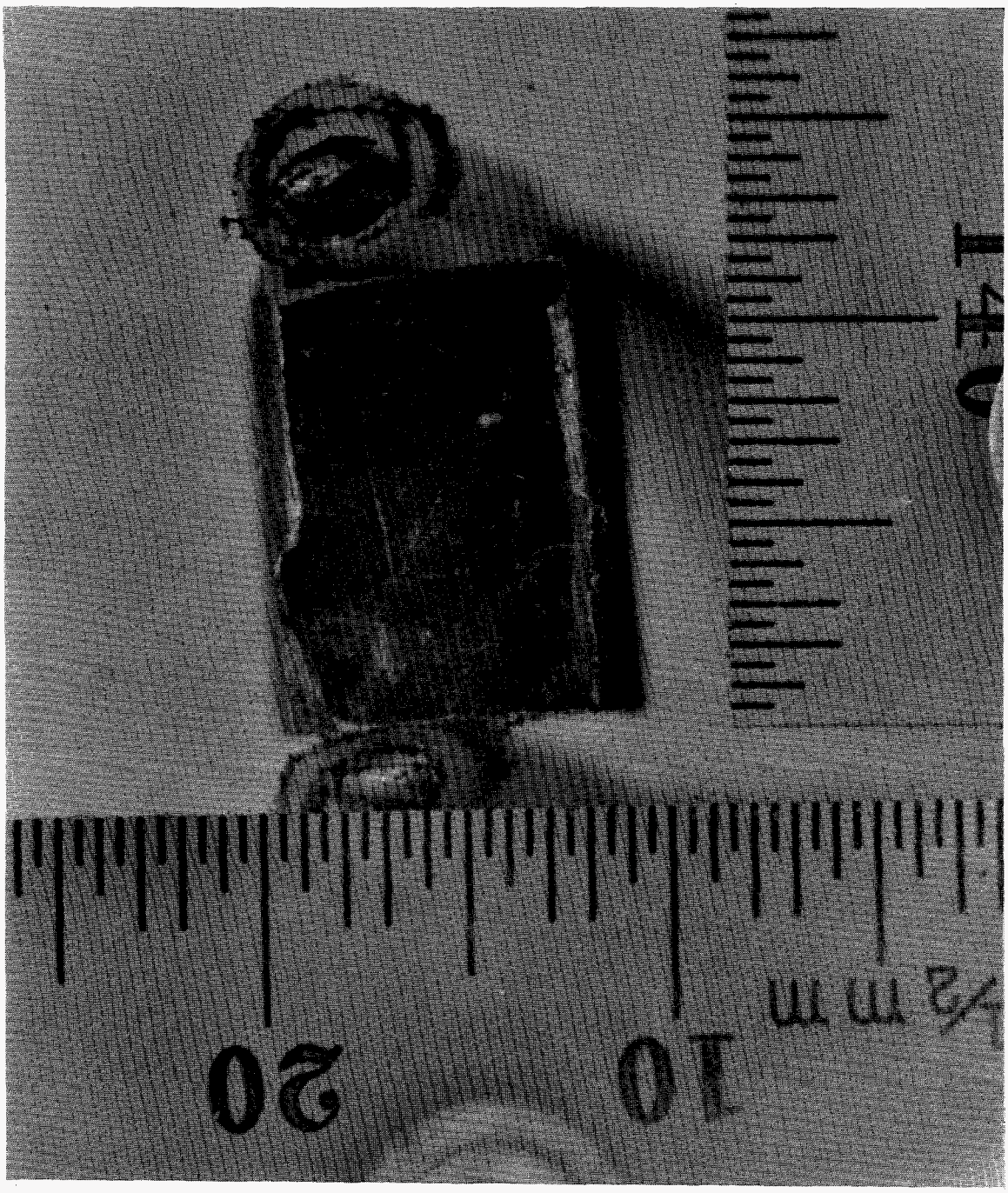


Figure B.18: Longitudinal Surface of Specimen SFEC5,4378-S2-E3A. Each division of the attached scale is 0.5 mm. The PTL log is Q-1401, Photo # 218.

Figure B.19: Longitudinal Surface of Specimen SFEC5,4378-S2-E3A. Each division of the attached scale is 0.5 mm. The PTL log is Q-1402, Photo # 219.

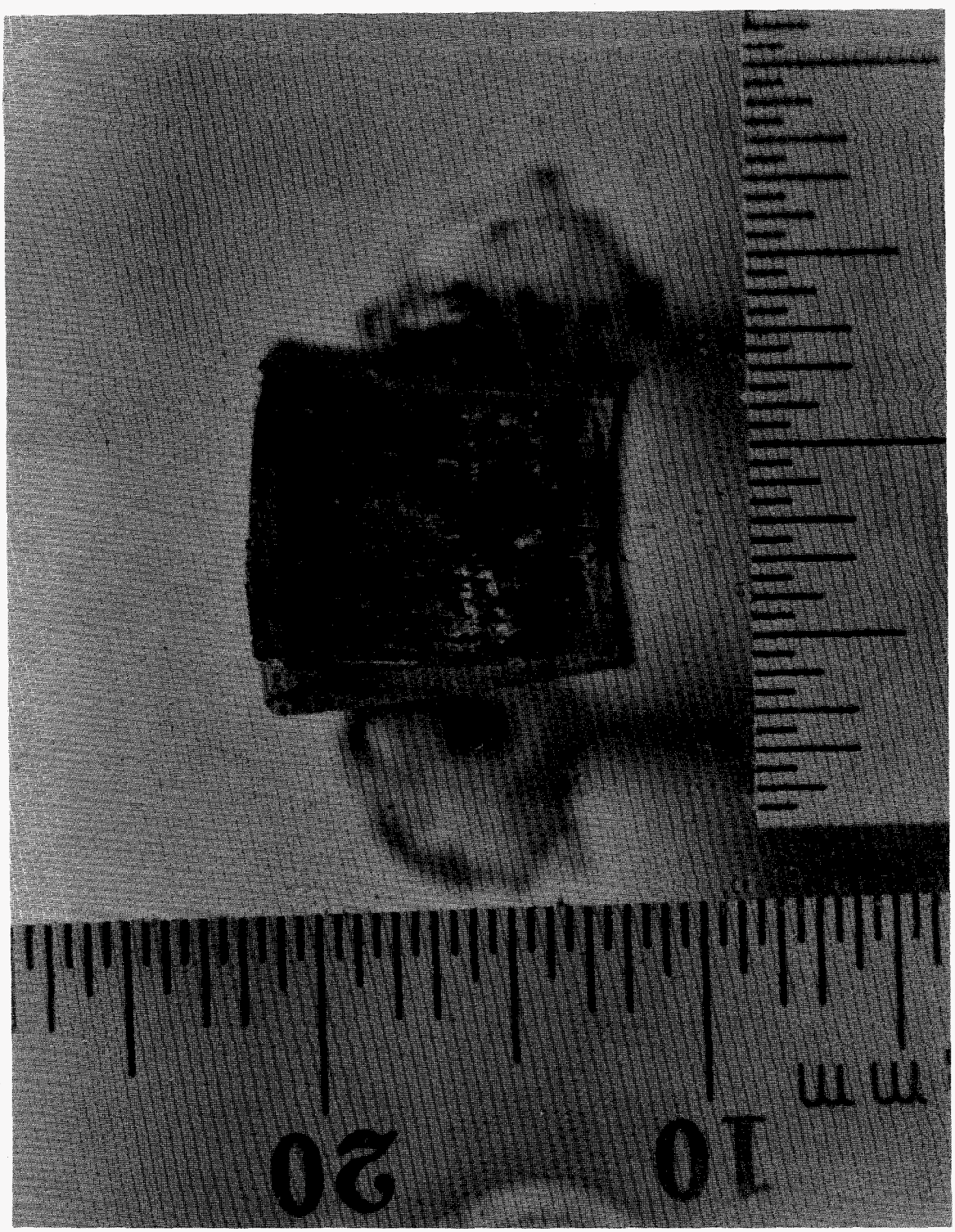


Figure B.20: View of the Bottom Plane of Specimen SFEC5,4378-S2-E3A. Each division of the attached scale is 0.5 mm. The PTL log is Q-1403, Photo # 220.

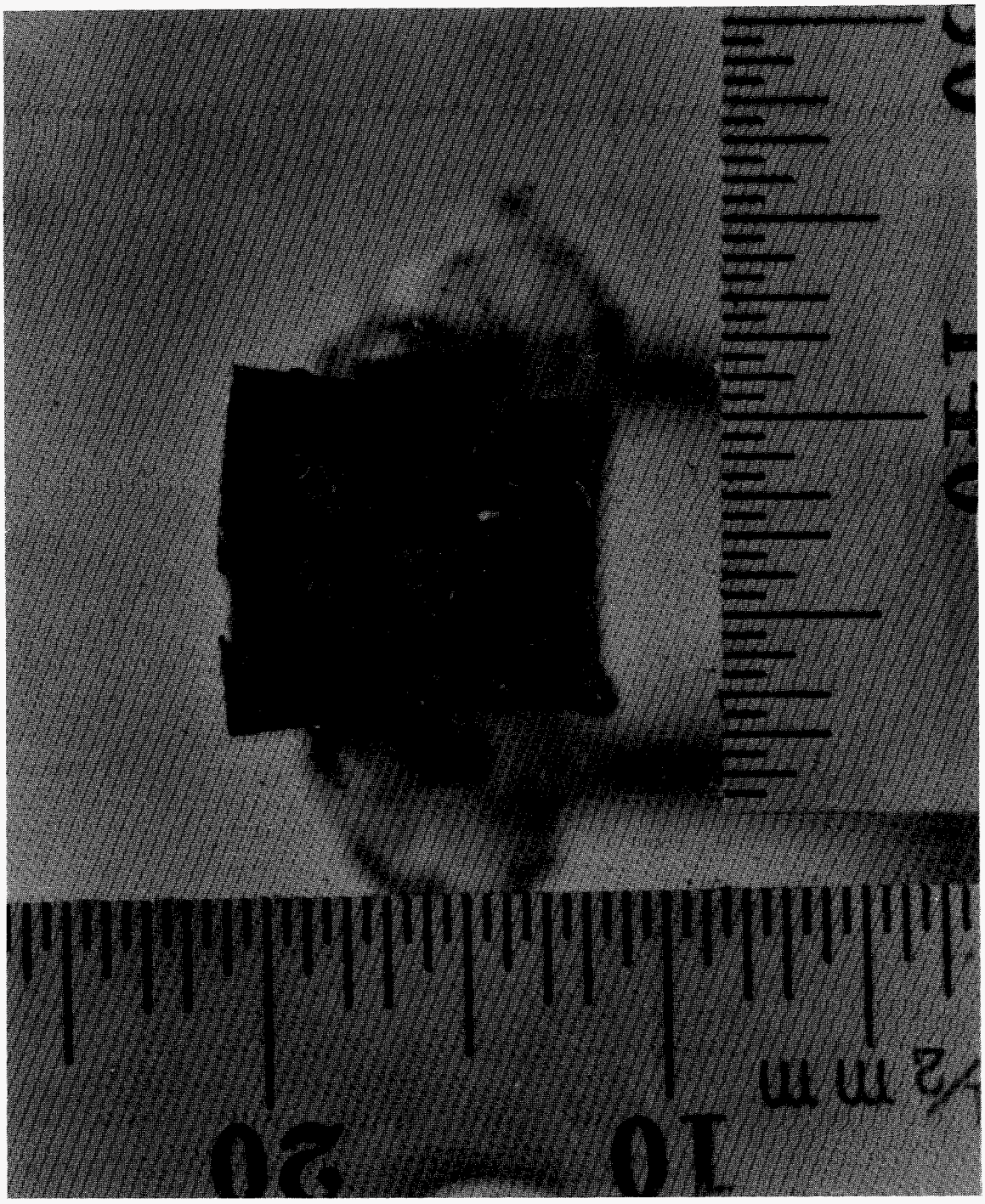


Figure B.21: View of the Top Plane of Specimen SFEC5,4378-S2-E3A. Each division of the attached scale is 0.5 mm. The PTL log is Q-1404, Photo # 221.

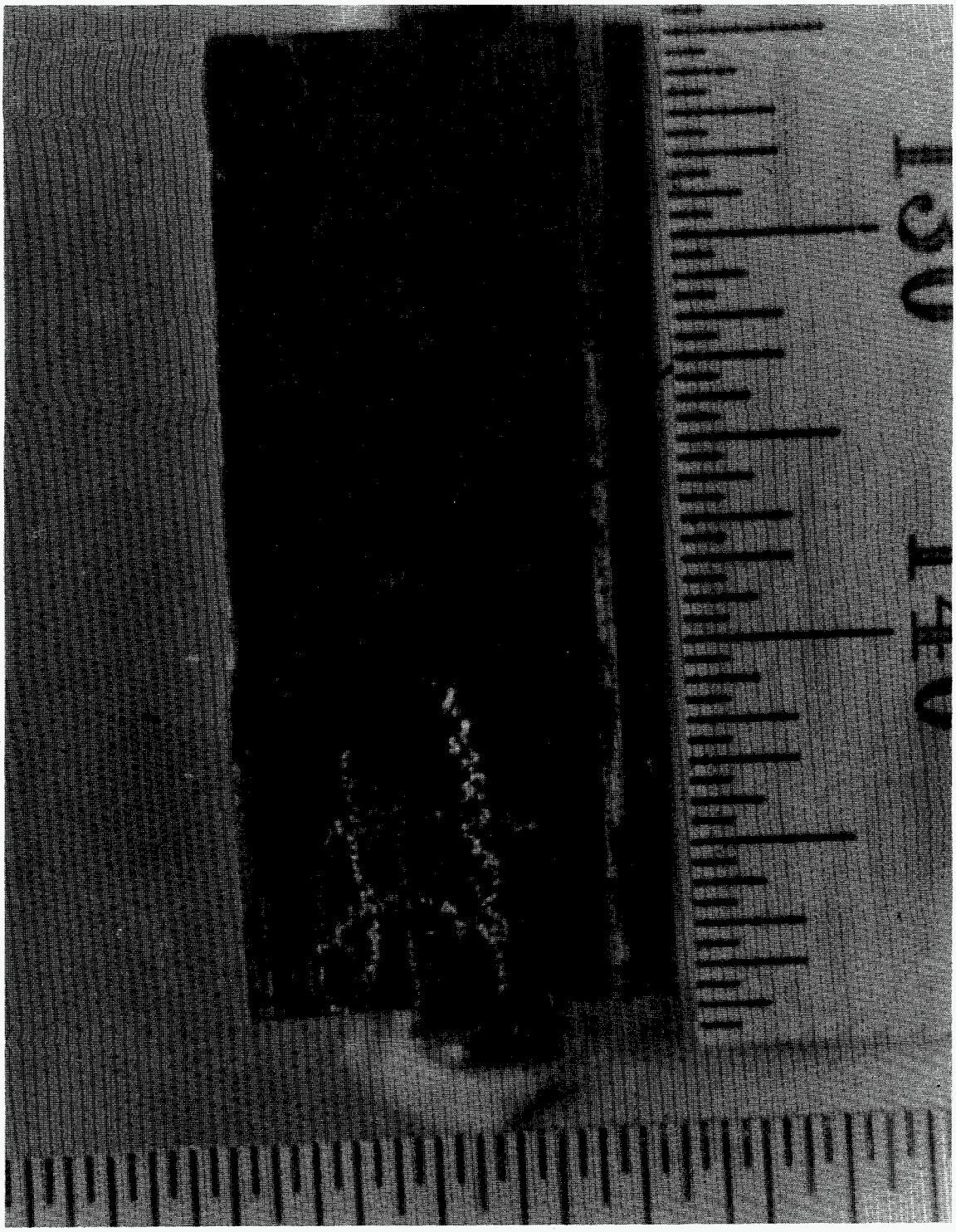


Figure B.22: Longitudinal Surface of Conditioned Specimen SFEC5,4378-S2-H. Each division of the attached scale is 0.5 mm. PTL log is Q-1513, Photo # 331.

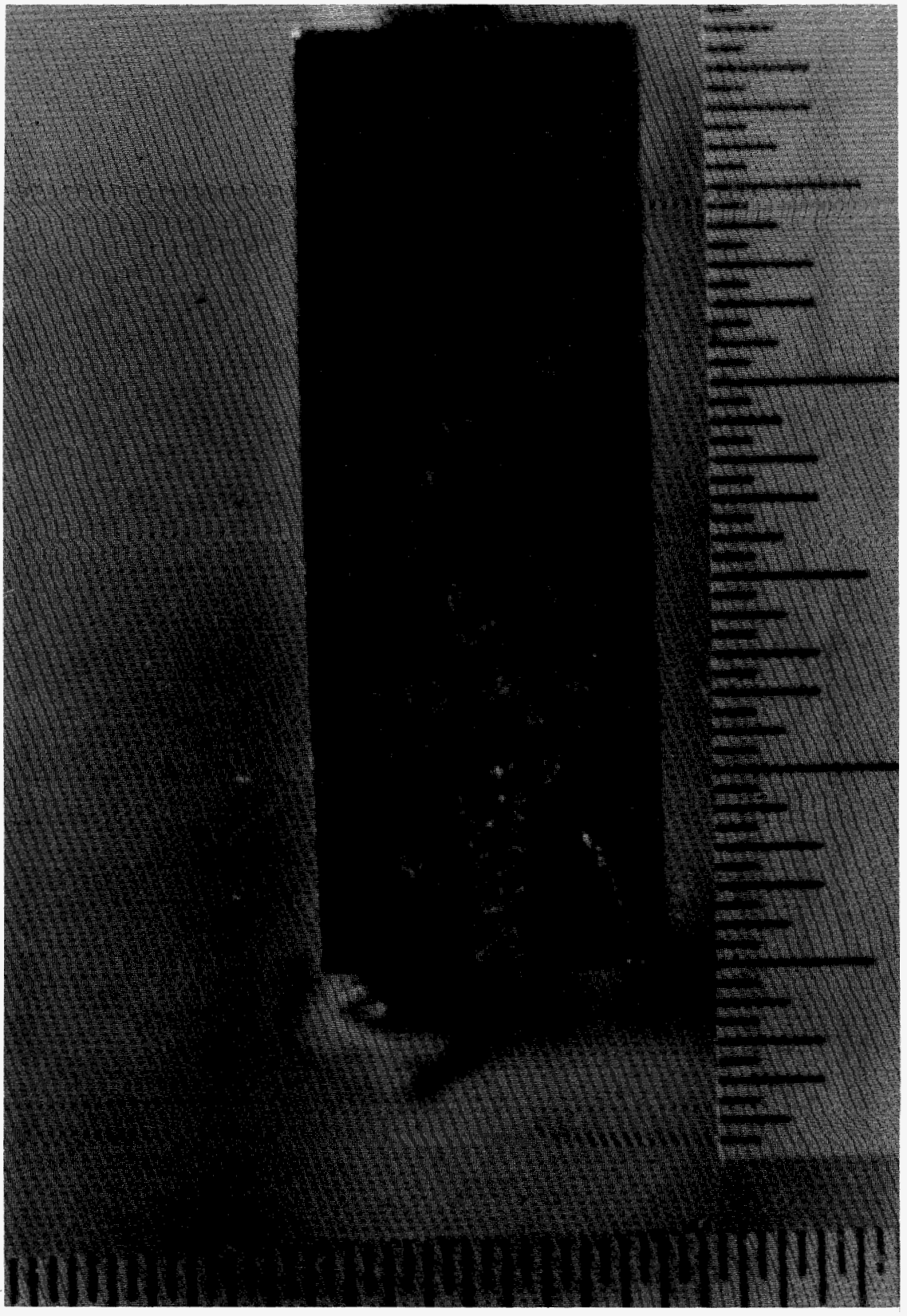


Figure B.23: Longitudinal Surface of Conditioned Specimen SFEC5,4378-S2-H. Each division of the attached scale is 0.5 mm. PTL log is Q-1514, Photo # 332.

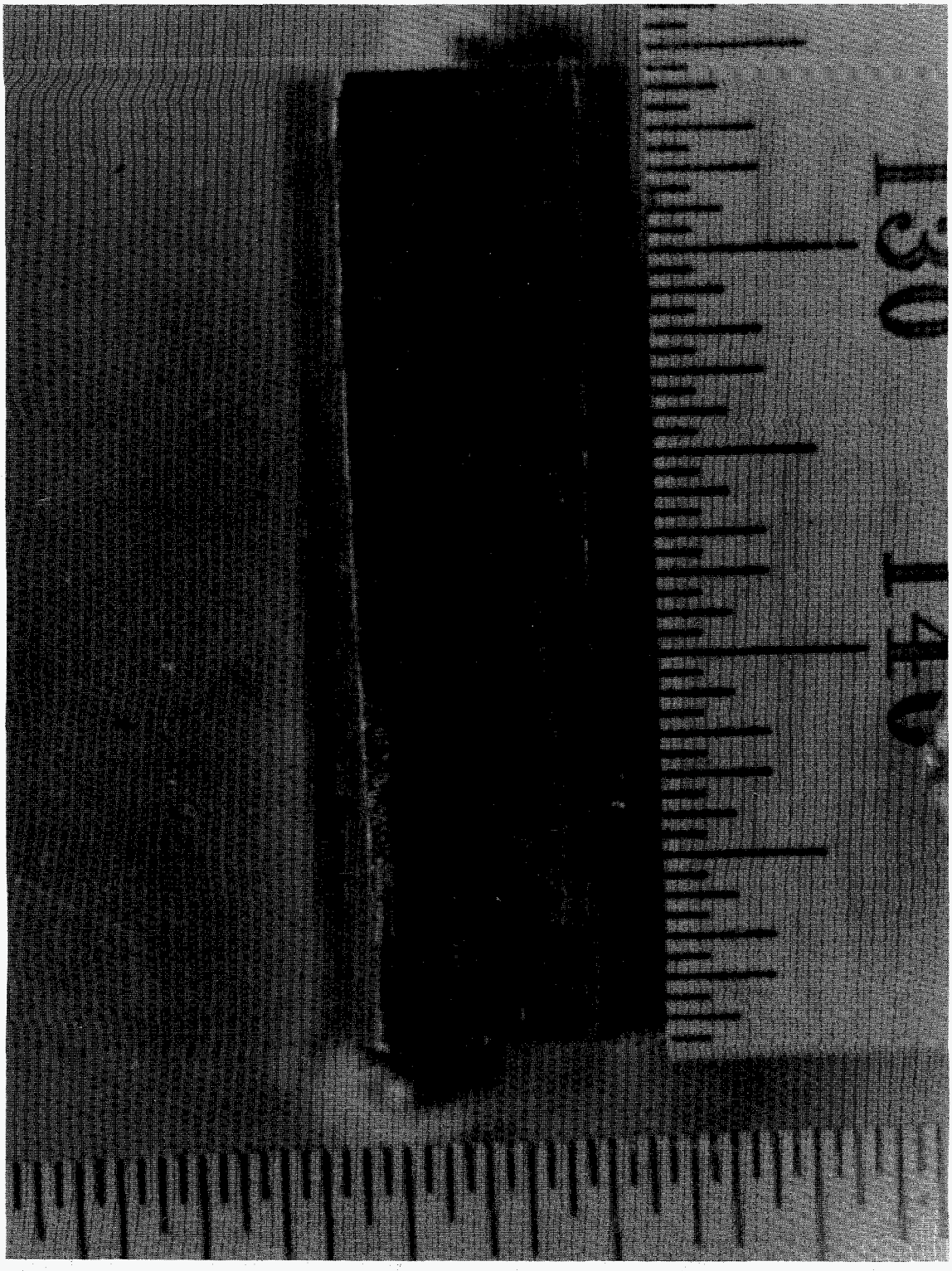


Figure B.24: Longitudinal Surface of Conditioned Specimen SFEC5,4378-S2-H. Each division of the attached scale is 0.5 mm. PTL log is Q-1515, Photo # 333.

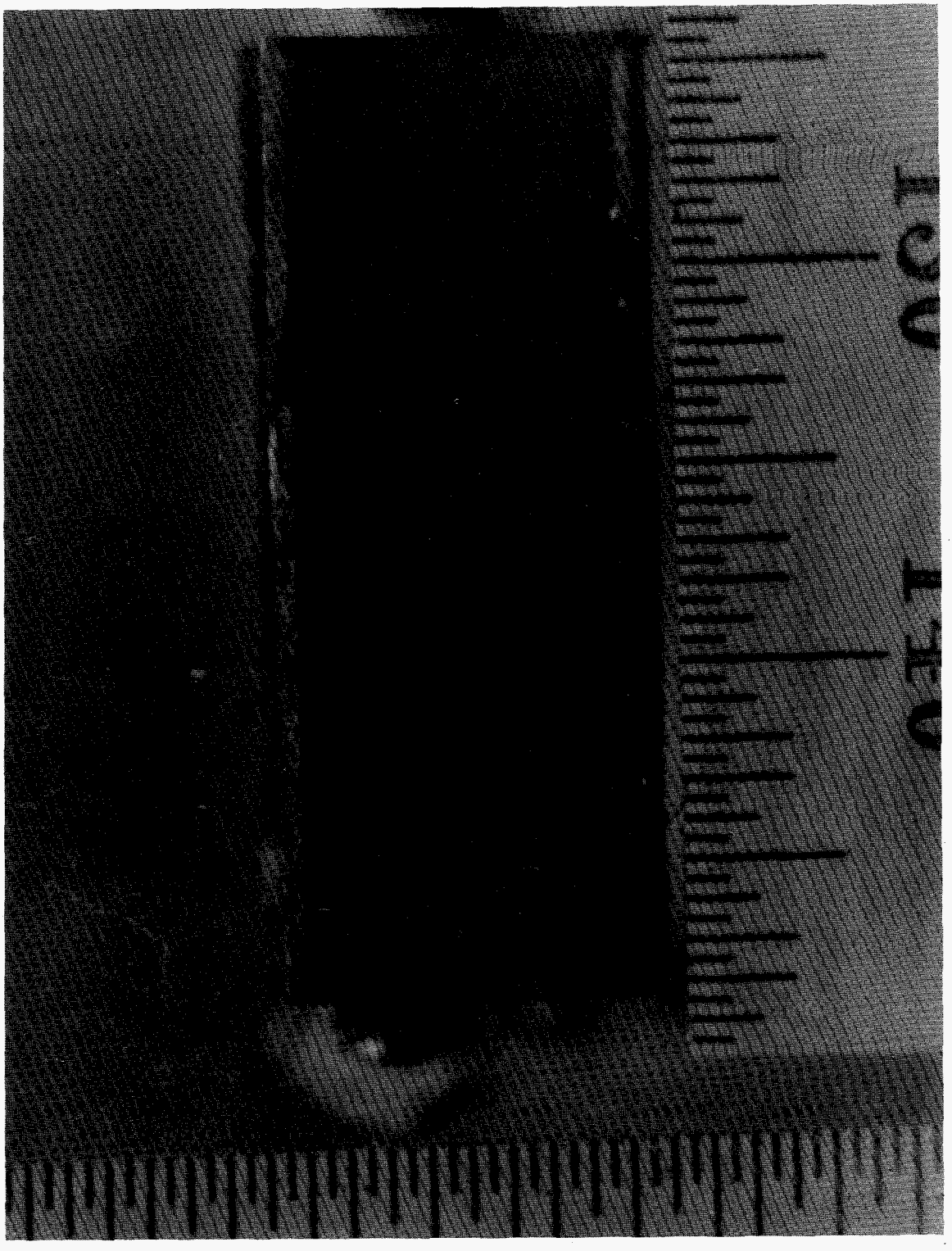


Figure B.25: Longitudinal Surface of Conditioned Specimen SFEC5,4378-S2-H. Each division of the attached scale is 0.5 mm. PTL log is Q-1516, Photo # 334.

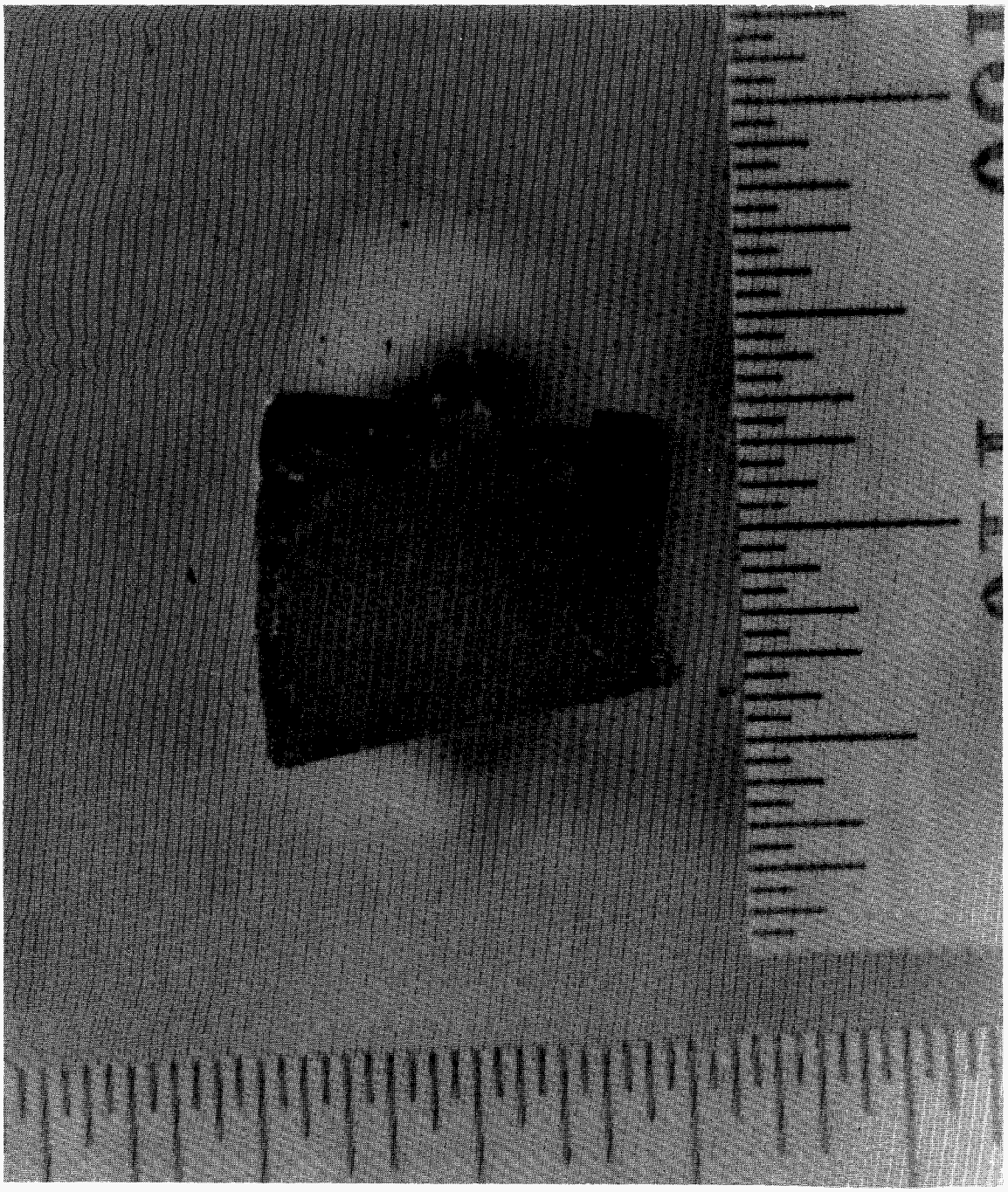


Figure B.26: Bottom Plane of Conditioned Specimen SFEC5,4378-S2-H. Each division of the attached scale is 0.5 mm. The PTL log is Q-1517, Photo # 335.

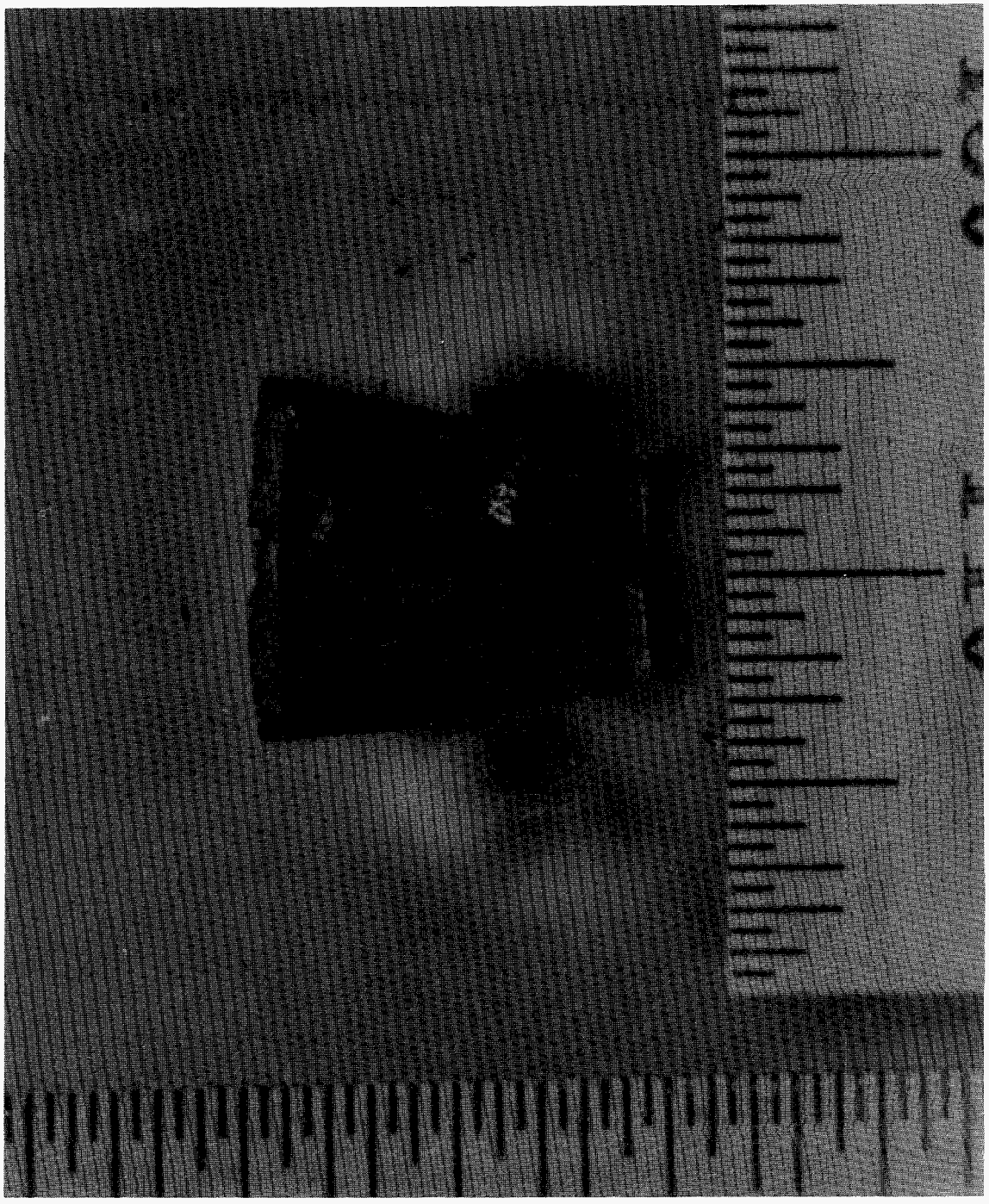


Figure B.27: Top Plane of Conditioned Specimen SFEC5,4378-S2-H. Each division of the attached scale is 0.5 mm. The PTL log is Q-1518, Photo # 336.

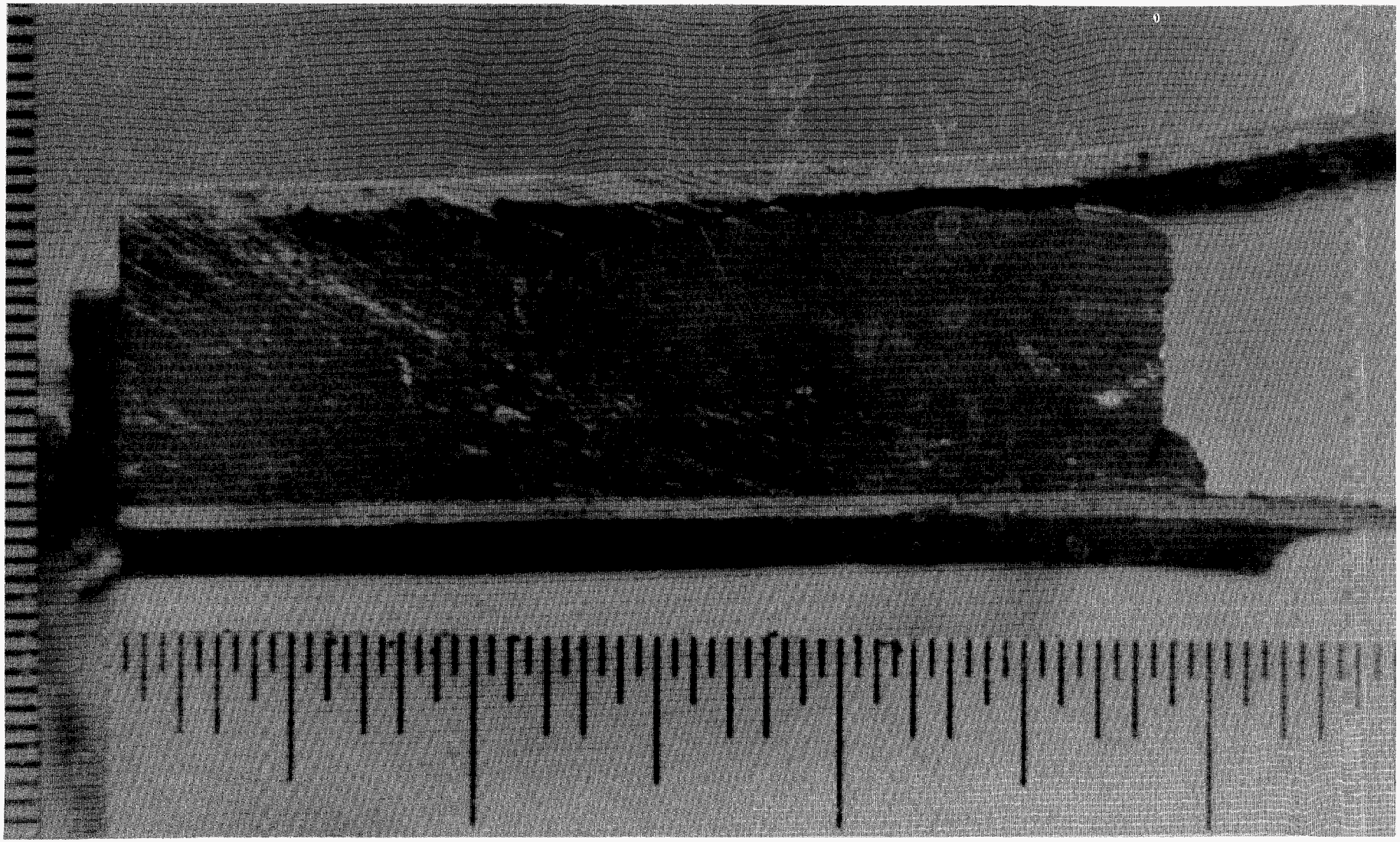


Figure B.28: Longitudinal Surface of As-Cut Specimen SFEC5,4378-S1A-D. Each division of the attached scale is 0.5 mm. PTL log is Q-1605, Photo # 422.

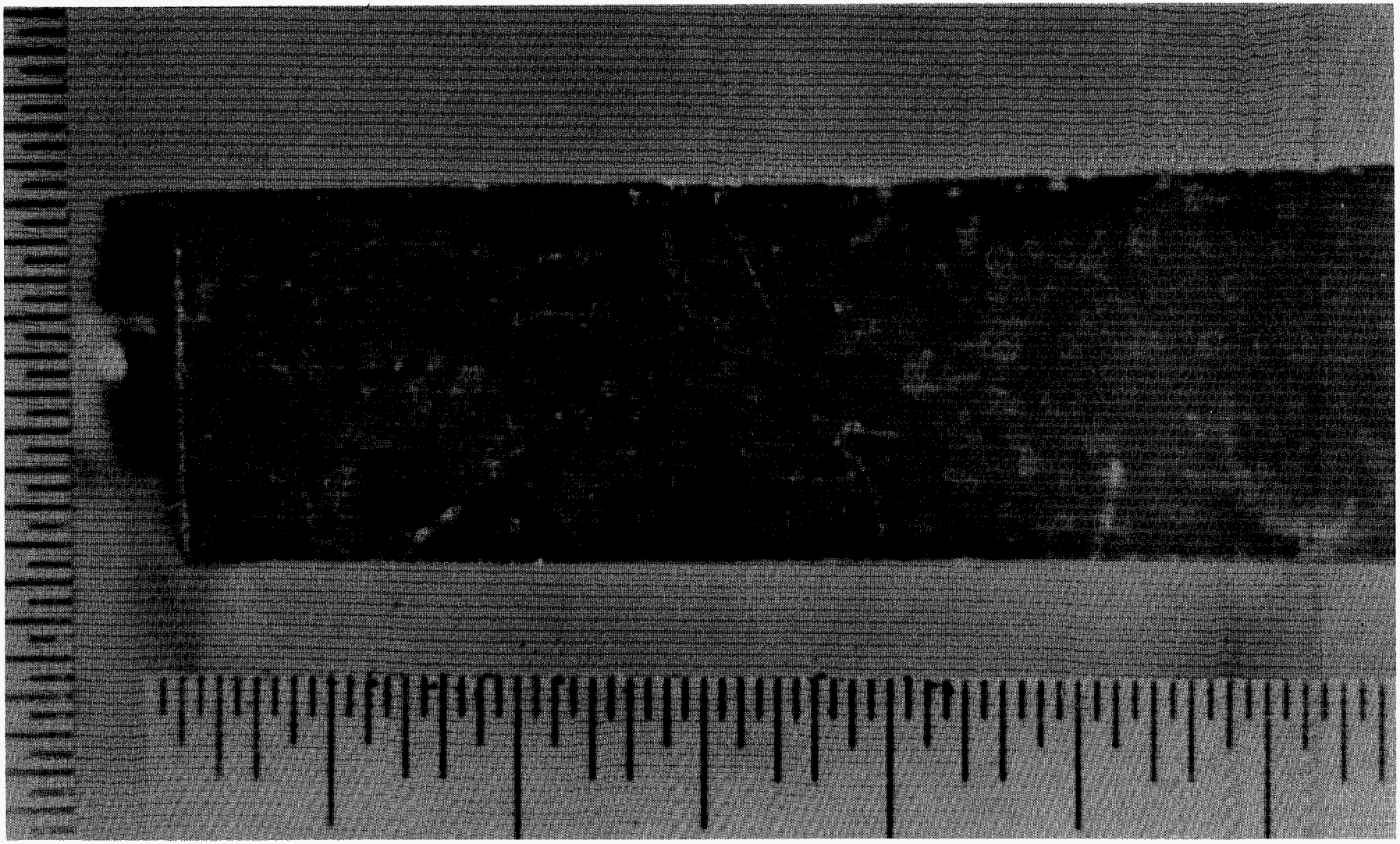


Figure B.29: Outside Cladding Surface of As-Cut Specimen SFEC5,4378-S1A-D. Each division of the attached scale is 0.5 mm. PTL log is Q-1606, Photo # 423.

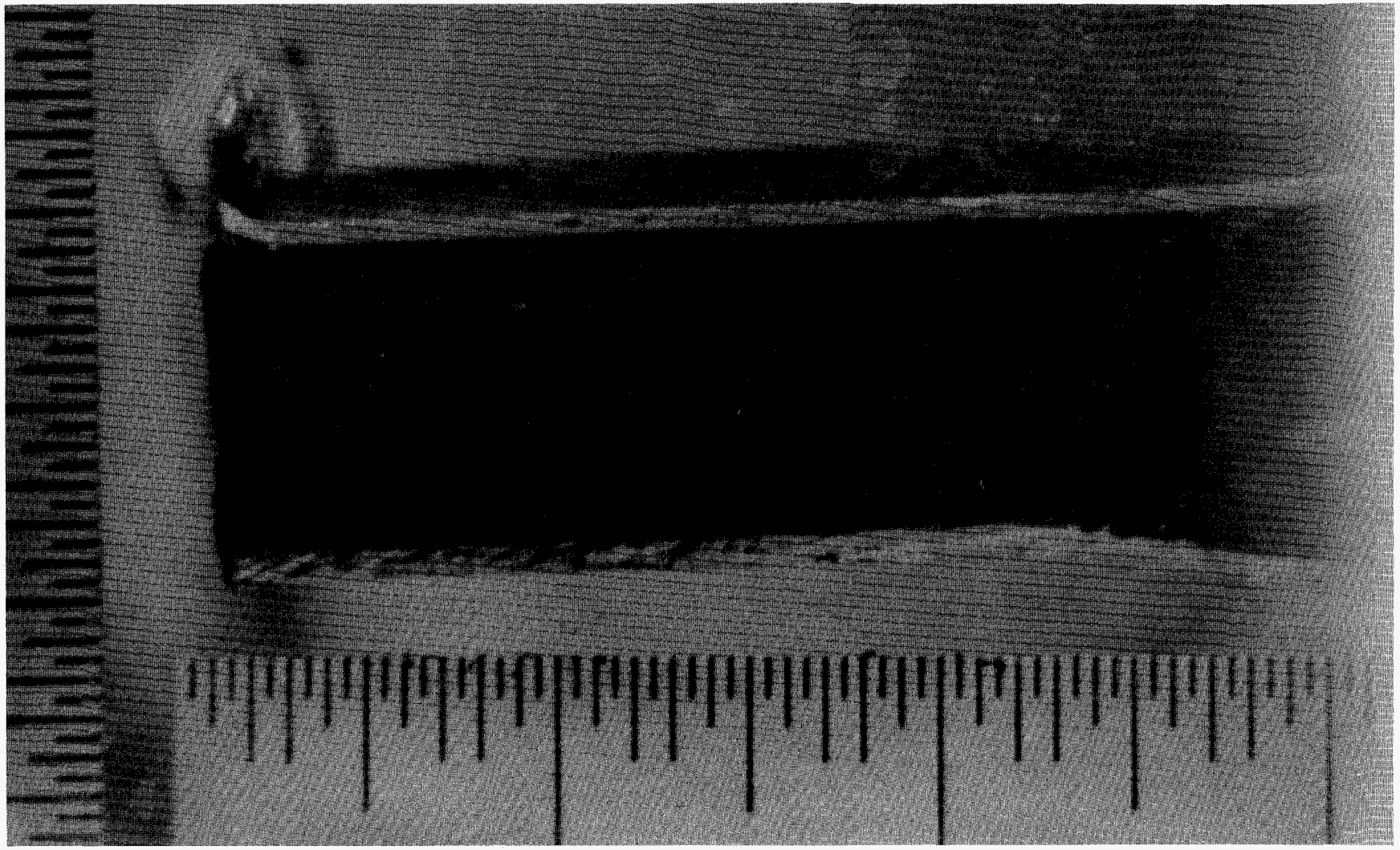


Figure B.30: The Other Longitudinal Surface of As-Cut Specimen SFEC5,4378-S1A-D. Each division of the attached scale is 0.5 mm. PTL log is Q-1607, Photo # 424.

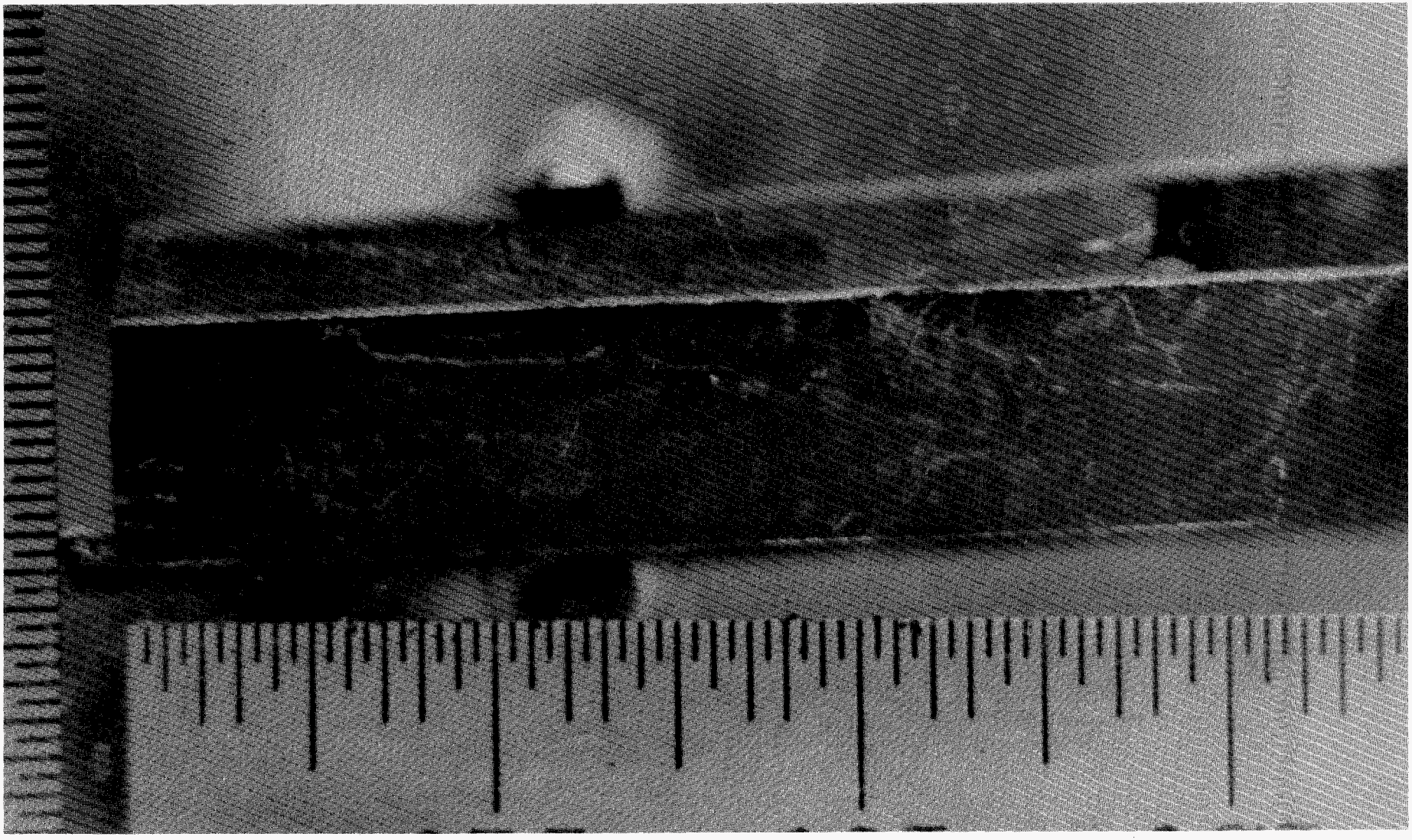


Figure B.31: Inner Cladding Surface of Specimen SFEC5,4378-S1A-D. Each division of the attached scale is 0.5 mm. PTL log is Q-1608, Photo # 425.

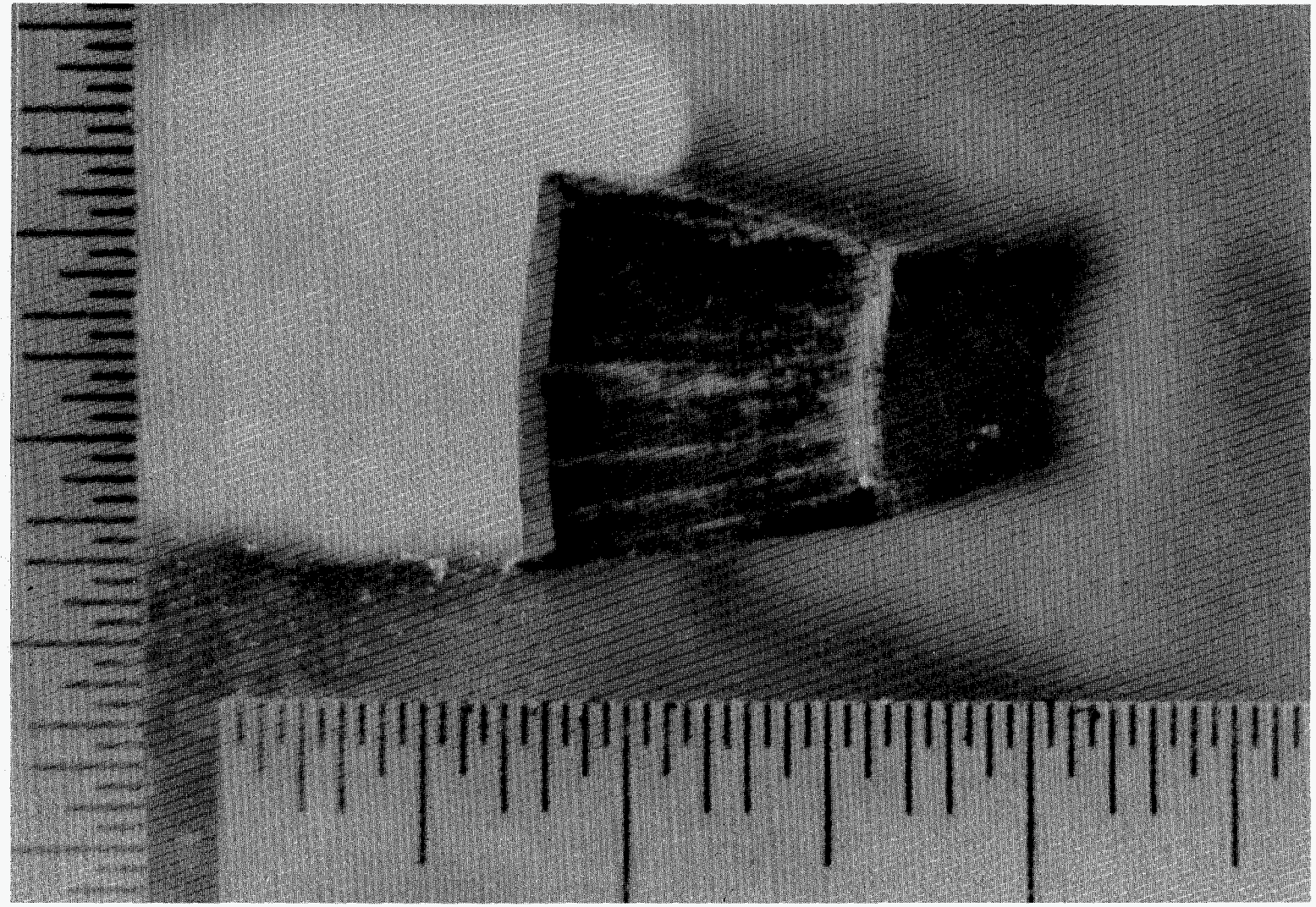


Figure B.32: Bottom Plane of As-Cut Specimen SFEC5,4378-S1A-D. Each division of the attached scale is 0.5 mm. The PTL log is Q-1609, Photo # 426.

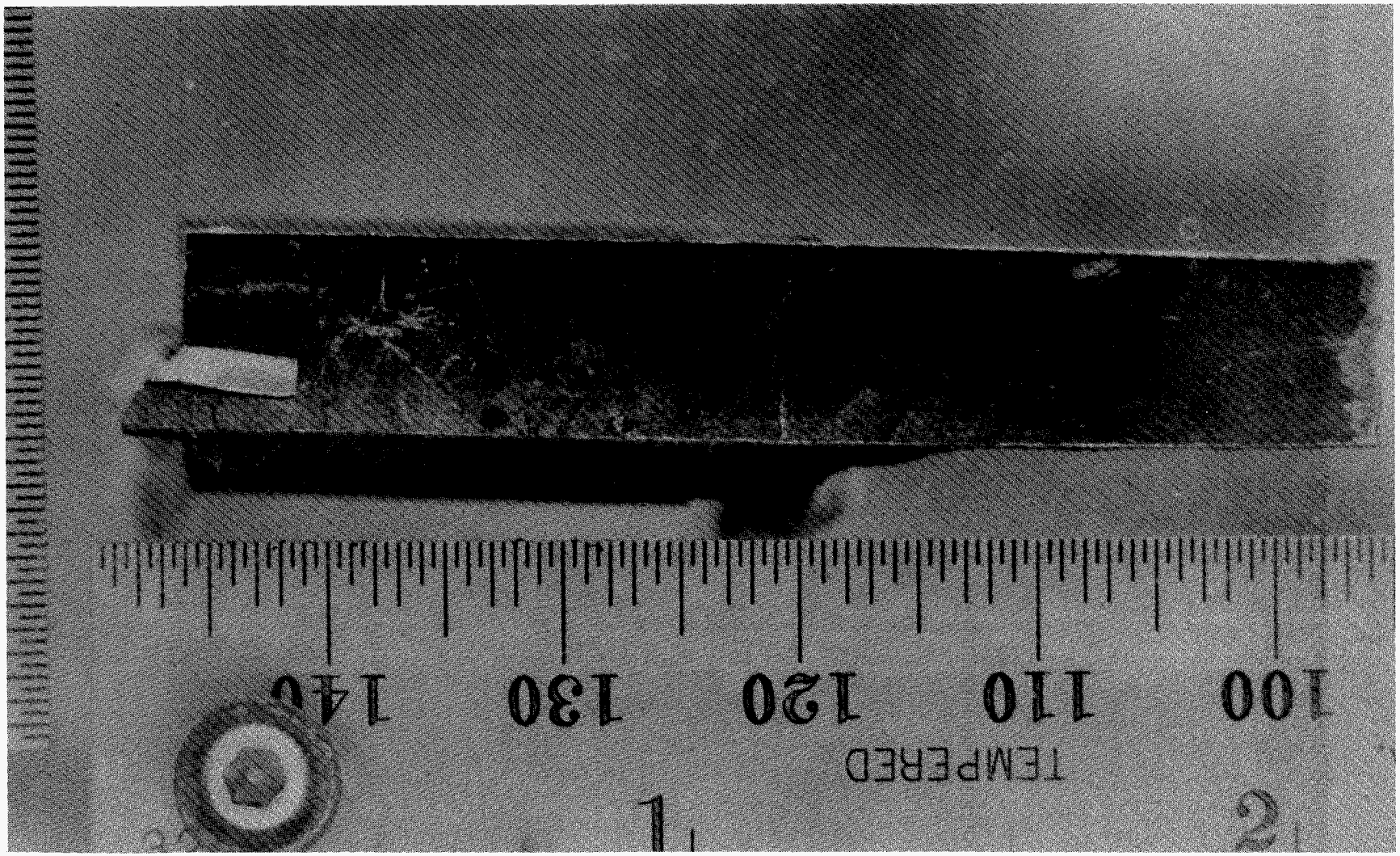


Figure B.33: Inner Cladding Surface of As-Cut Specimen SFEC5,4378-S1A-H. Each division of the attached scale is 0.5 mm. The PTL log is Q-1623, Photo # 440.

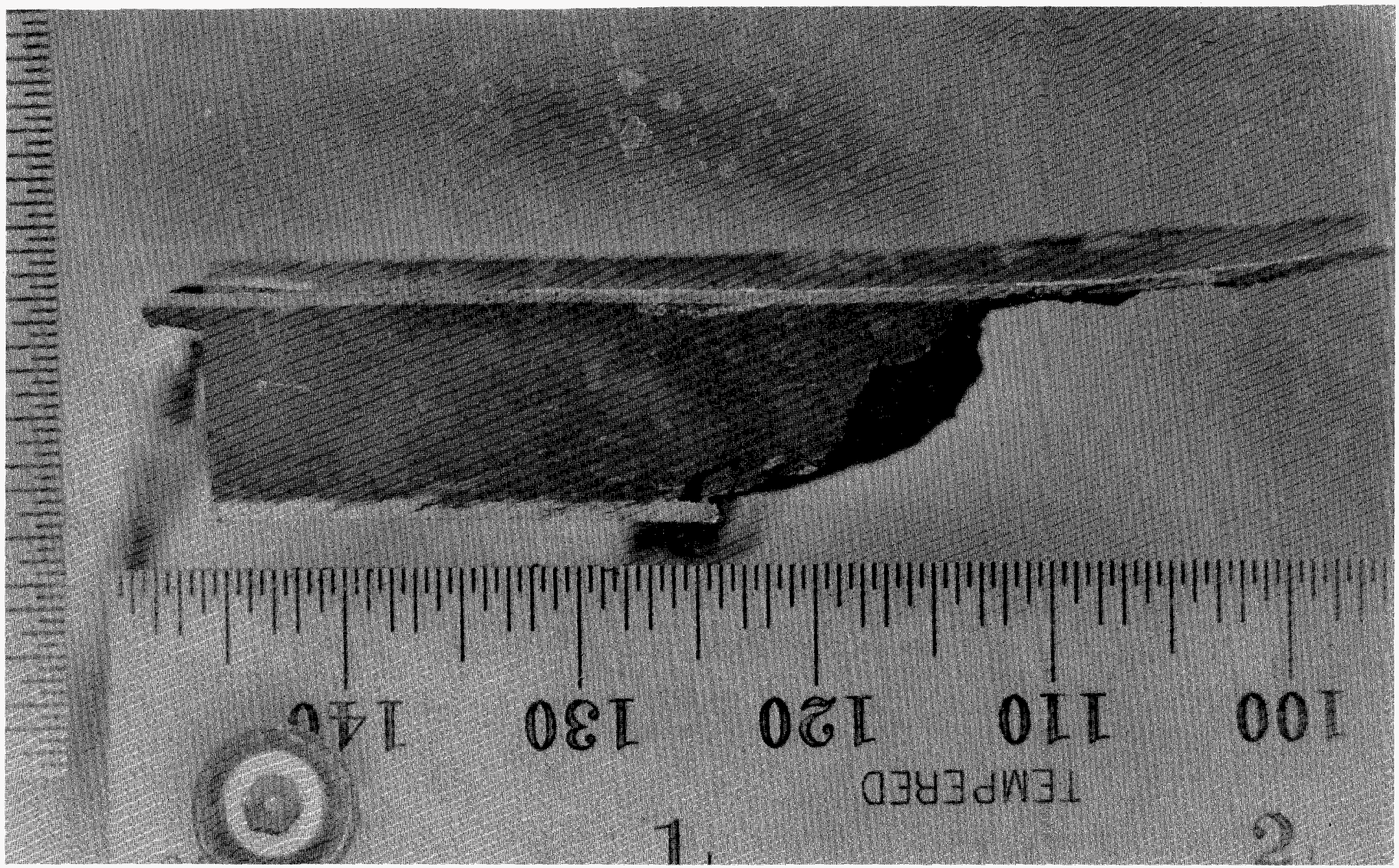


Figure B.34: Longitudinal Surface of As-Cut Specimen SFEC5,4378-S1A-H. Each division of the attached scale is 0.5 mm. PTL log is Q-1624, Photo # 441.

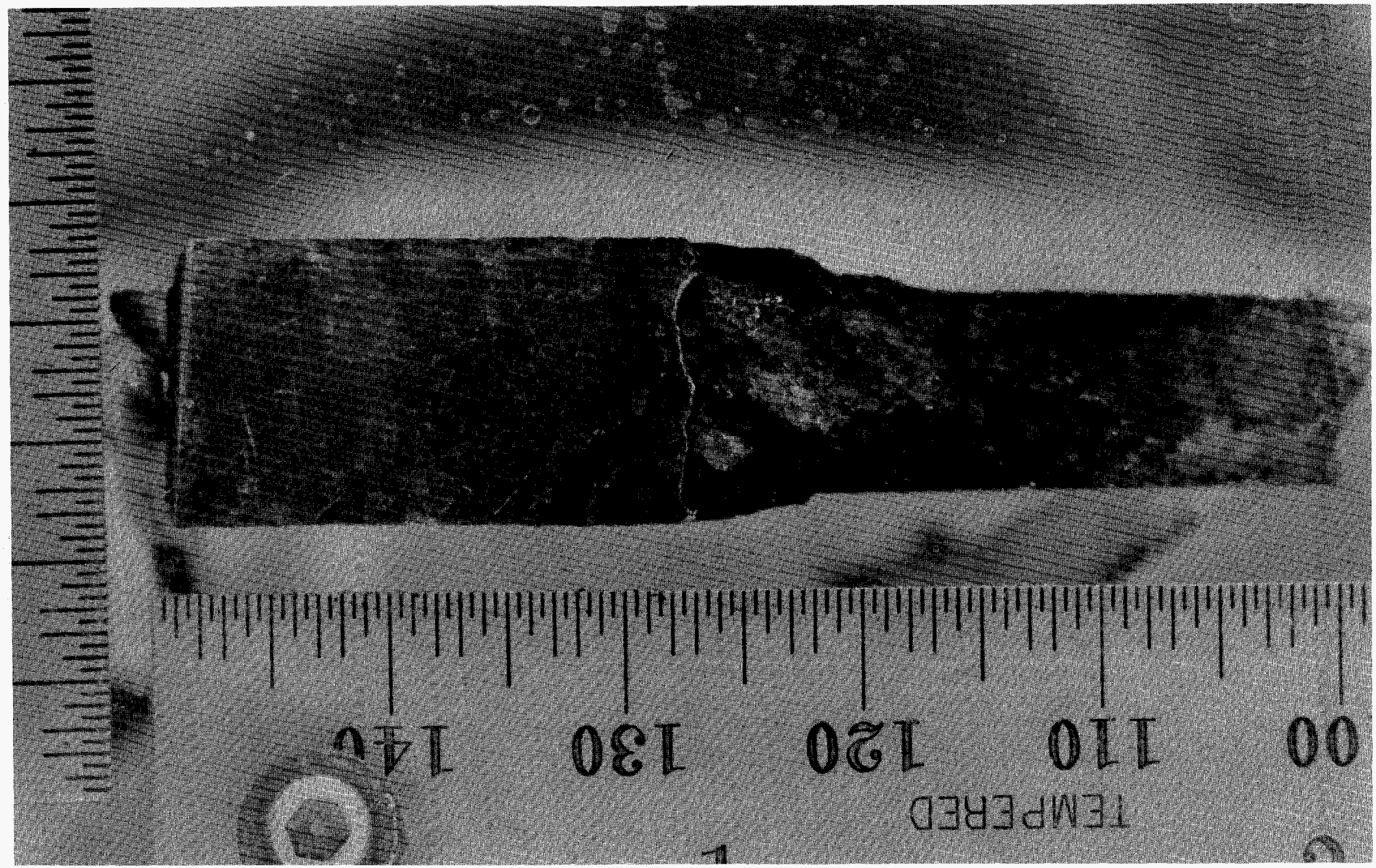


Figure B.35: Outer Cladding Surface of As-Cut Specimen SFEC5,4378-S1A-H. Each division of the attached scale is 0.5 mm. PTL log is Q-1625, Photo # 442.

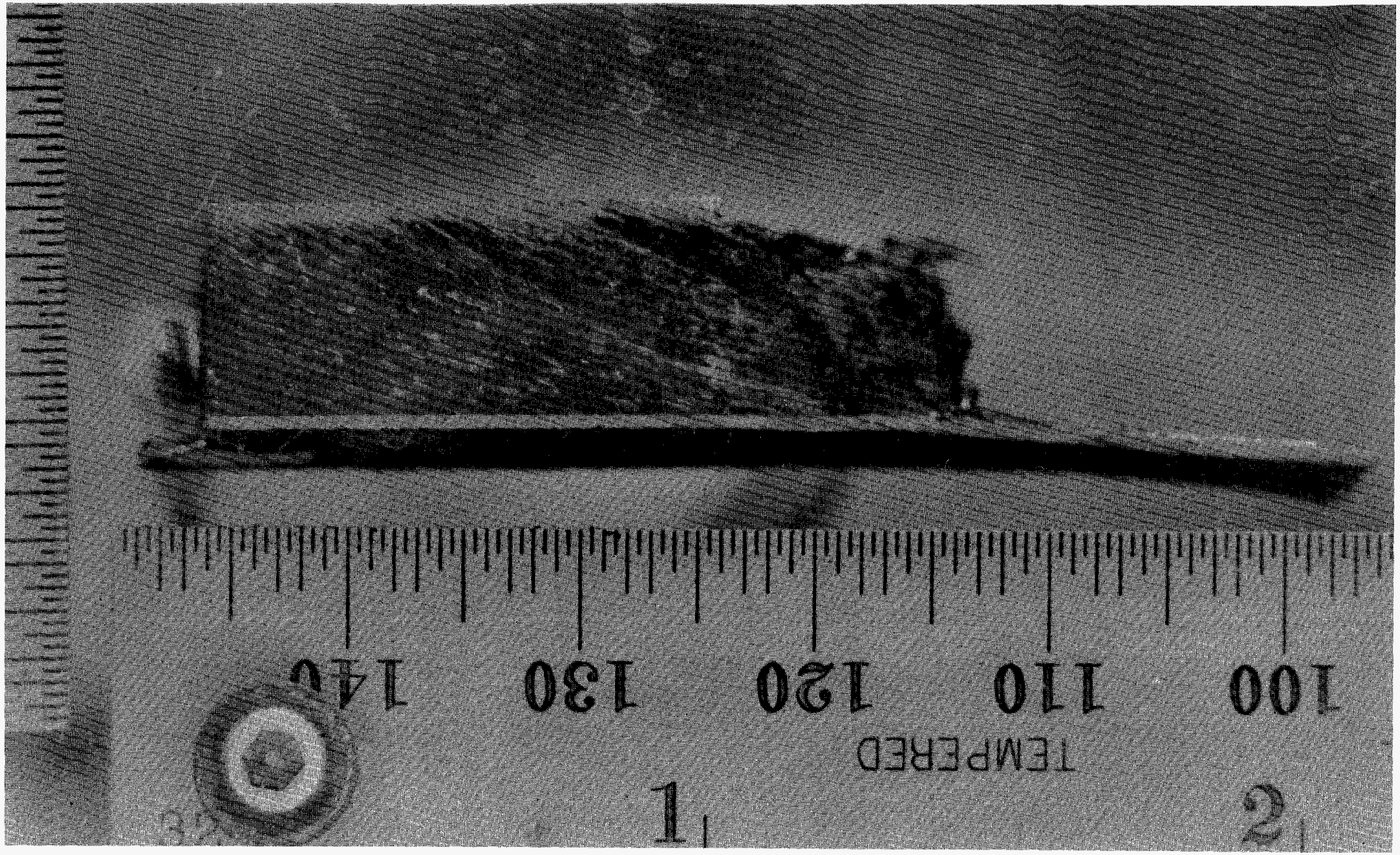


Figure B.36: The Other Longitudinal Surface of As-Cut Specimen SFEC5,4378-S1A-H. Each division of the attached scale is 0.5 mm. PTL log is Q-1626, Photo # 443.

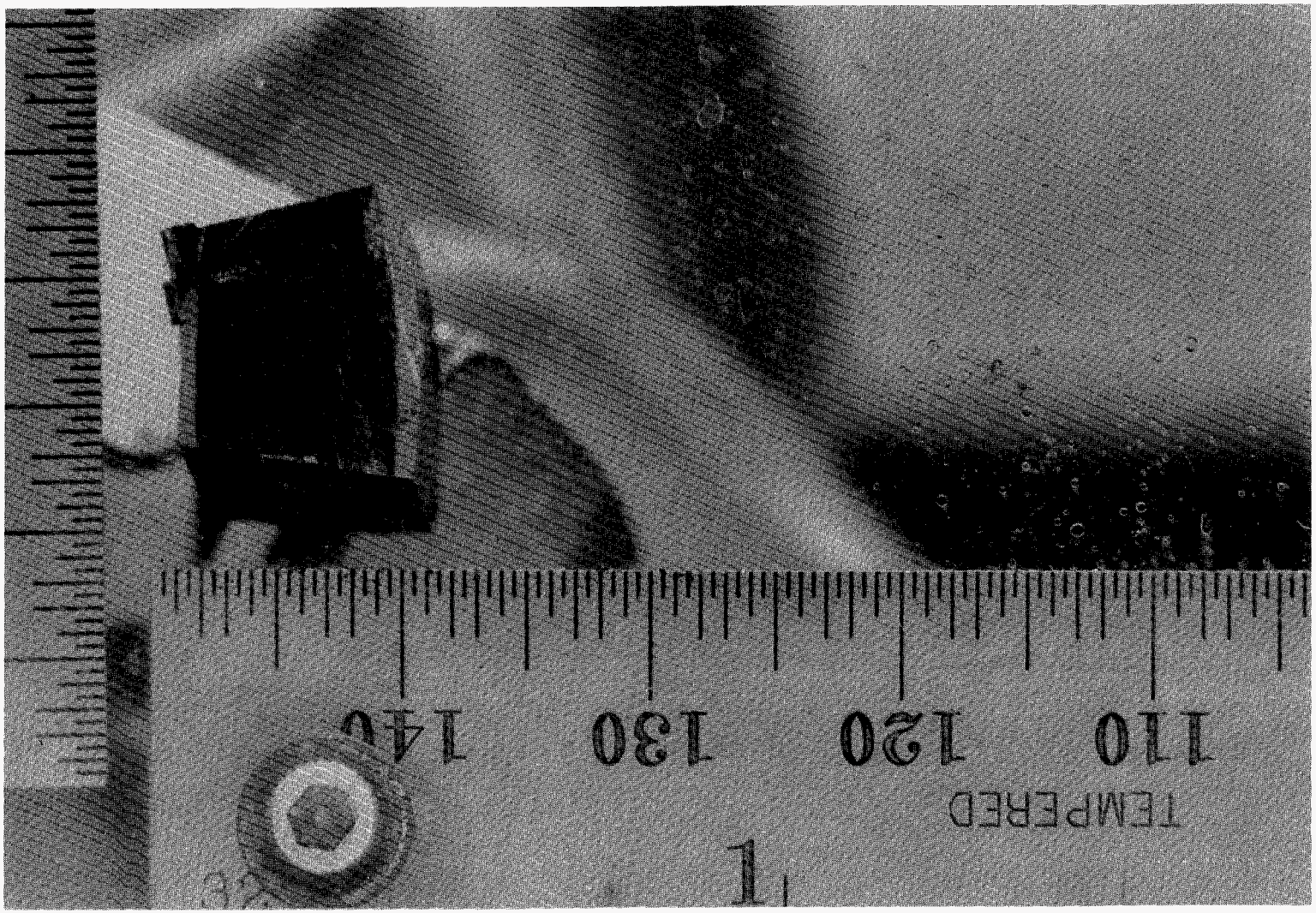


Figure B.37: Bottom Surface of As-Cut Specimen SFEC5,4378-S1A-H. Each division of the attached scale is 0.5 mm. PTL log is Q-1627, Photo # 444.

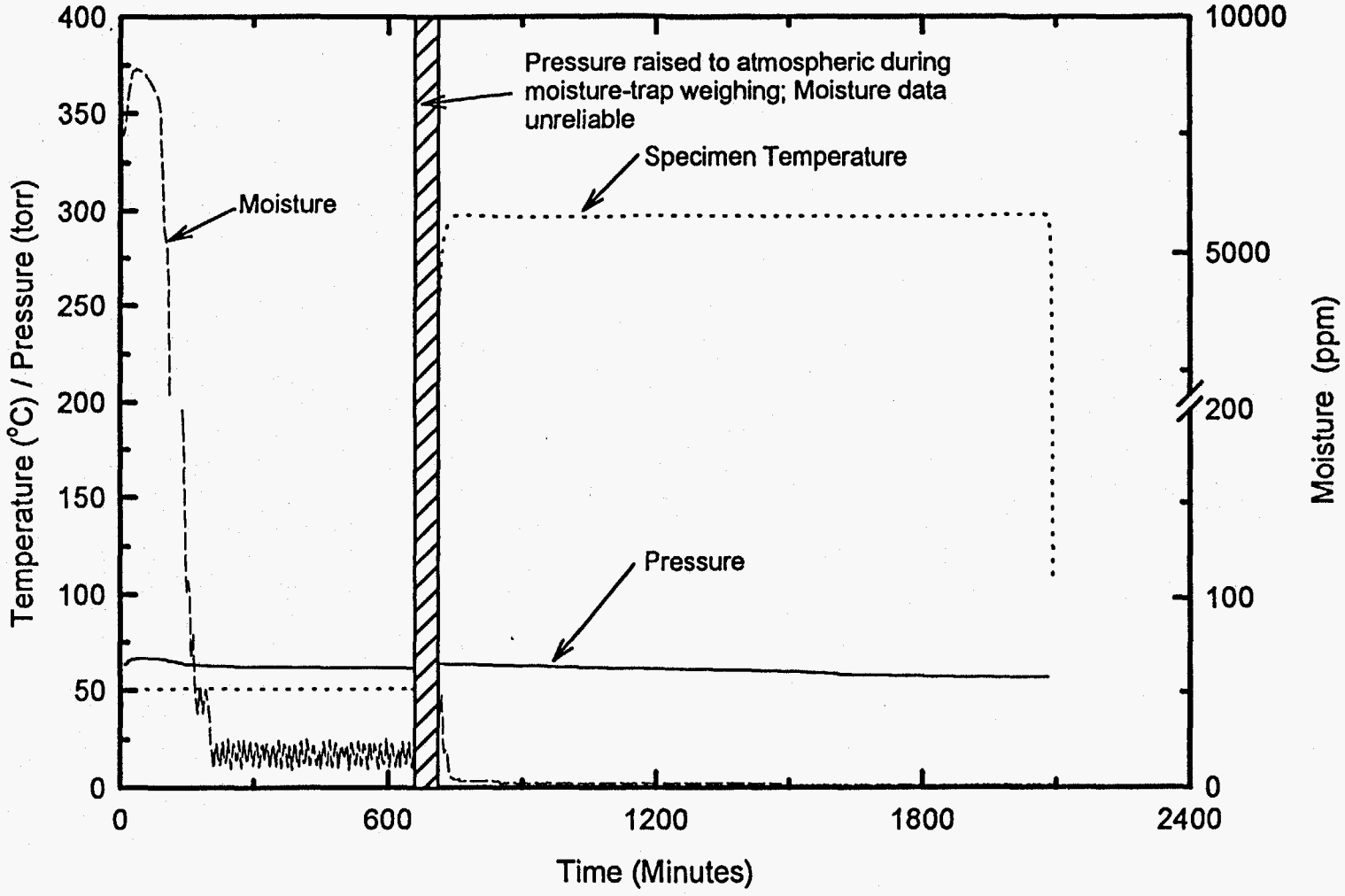


Figure B.38: Plots Of Moisture Content of the Off Gas Stream, Furnace Pressure and Specimen Temperature during the CVD and HVD Cycles of Furnace Run # 5.

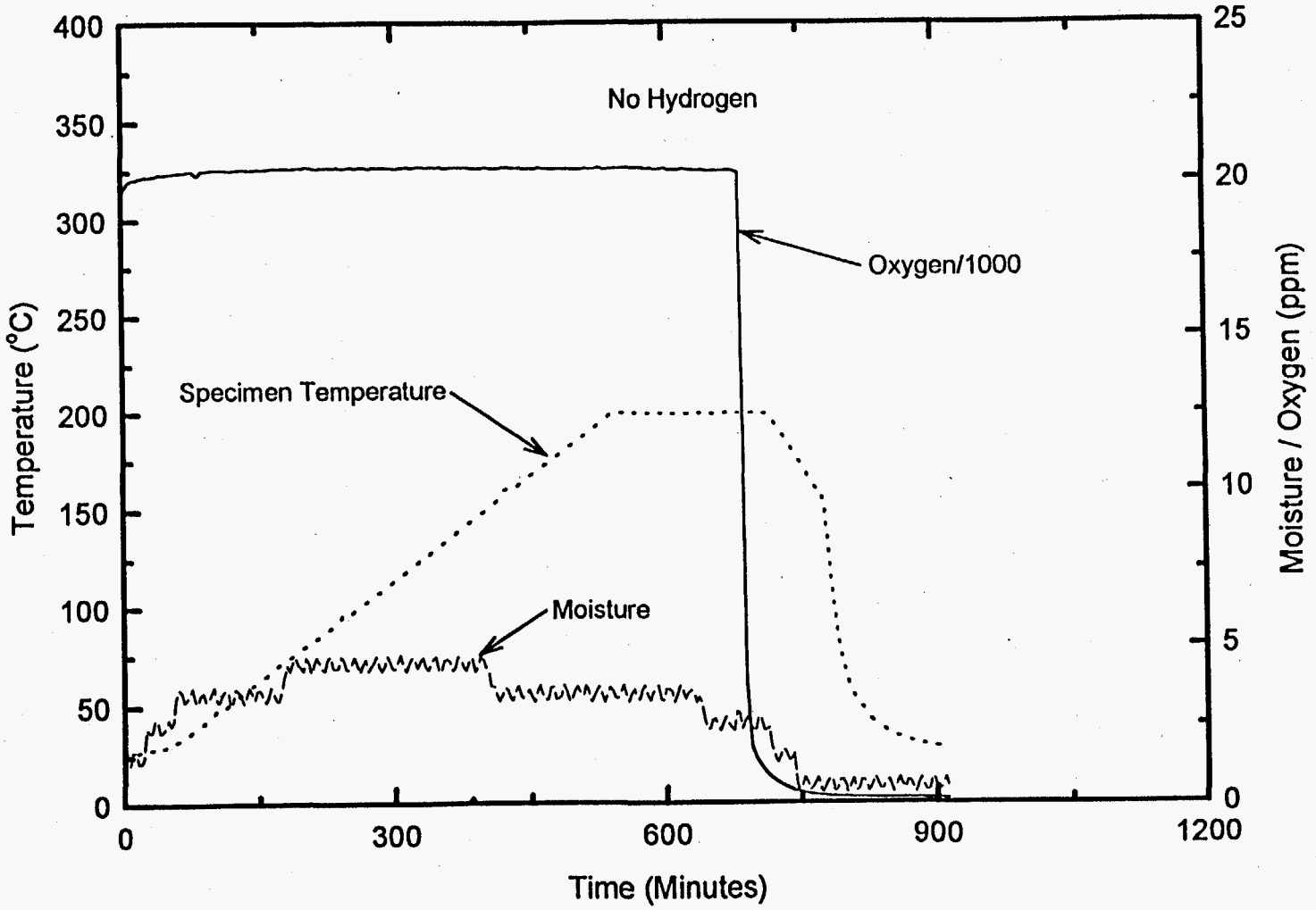


Figure B.39: Plots Of Moisture and Oxygen Contents in the Off Gas Stream, and Specimen Temperature during the Passivation Cycle of Furnace Run # 5.

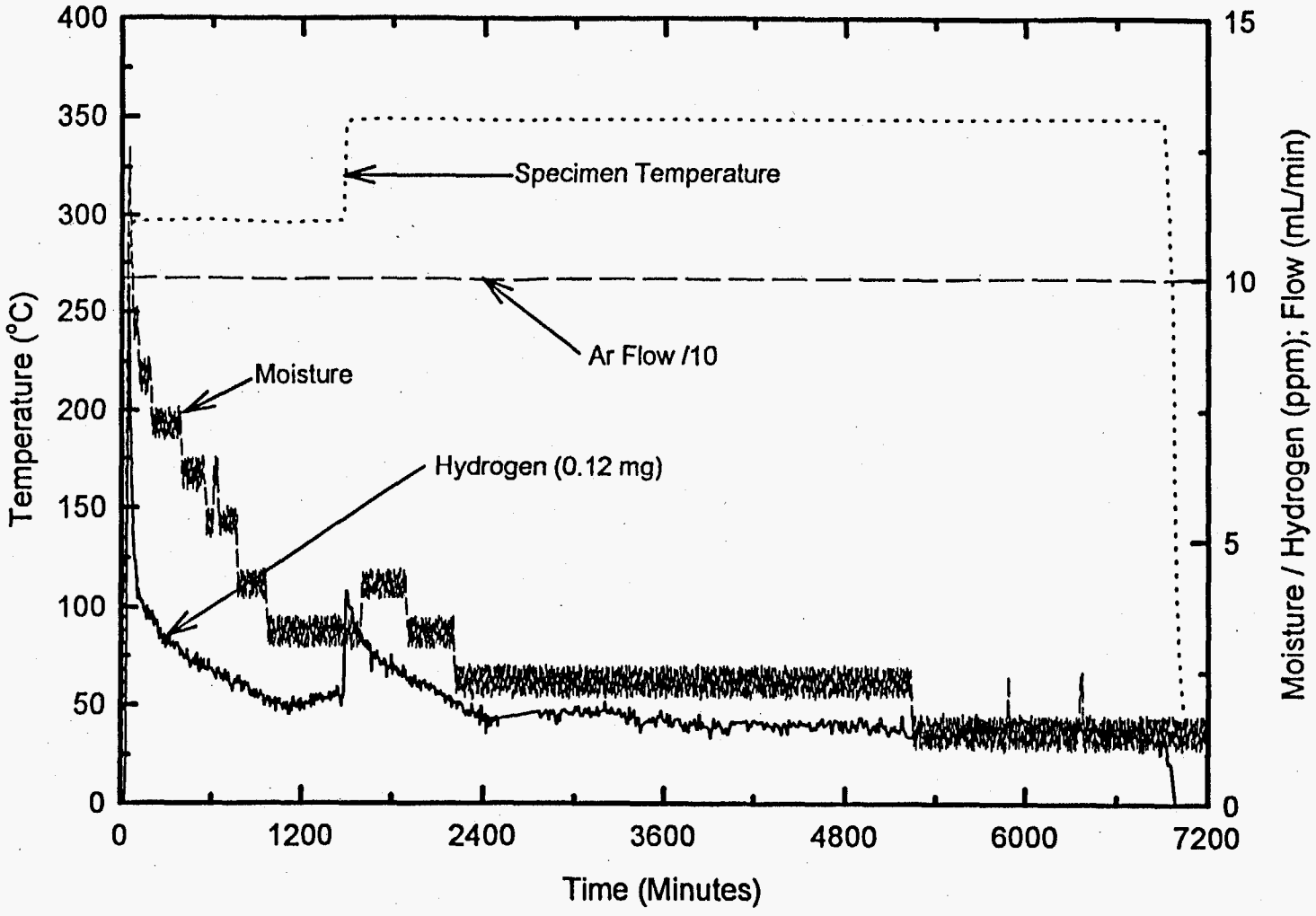


Figure B.40: Results of Hydrogen Release from a Decladded Fuel Specimen SFEC5,4378-S2-E1 Showing the History of Moisture and Hydrogen In the Off Gas Stream.

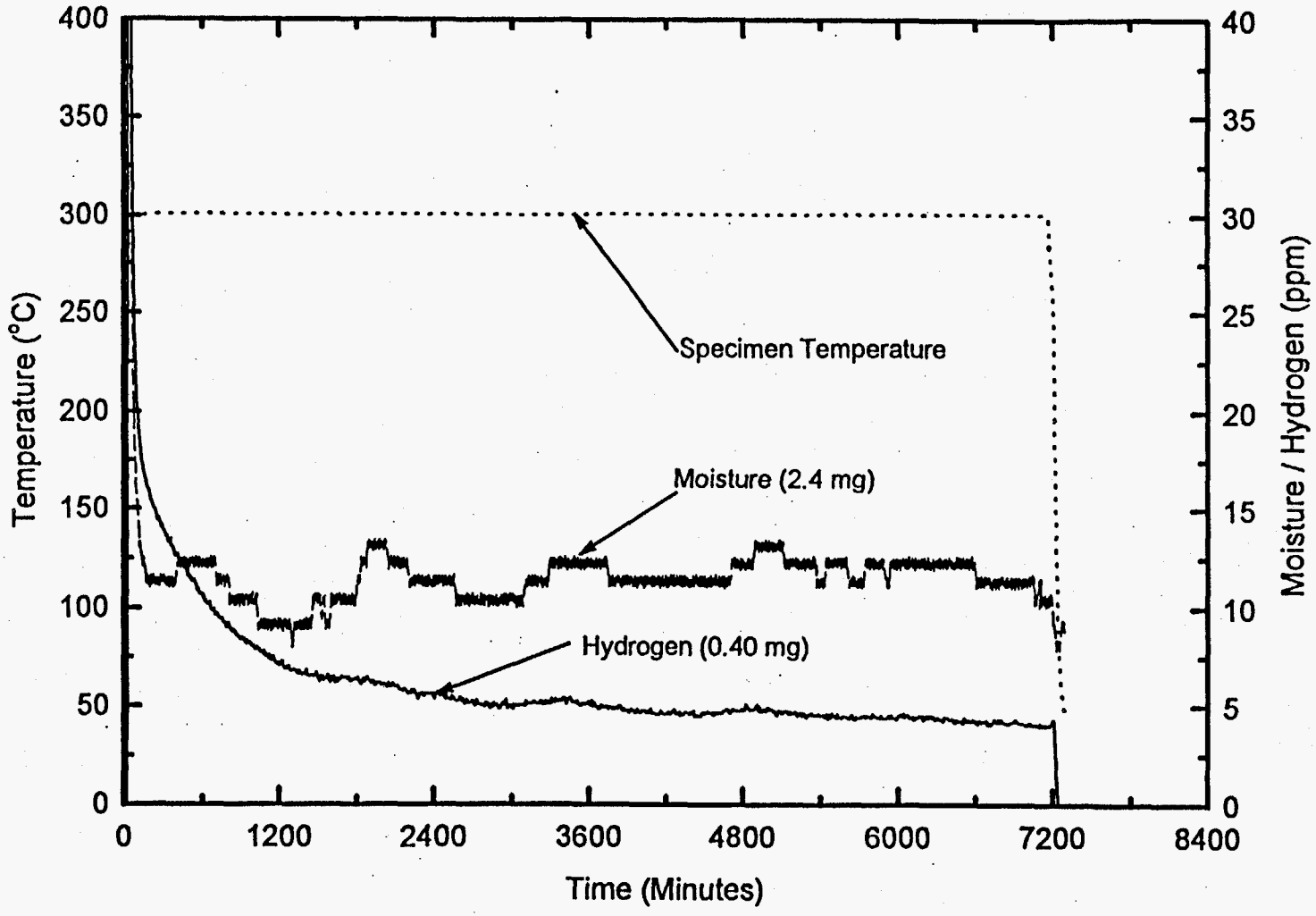


Figure B.41: Results of Hydrogen Release from a Piece of Cladding Material Broken Off From the Damaged End of SNF Element SFEC5,4378 Showing the History of Moisture and Hydrogen In the Off Gas Stream.

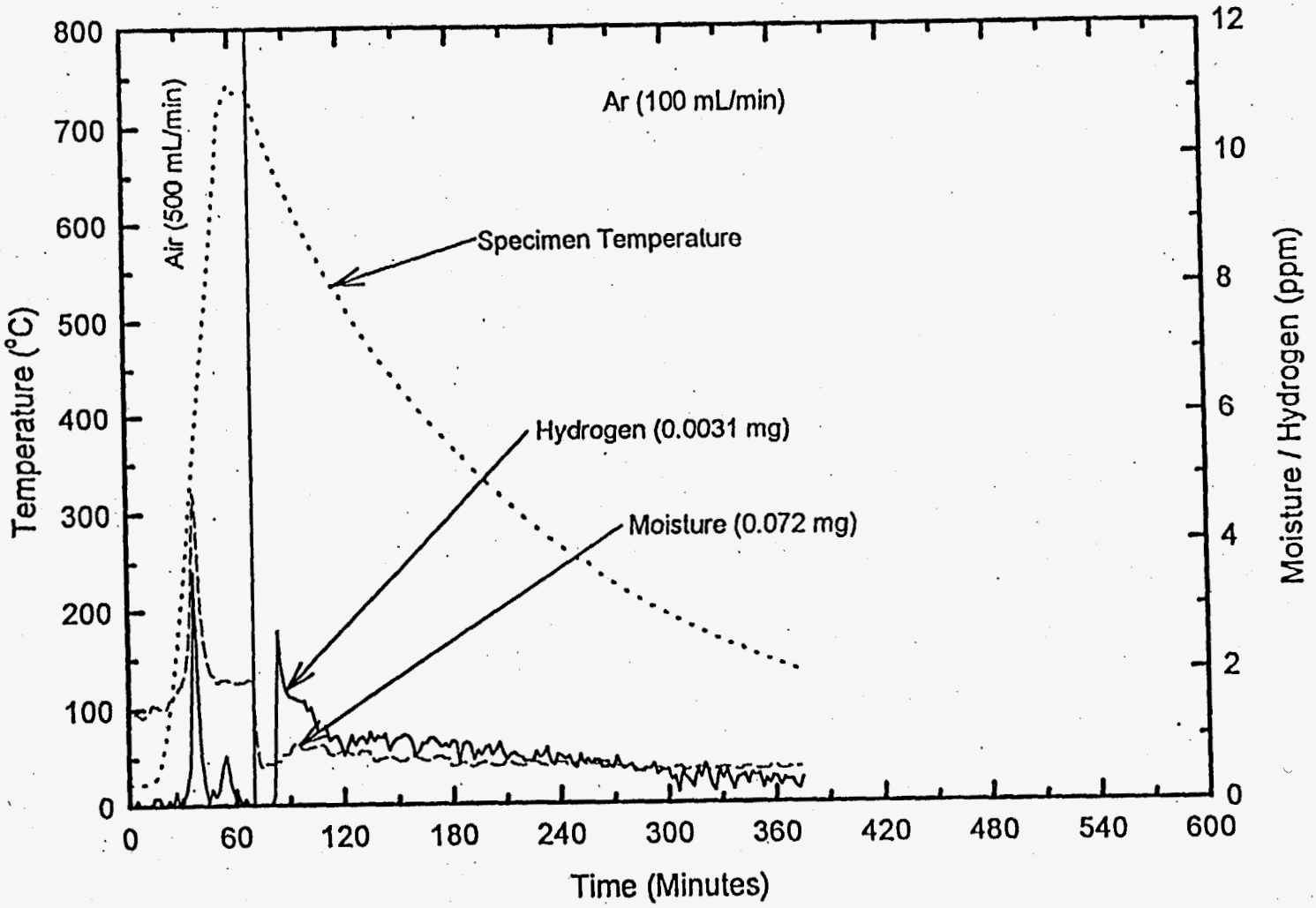


Figure B.42: Ignition Test Results From An As-Cut SNF Specimen SFEC5,4378-S2-E4A. The Vertical Line Indicates the Time When the Furnace Power Was Turned Off.

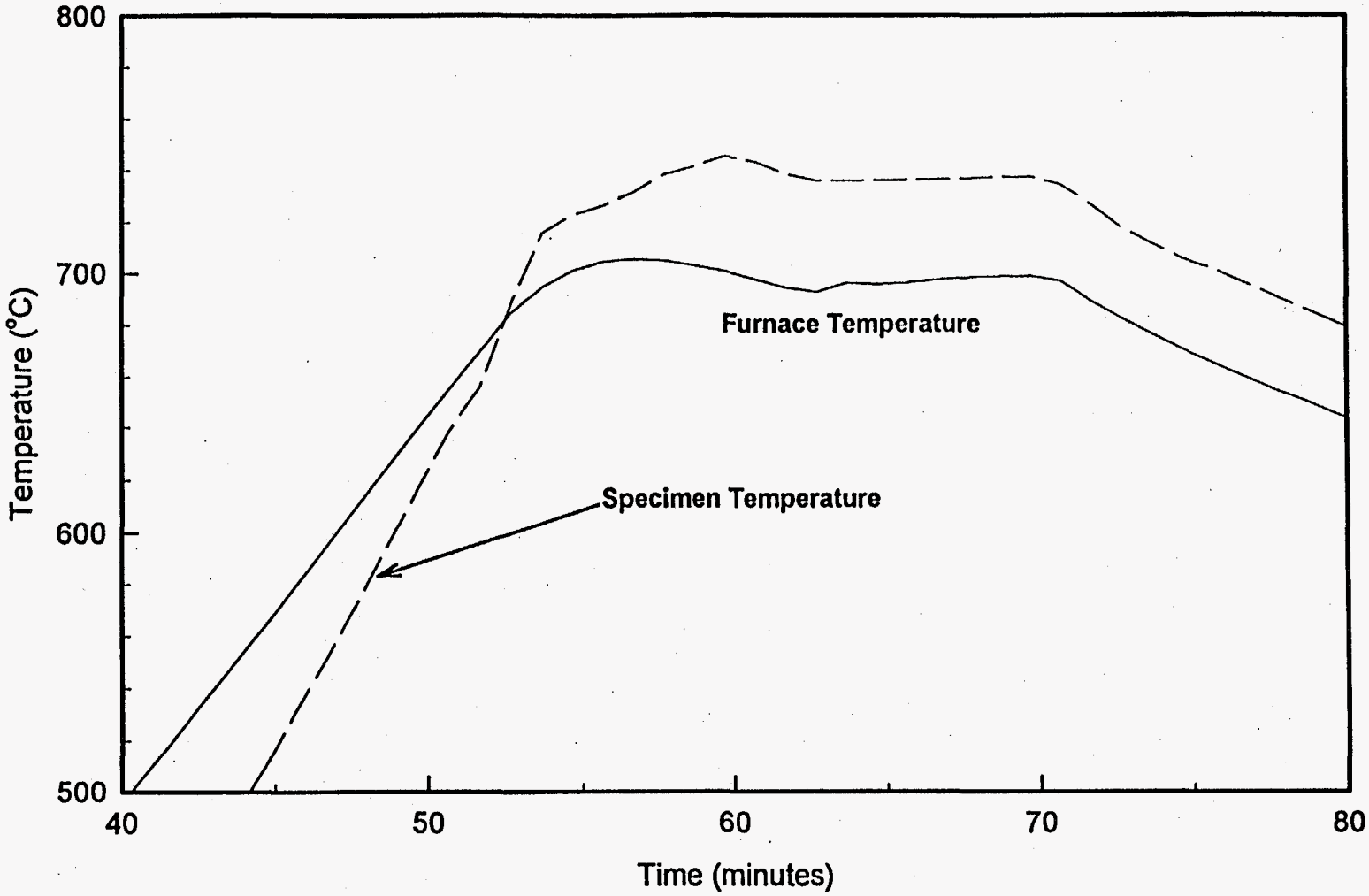


Figure B.43: An Expanded Plots of the Specimen and the Furnace Temperatures In the Region Where Ignition Of Figure B.42 is Expected to Occur.

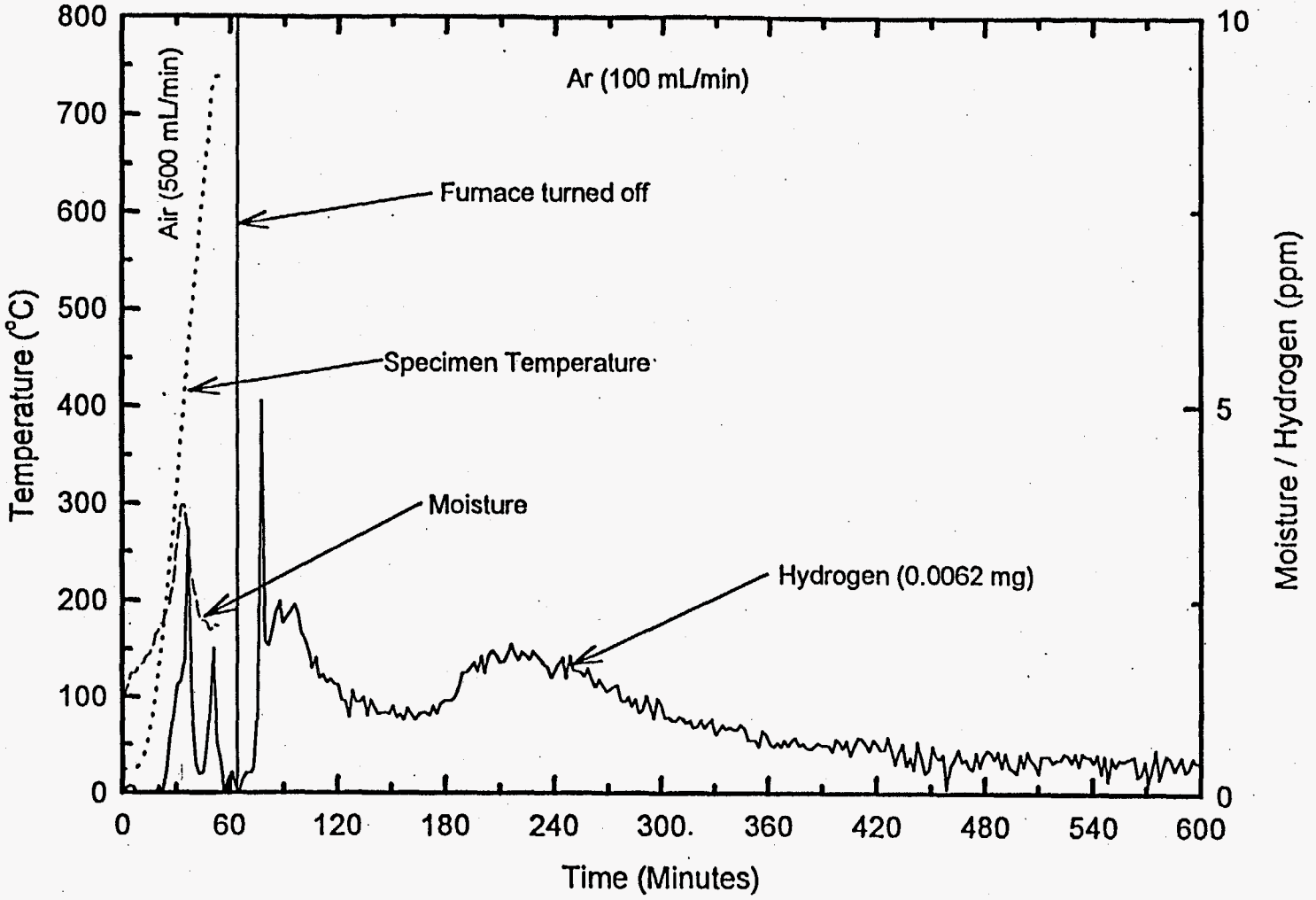


Figure B.44: Ignition Test Results From An ITA Process Conditioned Specimen SFEC5,4378-S2-H. The Vertical Line Indicates the Time When the Furnace Power Was Turned Off.

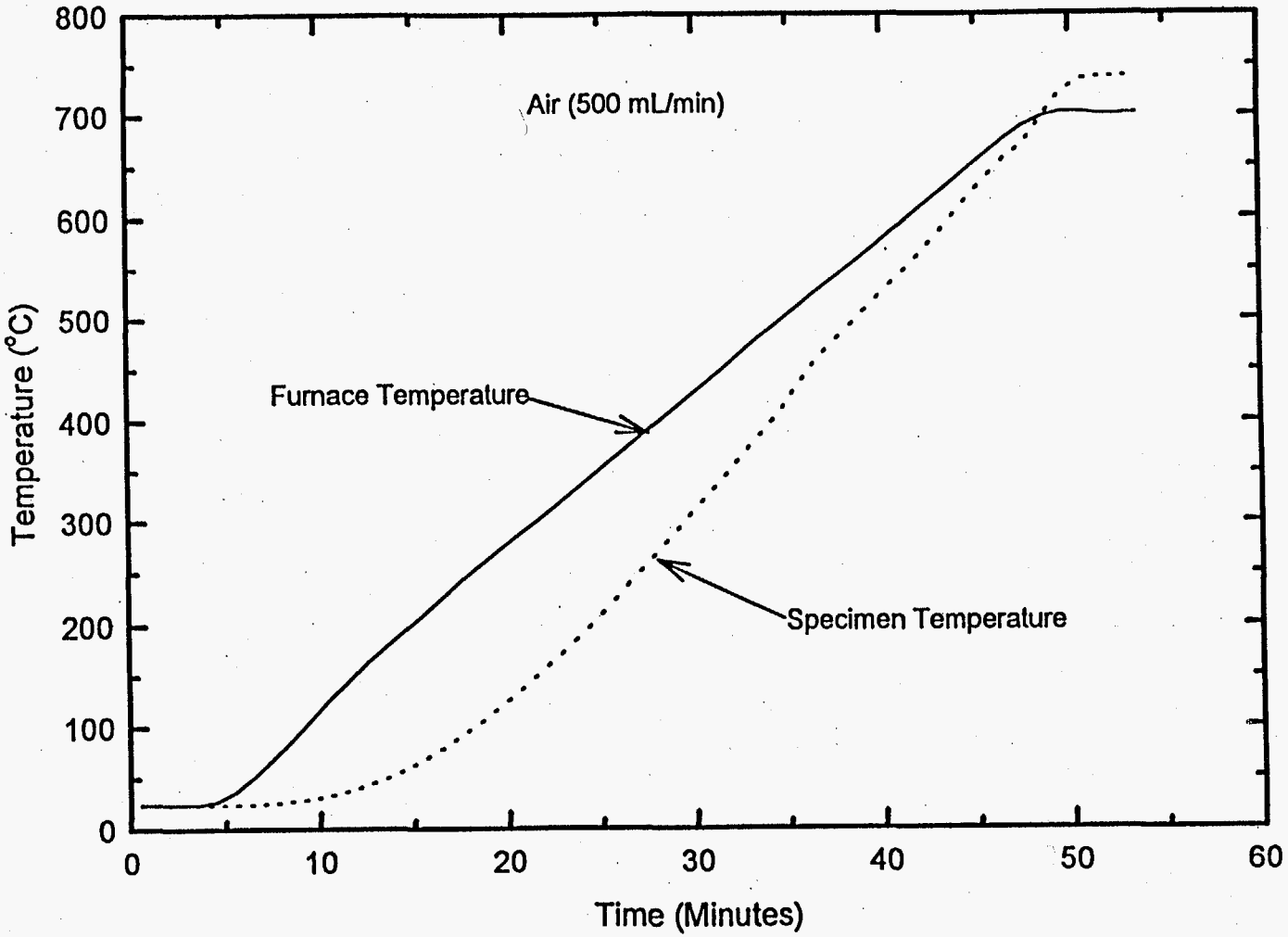


Figure B.45: An Expanded Plots of the Specimen and the Furnace Temperatures In the Region Where Ignition Of Figure B.44 is Expected to Occur.

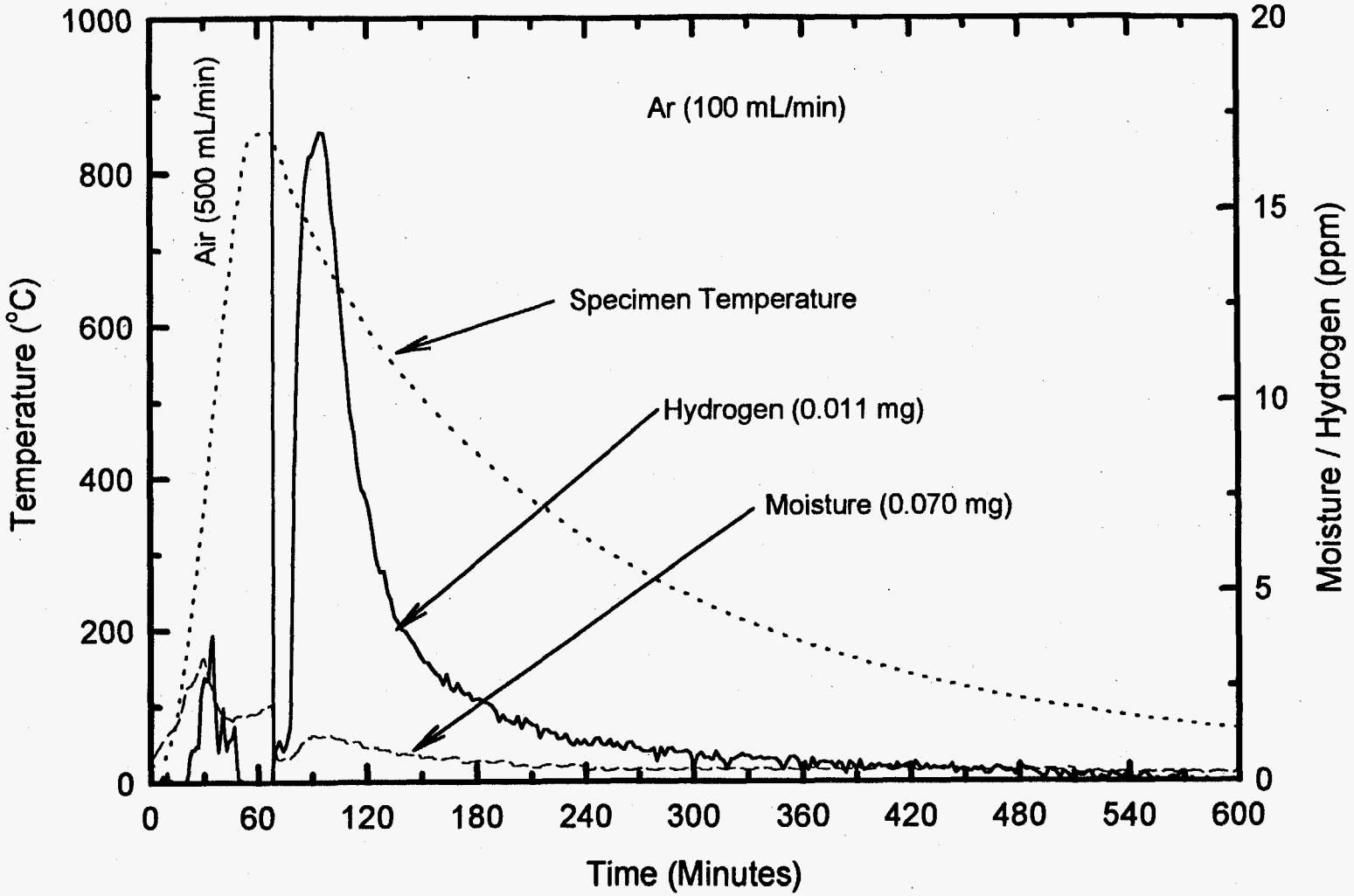


Figure B.46: Ignition Test Results of ITA Process Conditioned Specimen SFEC5,4378-S2-I. The Vertical Line Indicates the Time When the Furnace Power Was Turned Off.

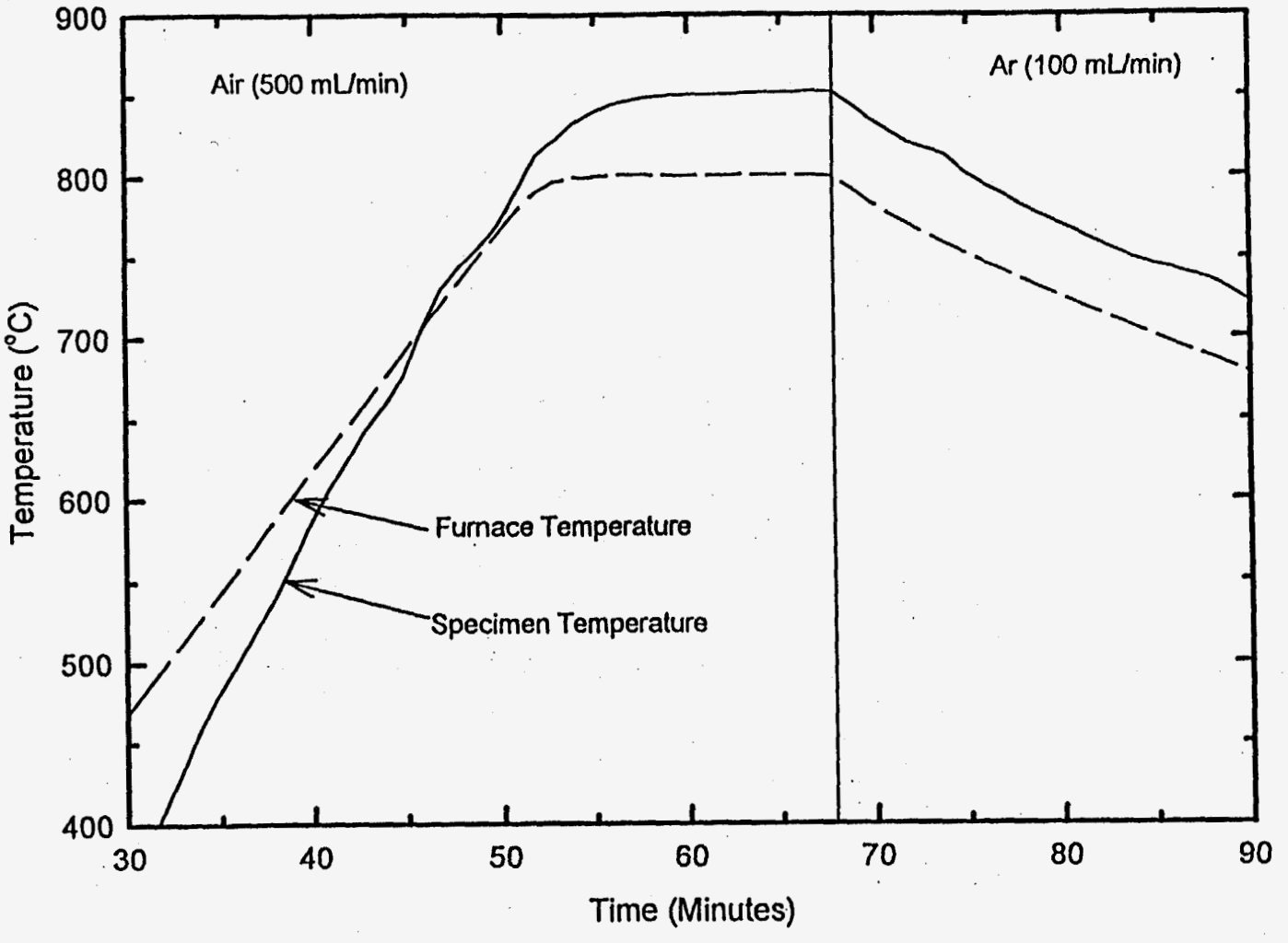


Figure B.47: An Expanded Plots of the Specimen and the Furnace Temperatures In the Region Where Ignition Of Figure B.46 is Expected to Occur.

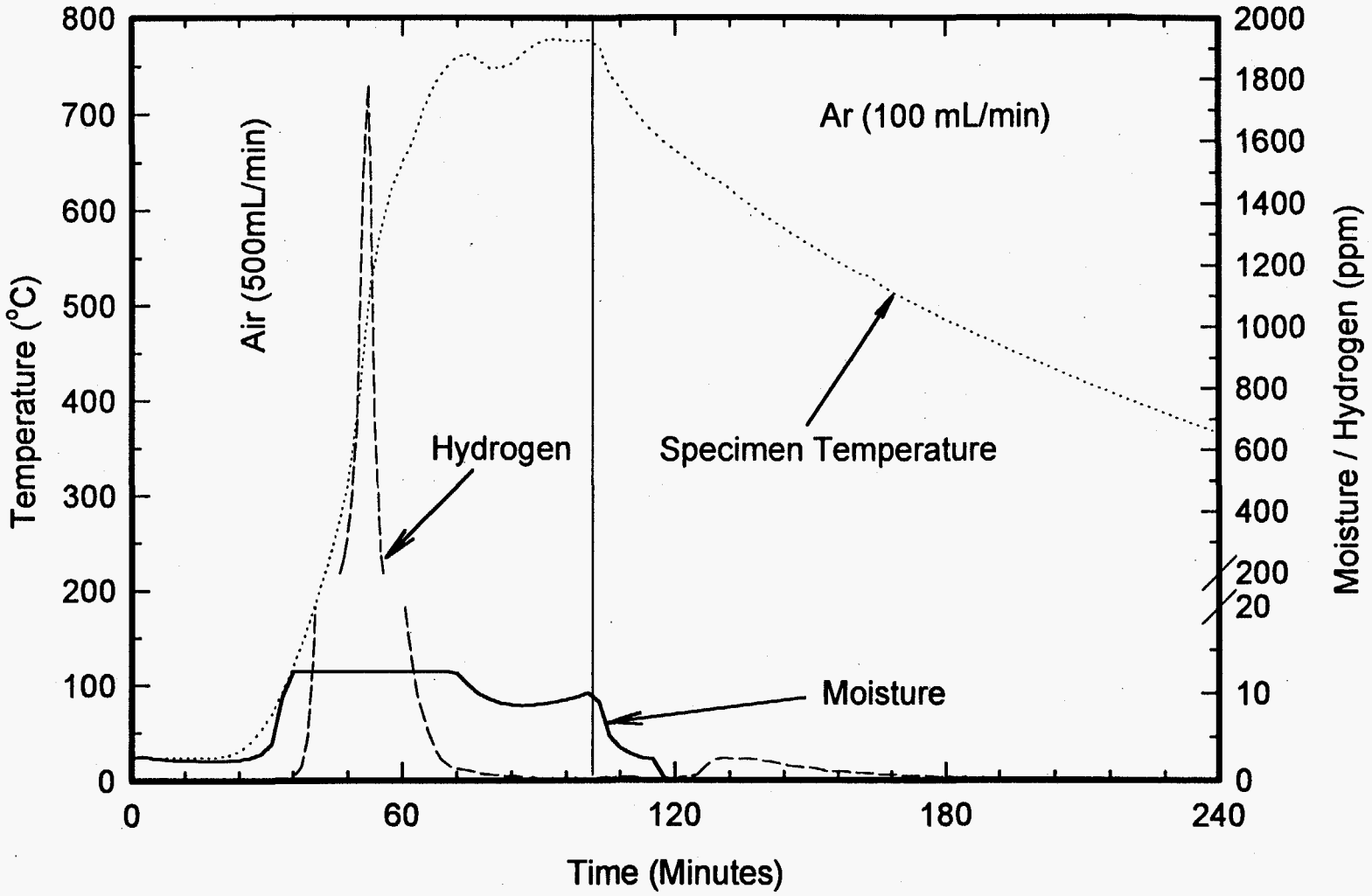


Figure B.48: Ignition Test Results of As-Cut SNF Specimen SFEC5,4378-S1A-D. The Vertical Line Indicates the Time When the Furnace Power Was Turned Off.

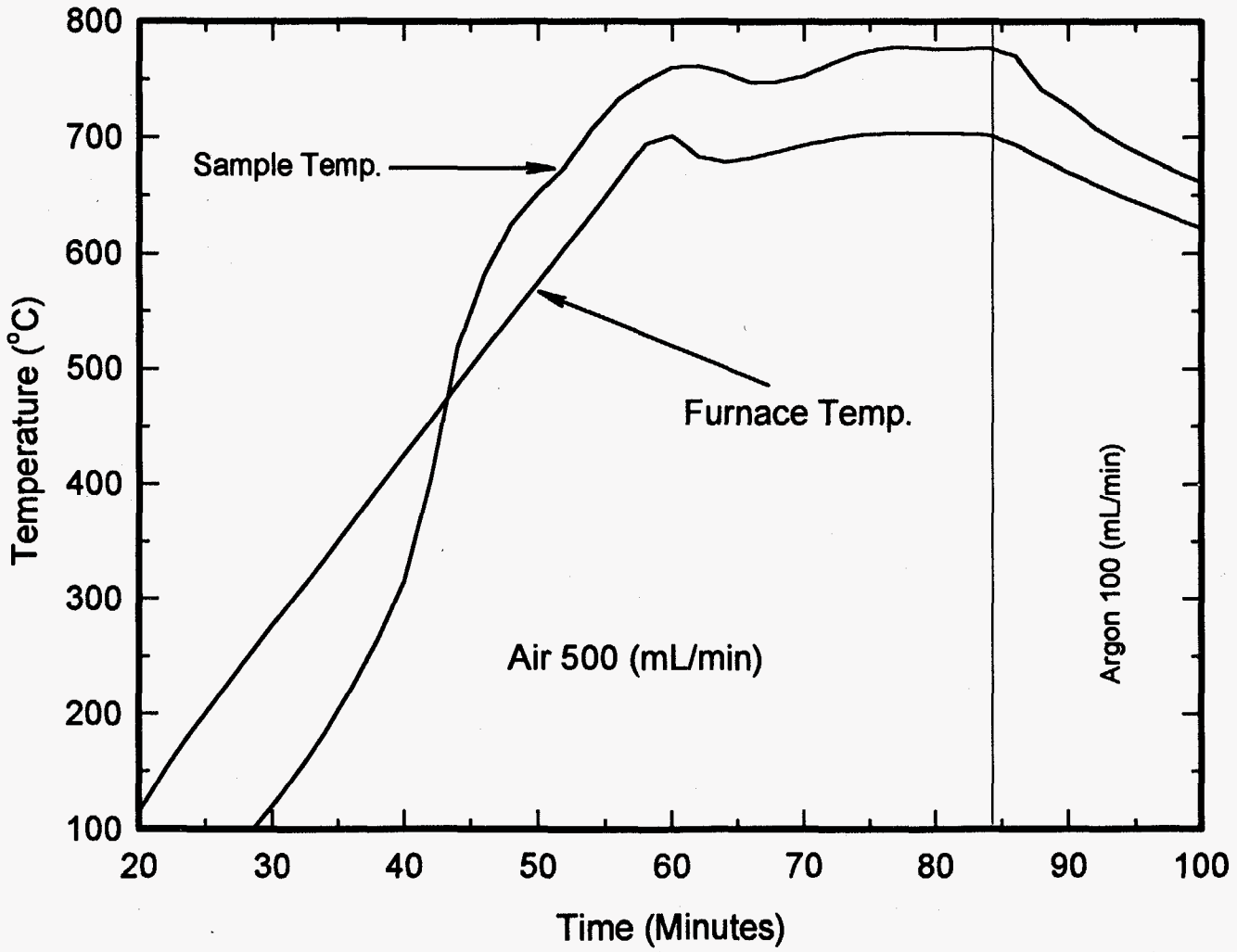


Figure B.49: An Expanded Plots of the Specimen and the Furnace Temperatures In the Region Where Ignition Of Figure B.48 is Expected to Occur.

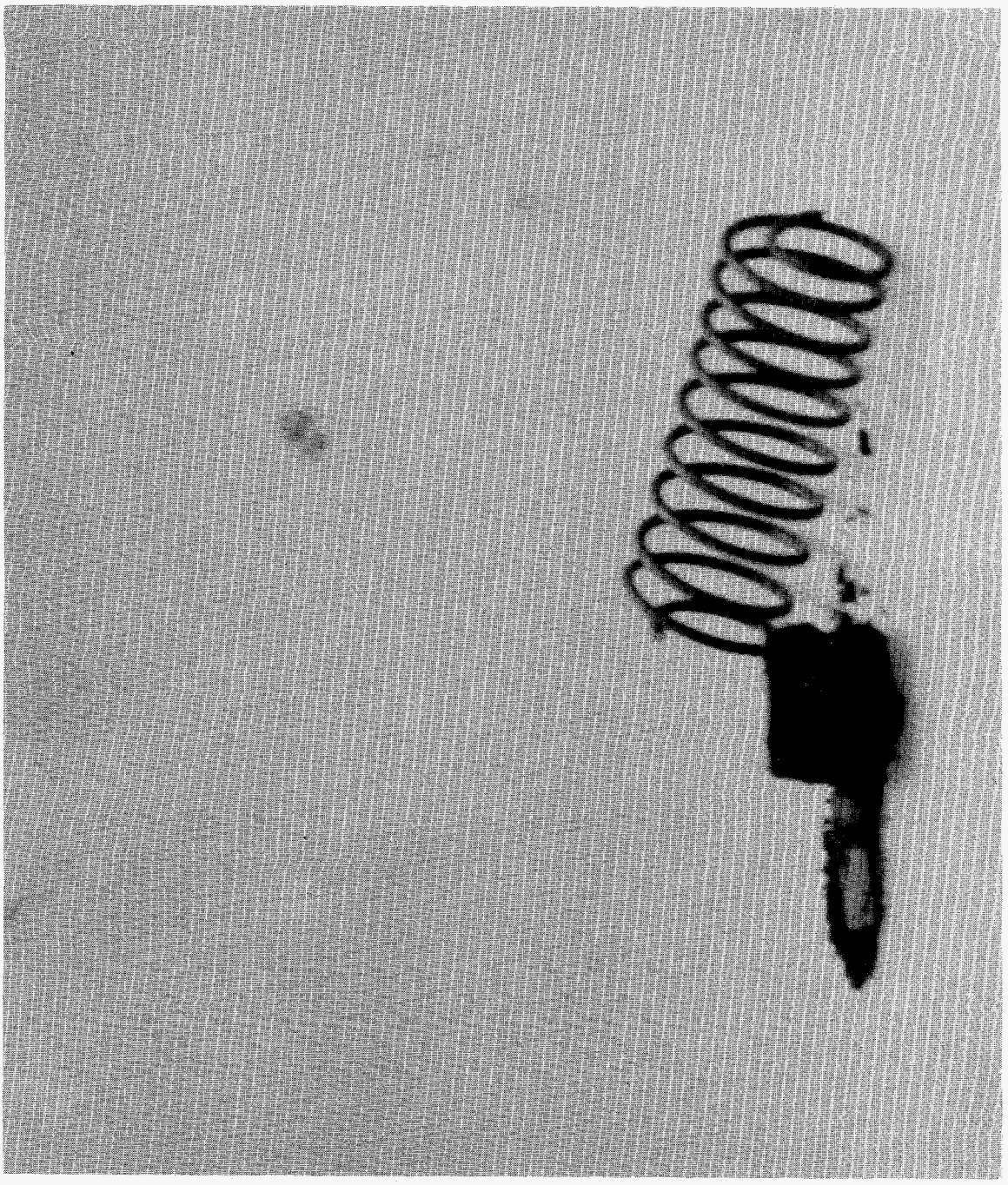


Figure B.50: Photograph of Specimen SFEC5,4378-S2-E4A Showing Highly Oxidized Specimen, the Residue from the Spalled Uranium Oxide and Spring Used to Keep Specimen In Contact With the K-Type Thermocouple Throughout the Test.

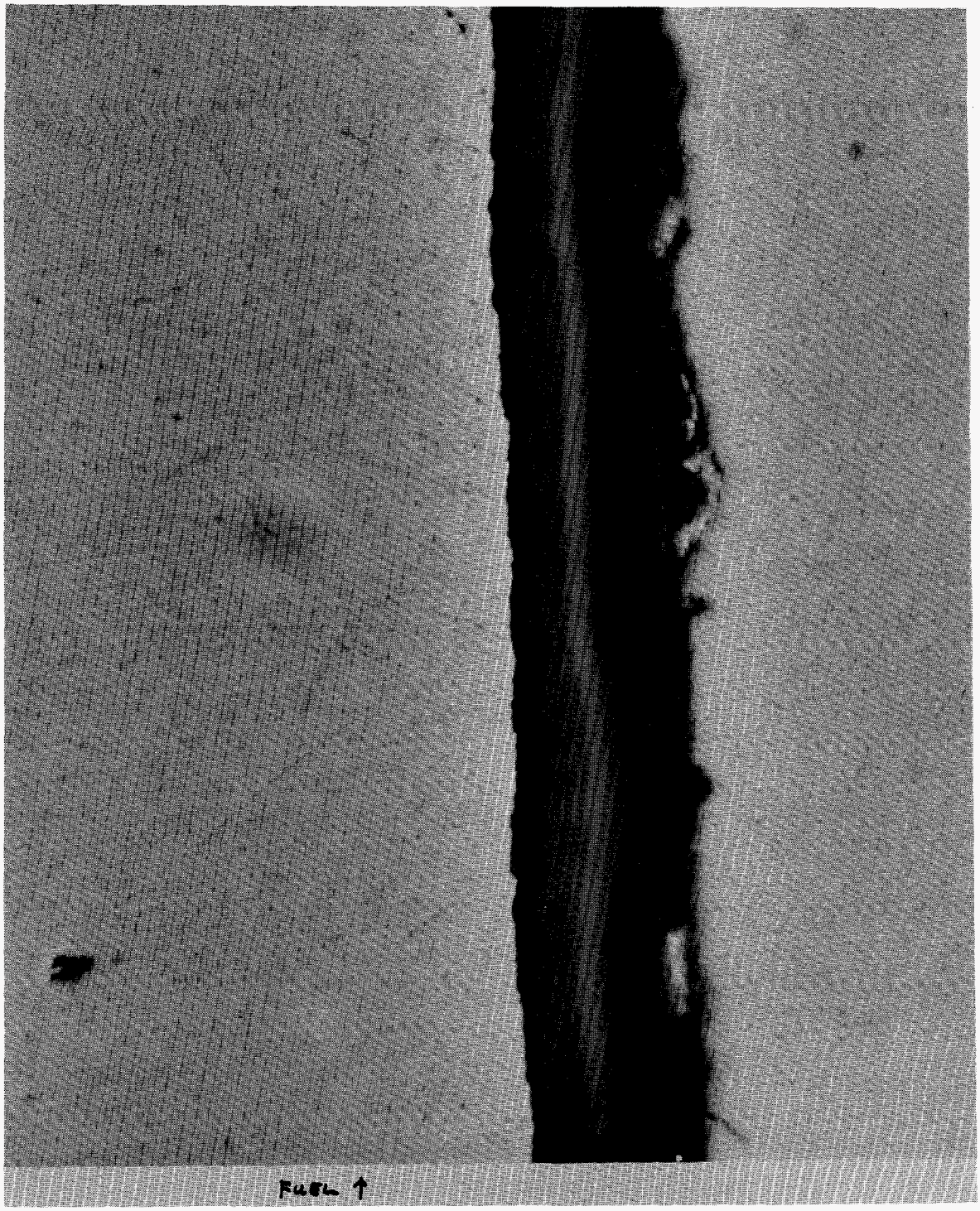


Figure B.51: Photomicrograph, 750X, of the As-Polished Conditioned Specimen SFEC5,4378-S2-J2 in the Longitudinal Plane. The photo shows one of the cut planes of the fuel (left side) and the mounting material (dark). PTL Log Q-1464, Photo # 283.

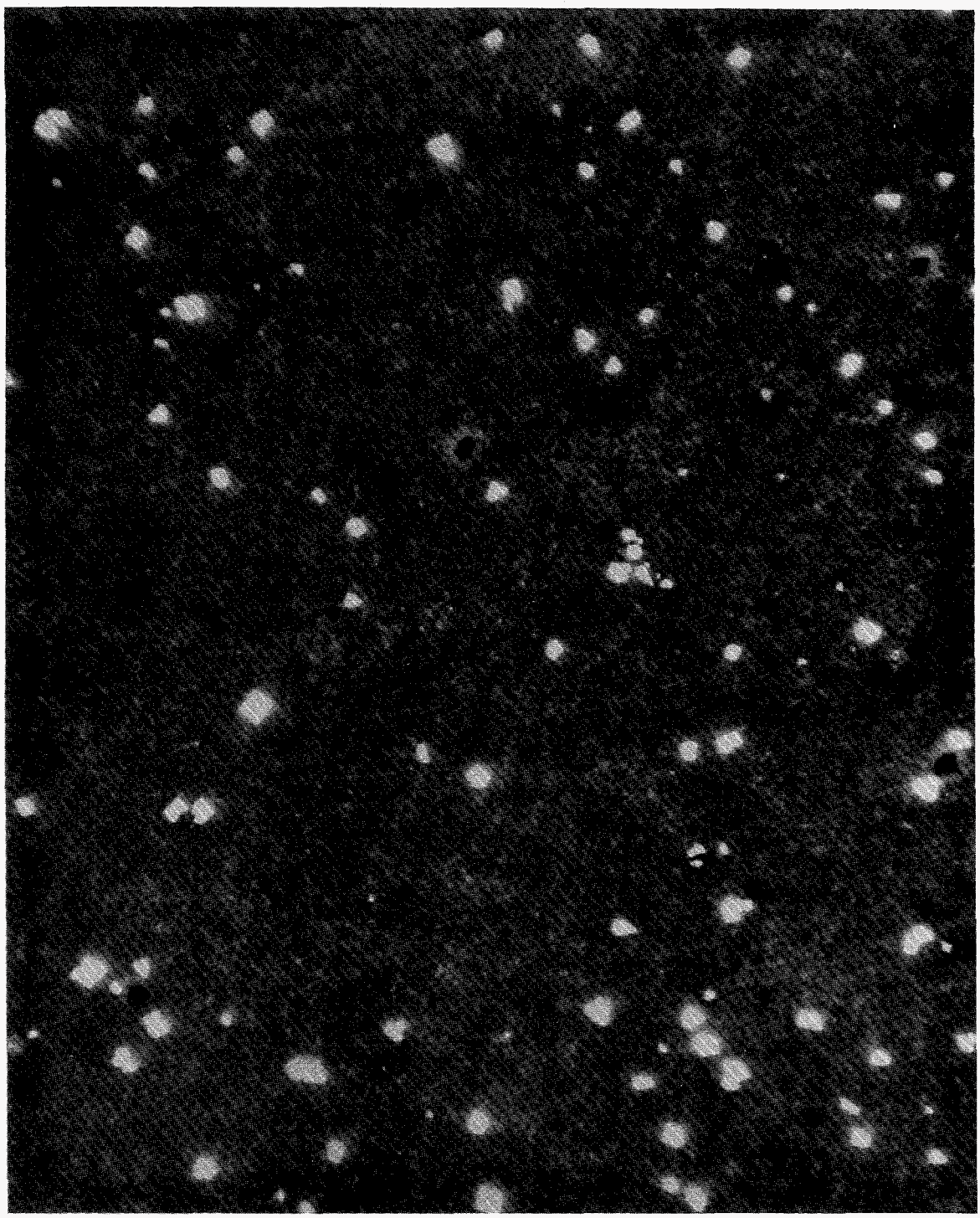


Figure B.52: Photomicrograph, 50X, of the As-Polished Conditioned Specimen SFEC5,4378-S2-J2 in the Longitudinal Plane. The photo illustrates the different phase structures on a heat tinted uranium surface. PTL Log Q-1471. Photo # 290.

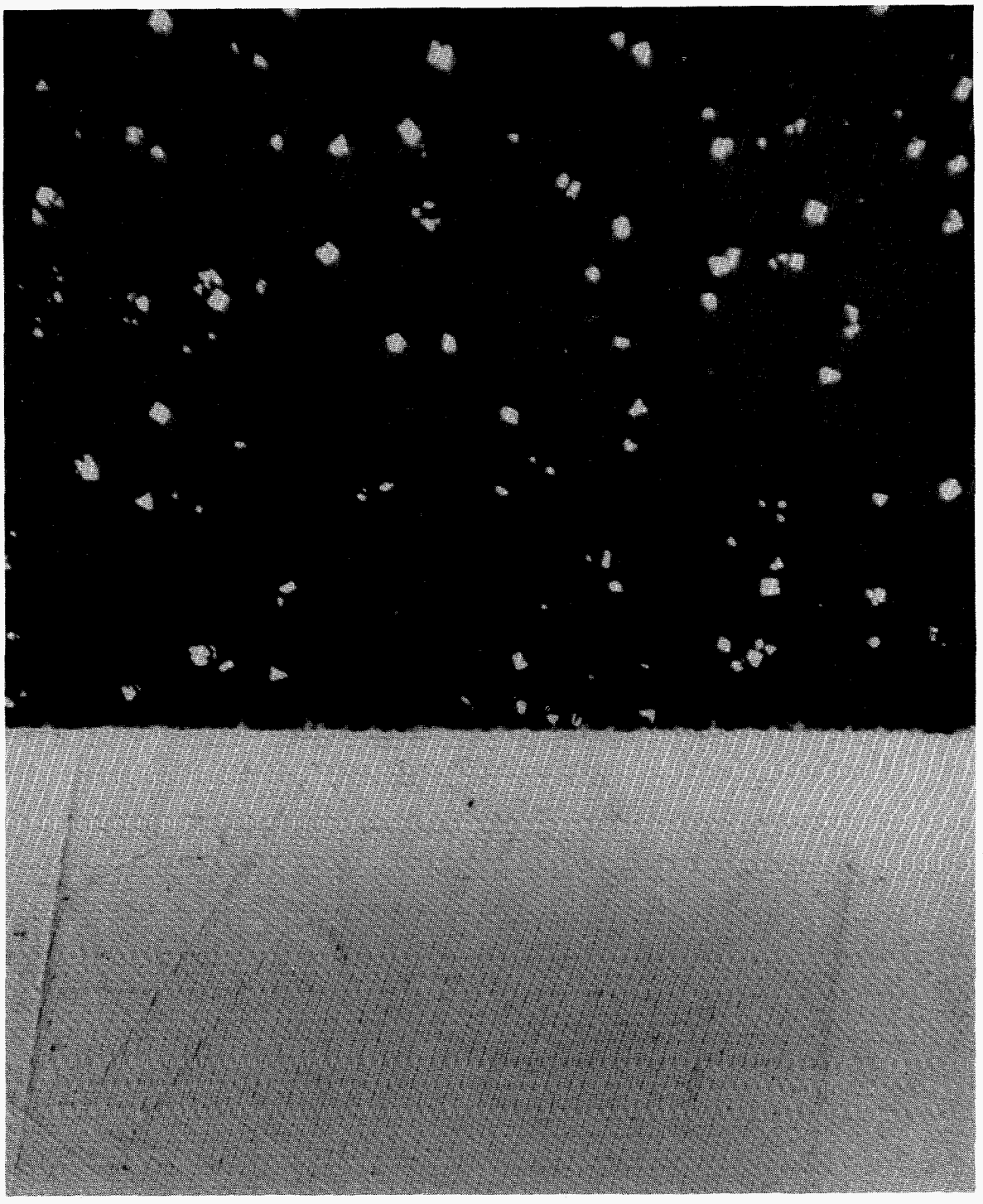


Figure B.53: Photomicrograph, 50X, of the As-Polished Conditioned Specimen SFEC5,4378-S2-J2 in the Longitudinal Plane. The photo shows the fuel/cladding boundary. PTL Log Q-1472, Photo # 291.

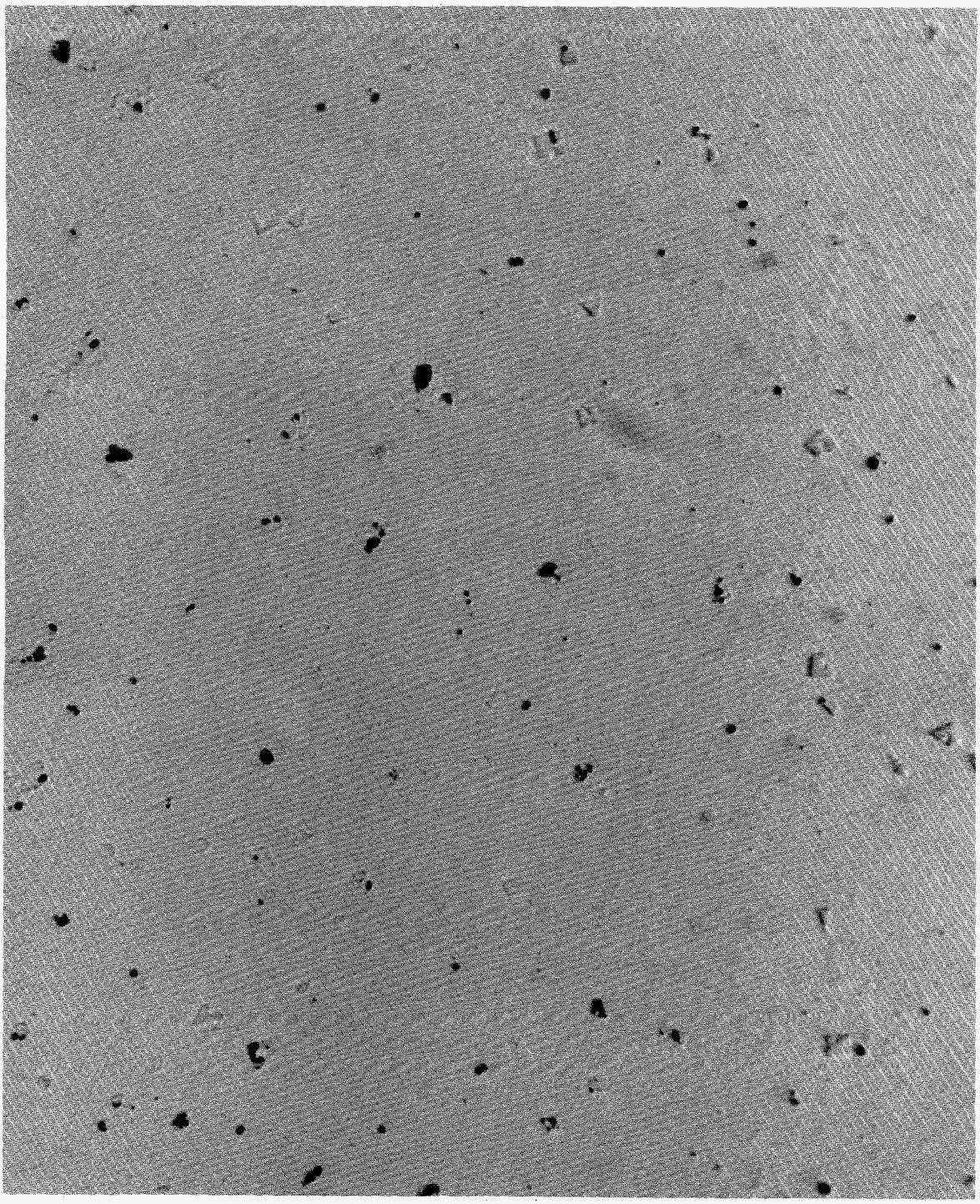


Figure B.54: Photomicrograph, 50X, of the Mid-Region of As-Polished Conditioned Specimen SFEC5,4378-S2-J1 in the Transverse Plane. The photo illustrates the different phase structures in the uranium matrix. The bright, almost regular geometric precipitates are most likely uranium carbide. PTL Log Q-1428, Photo # 247.

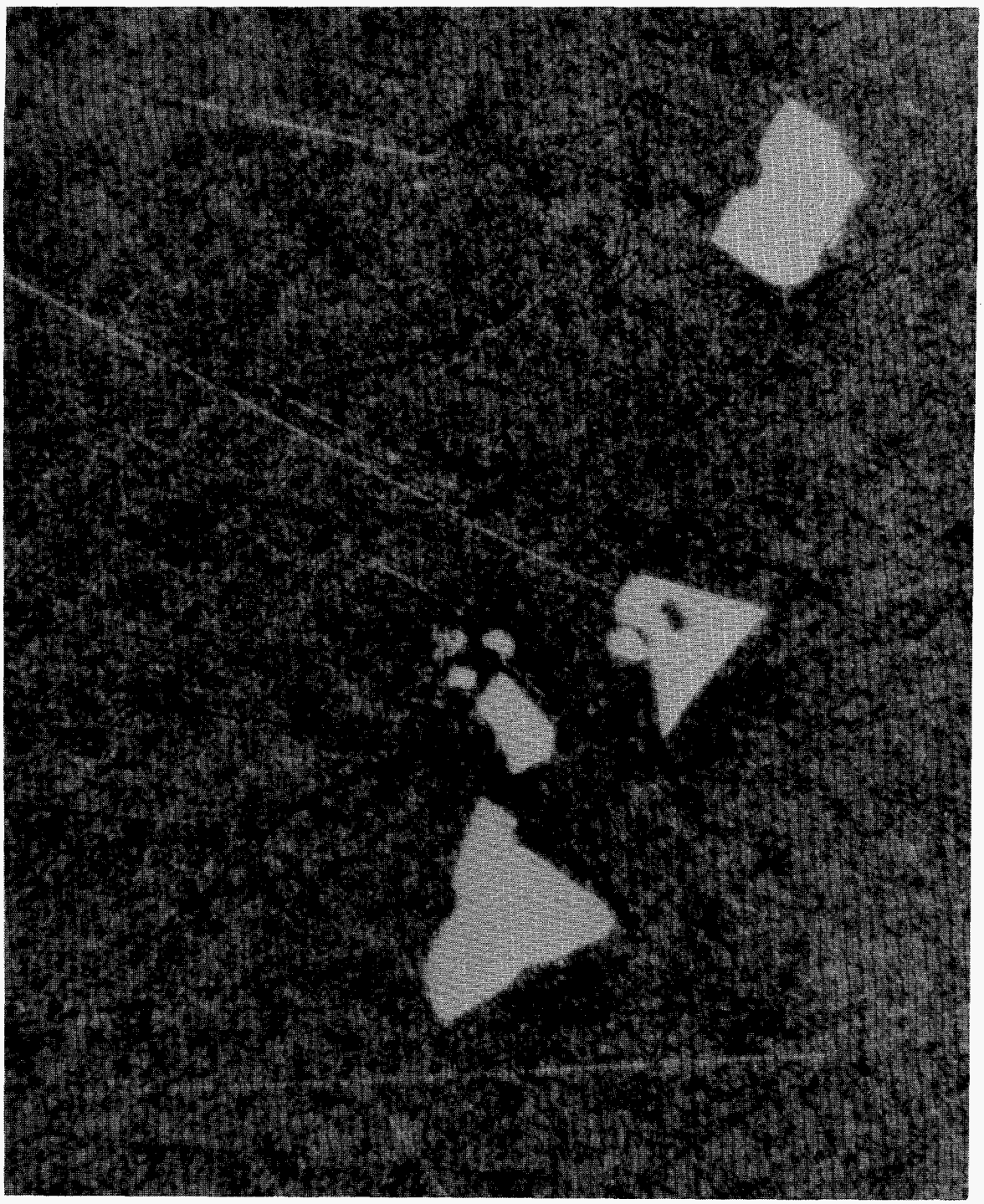


Figure B.55: Photomicrograph, 750X, of the Mid-Region of As-Polished Conditioned Specimen SFEC5,4378-S2-J1 in the Transverse Plane. The photo illustrates the different phase structures in the uranium matrix. The bright, almost regular geometric precipitates are most likely uranium carbide. PTL Log Q-1458, Photo # 277.

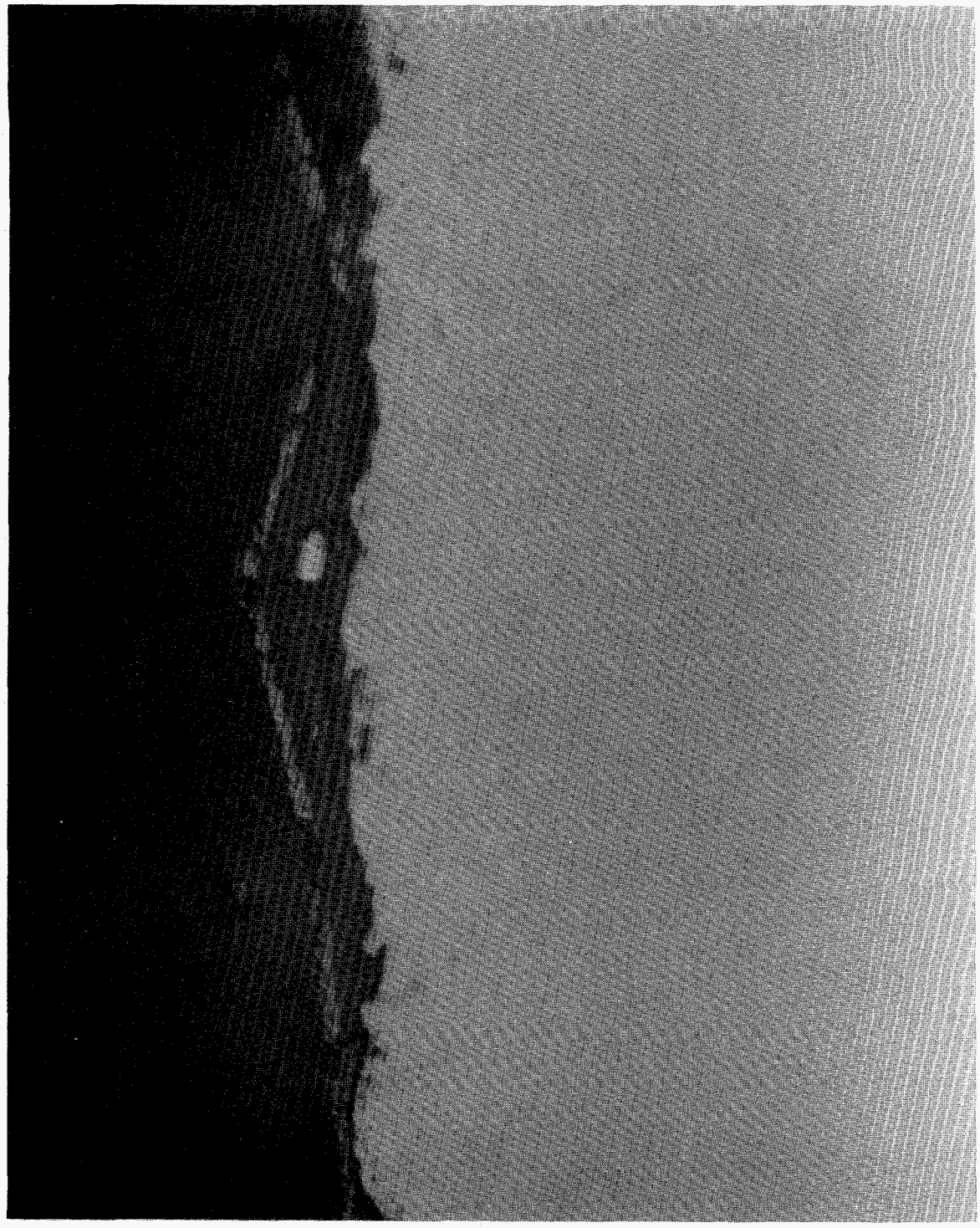


Figure B.56: Photomicrograph, 750X, of As-Polished Conditioned Specimen SFEC5,4378-S2-J1 in the Transverse Plane. The photo illustrates the loosely adhered oxide layer on the cut surface. PTL Log Q-1434, Photo # 253.

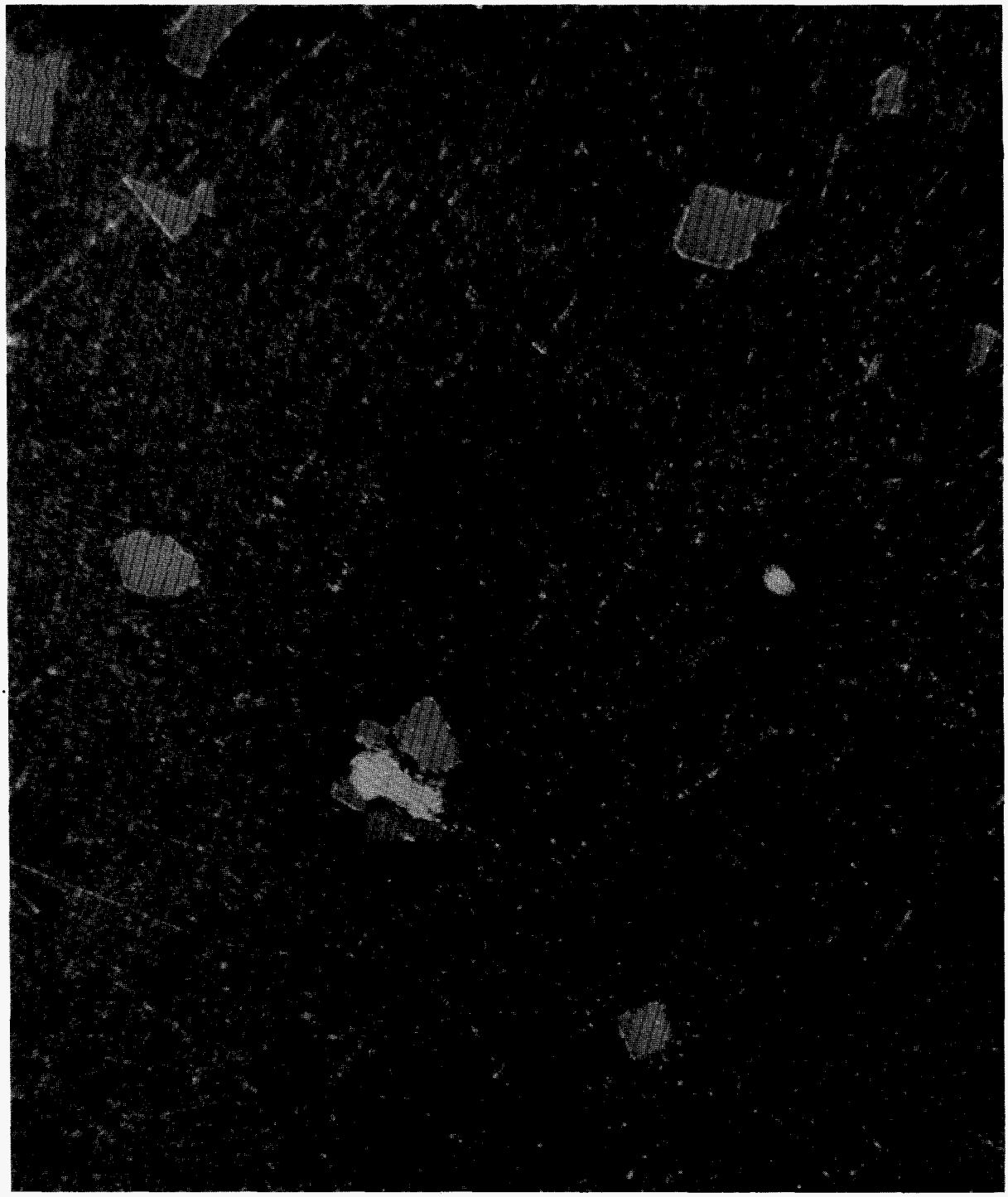


Figure B.57: Photomicrograph, 250X, of As-Polished Conditioned Specimen SFEC5,4378-S2-J1 in the Transverse Plane. This Figure shows a polarized image of the carbide inclusions in the uranium matrix. PTL Log Q-1456, Photo # 275.

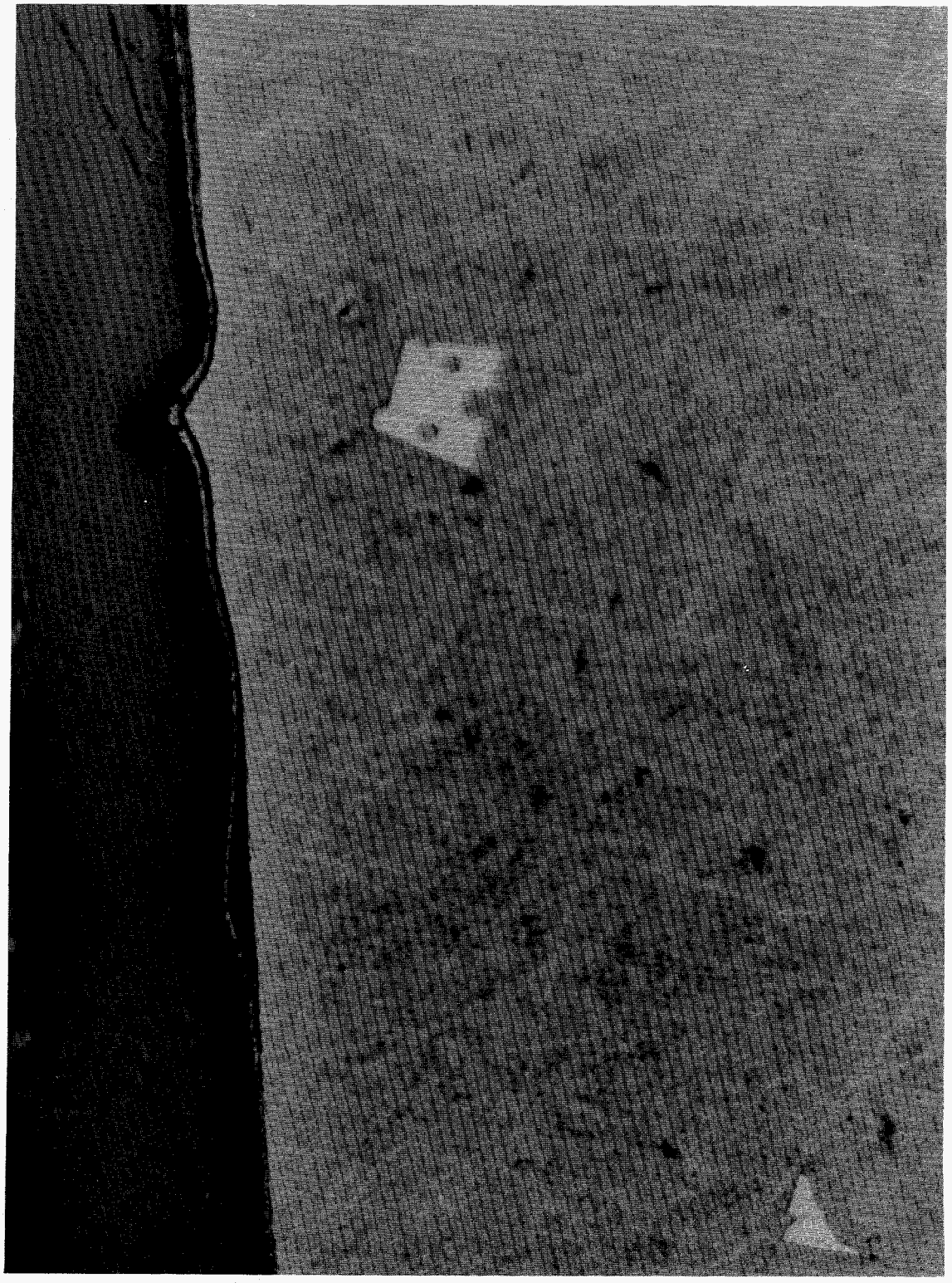


Figure B.58: Photomicrograph, 250X, of the As-Polished Conditioned Specimen SFEC5,4378-S2-B in the Longitudinal Plane. The photo shows the oxide layer/uranium metal interface. PTL Log Q-1509, Photo # 327.

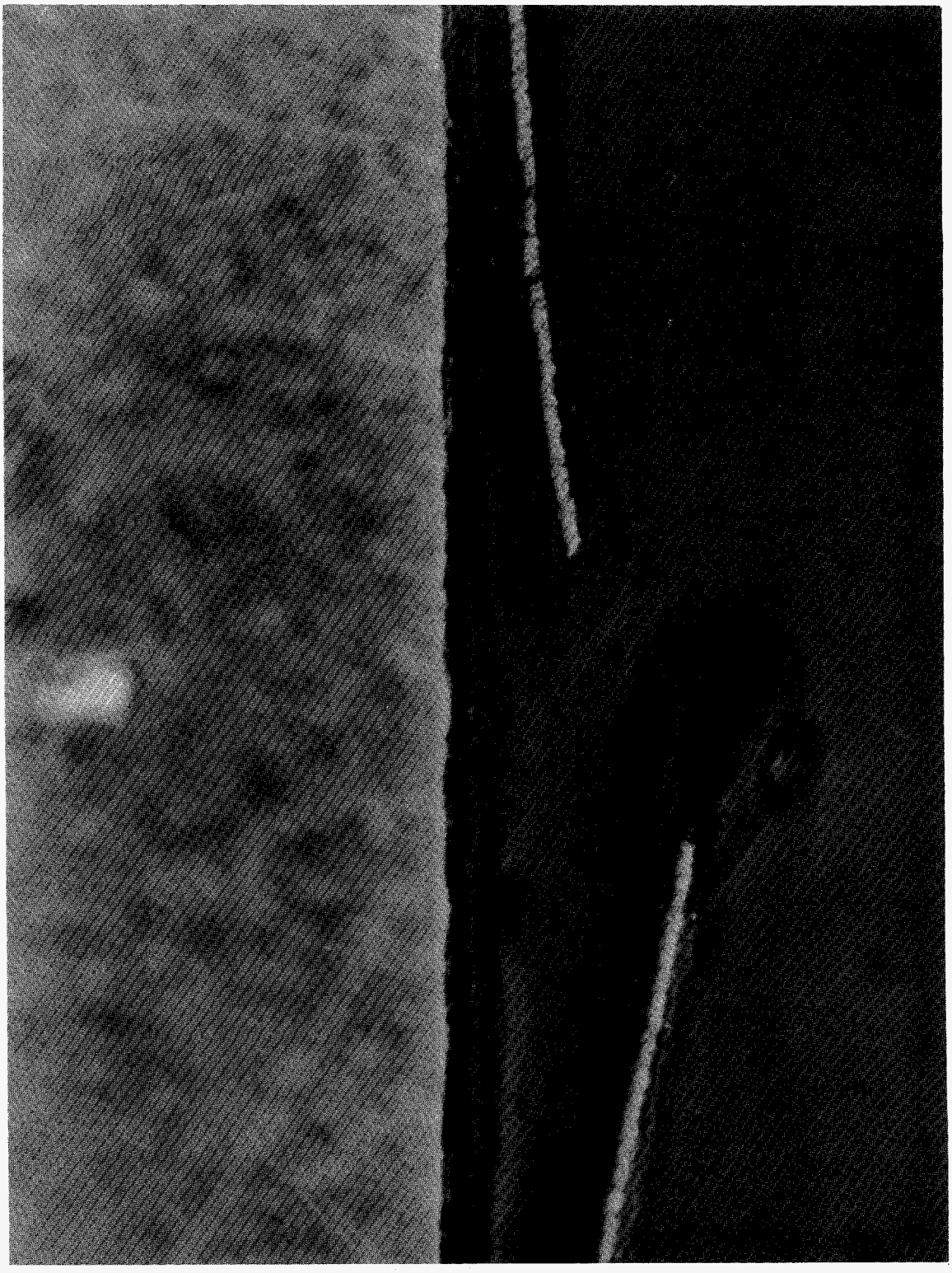


Figure B.59: Photomicrograph, 750X, of the As-Polished Conditioned Specimen SFEC5,4378-S2-B in the Longitudinal Plane, Illustrating the Spalling Off of the Oxide Layer. PTL Log Q-1511, Photo # 329.

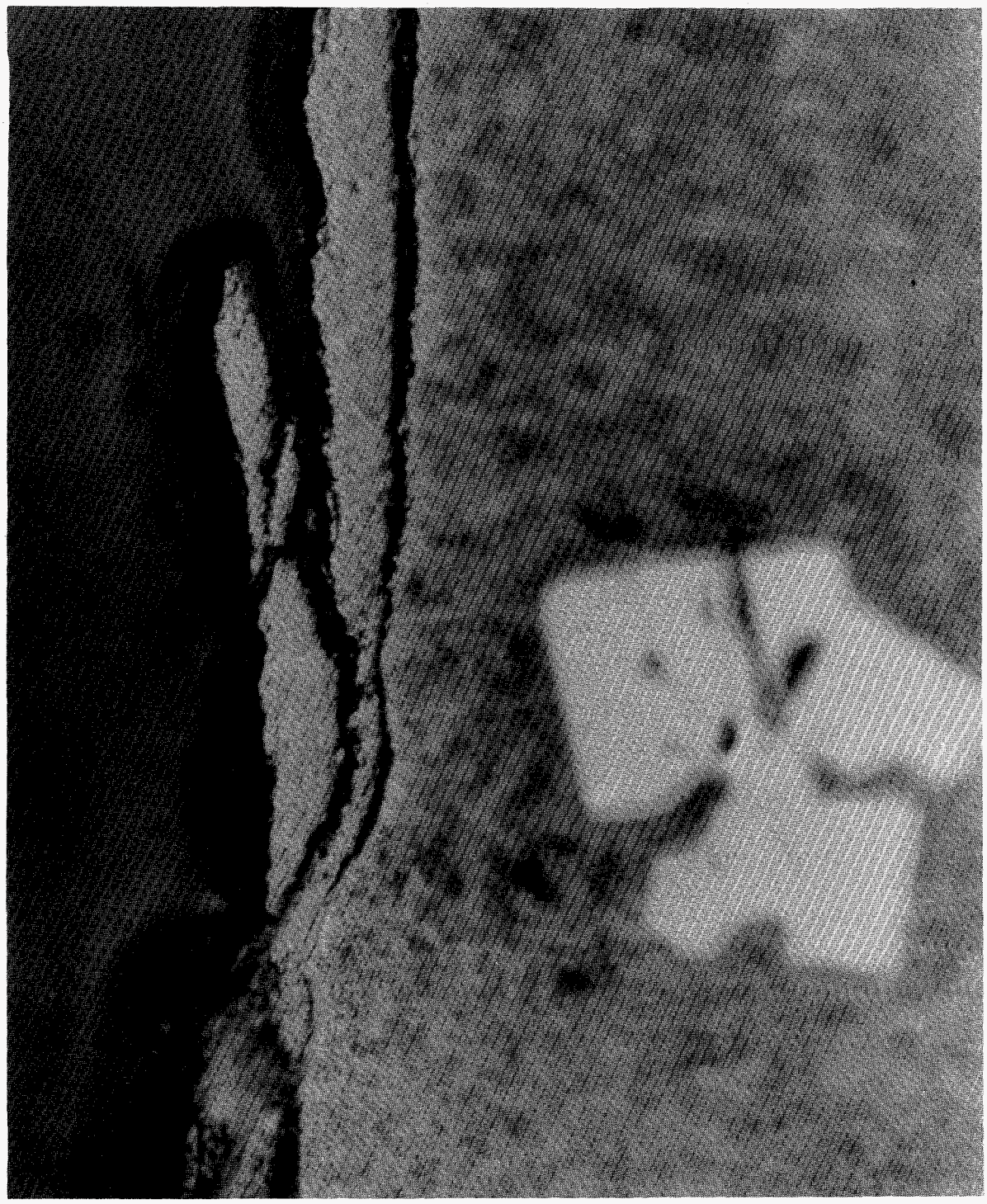


Figure B.60: Photomicrograph, 750X, of the As-Polished Conditioned Specimen SFEC5,4378-S2-B in theLongitudinal Plane. The photo shows another region of a loosely adhered oxide film on the metallic substrate. PTL Q-1512, Photo # 330.

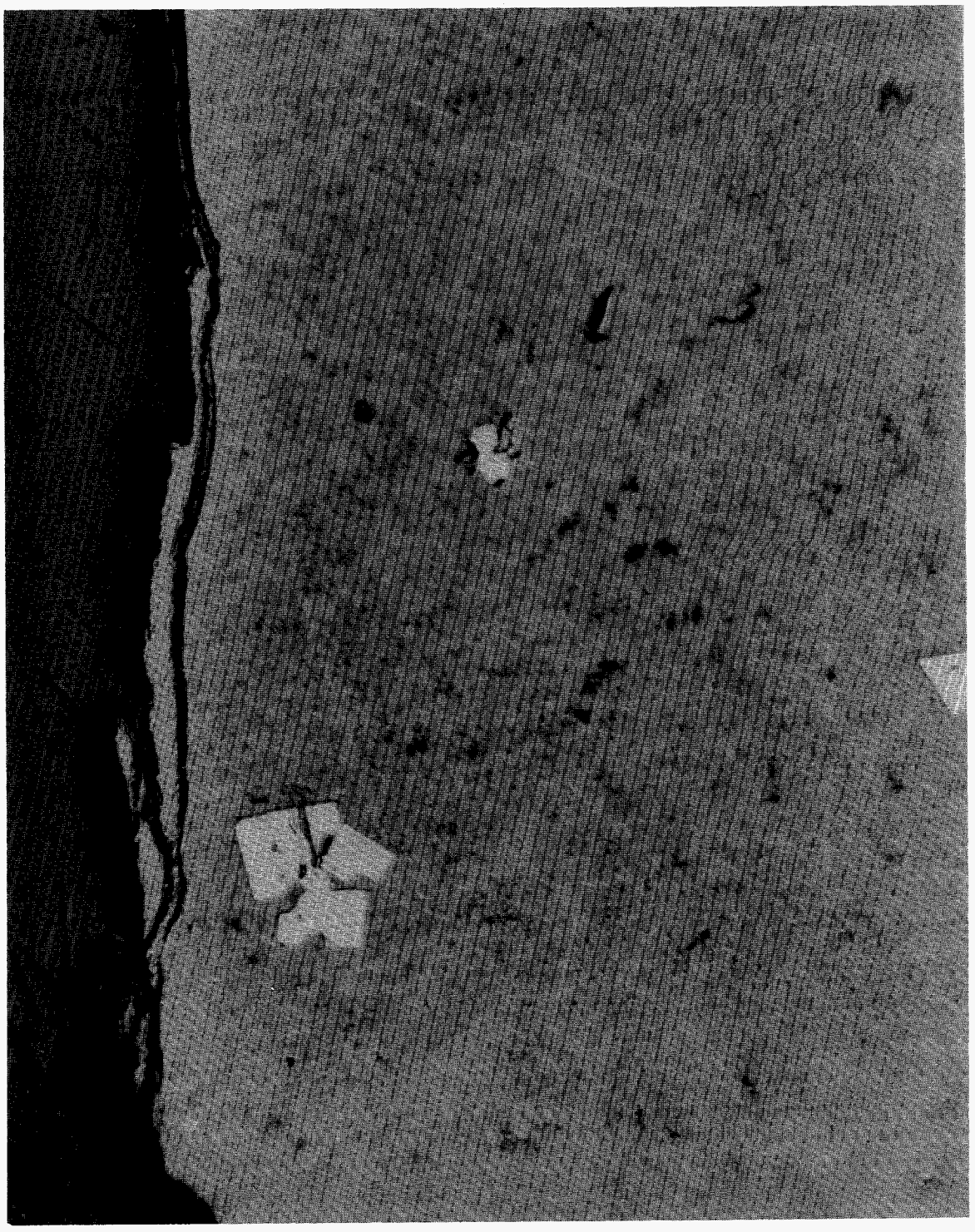


Figure B.61: Photomicrograph, 250X, of As-Polished Conditioned Specimen SFEC5,4378-S2-B in the Longitudinal Plane. The photo illustrates formation of loose oxide layer on the cut uranium surface. PTL Log Q-1507, Photo # 325.

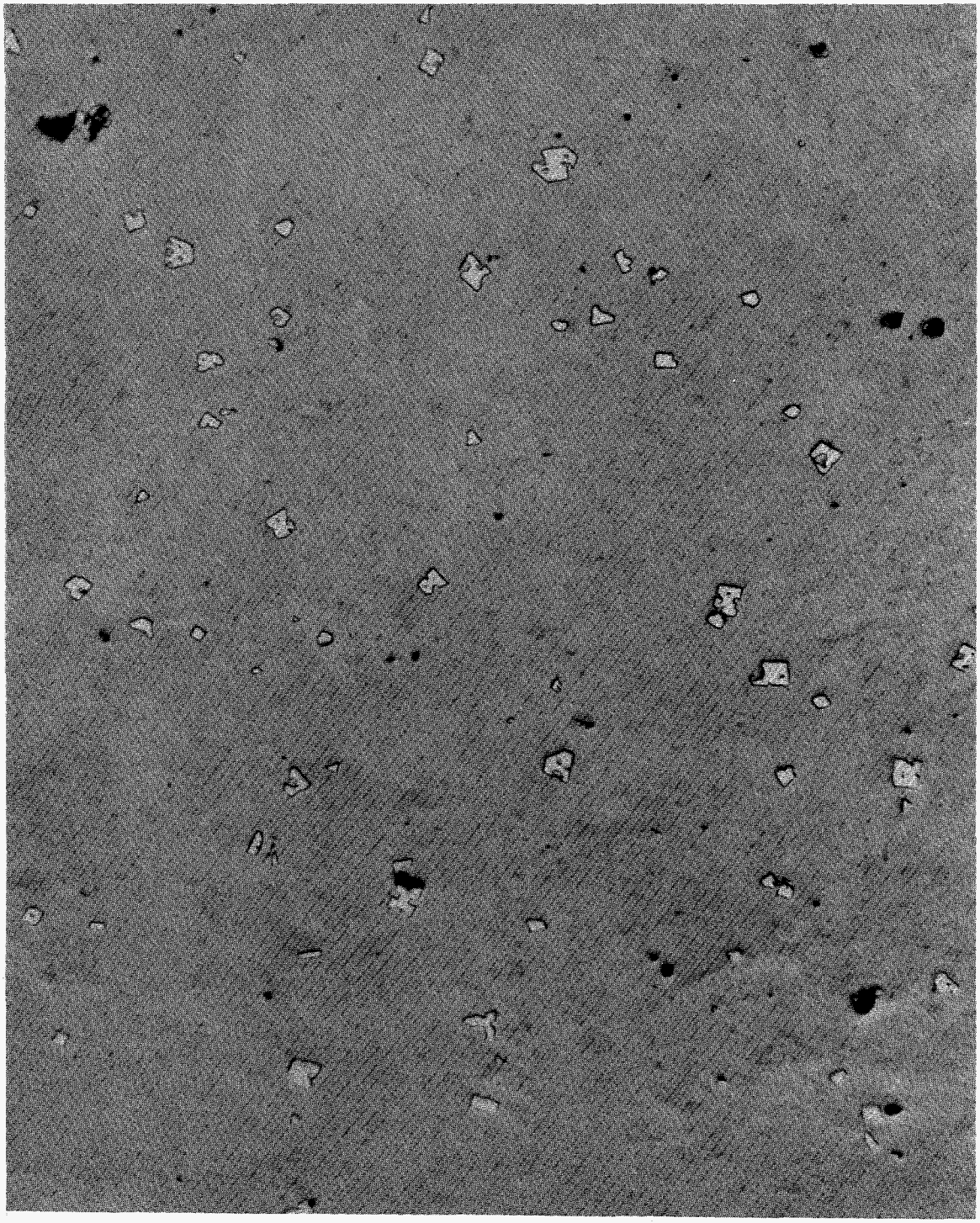


Figure B.62: Photomicrograph, 50X, of As-Polished Conditioned Specimen SFEC5,4378-S2-B in the Longitudinal Plane. The photo illustrates the different phase structures in the uranium matrix. PTL Log Q-1502, Photo # 320.

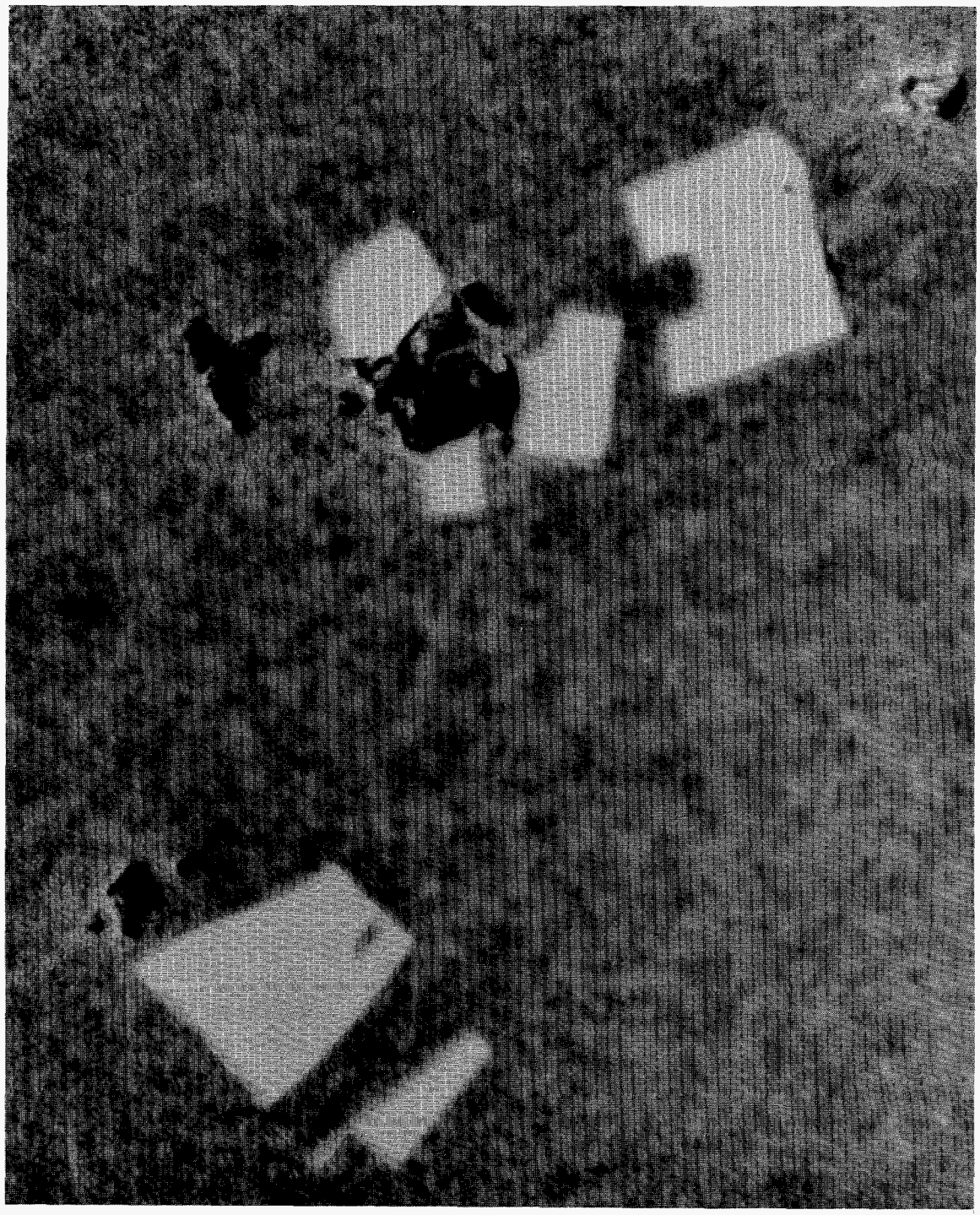


Figure B.63: Photomicrograph, 750X, of As-Polished Conditioned Specimen SFEC5,4378-S2-B in the Longitudinal Plane. The photo illustrates probable hydride inclusions as 'halo' and dark precipitates around the carbide inclusions. PTL Log Q-1497, Photo # 315.

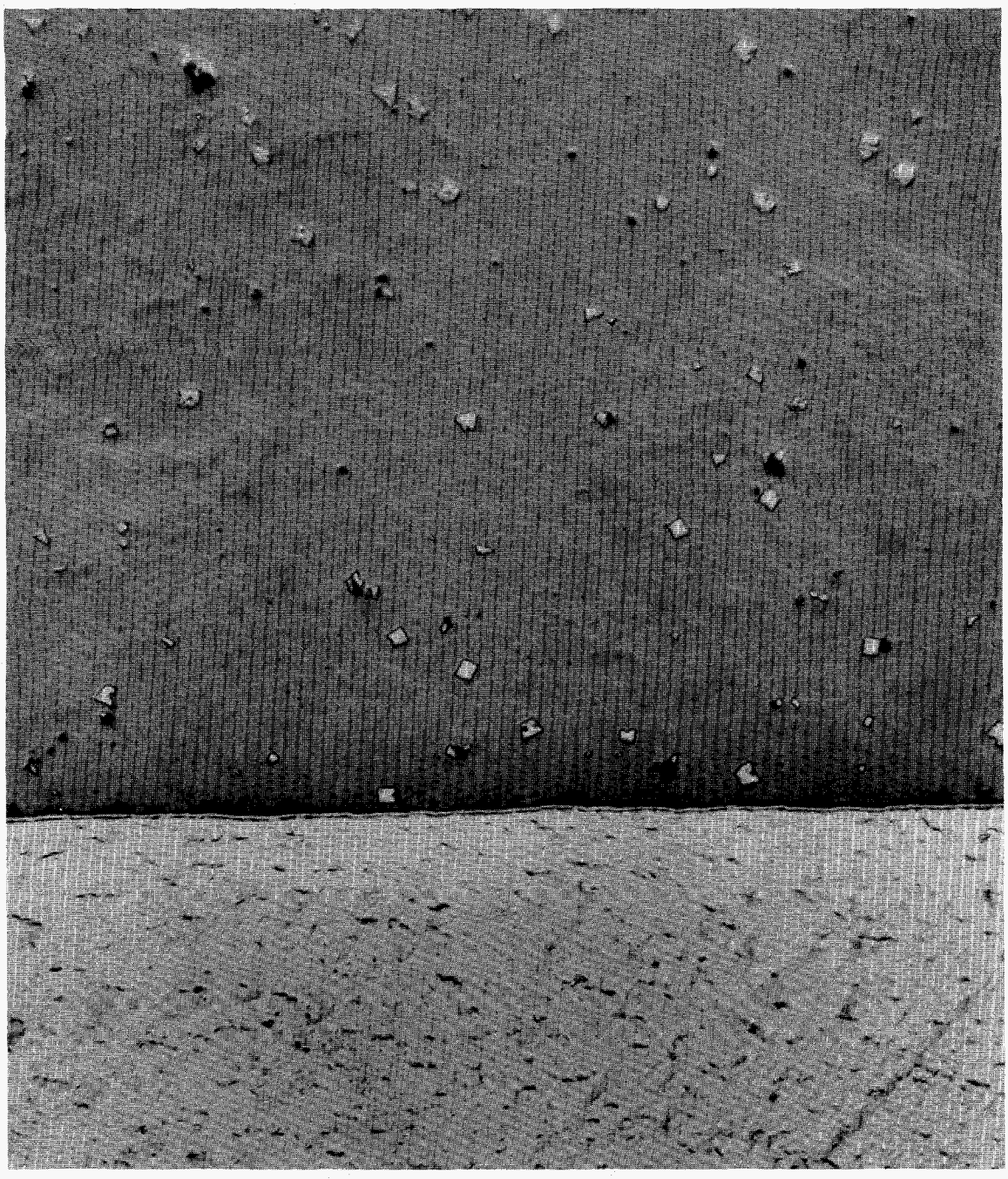


Figure B.64: Photomicrograph, 50X, of the Inner Cladding (Top)/Fuel (Bottom) Boundary of As-Polished Conditioned Specimen SFEC5,4378-S2-B in the Longitudinal Plane. The photo illustrates the inclusions in the uranium matrix. PTL Log Q-1505, Photo # 323.

Appendix C

SNF Specimen Identification and Status

Table C.1: SNF Specimen Status as of 20 Nov 95

Item No.	I.D. Number	Date Assigned	Description
1	SFEC5,4378	4-27-95	Dry weight and immersion density measured in F-cell.
2	SFEC5,4378-S1	5-10-95	Top 12-inch section of the fuel element in SFEC #5, 4378. Later sub-sectioned into 5-S1A and 5-S1B.
3	SFEC5,4378-S1-CLAD1	8-24-95 8-29-95 9-06-95	Cladding sample bent-off from mid-region (~130° - 180° orientation) of damaged area at top end of element; Weighed and used in **Furnace Run #8** (8/30); Weighed in I-cell; Disposition pending.
4	SFEC5,4378-S1-CLAD2	8-24-95	Cladding sample bent-off from left side (~180° - 260° orientation) of damaged area at top end of element. Later snapped into two length-wise sections, **-CLAD2-A** and **-CLAD2-B**.
5	SFEC5,4378-S1-CLAD2-A	8-28-95	Cladding sample snapped-off of **-CLAD2** (~ ½); Disposition pending.
6	SFEC5,4378-S1-CLAD2-B	8-28-95	Cladding sample snapped-off of **-CLAD2** (~ ½); Disposition pending.
7	SFEC5,4378-S1-EC	10-19-95 12-04-95	Top End Cap; Transferred to large storage basin (stored in SFEC #5).
8	SFEC5,4378-S1-L1	10-20-95 10-23-95	Oxidized fuel from damaged end of 5-S1 (~220° to 270°): Weighed in I-cell (36.0 g); stored under argon; Disposition pending.
9	SFEC5,4378-S1-L2	10-20-95 10-23-95	Oxidized fuel from damaged end of 5-S1 (~90° and 280°); Weighed in I-cell (4.9 g); stored under argon; Disposition pending.
10	SFEC5,4378-S1-L3	10-27-95	Fines and fall-out from sectioning of 5-S1 to obtain 5-S1A; Collection continued during sectioning of 5-S1A; Disposition pending.

Item No.	I.D. Number	Date Assigned	Description
11	SFEC5,4378-S1-PM1	9-05-95 9-11-95 10-02-95	Particulate matter (U_3O_8?) fallen from damage area of 5-S1 during collection of samples 5-S1-CLAD1 and 2; Weighed in I-cell; Transferred to Bldg. 325 for SEM and XRD analysis.
12	SFEC5,4378-S1A	10-23-95 11-15-95	Top 3 inches of Section 5-S1 after removing end cap. Began cutting into sub-sections after marking.
13	SFEC5,4378-S1A-A	11-28-95 01-24-96 01-29-96	~ 22° sector cut from 5-S1A; Transferred to Bldg. 325 for cutting into sections **-A1 & -A2**; Transferred new sections **5-S1A-A1 & -A2** to Bldg. 327.
14	SFEC5,4378-S1A-A1	01-29-96	22° sector, top half of **5-S1A-A**; Disposition pending.
15	SFEC5,4378-S1A-A2	01-29-96	22° sector, bottom half of **5-S1A-A**; Disposition pending.
16	SFEC5,4378-S1A-B	11-28-95 01-18-96 01-22-96	~ 22° sector cut from 5-S1A; Transferred to Bldg. 325 to be cut into sections **-B1 & -B2**; Transferred new sections **5-S1A-B1 & -B2** to Bldg. 327.
17	SFEC5,4378-S1A-B1	01-22-96 02-21-96 02-23-96	Transferred to Bldg. 327 (top half of **5-S1A-B**); Photographed for surface area measurement; Weighed in E-cell; **Furnace Run # 23** (conditioning test).
18	SFEC5,4378-S1A-B2	01-22-96 02-21-96 02-23-96	Transferred to Bldg. 327 (bottom half of **5-S1A-B**); Photographed for surface area measurement; Weighed in E-cell; **Furnace Run # 23** (conditioning test).
19	SFEC5,4378-S1A-C	11-28-95 01-09-96 01-10-96 01-10-96 01-18-96 01-23-96	~ 22° sector cut from 5-S1A; Weighed in I-cell; Photographed for surface area measurement; Used in **Furnace Run #18 [Conditioning Test #11]**; Photographed in G-cell; Weighed in I-cell.

Item No.	I.D. Number	Date Assigned	Description
20	SFEC5,4378-S1A-CD1	11-16-95	Cladding pieces that fell away during sectioning of 5-S1A. Disposition pending.
21	SFEC5,4378-S1A-D	11-29-95 12-18-95 01-05-96 01-05-96 01-08-96 01-09-96 01-10-96	~ 22° sector cut from 5-S1A; Transferred to Bldg. 325 to be cut prior to ignition testing; Transferred to Bldg. 327 (NOT sectioned); Photographed for surface area measurement; Weighed in I-cell; Used in **Furnace Run #17** [ignition test]; Residue photographed in G-cell and weighed in I-cell.
22	SFEC5,4378-S1A-E	11-29-95 01-04-96	~ 90° sector of SFEC5,4378-S1A; Cut from 5-S1A; Disposition pending.
23	SFEC5,4378-S1A-F	11-29-95 01-08-96	~ 22° sector of SFEC5,4378-S1A: Cut from 5-S1A; broke into pieces -F1 & -F2 while cutting.
24	SFEC5,4378-S1A-F1	01-08-96	Top section of SFEC5,4378-S1A-F; Disposition pending.
25	SFEC5,4378-S1A-F2	01-08-96 01-17-96 01-18-96 01-30-96 02-05-96	Bottom section of SFEC5,4378-S1A-F; Photographed for surface area measurement; Weighed in I-cell; Weighed in G-cell; used in **Furnace Run #20** (conditioning); Weighed in G-cell and I-cell.
26	SFEC5,4378-S1A-G	11-29-95 01-08-96 02-05-96 02-06-96 02-09-96 02-16-96 02-20-96	~ 22° sector of SFEC5,4378-S1A; Cut from 5-S1A; Photographed for surface area measurement; Transferred to G-cell for **Furnace Run #21** (conditioning); Weighed in G-cell for **Furnace Run #21** (conditioning); Weighed in G-cell; conducted **Furnace Run #22** (ignition); Photographed and weighed in G-cell.
27	SFEC5,4378-S1A-H	11-29-95 01-08-96 01-18-96 01-19-96 01-22-96 01-23-96	~ 22° sector of SFEC5,4378-S1A; Cut from 5-S1A; Photographed for surface area measurement; Weighed in I-cell; Conducted **Furnace Test #19** (ignition test); Photographed in G-cell and weighed in I-cell.

Item No.	I.D. Number	Date Assigned	Description
28	SFEC5,4378-S1A-I	11-29-95 01-08-96	~ 22° sector of SFEC5,4378-S1A; Cut from 5-S1A; Disposition pending.
29	SFEC5,4378-S1A-J	11-29-95 01-08-96	~ 90° sector of SFEC5,4378-S1A; Cut from 5-S1A; Disposition pending.
30	SFEC5,4378-S1AB	11-29-95 01-04-96	Two half-sections consisting of the bottom 1-inch of 5-S1A; Cut from 5-S1A; re-designated as 5-S1AB-1 & 5-S1AB-2.
31	SFEC5,4378-S1AB-1	01-04-96	0° to 180° sector of 5-S1AB; Disposition pending.
32	SFEC5,4378-S1AB-2	01-04-96	180° to 360° sector of 5-S1AB; Disposition pending.
33	SFEC5,4378-S1B	10-23-95 12-04-95	Bottom 9 inches of Section 5-S1 after removing 5-S1A; Transferred to large storage basin.
34	SFEC5,4378-S2	5-10-95	Middle, 1-inch, ring-section of the fuel element; Later sub-sectioned into specimens 5-S2-A thru -J.
35	SFEC5,4378-S2-A	5-12-95 5-24-95 7-21-95	~22° sector; Mounted transversely for metallographic examination; Heat tinted for hydride detection.

Item No.	I.D. Number	Date Assigned	Description
36	SFEC5,4378-S2-B	5-15-95 7-05-95 7-06-95 7-11-95 10-10-95 10-13-95	~22° sector; Photographed for surface area; Weighed in I-cell; used in **Furnace Run #2**; Weighed in I-cell after furnace run; Mounted longitudinally for metallographic examination; First heat tint for hydride detection.
37	SFEC5,4378-S2-C	5-15-95 5-25-95 8-01-95	~22° sector; Mounted longitudinally for metallographic examination; Heat tinted for hydride detection.
38	SFEC5,4378-S2-C1	7-11-95 7-19-95	Furnace residue from **Furnace Run #2**; Transferred to Bldg. 325 for XRD analysis.
39	SFEC5,4378-S2-D	5-16-95 7-07-95 7-18-95 11-09-95 11-10-95 11-13-95	~22° sector; Photographed for surface area; Weighed in I-cell and used in **Furnace Run #4**; Photographed for surface area; Weighed in I-cell; used in **Furnace Run #14 [Ignition Test #4]** Photographed in G-cell; weighed sample and residue (5-S2-FR8) in I-cell.
40	SFEC5,4378-S2-E	5-16-95	~90° sector; later subsectioned (4).
41	SFEC5,4378-S2-E1	8-02-95 8-17-95 8-21-95 8-22-95 8-23-95 8-29-95	~22° sector; Removed cladding; Photographed for surface area measurement; Weighed in I-cell after de-cladding; Used in **Furnace Run #7**; Weighed in I-cell with small particle of debris.
42	SFEC5,4378-S2-E1-CD1	8-17-95 10-06-95 10-09-95 10-10-95 10-11-95	Cladding cut from O.D. side of **5-S2-E1**; De-fueled in HNO_3 (20 wt.% solution); Photographed for surface area and weighed in I-cell; Used in **Furnace Run #10**; Weighed in I-cell; Archived in E-cell carousel.

Item No.	I.D. Number	Date Assigned	Description
43	SFEC5,4378-S2-E1-CD2	8-18-95 10-06-95 10-19-95	Cladding cut from I.D. side of **5-S2-E1**; De-fueled in HNO_3 (20 wt.% solution); Weighed in I-cell; Archived in E-cell carousel.
44	SFEC5,4378-S2-E2	8-03-95 8-21-95 8-23-95 9-27-95 10-09-95	~22° sector; Removed cladding; Photographed (8/23) & weighed (9/27) after de-cladding; Used in **Furnace Run #9**; Weighed in I-cell; Furnace residue is "5-S2-FR4".
45	SFEC5,4378-S2-E2-CD1	8-21-95 10-04-95 10-19-95	Cladding cut from O.D. side of **5-S2-E2**; De-fueled in HNO_3 (20 wt.% solution); Weighed in I-cell; Archived in E-cell carousel.
46	SFEC5,4378-S2-E2-CD2	8-21-95 10-04-95 10-10-95 10-19-95	Cladding cut from I.D. side of **5-S2-E2**; De-fueled in HNO_3 (20 wt.% solution); Photographed for surface area measurement; Weighed in I-cell; Archived in E-cell carousel.
47	SFEC5,4378-S2-E3	8-03-95 9-08-95	~22° sector; Subsectioned transversely into **-E3A and -E3B** for ignition testing.
48	SFEC5,4378-S2-E3A	9-08-95 9-12-95 10-24-95 10-25-95 10-27-95	~22° sector; top-half of **5-S2-E**; Photographed for surface area measurement; Weighed in I-cell for **Furnace Run #11 [Ignition Test #1]**; Used in **Ignition Test #1**; Weighed in I-cell; furnace residue is **"5-S2-FR5"**;
49	SFEC5,4378-S2-E3B	9-08-95 9-12-95	~22° sector; bottom-half of 5-S2-E3 (orientation NOT marked); Photographed for surface area. Disposition pending.
50	SFEC5,4378-S2-E4	8-03-95 9-08-95	~22° sector; Subsectioned transversely into **-E4A and -E4B** for ignition testing.

Item No.	I.D. Number	Date Assigned	Description
51	SFEC5,4378-S2-E4A	9-08-95 9-12-95 10-30-95 11-01-95 11-02-95	<22° sector; top-half of 5-S2-E4; Photographed for surface area measurement; Weighed in I-cell; Used in **Furnace Run #12 [Ignition Test #2]**; Photographed in G-cell; weighed in I-cell; Furnace residue is "**5-S2-FR6**";
52	SFEC5,4378-S2-E4B	9-08-95 9-13-95	<22° sector; bottom-half of 5-S2-E4 (orientation NOT marked); Photographed for surface area measurement. Disposition pending.
53	SFEC5,4378-S2-F	5-16-95	~90° sector; later subsectioned into **-F1 through -F4**.
54	SFEC5,4378-S2-F1	7-31-95 8-02-95 8-07-95 8-11-95 01-10-96 01-17-96	~22° sector; Photographed for surface area measurement; Weighed in I-cell; used in **Furnace Run #6**; Weighed in I-cell; furnace residue is "**5-S2-FR2**". Transferred to Bldg. 325 to be cut into **5-S2-F1A & -F1B**; New specimens **5-S2-F1A & -F1B** transferred to Bldg. 327.
55	SFEC5,4378-S2-F1A	01-17-96 02-23-96	Transferred to Bldg. 327 (top half of **5-S2-F1**); Weighed in G-cell for accountability; mounted sample for metallographic examination.
56	SFEC5,4378-S2-F1B	01-17-96 02-23-96	Transferred to Bldg. 327 (Bottom half of **5-S2-F1**); Weighed in G-cell for accountability; Metallographic examination is pending.
57	SFEC5,4378-S2-F2	8-01-95 8-03-95 8-07-95 8-11-95	~22° sector; Photographed for surface area measurement; Weighed in I-cell; used in **Furnace Run #6**; Weighed in I-cell; furnace residue is "**5-S2-FR2**".
58	SFEC5,4378-S2-F3	8-01-95 8-03-95 12-04-95	~22° sector; Photographed for surface area measurement; Transferred to Bldg. 325 for sub-sectioning and TGA.

Item No.	I.D. Number	Date Assigned	Description
59	SFEC5,4378-S2-F4	8-01-95 8-04-95 12-20-95 01-02-96	~22° sector; Photographed for surface area measurement. Weighed in I-cell; used in **Furnace Run #16**; Weighed in I-cell.
60	SFEC5,4378-S2-G	5-16-95 7-07-95 7-24-95 7-27-95	~22° sector; Photographed for surface area measurement; Weighed in I-cell; used in **Furnace Run #5**; Weighed in I-cell; Transfer to Bldg. 325 for pre-metallog. sectioning is pending.
61	SFEC5,4378-S2-H	5-16-95 6-27-95 6-28-95 7-06-95 10-31-95 11-02-95 11-03-95 11-06-95	~22° sector; Photographed for surface area measurement; Weighed in I-cell; used in **Furnace Run #1**; Weighed in I-cell after test; Photographed for surface area prior to **Ignition Test #3**; Weighed in I-cell; Used in **Furnace Run #13 [Ignition Test #3]**; Photogaphed in G-cell; weighed sample and residue (**5-S2-FR7**) in I-cell.
62	SFEC5,4378-S2-I	5-16-95 7-05-95 7-06-95 7-11-95 11-15-95 11-17-95 11-20-95	~22° sector; Photographed for surface area measurement; Weighed in I-cell; used in **Furnace Run #2**; Weighed in I-cell; Photographed for surface area measurement; Weighed in I-cell; used in **Furnace Run #15 [Ignition #3R]**; Photographed in G-cell; weighed in I-cell after ignition test.
63	SFEC5,4378-S2-J	5-16-95 6-27-95 6-28-95 7-06-95 8-23-95	~22° sector; Photographed for surface area measurement; Weighed in I-cell; used in **Furnace Run #1**; Weighed in I-cell; Sub-sectioned into **-J1 & -J2** for metallographic examination.
64	SFEC5,4378-S2-J1	8-23-95 8-31-95 9-22-95	~22° sector; top-half of 5-S2-J;weighed; Transversely mounted for post-run met. exam; Heat tinted for hydride detection.

Item No.	I.D. Number	Date Assigned	Description
65	SFEC5,4378-S2-J2	8-23-95 8-31-95 10-04-95	~22° sector; bottom-half of 5-S2-J; weighed in I-cell; Longitudinally mounted for post-run met. exam; Heat tinted for hydride detection.
66	SFEC5,4378-S2-FR2	8-11-95 8-17-95	Furnace residue found after **Furnace Run # 6**; Transferred to 325 Bldg. for XRD analysis.
67	SFEC5,4378-S2-FR3	9-20-95 9-28-95 10-02-95	Debris collected from first furnace tube after tube replaced; Weighed in I-cell; Transferred to Bldg. 325 for SEM and XRD analysis.
68	SFEC5,4378-S2-FR4	10-09-95 10-10-95 01-29-96	Residue from 5-S2-E2 found in boat after **Furnace Run #9**; Weighed in I-cell; Transferred to Bldg. 325 for XRD analysis.
69	SFEC5,4378-S2-FR5	10-26-95 10-30-95 01-29-96	Residue from 5-S2-E3A found in boat after **Furnace Run #11 [Ignition Test #1]**; Weighed in I-cell; Transferred to Bldg. 325 for XRD analysis.
70	SFEC5,4378-S2-FR6	11-02-95 11-02-95	Residue from 5-S2-E4A found in boat after **Furnace Run #12 [Ignition Test #2]**; Weighed in I-cell; Disposition pending.
71	SFEC5,4378-S2-FR7	11-06-95 11-06-95	Furnace residue from 5-S2-H after **Furnace Run #13 [Ignition Test #3]**; Weighed in I-cell; Disposition pending.
72	SFEC5,4378-S2-FR8	11-13-95 11-13-95	Furnace residue from 5-S2-D after **Furnace Run #14 [Ignition Test #4]**; Weighed in I-cell; Disposition pending.

Item No.	I.D. Number	Date Assigned	Description
73	SFEC5,4378-S2-FR9	11-20-95	Furnace residue from 5-S2-I after **Furnace Run #15 [Ignition Test #3R]**; Disposition pending.
74	SFEC5,4378-S2-FR10	11-20-95	Particulate residue from G-cell floor (miscellaneous sample droppings). Disposition pending.
75	SFEC5,4378-S3	5-11-95 12-04-95	Bottom 13-inch section of the fuel element; Transferred to large storage basin (in SFEC #5).
76	SFEC8,1990	02-08-96	Weighed in F-cell.
77	SFEC10,4366	12-15-95	Weighed in F-cell.
78	SFEC10,4366-1	12-06-95 01-12-96	Particulate from SFEC # 10 water, collected on Whatman 5 μm cellulose nitrate filter (~ 4 liters, no rinse); Transferred to Bldg. 325 for analysis.
79	SFEC10,4366-2	12-07-95 01-12-96	Particulate from SFEC # 10 D.I. water, collected on Whatman 5 μm cellulose nitrate filter (~ 2 liters, no rinse); Transferred to Bldg. 325 for analysis.
80	SFEC10,4366-3	12-07-95 01-12-96	Particulates from SFEC # 10 D.I. water, collected on Whatman 5 μm cellulose nitrate filter (~ 2 liters, no rinse); Transferred to Bldg. 325 for analysis.
81	SFEC10,4366-4	12-07-95 01-12-96	"Glob" of loose particulates scraped from bottom of element in SFEC # 10, collected on Whatman 5 μm cellulose nitrate filter; Transferred to Bldg. 325 for analysis.

Item No.	I.D. Number	Date Assigned	Description
82	SFEC10,4366-5	12-07-95 01-12-96	Particulates from flush of SFEC # 10 with D.I. water (~ 2 liters), collected on Whatman 5 μm cellulose nitrate filter; Transferred to Bldg. 325 for analysis.
83	SFEC10,4366-6	12-07-95 01-12-96	SFEC # 10 particulates from rinsing filter funnel with D.I. water, collected on Whatman 5 μm cellulose nitrate filter; Transferred to Bldg. 325 for analysis.
84	SFEC10,4366-L1	02-02-96	Loose fuel particles from damaged end collected prior to sectioning; Disposition pending.
85	SFEC10,4366-S1-A	*	~22° sector, top 1-inch.
86	SFEC10,4366-S1-B	*	~22° sector, top 1-inch.
87	SFEC10,4366-S1-C	*	~22° sector, top 1-inch.
88	SFEC10,4366-S1-D	*	~22° sector, top 1-inch.
89	SFEC10,4366-S2-A	*	~22° sector, ~1 to 2 inches below top end cap.
90	SFEC10,4366-S2-B	*	~22° sector, ~1 to 2 inches below top end cap.
91	SFEC10,4366-S2-C	*	~22° sector, ~1 to 2 inches below top end cap.
92	SFEC10,4366-S2-D	*	~22° sector, ~1 to 2 inches below top end cap.
93	SFEC10,4366-S3-A	*	~22° sector, ~2 to 3 inches below top end cap.

Item No.	I.D. Number	Date Assigned	Description
94	SFEC10,4366-S3-B	*	~22° sector, ~2 to 3 inches below top end cap.
95	SFEC10,4366-S3-C	*	~22° sector, ~2 to 3 inches below top end cap.
96	SFEC10,4366-S3-D	*	~22° sector, ~2 to 3 inches below top end cap.
97	SFEC10,4366-S3-E	*	~90° sector, ~2 to 3 inches below top end cap.
98	SFEC10,4366-S3-F	*	~22° sector, ~2 to 3 inches below top end cap.
99	SFEC10,4366-S3-G	*	~22° sector, ~2 to 3 inches below top end cap.
100	SFEC10,4366-S3-H	*	~22° sector, ~2 to 3 inches below top end cap.
101	SFEC10,4366-S3-I	*	~22° sector, ~2 to 3 inches below top end cap.
102	SFEC10,4366-S3-J	*	~90° sector, ~2 to 3 inches below top end cap.
103	SFEC10,4366-S4	*	Residual fuel element, ~3 to 12.5 inches from top end cap.
104	SFEC10,4366-S5-A	*	~22° sector, mid-length.
105	SFEC10,4366-S5-B	*	~22° sector, mid-length.
106	SFEC10,4366-S5-C	*	~22° sector, mid-length.
107	SFEC10,4366-S5-D	*	~22° sector, mid-length.
108	SFEC10,4366-S5-E	*	~22° sector, mid-length.

Item No.	I.D. Number	Date Assigned	Description
109	SFEC10,4366-S5-F	*	~22° sector, mid-length.
110	SFEC10,4366-S5-G	*	~22° sector, mid-length.
111	SFEC10,4366-S5-H	*	~22° sector, mid-length.
112	SFEC10,4366-S5-I	*	~22° sector, mid-length.
113	SFEC10,4366-S5-J	*	~22° sector, mid-length.
114	SFEC10,4366-S5-K	*	~22° sector, mid-length.
115	SFEC10,4366-S5-L	*	~22° sector, mid-length.
116	SFEC10,4366-S5-M	*	~22° sector, mid-length.
117	SFEC10,4366-S5-N	*	~22° sector, mid-length.
118	SFEC10,4366-S5-O	*	~22° sector, mid-length.
119	SFEC10,4366-S5-P	*	~22° sector, mid-length.

* denotes that the I. D. Number is "To Be Assigned".

C.1 Methodology Used to Assign the I.D. Numbers

The following methodology is recommended and/or has been applied, where practicable, to assign sample I.D. numbers for specimen identification and tracking.

Sample I.D. Number = (SFEC number) - (K-basin canister number) - (Section number as first cut from the element, or a **brief** mnemonic designator and sequence number if other than a section of the element) - (Subsection or subsample number, etc.) - (Sub-subsection, etc.) - etc. For example:

SFEC5,4378-S2-J2 = the second (J2) subsection cut from section **J**, where J was cut from Section **2** (S2) of the element that was removed from K-basin canister # **4378** and shipped in **SFEC #5** (This typically reduces to the short form "5-S2-J2".);

SFEC5,4378-S2-FR2 = the second sample of **R**esidue found in the **F**urnace (FR2) after the furnace run of a sample cut from section S2 of the element in SFEC #5 ...;

SFEC4,4321-S1-PM1 = the first sample of **P**articulate **M**atter collected from section S1, such as that which might fall out while cutting a piece of cladding from section S1;

SFEC4,4321-SV1 = the first sample of material from the element in SFEC #4, etc., which is collected in the sieve after pouring the canister water through the Sie**V**e (SV1);

SFEC4,4321-FT1 = the first Fil**T**er (FT1) from filtering water from canister SFEC #4;

SFEC4,4321-LD2 = the second sample of Liqui**D** (LD2) collected from SFEC #4;

SFEC5,4378-S1-CD2-B = the second subsection of cladding (B) taken from the second section of Cla**D**ding (CD2) which, in turn, was taken from the top section (S1) of the element in SFEC #5. The preferred short form is **5-S1-CD2-B**.

Appendix D

Test Instructions

CONTROLLED COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 7

Title: Sectioning of SNF Sample in SFEC #5 for Metallographic and Furnace Testing Specimens

Work Location: 327 (Building/Room or "General")	**TI No.:** SNF-CT-003
Author: John Abrefah	**Effective Date:** 05/01/95 **Supersedes Date:**

Signatures:

Author _____ (Signature) 05-01-95 (Date)

Technical Reviewer _____ (Signature) 5-2-95 (Date)

Approval:

Project Manager, HTSP _____ (Paul J. Turner) 5-2-95 (Date)

Concurrences:

PTL Manager _____ (James M. Seay) 5/2/95 (Date)

HSNFP Charact. Manager _____ (Ronald P. Omberg) 5/2/95 (Date)

Purpose/Scope The purpose of this test instruction is to direct the sectioning of the Spent Nuclear Fuel (SNF) samples shipped from 105-K basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF element SFEC5, 4378 (outer element with split open end cap).

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

1.0 Sectioning of the SNF Element SFEC5,4378

Element SFEC5,4378 will be sectioned using the milling machine in F-cell. Stainless steel specimen containers will be used to store the cut specimens under high purity argon.

Note: The SNF sample contained in the SFEC could be friable (i.e., easily crushed or damaged) due to irradiation and degradation in storage. Extreme care should be taken in handling to avoid breaking the fuel sample.

1.1 Provide video coverage of the activities as directed by cognizant scientist.

1.2 Prior to removing the SNF sample for the following activities, measure and record the cell's atmospheric conditions .

1.3 Remove the SNF element from the SFEC and mount it on the milling machine for the first cut.

1.4 Mark the 0° and 90° (clockwise) orientations along the entire length of the SNF element. The 0° should be at the same orientation as was used in the visual examinations.

1.5 Measure 14 inches (35.56 cm) from the undamaged end of the SNF element and mark that length with a visible pen or paint on the element (see the sectioning diagram in attachment 1, the first cut mark).

1.6 Measure 13 inches (33 cm) from the bottom (see the diagram in attachment 1, the second cut mark). Mark the measured length with a visible pen or paint.

1.7 Place the slotted template over the ring piece (SFEC5-2) of the fuel sample, aligning it with the 0° orientation mark on the ring sample.

CAUTION: There is the potential for the SNF sample to react with the cell's atmosphere. For observable energetic reaction in air, **DO NOT immerse the element in the SFEC water** but purge the element with Argon to preclude further sample degradation. Inform the cognizant scientist. If the reaction stops, re-insert the element in the SFEC. If not, continue the purging with the argon to control the burning of the SNF sample.

1.8 Mark 22.5°, 45°, 67.5°, 90° and 180° clockwise on the outside cladding of the ring with a visible pen or paint.

1.9 Place the template on the other half of the ring section and mark 22.5°, 45°, 67.5°, 90° counterclockwise. (See attachment 2).

1.10 Attach the adhesive K-type thermocouples (TCs) to both sides of the cutting location to measure temperature increases during the cutting.

1.11 Place the Argon trough box over the element.

1.12 Cut the SNF element at the marked location (first cut in the sectioning diagram) using the milling machine with vortex spray cooling. Start the cutting at a minimum speed and increase the speed as necessary to optimize the cutting time with minimal rise in temperature, at the direction of the cognizant scientist.

1.13 Let an operation assistant measure and record the temperature of the SNF sample at the begining of the cutting and every 15 minutes during the cutting.

1.14 Cut the 1 inch (2.54 cm) ring section (labelled SFEC5-2 in the diagram) from the piece with the undamaged end cap.

1.15 Insert sections labelled SFEC5-1 (on top with the cut surface down) and SFEC5-3 (at the bottom with the cut surface up) into the SFEC # 5.

1.16 Add de-ionized water to the SFEC until full and place the lid on. Store the SFEC in a vertical position.

2.0 Sectioning of the Ring Piece of SNF Element for Test Specimens

The ring section (labelled SFEC5-2 in attachment 2) of the SNF sample will be further sectioned along the longitudinal direction. The sectioning will provide a maximum of eight 22.5° sector specimens and two 90° sections.

2.1 Cut the ring section into two halves through the 0° and 180° marks.

2.2 Label the container with identification numbers such as SFEC5-4378-S2-A, SFEC5-4378-S2-B, SFEC5-4378-S2-C, etc, (i.e.,SFEC5 for SFEC #, 4378 for K basin canister #, S2 for sectioned piece and A,B,C, etc, for degree of the sector clockwise per sectioning SFEC5-2 diagram in attachment 2, i.e., A for 22.5°, B for 45°, etc.). Add the sectioned date to the labelling.

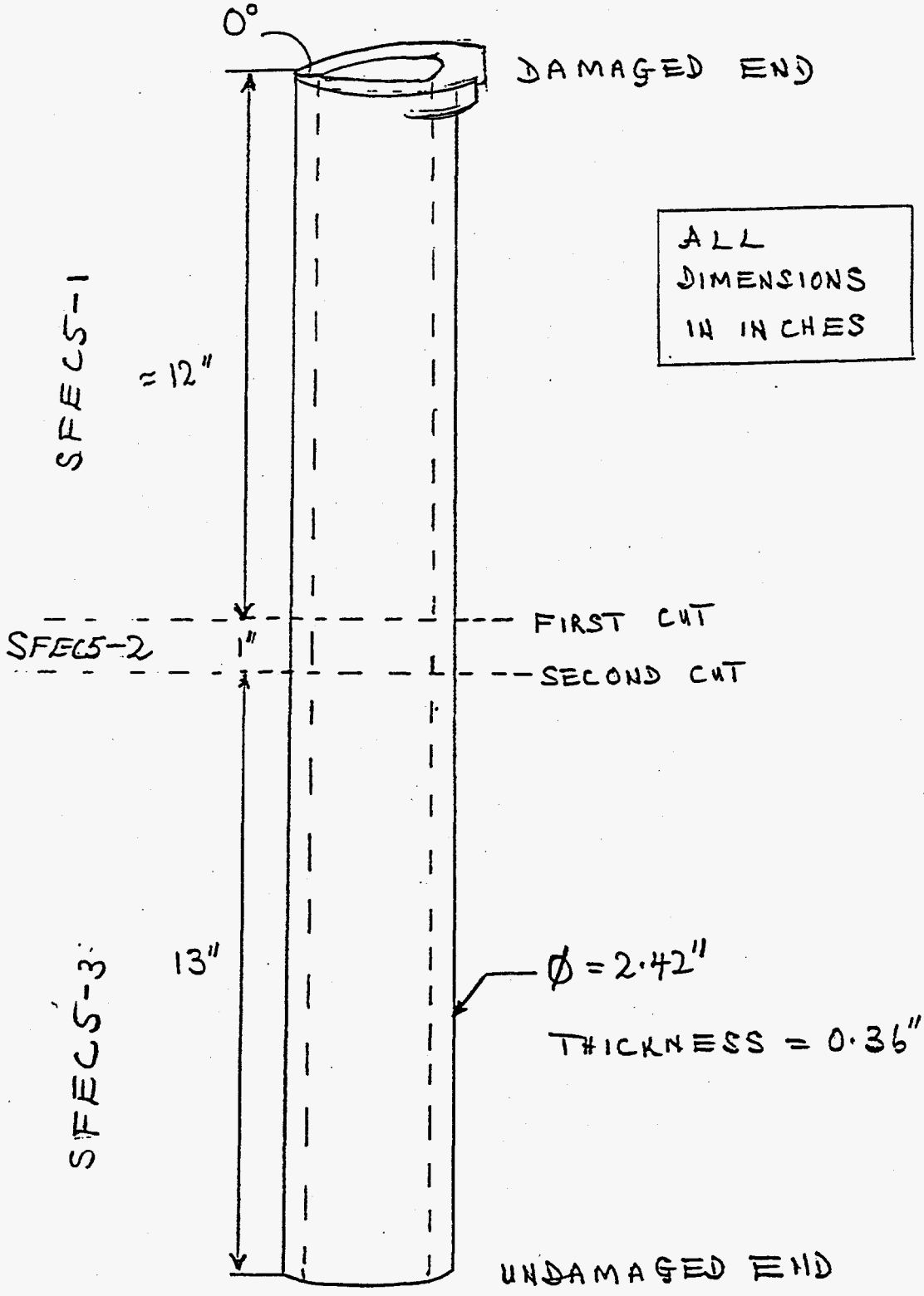
2.3 Mount one half-piece of the ring and section the four marked 22.5° sectors (shown in attachment 3) with the milling machine .

2.4 Place each specimen in the corresponding glass specimen container.

2.5 Repeat 2.2 - 2.4 for the other half-ring.

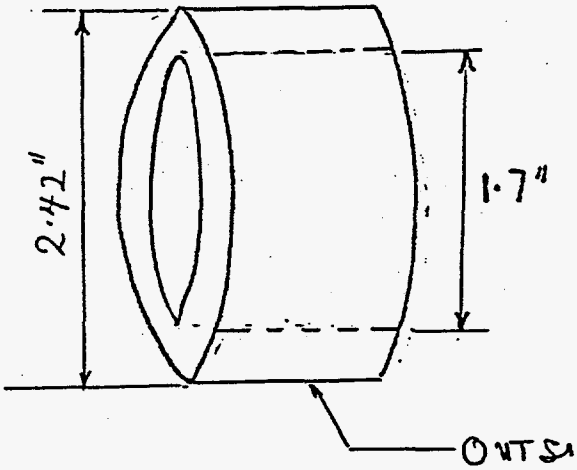
2.6 Place the glass containers in the stainless steel sample storage container. Evacuate the container with mechanical pump and backfill it with the high purity argon.

Attachment 1



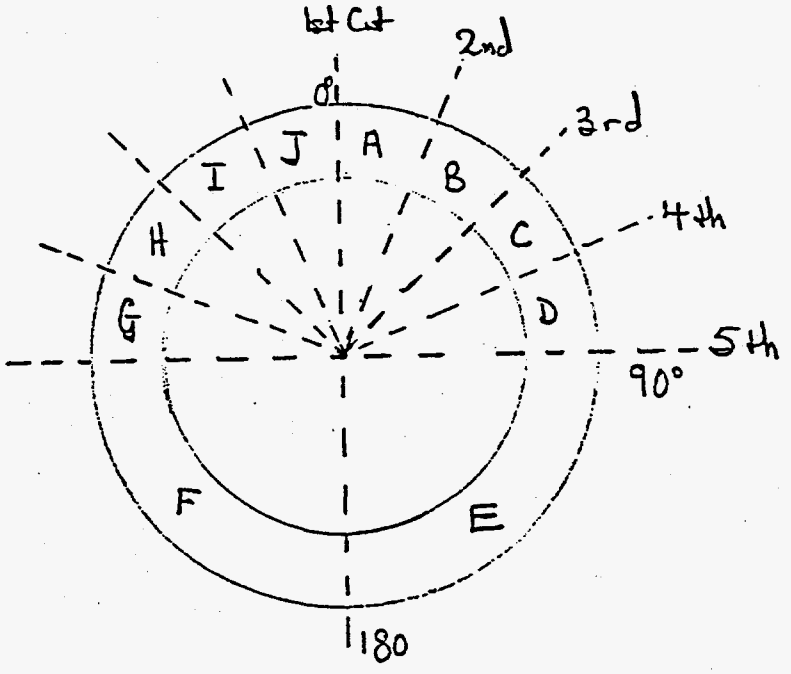
Attachment 2

SIDE VIEW OF SFEC5-2



ESTIMATED WEIGHT = 650g

SECTIONING OF SFEC 5-2



Data Sheet # 1

Technician: _____ **Date:** _____ **Cognizant Scientist** _____ **Date:** _____

SNF Temperature

SFEC #	Time (min)	Temperature (°C)	
		Thermocouple 1	Thermocouple 2

Hot Cell's Atmosphere: Temperature _____; Humidity _____;

Pressure Difference _____; Atm. Pressure _____

Test Instruction	Rev. No.: 0	Page 1 of 7

Title: Sectioning the Ruptured End of SNF Sample SFEC5,4378-S1

Work Location: 327 (Building/Room or "General")	**TI No.:** SNF-CT-006
Author: John Abrefah	**Effective Date:** 10/06/95 **Supersedes Date:**

Signatures:

Author _____ (Signature) 9-11-95 (Date)

Technical Reviewer _____ (Signature) 9-11-95 (Date)

Approval:

Project Manager, HTSP _____ (Dennis K Kreid) 9-13-95 (Date)

Concurrences:

PTL Manager _____ (James M. ~~Seay~~) 10/6/95 (Date)

HSNFP Charact. Manager _____ (Ronald P. Omberg) 10/6/95 (Date)

#1

Purpose/Scope The purpose of this test instruction is to direct the removal of the end cap from the ruptured end of the Spent Nuclear Fuel (SNF) sample SFEC5,4378-S1 and section the damaged end for specimens. These specimens will be used in metallographic examinations and for the furnace testing.

Applicability This test instruction is applicable to the ruptured end of SNF sample SFEC5,4378-S1 which was sectioned from the SNF element SFEC5,4378.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Pre-requisite Prior to removing the SNF sample from the SFEC for any sectioning activity, measure and record the cell's atmospheric conditions on attached data sheet.

1.0 Removal of the End Cap from SNF Sample SFEC5,4378-S1

The ruptured end cap of sample SFEC5,4378-S1 will be carefully removed using the milling machine in F-cell to expose the underlying material for further visual examination.

Note: The ruptured end of this SNF sample piece could be friable (i.e., easily crushed or damaged) due to irradiation and degradation in storage. Extreme care should be taken in handling it. Notify the cognizant scientist if the fuel sample breaks into pieces.

1.1 Remove the SNF element from the SFEC and mount it on the milling machine such that the damaged end cap can be gripped with a tool.

1.2 Before removing the end cap, place a plastic sheet or clean and use the trough under the damaged end to catch potentially loose particulates.

1.3 Attach an adhesive K-type thermocouple to the sample about an inch away from the end cap to measure temperature increases during the end cap removal activities.

1.4 Carefully remove the end cap by:

1.4.1 Cutting through the outside diameter cladding wall (approximately 25 mil thick) at the brazing/fuse-welded joint using the carbide blade.

1.4.2 Grip the end cap plate with a tool and/or manipulator and pull the end cap off at the direction of the project cognizant scientist .

1.4.3 If step 1.4.2 fail to separate the end cap, then proceed with the carbide blade to section the end cap from the SNF sample.

CAUTION: There is the potential for the SNF sample to react with the cell's atmosphere. If there is an observable energetic reaction in air, DO NOT immerse the element in water. Purge the element with argon to preclude further sample degradation. Inform the cognizant scientist. If the reaction stops, re-insert the element in the SFEC. If not, continue purging with argon to control or extinguish the burning of the SNF sample.

1.5 After removal of the end cap, mount sample SFEC5,4378-S1 for examination with the Opton telescope, and for photography at the direction of the project cognizant scientist.

Hold Point: Do not proceed with section 2.0 and 3.0 until directed by the cognizant scientist in writing.

2.0 Sectioning the Damaged End from Sample SFEC5,4378-S1

The damaged end of sample SFEC5,4378-S1 will be sectioned for specimens that will be used in the furnace testing, metallographic examinations and storage in an inert atmosphere for later testing.

2.1 Remove any loose piece(s) of fuel and store in a vial labeled SFEC5,4378-S1-L1 , L2..... at the direction of the project cognizant scientist.

2.2 Measure and mark the location to be cut with a sharpie permanent ink pen or an extra fine paint marker 3 inches from the damaged end of the specimen (Attachment 1). Record type and manufacturer of pen/marker used.

2.3 Place the specimen in the trough on the milling machine table and attach the K-type thermocouple to the sample about an inch away from the cutting location to monitor temperature changes during the cutting.

2.4 Cut the SNF element at the marked location using the milling machine with the carbide blade and argon vortex cooling. Start cutting at a minimum blade speed and increase the speed and/or feedrate as necessary to optimize the cutting time with minimal rise in temperature (e.g., $\leq$ 15°C rise above ambient).

2.5 Monitor the temperature of the SNF sample. Ensure that the sample temperature increase above the ambient stays below 15°C either by reducing the blade speed or feedrate or by redirecting the vortex nozzle to efficiently cool the sample.

2.6 Verify that the 0° orientation mark on the damaged end is visible. If not, then mark the 0° orientation on the three-inch (SFEC5,4378-S1A) piece with a sharpie permanent ink or fine point oil-based marker as shown in Attachment 1. Record the type and manufacturer of pen/marker used.

2.7 Perform visual examinations with the Opton telescope and photograph the cut surface of the specimen SFEC5,4378-S1A at the direction of the project cognizant scientist.

Hold Point: Do not proceed to section 3.0 until directed by the project cognizant scientist in writing.

3.0 Sectioning of the Damaged-End Piece Specimen

The three-inch long section (labeled SFEC5,4378-S1A in Attachment 1) of the SNF sample will be further sectioned along the longitudinal direction. The sectioning will provide eight 22.5° sector and two 90° sector specimens.

3.1 Place the slotted template over the damaged end (i.e., the top) of the piece, aligning it with the 0° orientation. Mark on the outside of the cladding 22.5°, 45°, 67.5°, 90° and 180° clockwise from the top with a visible pen or paint.

3.2 Place the template on the other half of the damaged end and mark 202.5°, 225°, 247.5°, 270° clockwise from the top starting from the 180° orientation. (See attachment 1).

3.3 Cut the SFEC5,4378-S1A ring section into two halves through the 0° and 180° marks.

3.4 Perform visual examinations of the longitudinal surfaces and take photographs of the cut surfaces of the two halves at the direction of the cognizant scientist.

Hold Point: Do not proceed to the following steps until directed by the cognizant scientist in writing.

3.5 Mount the two halves of specimen SFEC5,4378-S1A and cut a 1 inch piece from the damaged end or at location specified by the cognizant scientist (see attachment 1). Label the top half-ring pieces SFEC5,4378-S1AA and the bottom pieces SFEC5,4378-S1AB. Store the bottom pieces in an ultra high purity argon environment.

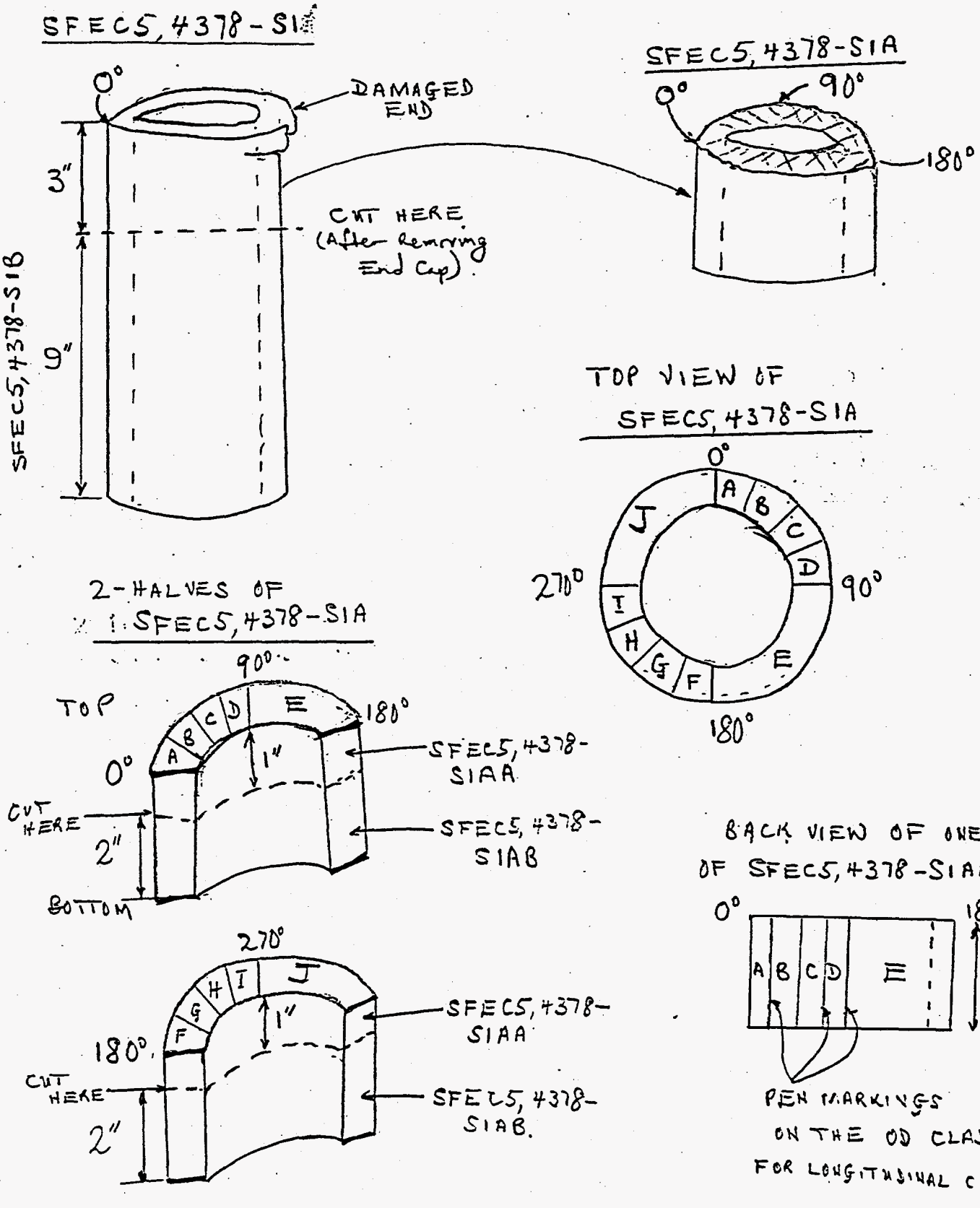
3.6 Section the top half-ring pieces (SFEC5,4378-S1AA) at the marked locations along the longitudinal directions to give eight 22.5° and to 90° specimens.

3.7 Label specimen vials with identification numbers such as '5-S1AA-A, '5-S1AA-B, 5-S1AA-C, etc, (i.e.,5 for SFEC #, S1AA for for the top piece of specimen SFEC5,4378-S1 and A,B,C, etc, for degree of the sector clockwise per sectioning SFEC5,4378-S1A diagram in Attachment 1, i.e., A for 22.5°, B for 45°, etc.).

3.8 Place each specimen in the corresponding labeled specimen vial.

3.9 Place the specimen vials in the labeled stainless steel sample storage container(s). Evacuate the sample storage container(s) per PTL procedure PTL-149 and backfill the container to about 5 psig with high purity argon (99.999%).

Attachment 1



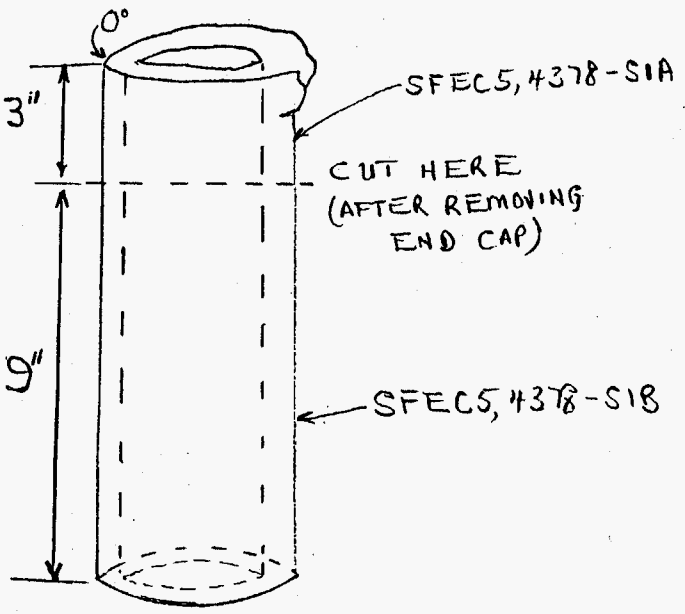
Data Sheet

F-Cell's Atmosphere

Temperature (°C)	Relative Humidity (%)	Pressure Differential (in H_2O)	Canyon Pressure Differential (in H_2O)	300-Area Atm. Pressure (in. Hg)	Date	Initials

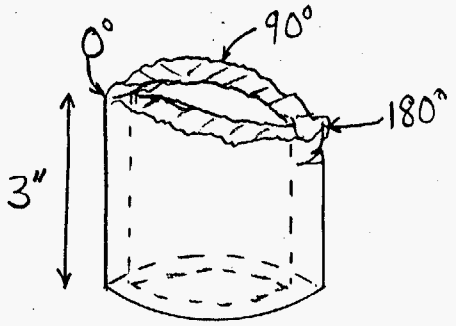
REVISED SECTIONING DIAGRAM FOR SFEC5, 4378-S1A.

SFEC5, 4378-S1



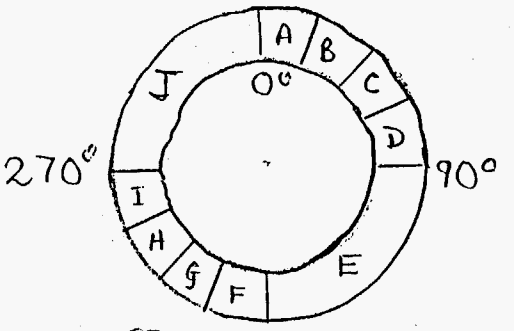
①

SIDEVIEW OF SFEC5, 4378-S1A



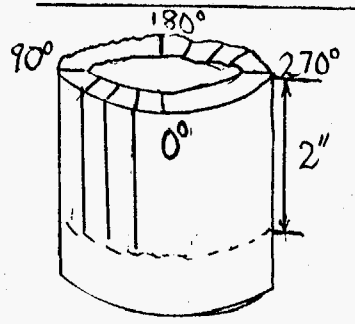
②

TOP VIEW OF SFEC5, 4378-S1A



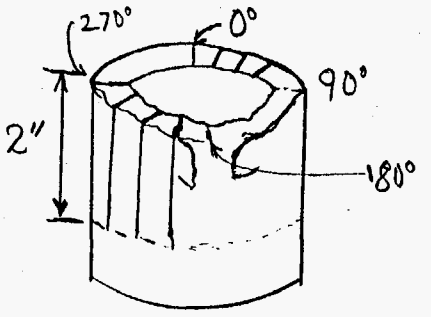
③

FRONT VIEW AT 0° SHOWING LONGITUDINAL CUTS AT 22.5° — 90°



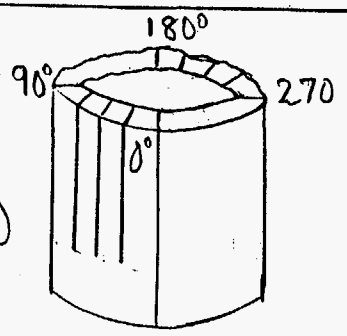
⑤

FRONT VIEW AT 180° SHOWING LONGITUDINAL CUTS AT 202.5° — 270°.



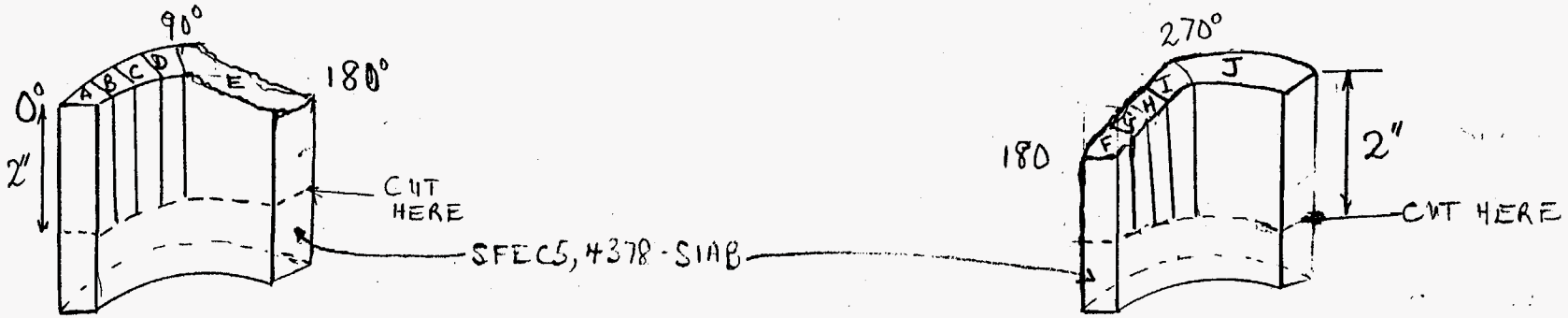
④

CUT SFEC5, 4378-S1A INTO 2 HALVES AT 0° AND 180° MARK



⑥

2- HALVES OF SPEC5, 4378 - S1A



CONTROLLED COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 10

Title: Vacuum Drying and Conditioning Testing of Initially Wet SNF Element SFEC5,4378 Specimens

Work Location: 327 (Building/Room or "General")	**TI No.:** SNF-CT-007
Author: John Abrefah	**Effective Date:** 7/5/95 **Supersedes Date:**

Signatures:

Author _____ (Signature) 7/5/95 (Date)

Technical Reviewer _____ (Signature) 7-5-95 (Date)

Approval:

Project Manager, HTSP Dennis Kreid (Denis K. Kreid) 7/6/95 (Date)

Concurrences:

PTL Manager _____ (James M. Seay) 7/6/95 (Date)

HSNFP Charact. Manager Bruce Mckenna for (Ronald P. Omberg) 7/7/95 (Date)

Purpose/Scope The purpose of this test instruction is to direct the drying and conditioning testing of Spent Nuclear Fuel (SNF) specimen in a controlled temperature and atmosphere furnace. The specimen was sectioned from SNF element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF specimens taken from the mid-length of the element SFEC5,4378.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) should read and follow the steps in the procedure PTL-151: *Operation of Conditioning Furnace System.*

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

A 22.5°-sector SNF specimen will be selected for loading into the resistively heated furnace shown schematically in Attachment 1.

1.1 Perform the *Equipment Readiness/Start Up* steps of the procedure PTL-151: *Operation of Conditioning Furnace System.* Record completion of each step with the operator's initials, time and date in the PTL log book.

1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 10 and 12 and opening valves 1, 5, 6, 7, 8, 9 and 11.

Before proceeding further in the procedure PTL-151 perform the following steps:

1.3 Dry the furnace system before loading the test specimen using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective then try the step 1.3.2 after discussions with the cognizant scientist otherwise disregard step 1.3.2.

1.3.1 Dry the furnace system by flowing high purity gas (e.g. 99.999% Ar) through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

Hold Point: Consult with the cognizant scientist before trying the alternate drying method in step 1.3.2 below and/or proceeding to the remaining steps of this test instruction.

1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

1.4 Identify and record the specimen identification code in block 9 or 28 on the data sheet of Attachment 2.

1.5 Use a calibrated ruler (record the calibration information in blocks 7 & 8 on the data sheet of Attachment 2) with an accuracy of ± 1mm to measure and record in blocks 12 through 18 and 21 through 27 on the data sheet of Attachment 2 the specimens dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp 'lips' that interfere with the measurements, sand them off with silicon carbide abrasive paper. Do not use a lubricant.

1.6 Weigh the specimen twice on a QA category one balance (record the balance calibration information in blocks 1, 2 & 3 on the data sheet of Attachment 2) with 0.1 mg sensitivity. Record the specimens weights in blocks 10 & 19, or 29 & 38 on the data sheet of Attachment 2.

Note: If the subsequent steps are performed immediately following step 1.3 then make sure the furnace system has cooled to the ambient hotcell temperature before loading the specimens. Minimize the time the furnace tube and gas lines will be open to air in the cell.

1.7 Mount the specimen in the sample holder such thatit is in the center of the uniform heated zone when loaded into the furnace. Ensure that the specimen is in contact with the specimen thermocouple.

1.8 Fill the sample holder with 5±0.2 ml of de-ionized water.

1.9 Load the sample holder into the furnace tube with extreme care to avoid loss of water and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

1.10 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the PTL log book.

2.0 Vacuum Drying Test

The specimen will be initially tested to determine the water content by driving off free and bound water first at low temperature vacuum followed by relatively high temperature vacuum. The moisture content of the off-gas will be detected by the moisture monitor, and the cumulative water will be trapped in the moisture traps. Before and after weights of the traps will be a measure of the water that came off.

2.1 Prior to beginning the drying test of the specimen, determine the weight of the moisture traps on a QA category one balance (record the balance calibration information in blocks 4, 5 & 6 on the data sheet of Attachment 2) with 10 mg sensitivity. Repeat each measurement and record their weights in blocks 47 & 51 and 49 & 53 on the data sheet of Attachment 2.

2.2 Pump the system down and control the system pressure to within the range of 50 to 100 Torr after closing valves 1, 2 and 11 and opening valves 5, 6, 7, 8, 9 and 10.

2.3 Configure valves 6 to 9 of Attachment 1 so that the pumping is through the two moisture traps (i.e., valves 6 to 9 opened).

2.4 Verify that all the analytical instruments (e.g., moisture monitor and pressure gauge) are responding properly by looking at the readings on the computer #1. Notify the cognizant scientist of any anomalous readings.

2.5 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the drying cycles. The heating tape upstream from the furnace will be turned off or on at the direction of the cognizant scientist.

2.6 Close valves 7 and 9 of Attachment 1 so that only moisture trap #1 is used in the low temperature vacuum drying cycle.

Note: Due to the swapping of moisture traps after the first low temperature vacuum drying cycle, the test must be scheduled such that the first cycle ends during regular working hours, otherwise arrange for that step to be performed during off hours.

2.7 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual):

- 2.7.1 to heat at a constant rate (e.g., between 10 and 15°C/min) to 50±2°C and hold the 50±2°C for 10 hours.
- 2.7.2 resume the constant heating rate after swapping the moisture traps in Step 2.9 (i.e., at the same rate as in Step 2.7.1) to 300±2°C and hold the 300±2°C for 24 more hours
- 2.7.3 then allow the furnace to cool to ambient temperature.

2.8 Record in block 55 on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that computer #1 is monitoring and recording the:

- 2.8.1 Water vapor concentration with the moisture monitor
- 2.8.2 Pressure with the pressure gauge
- 2.8.3 Furnace and specimen temperatures

2.9 After the cold vacuum dry cycle,close valves 6 to 8 and open valves 7 and 9 (Attachment 1) so that the moisture trap #2 is connected to the system.

2.10 Remove the moisture trap #1 during the second heating up rate to set the testing temperature at 300±2°C. Weigh the trap on a QA category one balance with 10 mg sensitivity. Repeat the weight measurement and record the weights in blocks 48 & 52 on the data sheet of Attachment 2.

2.11 After the second drying cycle, and with the furnace and specimen at ambient temperature, remove moisture trap #2 and weigh it twice on a QA category one balance with 10 mg sensitivity. Record the weights in blocks 50 & 54 on the data sheet of Attachment 2.

3.0 Conditioning Testing

The SNF passivation step in the furnace testing will involve exposing the test specimens to a gas mixture of 98% argon and 2% oxygen by volume at a flow rate of 100±2 cm^3/min.

3.1 Configure valves 6 to 9 (i.e. either valves 6 and 8 opened with valves 7 and 9 closed or valves 6 and 8 closed with valves 7 and 9 opened) so that gas flows through only one of the moisture traps.

3.2 Adjust the heating tape (downstream from the furnace) temperature to a constant value in the range of 50 to 100°C or the value specified by the cognizant scientist.

3.3 Purge the system with pure argon flow of 100±2 cm^3/min (i.e., by opening valves 1, 5, 6&8 or 7&9, and 11, but with valves 2,3,4, and 10 closed) for about 45 minutes and monitor the moisture content in the system. If the moisture monitor indicates high concentration (i.e., greater than -50°C dew point) then consult with the cognizant scientist before proceeding to the next step.

3.4 Set the argon and oxygen flow controllers to give the flows indicated above (i.e., 98% argon, 2% oxygen at a flow rate of 100±2 cm^3/min).

3.5 Switch the gas flow to the GC through the bypass line (i.e., opening valve 4 and closing valves 1, 2, 3, and 11 of Attachment 1). Record in block 56 on the data sheet of Attachment 2 the switch over time. Program computer #2 to monitor and record the oxygen concentration in the gas stream every 2 minutes for a period of at least 15 minutes after equilibration to establish the baseline.

3.6 Switch the gas flow to the furnace by opening valve 1, 5, and 11, and by closing valves 2, 3, 4 and 10. Record in block 57 on the data sheet of Attachment 2. Program computer #2 to monitor and record the oxygen concentration in the gas stream measured by the GC every 5 minutes until a constant oxygen concentration is established. At that constant concentration, monitor the gas stream for at least 30 minutes to establish the baseline concentration.

3.7 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual) to heat at a constant rate (e.g., between 12 to 15°C/min) to 150±2°C and hold at that temperature indefinitely. (Note that furnace cooling will be initiated manually after about 10 hours as described in Step 3.8.) Verify that the computers are monitoring and recording the:

- 3.7.1 Moisture concentration using the moisture monitor for every 5 minutes.
- 3.7.2 Oxygen and hydrogen concentrations using the gas chromatograph for every 5 minutes.
- 3.7.3 Furnace and specimen temperatures every 5 minutes.

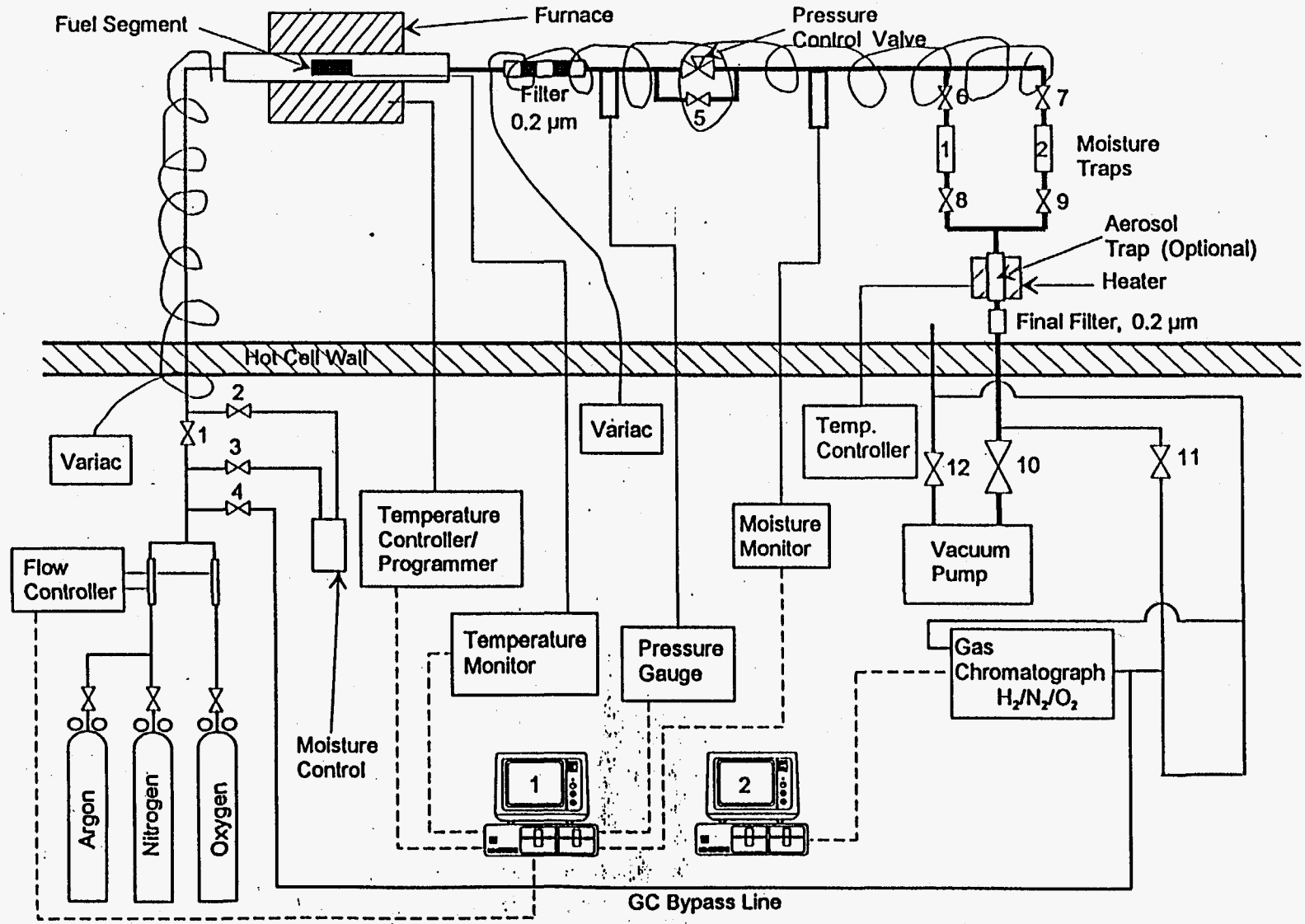
3.8 After 10 hours of conditioning of the specimens, turn off the oxygen flow and the furnace, and allow the specimen to cool to ambient temperature in flowing argon. Record in block 58 on the data sheet of Attachment 2 the time oxygen gas is turned off.

3.9 Open the furnace and carefully remove the specimens for weighing using a QA category one balance with 0.1 mg sensitivity. Weigh each specimen twice and record their weights in blocks 11 & 20 and 30 & 39 on the data sheet of Attachment 2. Record other observations in the PTL log book and on a sheet to be pasted in the Laboratory Record Book (LRB).

3.10 Perform the test shut down steps in the procedure PTL-151. Record completion of each step with the operator's initials, date and time in the PTL log book.

3.11 At the direction of the cognizant scientist, remove and store the two 0.2 μm filters for possible radionuclide analyses at a later date. Record the storage location and date in the PTL log book.

Schematic Of The Furnace System



Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
4 - Moisture Trap Balance Description	5 - Moisture Trap Balance Calibration Number	6 - Calibration Expiration Date
	7 - Ruler Calibration Number	8 - Ruler Calibration Date

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	10	11	12	13	14	15	16	17	18
2	19	20	21	22	23	24	25	26	27

Measurement Number	28 - Specimen Identification Code								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	29	30	31	32	33	34	35	36	37
2	38	39	40	41	42	43	44	45	46

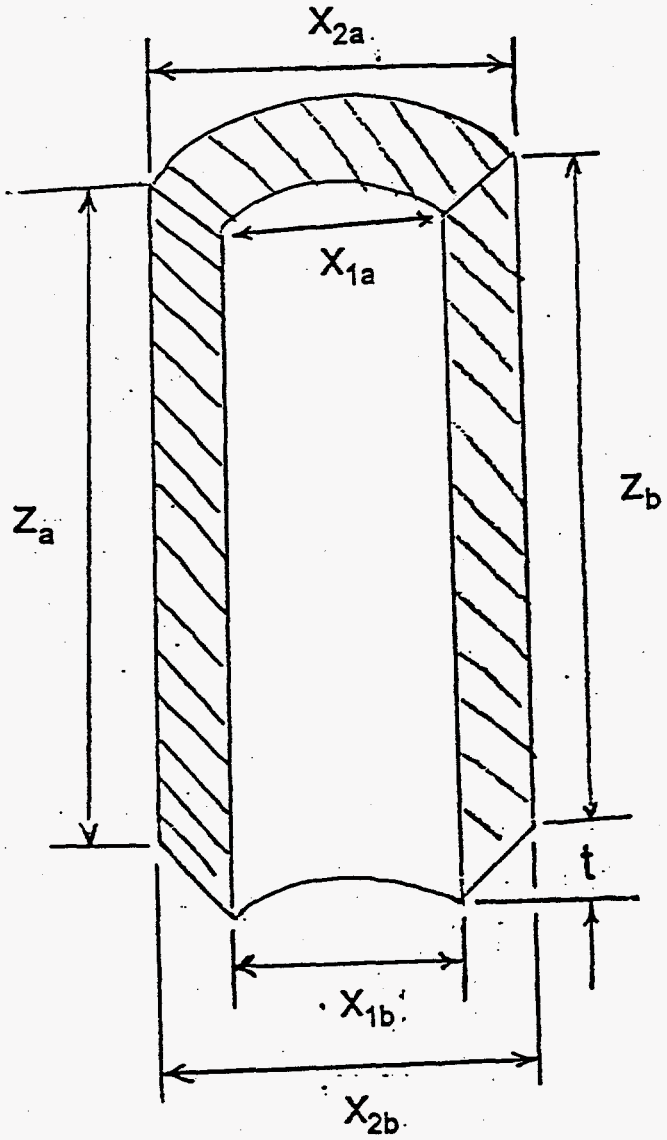
Measurement Number	Moisture Trap #1		Moisture Trap #2	
	Weight Before Test (g)	Weight After Test (g)	Weight Before Test (g)	Weight After Test (g)
1	47	48	49	50
2	51	52	53	54

SYSTEM STATUS

55 - Time/Date	56 - Time/Date	57 - Time/Date	58 - Time/Date

Attachment 3

Specimen Dimensions



CONTROLLED COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 4
Title: Sectioning of SNF Specimens SFEC5,4378-S2-E & F		
Work Location: 327 (Building/Room or "General")	**TI No.: SNF-CT-008**	
Author: John Abrefah	**Effective Date:** 7/26/95 **Supersedes Date:**	

Signatures:

Author _____ 7/26/95
(Signature) (Date)

Technical Reviewer _____ 7-26-95
(Signature) (Date)

Approval:

Project Manager, HTSP Denis Kreid 7/26/95
(Denis K. Kreid) (Date)

Concurrences:

HSNFP Charact. Manager R P Omberg 7/26/95
(Ronald P. Omberg) (Date)

Purpose/Scope The purpose of this test instruction is to direct the sectioning of additional test specimens from the two 90°-sector specimens SFEC5,4378-S2-E & F in storage.

Applicability This test instruction is applicable to SNF specimens SFEC5, 4378-S2-E & F.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

1.0 Sectioning of the SNF Specimens SFEC5,4378-S2-E & F

Specimens SFEC5,4378-S2-E & F will be sectioned using the milling machine in F-cell. Stainless steel specimen containers will be used to store the cut specimens under high purity argon.

1.1 Provide video coverage of the activities as directed by cognizant scientist.

1.2 Prior to removing the SNF specimens for the following activities, measure and record the cell's atmospheric conditions .

CAUTION: There is the potential for the SNF sample to react with the cell's atmosphere. If an observable energetic reaction in air occurs, **DO NOT immerse the specimen in water** but purge it with argon to preclude further sample degradation and/or control the burning. Inform the cognizant scientist.

1.3 Place a slotted template over the 90°-sector specimen SFEC5,4378-S2-E, aligning it with the 90° orientation mark on the specimen (see Attachment 1)

1.4 Place a mark at 22.5°, 45° and 67.5°, from the 90° orientation with a visible pen or paint on the cladding of specimen SFEC5,4378-S2-E. The marked specimens will be given an identification label such as SFEC5,4378-S2-EX where X is 1, 2, 3 or 4 in the clockwise direction.

1.5 Place the template on the 90°-sector specimen SFEC5,4378-S2-F and mark with a visible pen or paint at 22.5°, 45° and 67.5° from the 180° orientation. Each sector will be labeled as SFEC5,4378-S2-FX where X is 1, 2, 3 or 4 in the clockwise direction from the 180° orientation. (See Attachment 1).

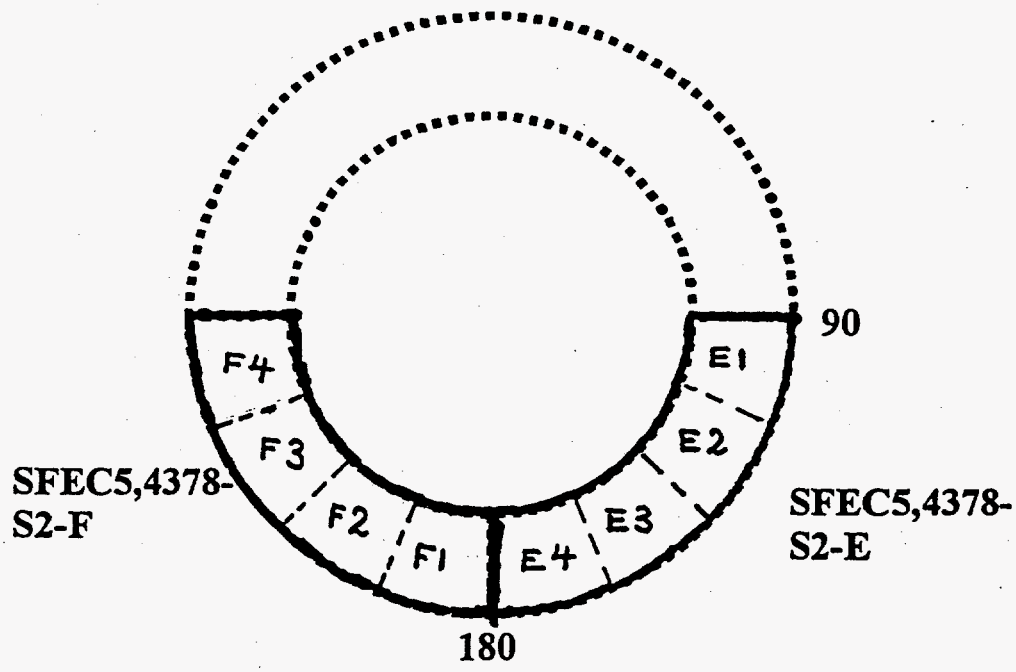
1.6 Attach adhesive K-type thermocouple to the specimen, if possible away from the cutting location to measure temperature increases during the cutting. Limit temperature increase to below 15°C above ambient.

1.7 Mount each 90°-sector specimen and cut the maximum possible number of 22.5°-sector specimens at the marked locations using the milling machine with argon vortex cooling. Start the cutting at a minimum speed and increase the speed as necessary to optimize the cutting time with minimal rise in temperature (< 15°C above ambient temperature), at the direction of the cognizant scientist.

1.8 Label specimen containers with identification numbers corresponding to steps 1.4 and 1.5 and place each specimen in the corresponding specimen container before the next specimen is cut.

1.9 Place the specimen containers in the stainless steel sample storage container. Evacuate the container with mechanical pump and backfill it with high purity argon.

Attachment 1



CONTROLLED COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 7
Title: Sectioning and Mounting of SNF Specimens SFEC5,4378-S2-B & J		
Work Location: 327 (Building/Room or "General")	**TI No.: SNF-CT-009**	
Author: John Abrefah	**Effective Date:** 8/9/95 **Supersedes Date:**	

Signatures:

Author _____ (Signature) 8-9-95 (Date)

Technical Reviewer _____ (Signature) 8-9-95 (Date)

Approval:

Project Manager, HTSP Dennis Kreid (Denis K. Kreid) 8-9-95 (Date)

Concurrences:

PTL Manager _____ (James M. Seay) 8/11/95 (Date)

HSNFP Charact. Manager _____ (Ronald P. Omberg) 8/11/95 (Date)

Purpose/Scope The purpose of this test instruction is to direct the sectioning, mounting and polishing of conditioned test specimens SFEC5,4378-S2-J & B for metallographic examinations.

Applicability This test instruction is applicable to SNF specimens SFEC5, 4378-S2-B & J.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

1.0 Sectioning of the SNF Specimens SFEC5,4378-S2-B & J

Specimens SFEC5,4378-S2-B & J will be sectioned using the a slow speed cut-off saw with diamond blade (e.g., the Isomet or LECO). Stainless steel specimen containers will be used to store the cut specimens under high purity argon.

CAUTION: There is the potential for the SNF sample to react with the cell's atmosphere. If an observable energetic reaction in air occurs, **DO NOT immerse the specimen in water** but purge it with argon to preclude further sample degradation and/or control the burning. Inform the cognizant scientist.

1.1 Cut each specimen (-S2-B or -S2-J) at approximately the mid-point as illustrated in the Attachments 1 & 2 using the Isomet or LECO. Use oil-base Hyprez coolant or equivalent for cooling. Cut at low speed to avoid heat generation and sample alteration. Put a mark on the OD cladding side at the top of specimens 5-S2-B2 and 5-S2-J2 to mark the orientation after cutting.

1.2 Label specimen containers with identification numbers 5-S2-B1, 5-S2-B2, 5-S2-J1 and 5-S2-J2 and place each specimen in the corresponding specimen container.

1.3 Place the specimen containers in the stainless steel sample storage container. Evacuate the container with mechanical pump and backfill it with high purity (99.999) argon at least three times. Finally backfill container to about 5 psig with the high purity argon.

2.0 Specimen Mounting, Polishing for Examinations

Specimens, sectioned from SFEC5,4378-S2-B & J will be mounted both on the transverse and longitudinal-section planes for metallographic examinations.

2.1 Mount specimens 5-S2-B1 and 5-S2-J1 such that the transverse planes are available for grinding, polishing and etching (see the illustration in Attachments 1 & 2). Use either polyester "boat resin" (e.g., Titan Fiberlay Clear Cast resin or Fiberlay Finishing resin) and/or Buehler Epo-thin epoxy resin, or equivalent for the mount. **Ensure edge retention of the uranium cut surfaces so that oxide thickness can be measured.**

2.2 Mark the top orientation of the specimen on the mounting ring. This orientation should be the same direction as the top of SFEC5,4378-S2-B.

2.3 Mount specimens 5-S2-B2 and 5-S2-J2 such that the longitudinal surfaces are available for grinding, polishing and etching (see the illustration in Attachments 1 & 2). Use either polyester "boat resin" (e.g., Titan Fiberlay Clear Cast resin or Fiberlay Finishing resin) and/or Buehler Epo-thin epoxy resin, or equivalent for this mount. **Ensure edge retention of the uranium cut surfaces so that oxide thickness can be measured.**

2.4 Mark the radial orientation of the specimen on the mounting ring. This direction should be the specimen's degree orientation (e.g., for specimen SFEC5,4378-S2-B2 per sectioning diagram, the markings will be 22° and 45°). Also mark the top orientation of the specimen.

Note: If specimens SFEC5,4378-S2-B & J cannot be cut in half per steps 1.1 through 1.3 then mount the whole specimen SFEC5,4378-S2-J per step 2.1 and the whole specimen SFEC5,4378-S2-B per step 2.3.

2.5 Progressively, grind the top surface of the transverse specimen and the 45° side on B and 0° on J surface of the longitudinal specimen on 180 followed by 240 grit silicon carbide (SiC) abrasive paper.

2.6 Take the specimens through 600 grit SiC abrasive paper followed by either 800 to 1200 grit SiC abrasive paper or 1/0 to 3/0 emery paper, using a lubricant of paraffin in kerosene (50 g paraffin per liter of kerosene) or Hyprez OS Type III, or Hyprez OS Type IV, or equivalent.

2.7 Polish on Leco Pan-K, "Gold Polishing" cloth (e.g., Glennel Corp. gold label polishing cloth), or equivalent using the following steps:

2.7.1 4 - 8 microns diamond paste suspended in Hyprez O.S. Type IV lubricant, at approximately 40 to 60% on the Ratiotrol.

2.7.2 1 - 2 microns diamond paste suspended in Hyprez O.S. Type IV lubricant, at approximately 40 to 60% on the Ratiotrol.

2.7.3 0.05 micron Buehler micro polish II, or equivalent,suspended in a 2% solution of chromic acid and de-ionized water on a micro cloth using the Rotor or the Vibra polisher for 30 seconds to 5 minutes (or use silica slurry on the micro cloth).

2.8 Examine the polished surface structure (see Attachment 3 for illustration of uranium hydride agglomerates) and take photomicrographs in the following sequence:

2.8.1 Fuel Surface Structure: 50X micro; and 50 - 750X photomicrographs as selected by the cognizant scientist.

2.8.2 Fuel/Cladding Interface: 50X micro and 50-750X selected photomicrographs by the cognizant scientist.

2.8.3 Fuel edge: 50X micro; and 50 - 750X photomicrographs as selected by the cognizant scientist.

Get the cognizant scientist approval to proceed to the following steps.

2.9 Oxidize the polished surfaces under a heated lamp (e.g., 500 Watt halogen lamp with the specimen surface about 2 to 3 inches away from the lens) for 15 to 30 minutes to get the 'halo' effect (see Attachment 3 for an illustration of the 'halo' effect).

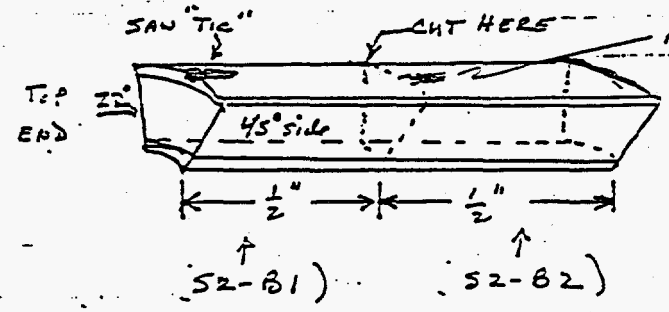
2.10 Examine the oxidized specimens in the following sequence:

2.10.1 Fuel Surface Structure: 50X micro; and 50 - 750X photomicrographs as selected by the cognizant scientist.

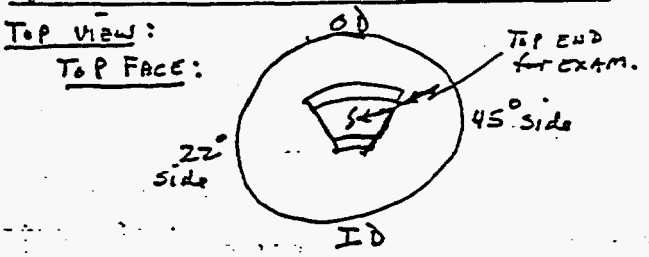
2.10.2 Fuel/Cladding Interface: 50X micro and 50-750X selected photomicrographs by the cognizant scientist.

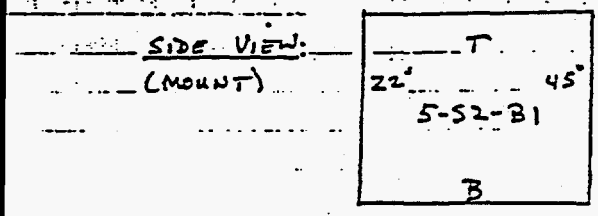
Attachment 1

Specimen SFEC5-4378-S2-B: (5-S2-B)

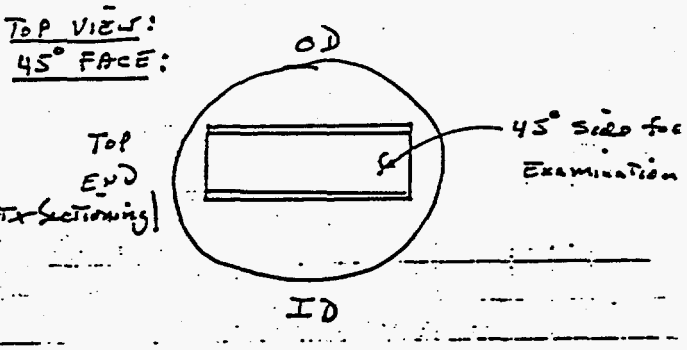


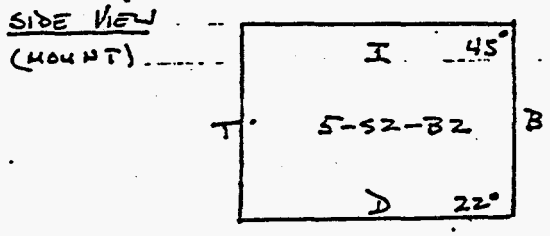
Specimen SFEC5-4378-S2-B1:





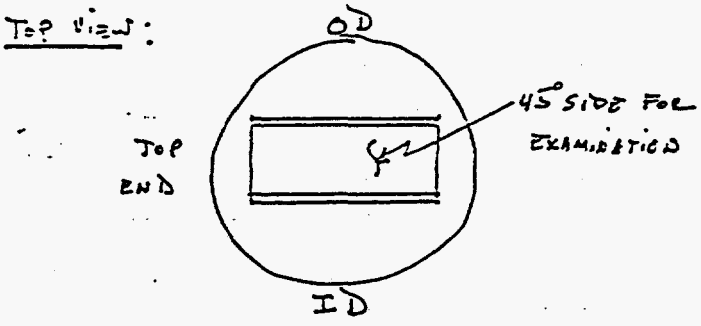
Specimen SFEC5-4378-S2-B2:

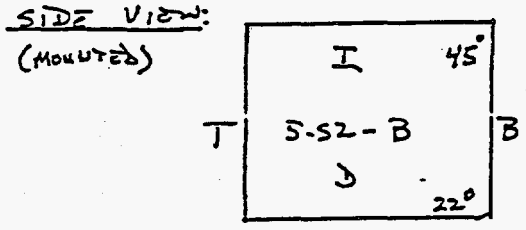




If Specimen Cannot be Cut in Half as Above, Mount the whole Specimen For Examination of its 45° SIDE As Below

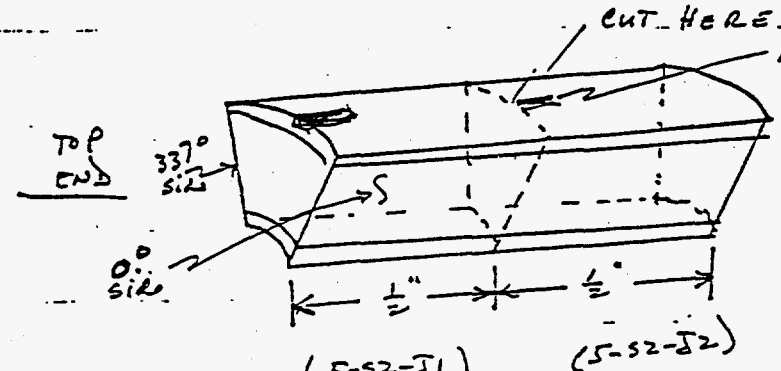
Specimen SFEC5-4378-S2-B:



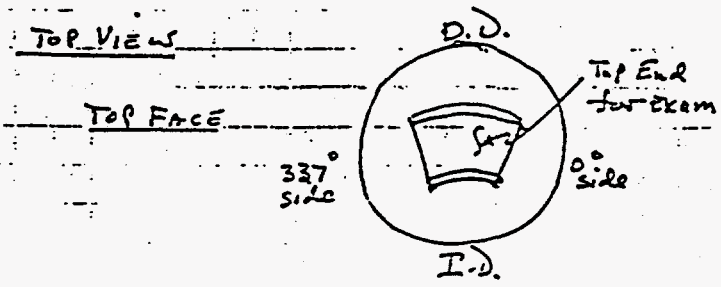


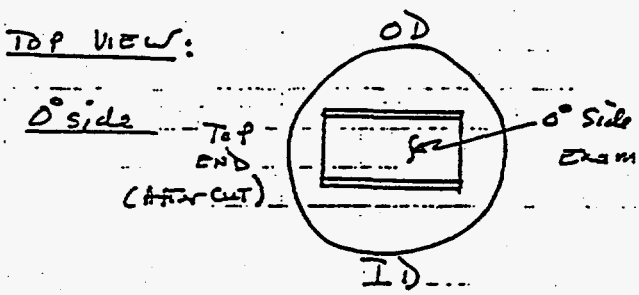
Attachment 2

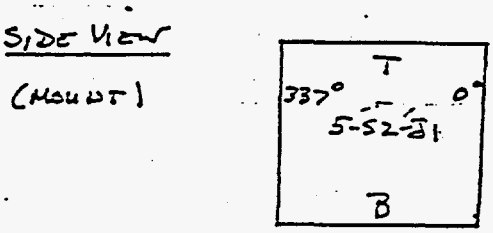
Specimen SFEC5-4378-52-J: (5-52-J)

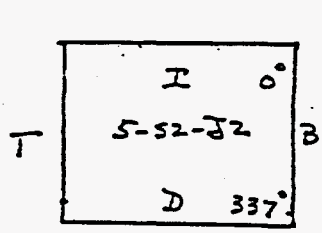


Specimen SFEC5-4378-52-J1: Specimen SFEC-4378-52-J



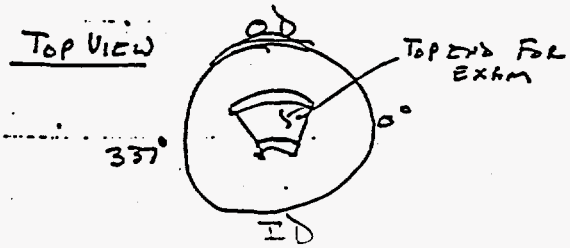


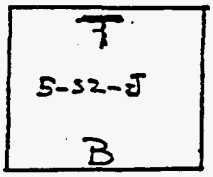




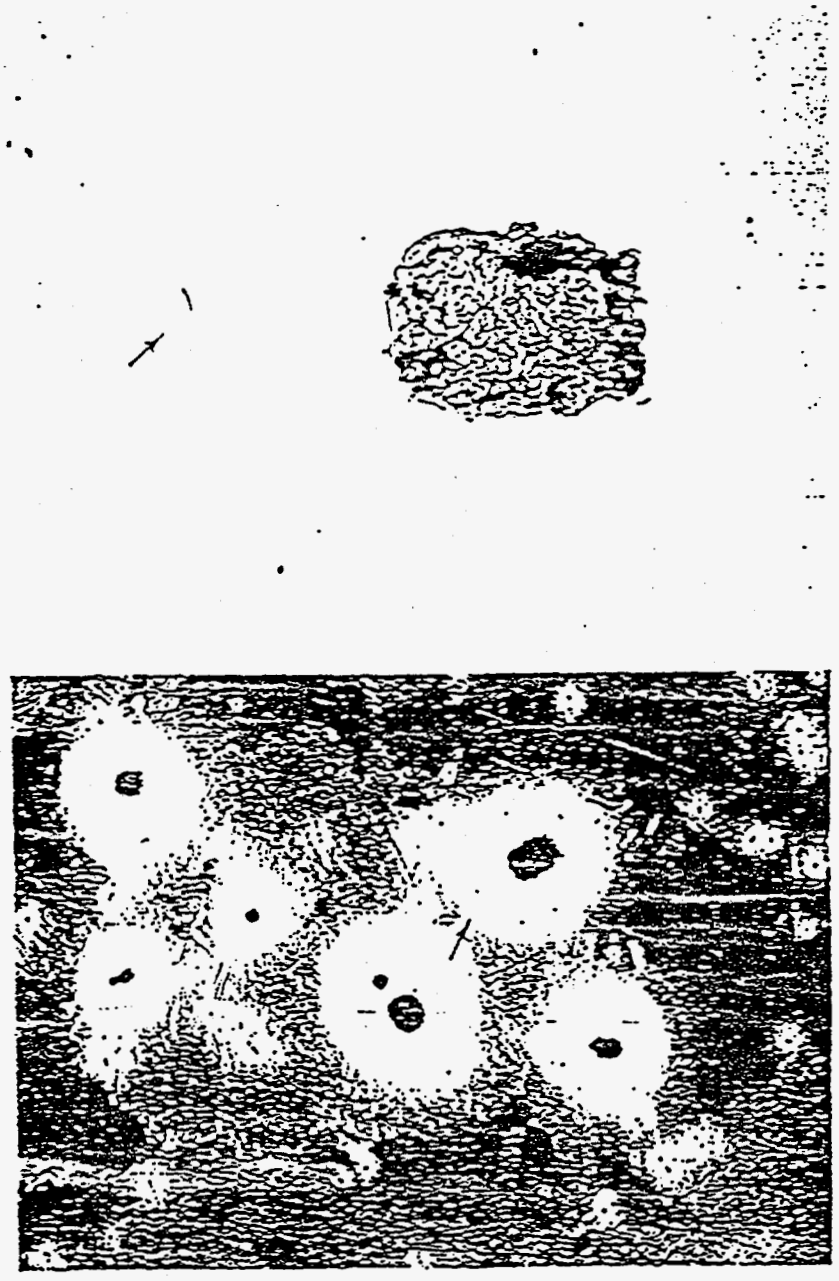
If Specimen cannot be cut in half as above, mount the whole specimen for examination of its TOP END (TRANSVERSE)

Specimen SFEC5-4378-52-J:





Attachment 3



Photomicrographs, 500X top, 100X bottom, illustrating the large uranium hydride agglomerates found in one of the Jominy specimens. Note normal size hydride to left of agglomerate in top and bottom photos, also the characteristic "halos" in the bottom photo. Samples in as polished condition; bottom photo taken after heat tinting for 15 minutes.

CONTROLLED COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 5

Title: Decladding SNF Specimens SFEC5,4378-S2-E1 & E2 and 'Cutting' Cladding Specimen

Work Location: 327 (Building/Room or "General")	**TI No.: SNF-CT-010**
Author: John Abrefah	**Effective Date:** 8/9/95 **Supersedes Date:**

Signatures:

Author _____ 8-9-95
(Signature) (Date)

Technical Reviewer _____ 8-9-95
(Signature) (Date)

Approval:

Project Manager, HTSP Denis Kreid 8-10-95
(Denis K. Kreid) (Date)

Concurrences:

PTL Manager _____ 8/11/95
(James M. Seay) (Date)

HSNFP Charact. Manager _____ 8-10-95
(Ronald P. Omberg) (Date)

Purpose/Scope The purpose of this test instruction is to direct decladding of specimens SFEC5,4378-S2-E1 & E2 and 'cutting' cladding specimen from the damaged end of SFEC5,4378-S1 for furnace testing.

Applicability This test instruction is applicable to SNF specimens SFEC5, 4378-S2-E1 & E1 and SFEC5,4378-S1.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

1.0 Decladding of the SNF Specimens SFEC5,4378-S2-E1 & E2

Specimens SFEC5,4378-S2-E1 & E2 will be decladded either by cutting with a saw blade or by grinding. Stainless steel specimen containers will be used to store the prepared specimens under high purity (99.999%) argon.

CAUTION: There is a potential for the SNF sample to react with the cell's atmosphere. If an observable energetic reaction in air occurs, **DO NOT immerse the specimen in water** but purge it with argon to preclude further sample degradation and/or control the burning. Inform the cognizant scientist.

1.1 Either cut or grind off the outer and inner cladding of specimens SFEC5,4378-S2-E1 & E2. If it is more practical to cut these specimens into two pieces before the decladding step then cut the specimens at approximately the mid-point (see the illustration in Attachment 1). Use oil base Hyprez coolant in all cutting activities.

The cognizant scientist or his designate must be present during the decladding process.

1.2 Place the decladded specimens in their respective specimen containers and store in the stainless steel sample storage container. Evacuate the container with mechanical pump and backfill it with high purity (99.999%) argon at least three times. Finally, backfill the container to about 5 psig with high purity argon.

2.0 'Cutting' a Piece of Cladding Specimen

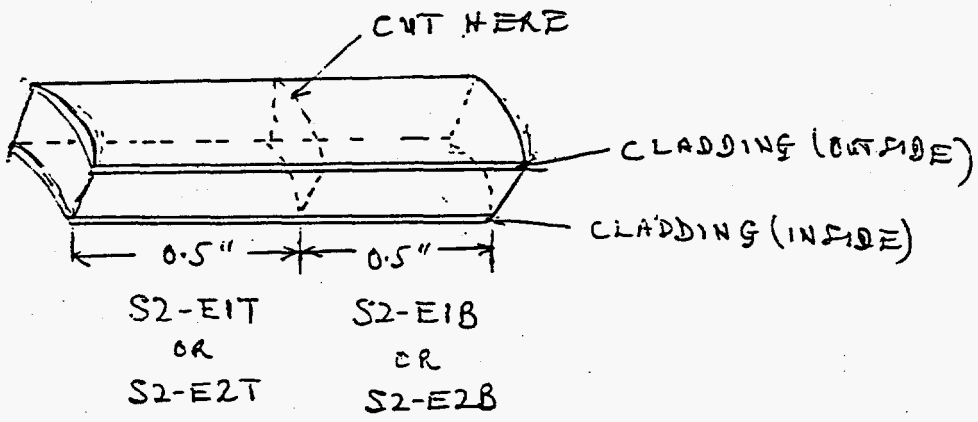
A cladding specimen will be taken from the peeled off part of cladding material at the damaged end of specimen SFEC5,4378-S1 for the furnace testing.

2.1 Mount specimen SFEC5,4378-S1on the milling machine and slowly cut, with the carbide blade, the cladding piece sticking out at the damaged end (see Attachment 2). Use the argon vortex to cool the specimen during the cutting operation. Ensure that the specimen will be retrieved after cutting.

2.2 Label a storage container SFEC5,4378-CLAD and place the specimen into the container.

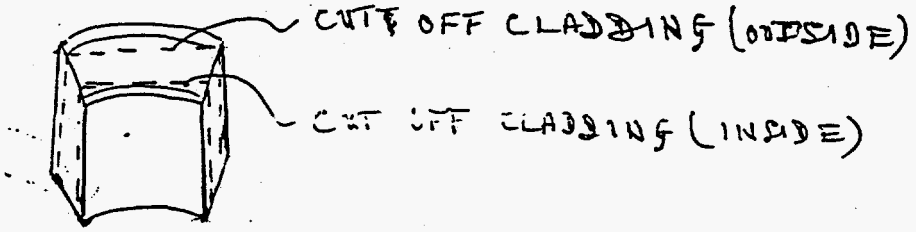
Attachment 1

SPECIMEN SFEC5.4378-S2-E1 AND E2



FOR SPECIMEN S2-EXT OR S2-EXB (where X = 1 OR 2)

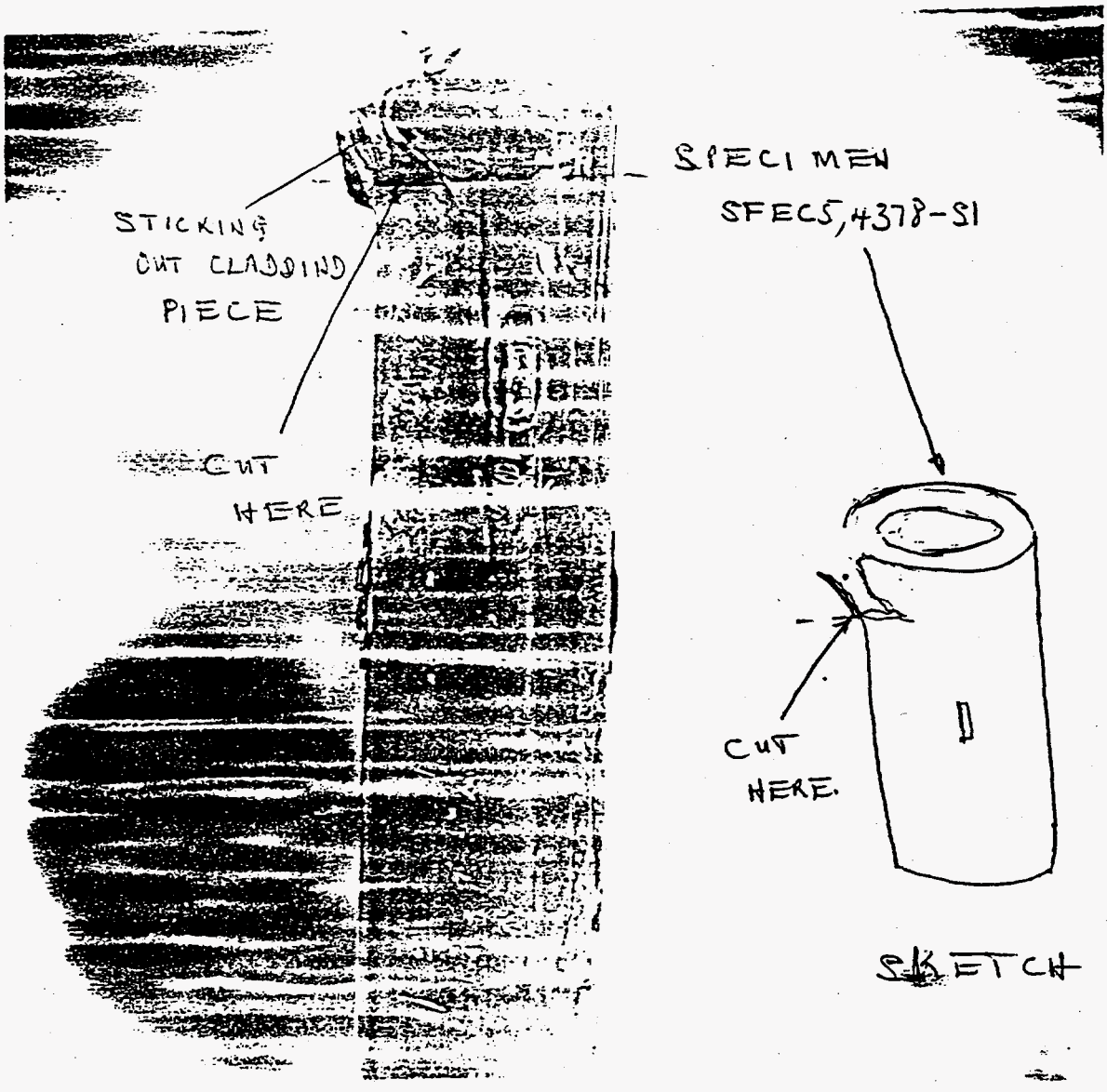
TOP VIEW



OR.

GRIND THE INSIDE AND OUTSIDE CLADDING MATERIAL OFF.

Attachment 2



CONTROLLED
COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 8
Title: Ignition testing of Specimen SFEC5,4378-S2-E3A		
Work Location: 327 (Building/Room or "General")	**TI No.: SNF-CT-012**	
Author: John Abrefah	**Effective Date:** 10/10/95 **Supersedes Date:**	

Signatures:

Author _____ 10-10-95
(Signature) (Date)

Technical Reviewer _____ 10-10-95
(Signature) (Date)

Concurrences:

PTL Manager _____ 10/18/95
(James M. Seay) (Date)

HSNFP Charact. Manager _____ 10/19/95
(Ronald P. Omberg) (Date)

Purpose/Scope The purpose of this test instruction is to direct the ignition testing (ignition test # 1) of Spent Nuclear Fuel (SNF) specimen SFEC5,4378-S2-E3A in a controlled temperature and atmosphere furnace. The specimen was sectioned from SNF element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF specimen SFEC5,4378-S2-E3A taken from the mid-length of the element SFEC5,4378.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) shall read and follow the steps in the procedure PTL-151: *Operation of Conditioning Furnace System.*

Equipment Reconfiguration Replace alumina tube with silicon carbide tube.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

SNF specimen SFEC5,4378-S2-E3A will be loaded into the resistively heated furnace shown schematically in Attachment 1. The silicon carbide furnace tube is preferred for this testing. If the furnace system is in standby condition, then skip steps 1.1 through 1.3 (if steps 1.1 through 1.3 are skipped, enter "N/A" in the appropriate boxes on the check list).

1.1 Perform the *Equipment Readiness/Start Up* steps of procedure PTL-151: *Operation of Conditioning Furnace System.* Record completion of each step with the operator's initials, time and date in the PTL log book.

1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 10 and 12 and opening valves 1, 5, 6, 7, 8, 9 and 11.

Before proceeding further in procedure PTL-151 perform the following steps:

1.3 Dry the furnace system before loading the test specimen using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective (i.e. moisture monitor reads > 5 ppm), then try the step 1.3.2 after discussions with the cognizant scientist. Otherwise, disregard step 1.3.2.

1.3.1 Dry the furnace system by flowing ultra high purity gas (e.g. 99.999% Ar) through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer. When a constant concentration is reached, stop the drying process.

Hold Point: Consult with the cognizant scientist before performing the alternate drying method in step 1.3.2 below and/or before proceeding with the remaining steps of this test instruction.

1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

1.4 Use a calibrated ruler (record the calibration information in blocks **7 & 8** on the data sheet of Attachment 2) with an accuracy of ± 1mm to measure and record in blocks **12** through **18** and **21** through **27** on the data sheet of Attachment 2 the specimen dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp "burrs" that interfere with the measurements, sand them off with silicon carbide abrasive paper. Do not use a lubricant.

1.5 Weigh the specimen twice on a QA Category 1 balance (record the balance calibration information in blocks **1, 2 & 3** on the data sheet of Attachment 2) with 0.1 mg sensitivity. Record the specimen weight in blocks **10 & 19** on the data sheet of Attachment 2.

1.6 Weigh the moisture trap that will be used on a QA category 1 balance (record the balance calibration information in blocks **4, 5 & 6** on the data sheet of Attachment 2) with 10 mg sensitivity. Repeat each measurement and record their weights in blocks **28 & 30** on data sheet of Attachment 2.

Note: If the subsequent steps are performed immediately following step 1.3, then make sure that the furnace system has cooled to the ambient hot cell temperature before loading the specimens. Minimize the time the furnace tube and gas lines are open to air in the hot cell.

1.7 Mount the specimen in the sample holder such that the specimen is in the center of the uniform heated zone when loaded into the furnace. Ensure that the specimen is in contact with the specimen thermocouple.

1.8 Load the sample holder into the furnace tube with extreme care, and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

1.9 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the PTL log book.

2.0 Ignition Test

The specimen will be heated to the point of ignition in a flowing dry air atmosphere and then quenched by purging the system with high purity argon. The moisture content of the off-gas will be detected by the moisture monitor, and the cumulative amount of water evolved will be trapped in the moisture traps. Before and after weights of the traps will be a measure of the water that came off. The hydrogen in the gas stream will be monitored.

2.1 Open the dry air cylinder and configure the valves so that the dry air flows through the system. Establish a dry air flow of 500 cc/min.

2.2 Configure valves 6 to 9 (i.e., either valves 6 and 8 opened with valves 7 and 9 closed or valves 6 and 8 closed with valves 7 and 9 opened) so that gas flows through only one of the moisture traps during the testing.

2.3 Verify that all the analytical instruments (e.g., moisture monitor and pressure gauge) are responding properly by looking at the readings on computer #1. Notify the cognizant scientist of any anomalous readings.

2.4 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the testing. The heating tape upstream from the furnace can be left turned off.

2.5 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual):

2.5.1 to heat at a constant rate of 15°C/min to 700±5°C and hold the 700±5°C for 15 minutes.

2.5.2 then allow the furnace to cool to ambient temperature.

2.6 Record in block 32 on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that computer #1 is monitoring and recording the:

2.6.1 Water vapor concentration with the moisture monitor
2.6.2 Pressure with the pressure gauge
2.6.3 Furnace and specimen temperatures

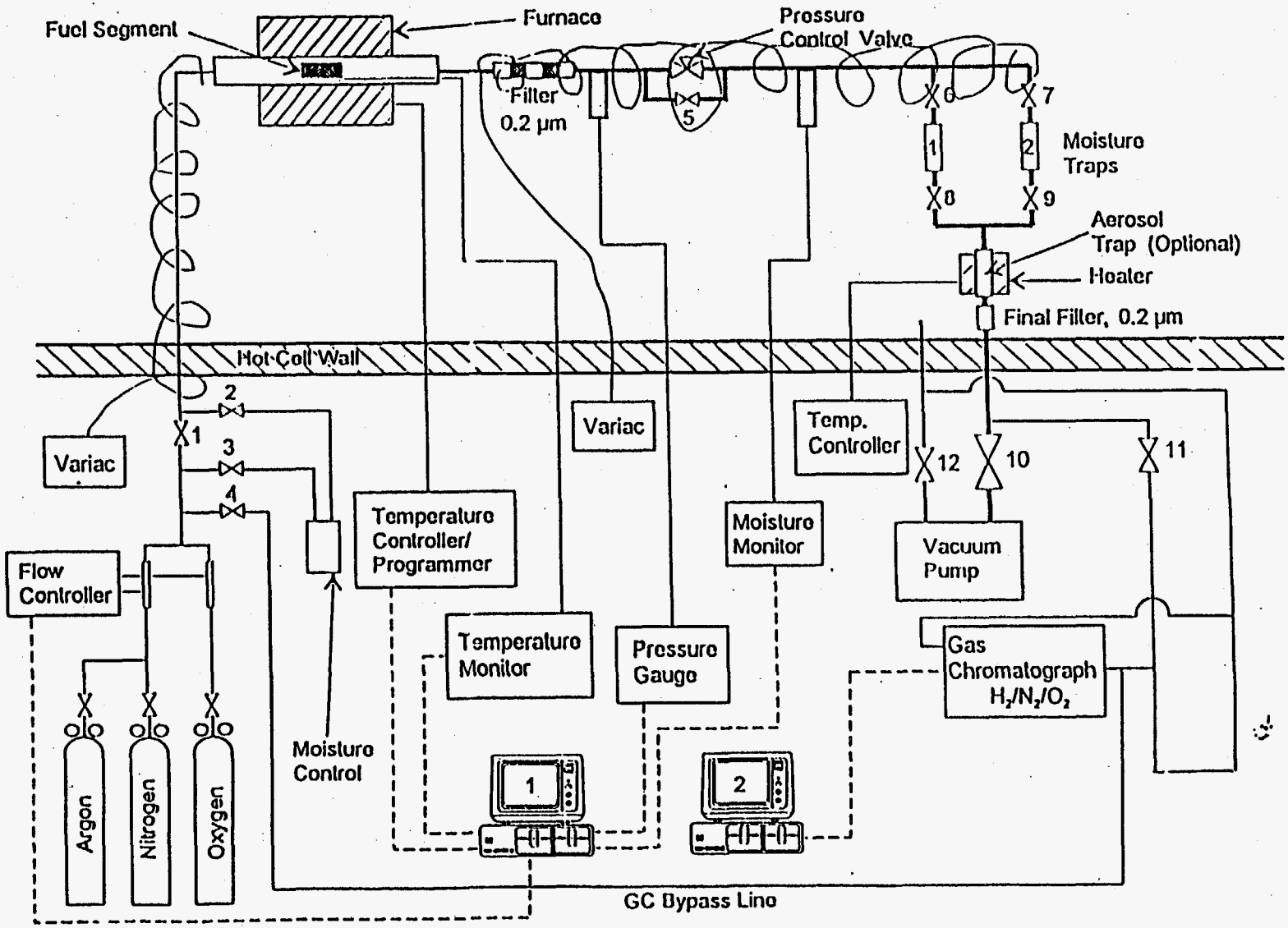
2.7 The operator shall continuously monitor the specimen temperature above 250°C. If the specimen temperature shows signs of rapid change (see Attachment 3) then switch the gas supply from dry air to ultra high purity argon to quench the reaction. Shut down the furnace manually with argon flowing through the system.

2.8 Remove the moisture trap that was used and weigh it on a QA category one balance with 10 mg sensitivity. Repeat the weight measurement and record the weights in blocks 29 & 31 on the data sheet of Attachment 2.

2.9 Open the furnace and carefully remove the specimen. Weigh the specimen twice using a QA Category 1 balance with 0.1 mg sensitivity. Record the weights in blocks 11 & 20 on the data sheet of Attachment 2. Record other observations in the PTL log book for inclusion in the project Laboratory Record Book (LRB).

2.10 Perform the test shut down steps that leave the furnace system in standby condition, that is, at ambient temperature with argon flow rate of about 100 cc/min.

Schematic Of The Furnace System



Attachment 2

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
4 - Moisture Trap Balance Description	5 - Moisture Trap Balance Calibration Number	6 - Calibration Expiration Date
	7 - Ruler Calibration Number	8 - Ruler Calibration Date

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S2-E3A								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	10	11	12	13	14	15	16	17	18
2	19	20	21	22	23	24	25	26	27

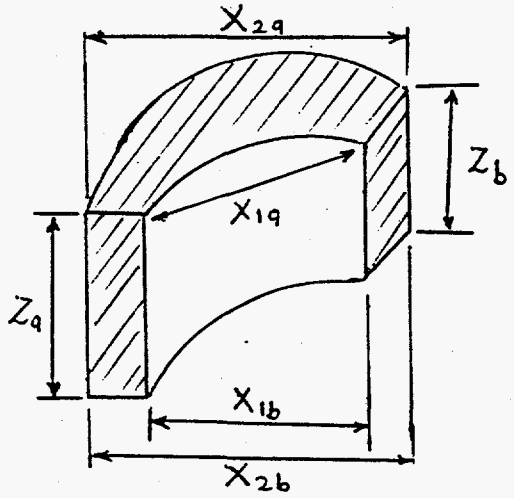
Measurement Number	Moisture Trap #1	
	Weight Before Test (g)	Weight After Test (g)
1	28	29
2	30	31

SIGNATURE/VERIFICATION BLOCKS

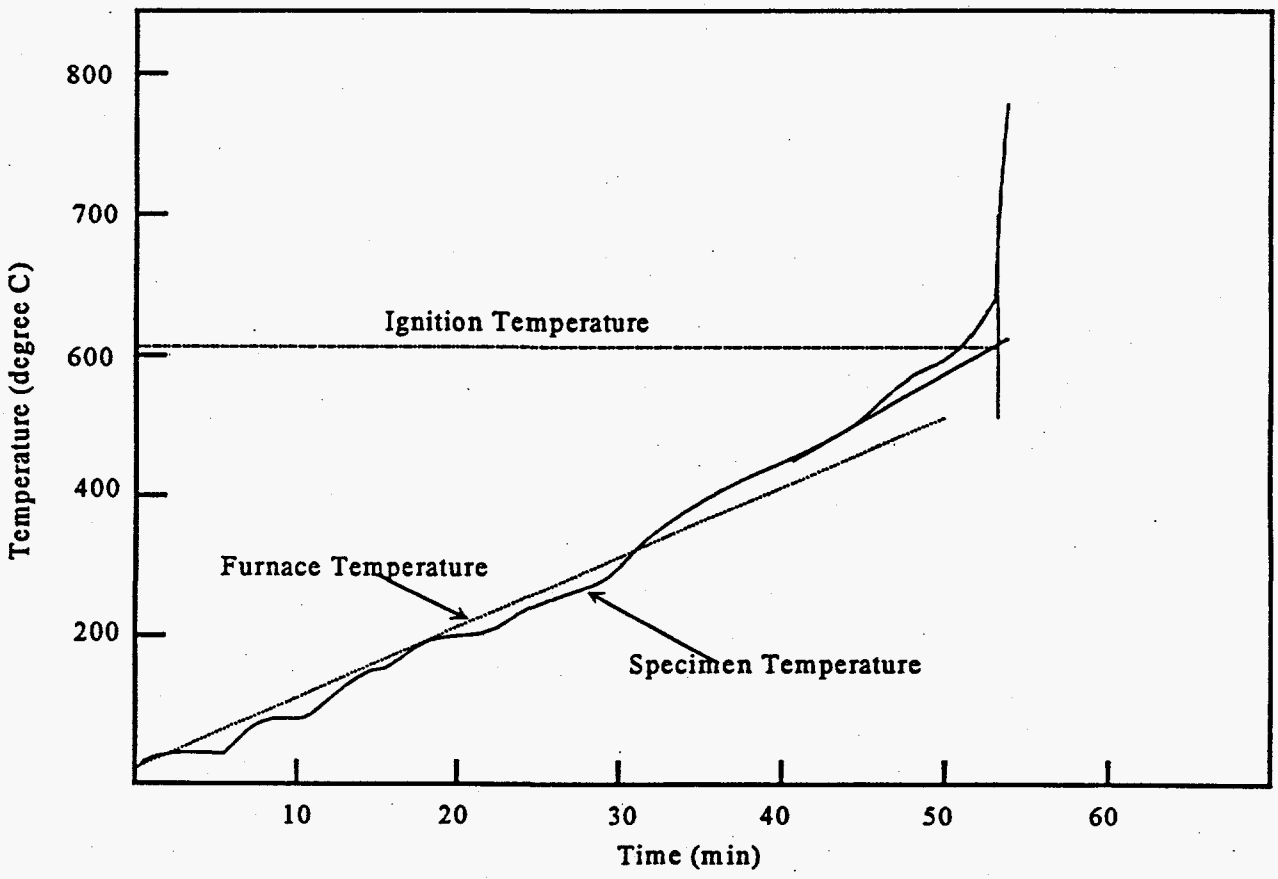
32

Attachment 3

Specimen Dimensions



Ignition Behavior of Metallic Uranium in Oxygen at Constant Heating Rate



CONTROLLED COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 8
Title: Hydrogen Release Test of Specimen SFEC5,4378-S2-E2		
Work Location: 327 (Building/Room or "General")	**TI No.: SNF-CT-013**	
Author: John Abrefah	**Effective Date:** 9/19/95 **Supersedes Date:**	

Signatures:

Author _____ (Signature) 9-20-95 (Date)

Technical Reviewer _____ (Signature) 9-20-95 (Date)

Concurrences:

PTL Manager _____ (James M. Scay) 9/20/95 (Date)

HSNFP Charact. Manager _____ (Ronald P. Omberg) 9/22/95 (Date)

Purpose/Scope The purpose of this test instruction is to direct the testing of hydrogen release (furnace run # 9) from Spent Nuclear Fuel (SNF) specimen SFEC5,4378-S2-E2. The specimen was sectioned from SNF element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF specimen SFEC5,4378-S2-E2 taken from the mid-length of the element SFEC5,4378. This specimen was decladded by cutting off the outer and inner cladding using a slow speed saw (see TI # SNF-CT-0010). The specimen has been stored under argon atmosphere in a sealed container.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) should read and follow the steps in procedure PTL-151: *Operation of Conditioning Furnace System.*

Equipment Reconfiguration The furnace system was reconfigured to reduce moisture content in the gas stream following an observation in the furnace run # 8 of higher moisture content. The steps involved insertion of a moisture trap in the upstream line close to the gas supply, a new furnace tube and upstream gas line with a heating tape.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

SNF specimen SFEC5,4378-S2-E2 will be loaded into the resistively heated furnace shown schematically in Attachment 1 to conduct hydrogen release test. If the furnace system is in standby condition then skip steps 1.1 through 1.3

1.1 Perform the ***Equipment Readiness/Start Up*** steps of the procedure PTL-151: *Operation of Conditioning Furnace System.* Record completion of each step with the operator's initials, time and date in the PTL log book.

1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 6, 8, 10 and 12 and opening valves 1, 5, 7, 9 and 11.

Note: For evacuating the system, CLOSE valves 2, 3, 4, and 11, and OPEN valves 1, 5, 6, 7, 8, 9, 10 and 12.

1.3 Dry the furnace system before loading the test specimens using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective then try the step 1.3.2 after discussions with the cognizant scientist otherwise disregard step 1.3.2.

1.3.1 Dry the furnace system by flowing high purity gas (e.g. 99.999% Ar) through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being acquired by the computer and when a constant concentration is reached, stop the drying process.

Hold Point: Consult with the cognizant scientist before performing the alternate drying method in step 1.3.2 below and/or before proceeding to the remaining steps of this test instruction.

1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being acquired by the computer and when a constant concentration is reached, stop the drying process.

1.4 Use a calibrated ruler (record the calibration information in blocks 7 & 8 on the data sheet of Attachment 2) with an accuracy of ± 1mm to measure and record in blocks 12 through 27 on the data sheet of Attachment 2, the specimen dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp 'burrs' that interfere with the measurements, gring them off with silicon carbide abrasive paper. **Do not use a lubricant.**

1.5 Weigh the specimen twice on a QA category one balance (record the balance calibration information in blocks 1 through 3 on the data sheet of Attachment 2) with 0.1 mg sensitivity and record its weight in blocks 10 & 19 on the data sheet of Attachment 2.

1.6 Mount the specimen in the sample holder such that the specimen will be in the center of the uniform heated zone when loaded into the furnace. Ensure that the specimen is in contact with the specimen thermocouple.

1.7 Load the sample holder into the furnace tube and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

1.8 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the PTL log book.

1.9 Prior to beginning the test, determine the baseline moisture content in the system. Ensure the moisture content is less than 1 ppm. Then, dry the specimen in a vacuum of about 10 Torr or less and heating the furnace system including the specimen to about 50°C.

2.0 Hydrogen Release Test

The specimen will be initially heated to release most of the hydrogen. If the time required to achieve total release of most of the hydrogen is greater than specified in step 2.5.2 (i.e., 48 hrs) then consult with the cognizant scientist before shutting down the test.

2.1 Purge the system with high purity (e.g. 99.999%) argon gas for a period of at least 30 minutes. Use an argon flow rate of 100±2 cm^3/min.

2.2 Verify that all the analytical instruments (e.g., GC, moisture monitor, pressure gauge, mass flow controllers and the computers) are responding properly by looking at the readings on both computers. Notify the cognizant scientist of any anomalous readings.

2.3 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the testing. The heating tape upstream from the furnace will be turned off or on at the direction of cognizant scientist.

2.4 Configure valves 6 to 9 of Attachment 1 (i.e., **either** valves 6 and 8 opened with valves 7 and 9 closed **or** valves 6 and 8 closed with 7 and 9 opened) so that gas flows through only one of the moisture traps.

2.5 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual):

2.5.1 to heat the furnace at a constant rate, within the range of 10 to 15°C/min, to 300±2°C

2.5.2 hold the 300±2°C for at least 48 hours or until the hydrogen in the off gas stream is below 1 ppm.

2.5.3 then allow the furnace to cool to ambient temperature.

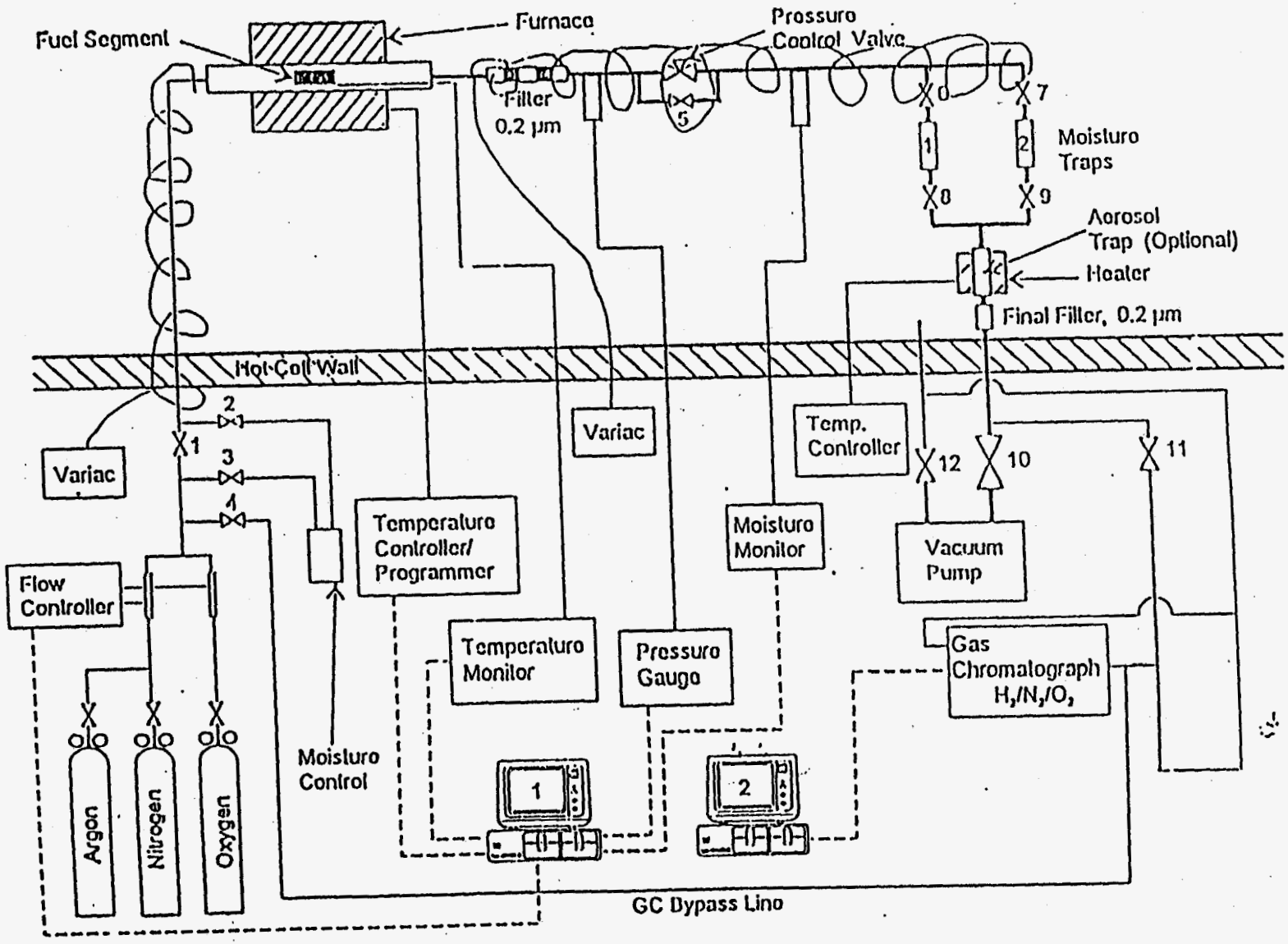
2.6 Record in block **28** on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that the computers are monitoring and recording the:

2.6.1 Water vapor concentration with the moisture monitor
2.6.2 Hydrogen and oxygen concentrations with the GC
2.6.3 Furnace and specimen temperatures

2.7 Open the furnace and carefully remove the specimen for weighing using a QA category one balance with 0.1 mg sensitivity. Weigh the specimen twice and record its weight in blocks **11 & 20** on the data sheet of Attachment 2. Record other observations in the PTL log book.

2.8 Perform the test shut down steps that leave the furnace system in standby condition, that is, at ambient temperature with an argon flow rate of about 100 cc/min.

Schematic Of The Furnace System



Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
4 - Moisture Trap Balance Description	5 - Moisture Trap Balance Calibration Number	6 - Calibration Expiration Date
	7 - Ruler Calibration Number	8 - Ruler Calibration Date

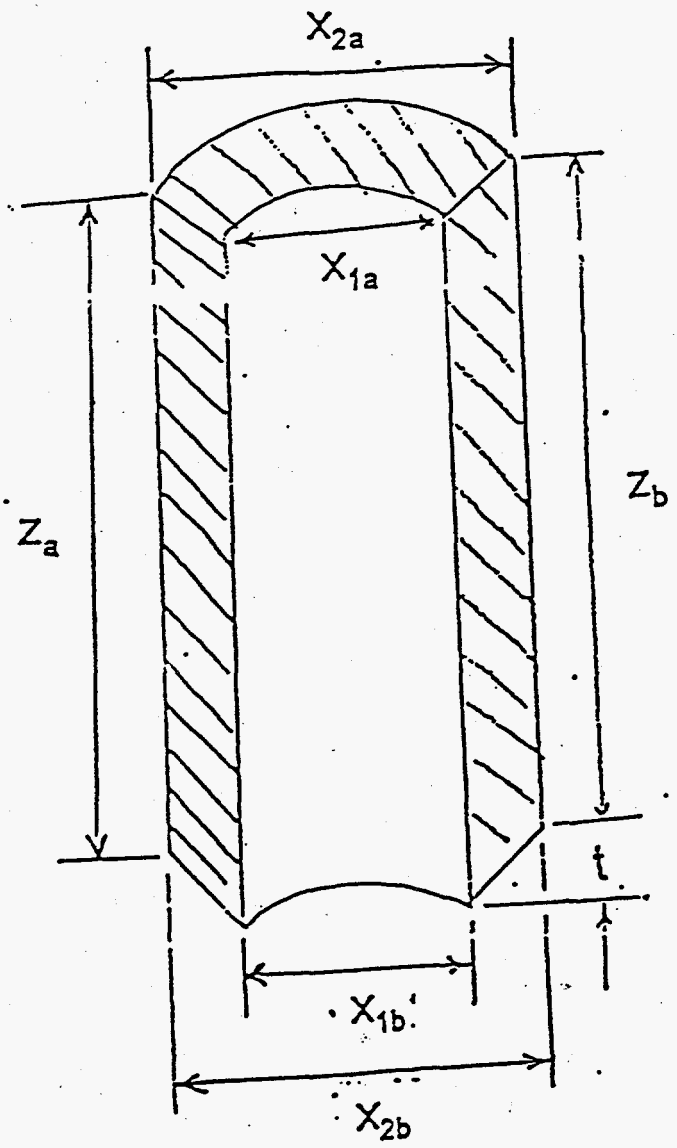
SPECIMEN MEASUREMENTS

Measurement Number	9 - Specimen Identification Code SFEC5,4378-S2-E2								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	10	11	12	13	14	15	16	17	18
2	19	20	21	22	23	24	25	26	27

SYSTEM STATUS

28 - Time/Date

Specimen Dimensions



CONTROLLED COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 8

Title: Hydrogen Release Test of Cladding Specimen SFEC5,4378-S2-E1-CD1 (Run # 10)

Work Location: 327 (Building/Room or "General")	**TI No.:** SNF-CT-014
Author: John Abrefah	**Effective Date:** 10/09/95 **Supersedes Date:**

Signatures:

Author _____ 10-09-95
(Signature) (Date)

Technical Reviewer _____ 10-9-95
(Signature) (Date)

Concurrences:

PTL Manager _____ 10/9/95
(James M. Seay) (Date)

HSNFP Charact. Manager _____ 10/9/95
(Ronald P. Omberg) (Date)

Purpose/Scope The purpose of this test instruction is to direct the testing of hydrogen release (furnace run # 10) from a cladding specimen SFEC5,4378-S2-E1-CD1. The specimen was sectioned from Spent Nuclear Fuel (SNF) element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to cladding specimen SFEC5,4378-S2-E1-CD1 taken from the mid-length of the element SFEC5,4378. This specimen was de-fueled by dissolving the fuel material from the cladding using a nitric acid solution.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) should read and follow the steps in procedure PTL-151: *Operation of Conditioning Furnace System.*

Equipment Reconfiguration The same as in TI # SNF-CT-013.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

Cladding specimen SFEC5,4378-S2-E1-CD1 will be loaded into the resistively heated furnace shown schematically in Attachment 1 to conduct the hydrogen release test. If the furnace system is in standby condition then skip steps 1.1 through 1.3

1.1 Perform the *Equipment Readiness/Start Up* steps of the procedure PTL-151: *Operation of Conditioning Furnace System.* Record completion of each step with the operator's initials, time and date in the PTL log book.

1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 6, 8, 10 and 12 and opening valves 1, 5, 7, 9 and 11.

Note: For evacuating the system, CLOSE valves 2, 3, 4, and 11, and OPEN valves 1, 5, 6, 7, 8, 9, 10 and 12.

1.3 Dry the furnace system before loading the test specimens using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective then try the step 1.3.2 after discussions with the cognizant scientist otherwise disregard step 1.3.2.

1.3.1 Dry the furnace system by flowing high purity (99.999%) argon gas through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being acquired by the computer and when a constant concentration is reached, stop the drying process.

Hold Point: Consult with the cognizant scientist before performing the alternate drying method in step 1.3.2 below and/or before proceeding to the remaining steps of this test instruction.

1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being acquired by the computer and when a constant concentration is reached, stop the drying process.

1.4 Use a calibrated ruler (record the calibration information in blocks 4 & 5 on the data sheet of Attachment 2) with an accuracy of ± 1mm to measure and record in blocks 9 through 11 and 14 through 16 on the data sheet of Attachment 2, the specimen dimensions shown in Attachment 3. Repeat each dimensional measurement.

1.5 Weigh the specimen twice on a QA Category 1 balance (record the balance calibration information in blocks 1 through 3 on the data sheet of Attachment 2) with 0.1 mg sensitivity and record its weight in blocks 7 & 12 on the data sheet of Attachment 2.

1.6 Mount the specimen in the sample holder such that the specimen will be in the center of the uniform heated zone when loaded into the furnace. Ensure that the specimen is in contact with the specimen thermocouple.

1.7 Load the sample holder into the furnace tube and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

1.8 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the PTL log book.

1.9 Prior to beginning the test, determine the baseline moisture content in the system. Ensure the moisture content is less than 1 ppm. Then, dry the specimen in a vacuum of about 10 Torr or less and heating the furnace system including the specimen to about 50°C.

2.0 Hydrogen Release Test

The specimen will be initially heated to release most of the hydrogen. If the time required to achieve release of most of the hydrogen in the specimen is greater than specified in step 2.5.2 (i.e., 48 hrs) then consult with the cognizant scientist before shutting down the test.

2.1 Purge the system with high purity (e.g. 99.999%) argon gas for a period of at least 30 minutes. Use an argon flow rate of 100±2 cm^3/min.

2.2 Verify that all the analytical instruments (e.g., GC, moisture monitor, pressure gauge, mass flow controllers and the computers) are responding properly by looking at the readings on both computers. Notify the cognizant scientist of any anomalous readings.

2.3 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the testing. The heating tape upstream from the furnace will be turned off or on at the direction of cognizant scientist.

2.4 Configure valves 6 to 9 of Attachment 1 at the direction of the cognizant scientist (i.e., **either** valves 6 and 8 opened with valves 7 and 9 closed **or** valves 6 and 8 closed with 7 and 9 opened) so that gas flows through only one of the moisture traps.

2.5 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual):

2.5.1 to heat the furnace at a constant rate, within the range of 10 to 15°C/min, to 300±2°C

2.5.2 hold the 300±2°C for at least 48 hours or until the hydrogen in the off gas stream is below 1 ppm.

2.5.3 then allow the furnace to cool to ambient temperature.

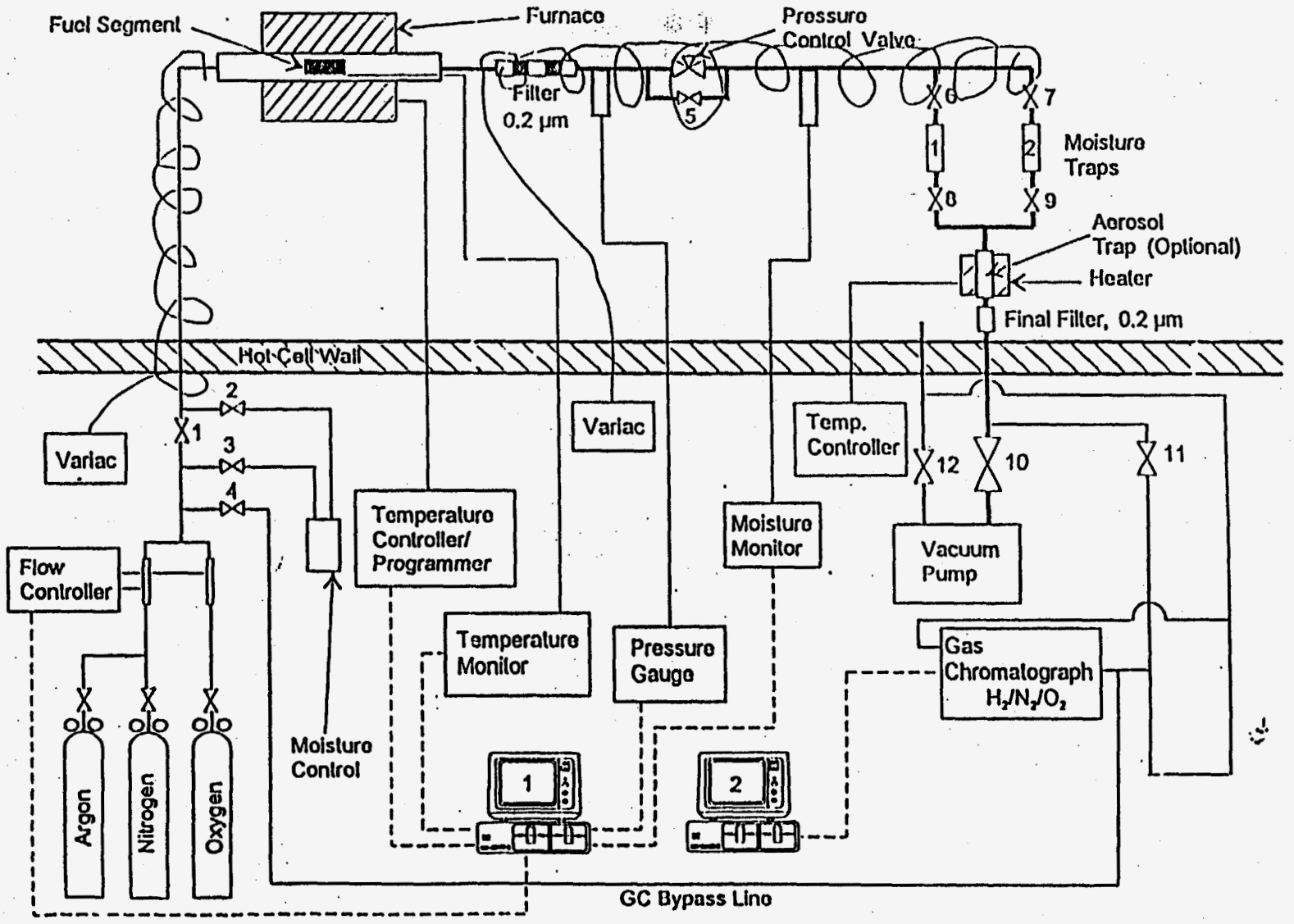
2.6 Record in block 17 on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that the computers are monitoring and recording the:

2.6.1 Water vapor concentration with the moisture monitor
2.6.2 Hydrogen and oxygen concentrations with the GC
2.6.3 Furnace and specimen temperatures

2.7 Open the furnace and carefully remove the specimen for weighing using a QA Category 1 balance with 0.1 mg sensitivity. Weigh the specimen twice and record its weight in blocks 8 & 13 on the data sheet of Attachment 2. Record other observations in the PTL log book.

2.8 Perform the test shut down steps that leave the furnace system in standby condition, that is, at ambient temperature with an argon flow rate of about 100 cc/min.

Schematic Of The Furnace System



Attachment 2

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
	4 - Ruler Calibration Number	5 - Ruler Calibration Date

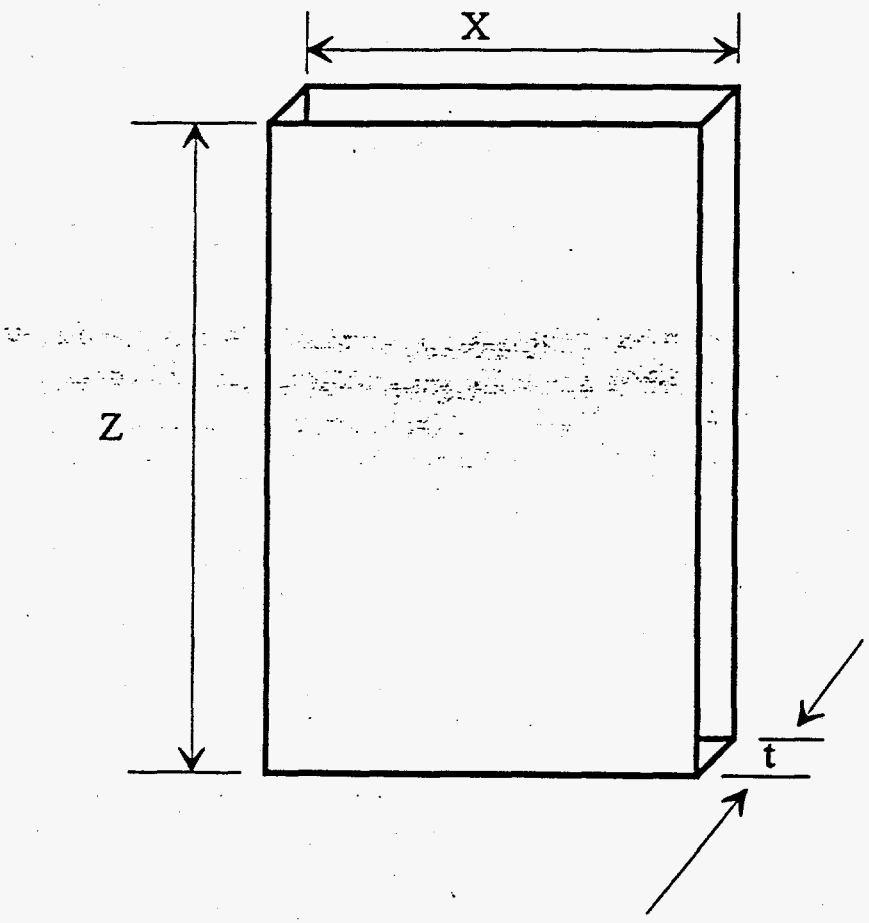
SPECIMEN MEASUREMENTS

Measurement Number	6 - Specimen Identification Code SFEC5,4378-S2-E1-CD1				
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)		
			Length, Z	Thickness, t	Width, X
1	7	8	9	10	11
2	12	13	14	15	16

SYSTEM STATUS

17 - Time/Date

Specimen Dimensions



CONTROLLED
COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 8
Title: Ignition testing of Specimen SFEC5,4378-S2-E4A		
Work Location: 327 (Building/Room or "General")	**TI No.: SNF-CT-015**	
Author: John Abrefah	**Effective Date:** 10/23/95 **Supersedes Date:**	

Signatures:

Author _____ (Signature) 10-19-95 (Date)

Technical Reviewer _____ (Signature) 10-19-95 (Date)

Concurrences:

PTL Manager _____ (James M. Seay) 10/25/95 (Date)

HSNFP Charact. Manager _____ (Ronald P. Omberg) 10/27/95 (Date)

Purpose/Scope The purpose of this test instruction is to direct the ignition testing (ignition test # 2) of Spent Nuclear Fuel (SNF) specimen SFEC5,4378-S2-E4A in a controlled temperature and atmosphere furnace. The specimen was sectioned from SNF element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF specimen SFEC5,4378-S2-E4A taken from the mid-length of the element SFEC5,4378.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) shall read and follow the steps in the procedure PTL-151: *Operation of Conditioning Furnace System.*

Equipment Reconfiguration Replace alumina tube with silicon carbide tube.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

SNF specimen SFEC5,4378-S2-E4A will be loaded into the resistively heated furnace shown schematically in Attachment 1. The silicon carbide furnace tube is preferred for this testing. If the furnace system is in standby condition, then skip steps 1.1 through 1.3 (if steps 1.1 through 1.3 are skipped, enter "N/A" in the appropriate boxes on the check list).

1.1 Perform the *Equipment Readiness/Start Up* steps of procedure PTL-151: *Operation of Conditioning Furnace System.* Record completion of each step with the operator's initials, time and date in the PTL log book.

1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 10 and 12 and opening valves 1, 5, 6, 7, 8, 9 and 11.

Before proceeding further in procedure PTL-151 perform the following steps:

1.3 Dry the furnace system before loading the test specimen using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective (i.e. moisture monitor reads > 5 ppm), then try the step 1.3.2 after discussions with the cognizant scientist. Otherwise, disregard step 1.3.2.

1.3.1 Dry the furnace system by flowing ultra high purity gas (e.g. 99.999% Ar) through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer. When a constant concentration is reached, stop the drying process.

Hold Point: Consult with the cognizant scientist before performing the alternate drying method in step 1.3.2 below and/or before proceeding with the remaining steps of this test instruction.

1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

1.4 Use a calibrated ruler (record the calibration information in blocks 7 & 8 on the data sheet of Attachment 2) with an accuracy of ± 1mm to measure and record in blocks 12 through 18 and 21 through 27 on the data sheet of Attachment 2 the specimen dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp "burrs" that interfere with the measurements, sand them off with silicon carbide abrasive paper. Do not use a lubricant.

1.5 Weigh the specimen twice on a QA Category 1 balance (record the balance calibration information in blocks 1, 2 & 3 on the data sheet of Attachment 2) with 0.1 mg sensitivity. Record the specimen weight in blocks 10 & 19 on the data sheet of Attachment 2.

1.6 Weigh the moisture trap that will be used on a QA category 1 balance (record the balance calibration information in blocks 4, 5 & 6 on the data sheet of Attachment 2) with 10 mg sensitivity. Repeat each measurement and record their weights in blocks 28 & 30 on data sheet of Attachment 2.

Note: If the subsequent steps are performed immediately following step 1.3, then make sure that the furnace system has cooled to the ambient hot cell temperature before loading the specimens. Minimize the time the furnace tube and gas lines are open to air in the hot cell.

1.7 Mount the specimen in the sample holder such that the specimen is in the center of the uniform heated zone when loaded into the furnace. Ensure that the specimen is in contact with the specimen thermocouple.

1.8 Load the sample holder into the furnace tube with extreme care, and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

1.9 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the PTL log book.

2.0 Ignition Test

The specimen will be heated to the point of ignition in a flowing dry air atmosphere and then quenched by purging the system with high purity argon. The moisture content of the off-gas will be detected by the moisture monitor, and the cumulative amount of water evolved will be trapped in the moisture traps. Before and after weights of the traps will be a measure of the water that came off. The hydrogen in the gas stream will be monitored.

2.1 Open the dry air cylinder and configure the valves so that the dry air flows through the system. Establish a dry air flow of 500 cc/min.

2.2 Configure valves 6 to 9 (i.e., either valves 6 and 8 opened with valves 7 and 9 closed or valves 6 and 8 closed with valves 7 and 9 opened) so that gas flows through only one of the moisture traps during the testing.

2.3 Verify that all the analytical instruments (e.g., moisture monitor and pressure gauge) are responding properly by looking at the readings on computer #1. Notify the cognizant scientist of any anomalous readings.

2.4 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the testing. The heating tape upstream from the furnace can be left turned off.

2.5 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual):

2.5.1 to heat at a constant rate of 15°C/min to 700±5°C and hold the 700±5°C for 15 minutes.

2.5.2 then allow the furnace to cool to ambient temperature.

2.6 Record in block 32 on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that computer #1 is monitoring and recording the:

2.6.1 Water vapor concentration with the moisture monitor
2.6.2 Pressure with the pressure gauge
2.6.3 Furnace and specimen temperatures

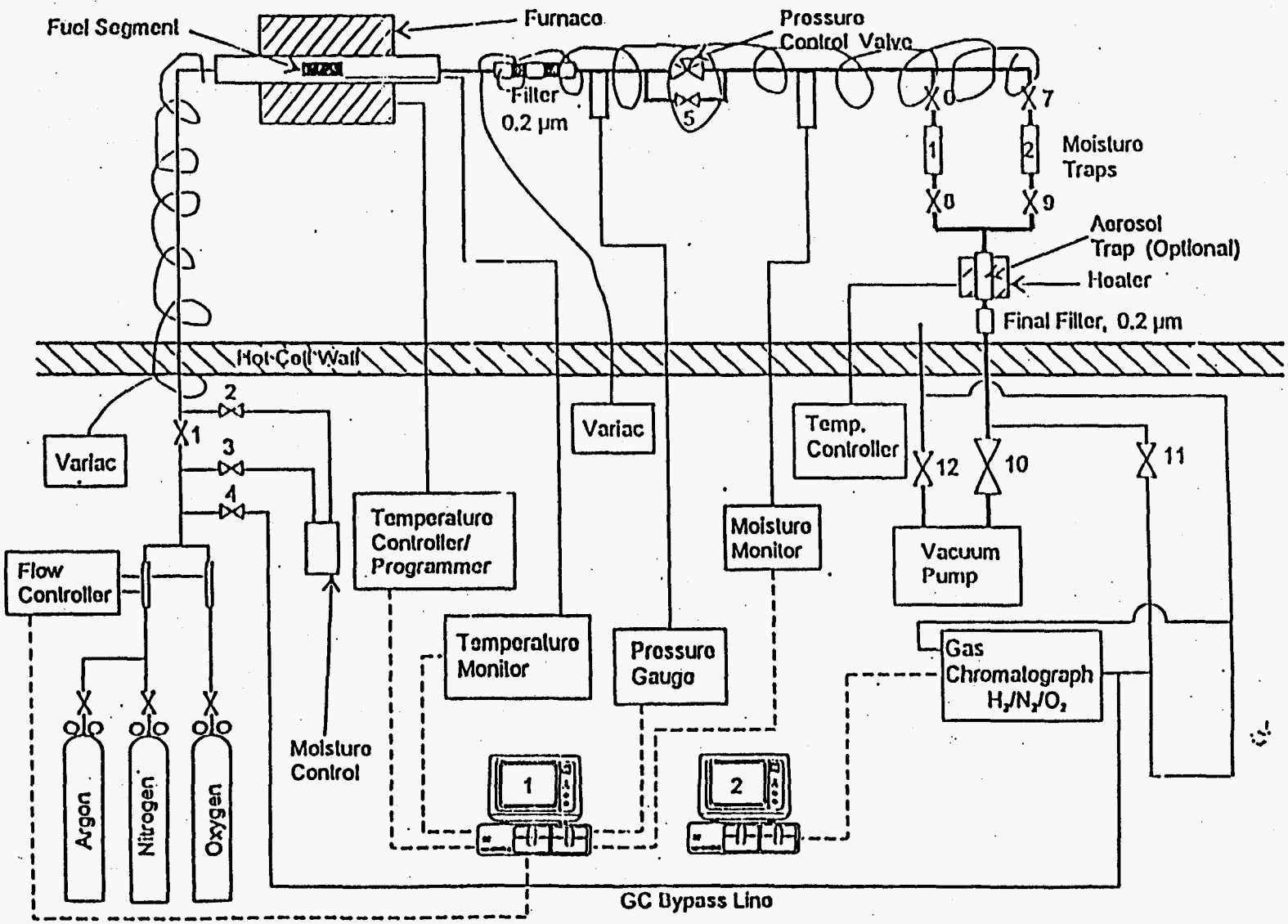
2.7 The operator shall continuously monitor the specimen temperature above 250°C. If the specimen temperature shows signs of rapid change (see Attachment 3) then switch the gas supply from dry air to ultra high purity argon (flow rate of about 1 liter/min) to quench the reaction. Shut down the furnace manually with argon flowing through the system.

2.8 Remove the moisture trap that was used and weigh it on a QA category one balance with 10 mg sensitivity. Repeat the weight measurement and record the weights in blocks 29 & 31 on the data sheet of Attachment 2.

2.9 Open the furnace and carefully remove the specimen. Weigh the specimen twice using a QA Category 1 balance with 0.1 mg sensitivity. Record the weights in blocks 11 & 20 on the data sheet of Attachment 2. Record other observations in the PTL log book for inclusion in the project Laboratory Record Book (LRB).

2.10 Perform the test shut down steps that leave the furnace system in standby condition, that is, at ambient temperature with argon flow rate of about 100 cc/min.

Schematic Of The Furnace System



Attachment 2

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
4 - Moisture Trap Balance Description	5 - Moisture Trap Balance Calibration Number	6 - Calibration Expiration Date
	7 - Ruler Calibration Number	8 - Ruler Calibration Date

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S2-E4A								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	10	11	12	13	14	15	16	17	18
2	19	20	21	22	23	24	25	26	27

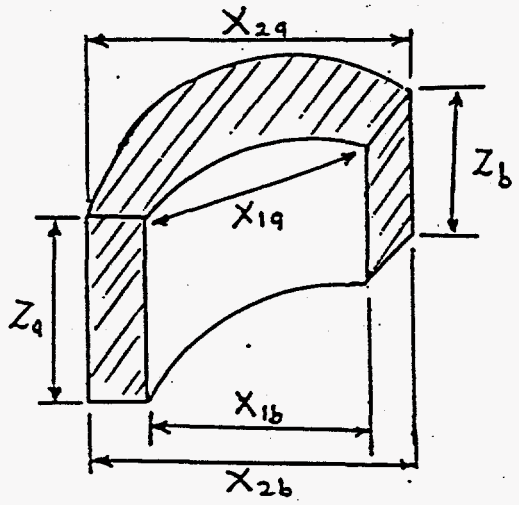
Measurement Number	Moisture Trap #1	
	Weight Before Test (g)	Weight After Test (g)
1	28	29
2	30	31

SIGNATURE/VERIFICATION BLOCKS

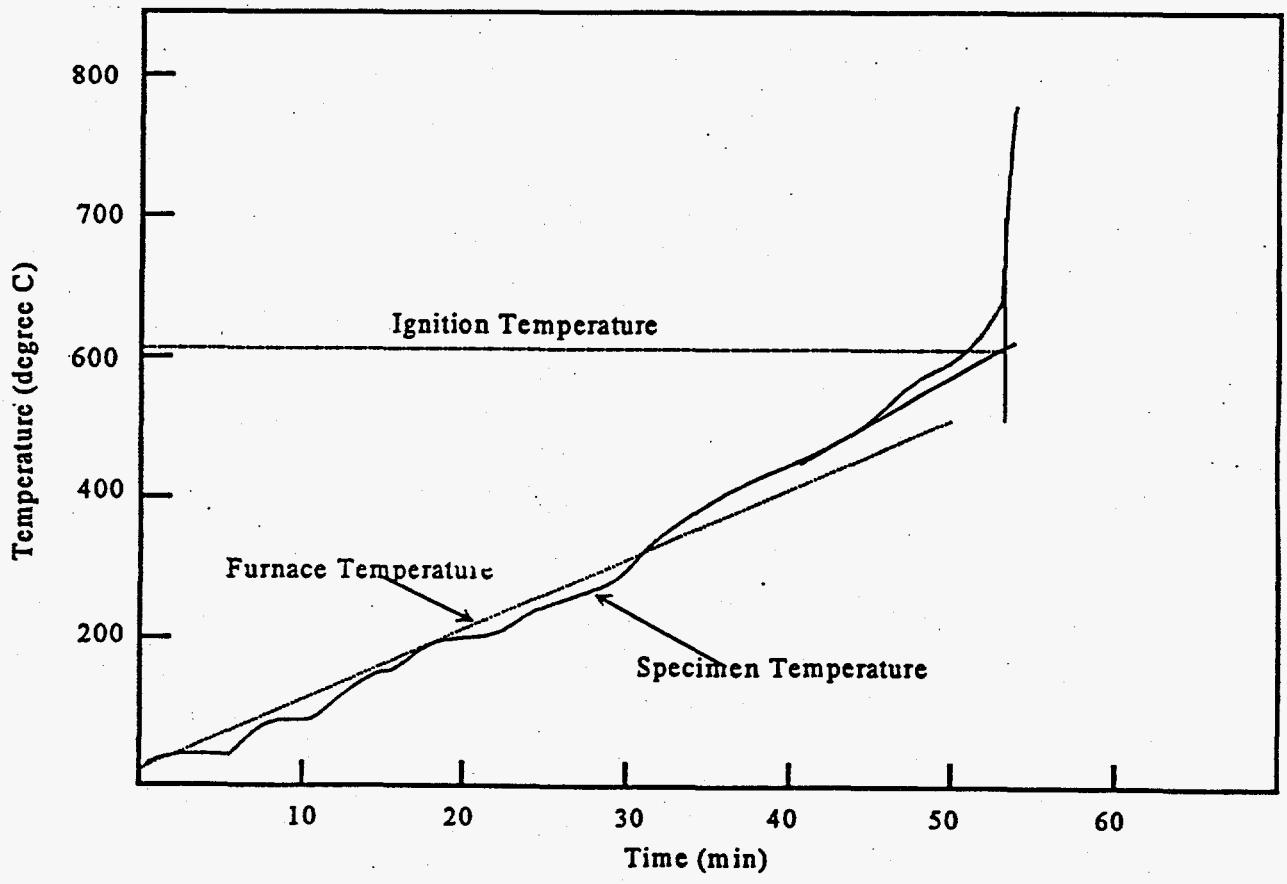
32

Attachment 3

Specimen Dimensions



Ignition Behavior of Metallic Uranium in Oxygen at Constant Heating Rate



CONTROLLED
COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 8
Title: Ignition testing of Specimen SFEC5,4378-S2-H		
Work Location: 327 (Building/Room or "General")	**TI No.: SNF-CT-016**	
Author: John Abrefah	**Effective Date:** 11/01/95 **Supersedes Date:**	

Signatures:

Author _____ (Signature) 10-19-95 (Date)

Technical Reviewer _____ (Signature) 10-19-95 (Date)

Concurrences:

PTL Manager _____ (James M. Seay) 10/26/95 (Date)

HSNFP Charact. Manager _____ (Ronald P. Omberg) 10/27/95 (Date)

Purpose/Scope The purpose of this test instruction is to direct the ignition testing (ignition test # 3) of post-conditioned Spent Nuclear Fuel (SNF) specimen SFEC5,4378-S2-H in a controlled temperature and atmosphere furnace. The specimen was sectioned from SNF element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF specimen SFEC5,4378-S2-H taken from the mid-length of the element SFEC5,4378 and conditioned in a controlled atmosphere furnace (TI #: SNF-CT-005).

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) shall read and follow the steps in the procedure PTL-151: *Operation of Conditioning Furnace System.*

Equipment Reconfiguration Replace alumina tube with silicon carbide tube.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

SNF specimen SFEC5,4378-S2-H will be loaded into the resistively heated furnace shown schematically in Attachment 1. The silicon carbide furnace tube is preferred for this testing. If the furnace system is in standby condition, then skip steps 1.1 through 1.3 (if steps 1.1 through 1.3 are skipped, enter "N/A" in the appropriate boxes on the check list).

1.1 Perform the ***Equipment Readiness/Start Up*** steps of procedure PTL-151: *Operation of Conditioning Furnace System.* Record completion of each step with the operator's initials, time and date in the PTL log book.

1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 10 and 12 and opening valves 1, 5, 6, 7, 8, 9 and 11.

Before proceeding further in procedure PTL-151 perform the following steps:

1.3 Dry the furnace system before loading the test specimen using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective (i.e. moisture monitor reads > 5 ppm), then try the step 1.3.2 after discussions with the cognizant scientist. Otherwise, disregard step 1.3.2.

1.3.1 Dry the furnace system by flowing ultra high purity gas (e.g. 99.999% Ar) through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer. When a constant concentration is reached, stop the drying process.

Hold Point: Consult with the cognizant scientist before performing the alternate drying method in step 1.3.2 below and/or before proceeding with the remaining steps of this test instruction.

1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

1.4 Use a calibrated ruler (record the calibration information in blocks 7 & 8 on the data sheet of Attachment 2) with an accuracy of ± 1mm to measure and record in blocks 12 through 18 and 21 through 27 on the data sheet of Attachment 2 the specimen dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp "burrs" that interfere with the measurements, sand them off with silicon carbide abrasive paper. Do not use a lubricant.

1.5 Weigh the specimen twice on a QA Category 1 balance (record the balance calibration information in blocks 1, 2 & 3 on the data sheet of Attachment 2) with 0.1 mg sensitivity. Record the specimen weight in blocks 10 & 19 on the data sheet of Attachment 2.

1.6 Weigh the moisture trap that will be used on a QA category 1 balance (record the balance calibration information in blocks 4, 5 & 6 on the data sheet of Attachment 2) with 10 mg sensitivity. Repeat each measurement and record their weights in blocks 28 & 30 on data sheet of Attachment 2.

Note: If the subsequent steps are performed immediately following step 1.3, then make sure that the furnace system has cooled to the ambient hot cell temperature before loading the specimens. Minimize the time the furnace tube and gas lines are open to air in the hot cell.

1.7 Mount the specimen in the sample holder such that the specimen is in the center of the uniform heated zone when loaded into the furnace. Ensure that the specimen is in contact with the specimen thermocouple.

1.8 Load the sample holder into the furnace tube with extreme care, and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

1.9 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the PTL log book.

2.0 Ignition Test

The specimen will be heated to the point of ignition in a flowing dry air atmosphere and then quenched by purging the system with high purity argon. The moisture content of the off-gas will be detected by the moisture monitor, and the cumulative amount of water evolved will be trapped in the moisture traps. Before and after weights of the traps will be a measure of the water that came off. The hydrogen in the gas stream will be monitored.

2.1 Open the dry air cylinder and configure the valves so that the dry air flows through the system. Establish a dry air flow of 500 cc/min.

2.2 Configure valves 6 to 9 (i.e., either valves 6 and 8 opened with valves 7 and 9 closed or valves 6 and 8 closed with valves 7 and 9 opened) so that gas flows through only one of the moisture traps during the testing.

2.3 Verify that all the analytical instruments (e.g., moisture monitor and pressure gauge) are responding properly by looking at the readings on computer #1. Notify the cognizant scientist of any anomalous readings.

2.4 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the testing. The heating tape upstream from the furnace can be left turned off.

2.5 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual):

2.5.1 to heat at a constant rate of 15°C/min to 700±5°C and hold the 700±5°C for 15 minutes.

2.5.2 then allow the furnace to cool to ambient temperature.

2.6 Record in block 32 on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that computer #1 is monitoring and recording the:

2.6.1 Water vapor concentration with the moisture monitor
2.6.2 Pressure with the pressure gauge
2.6.3 Furnace and specimen temperatures

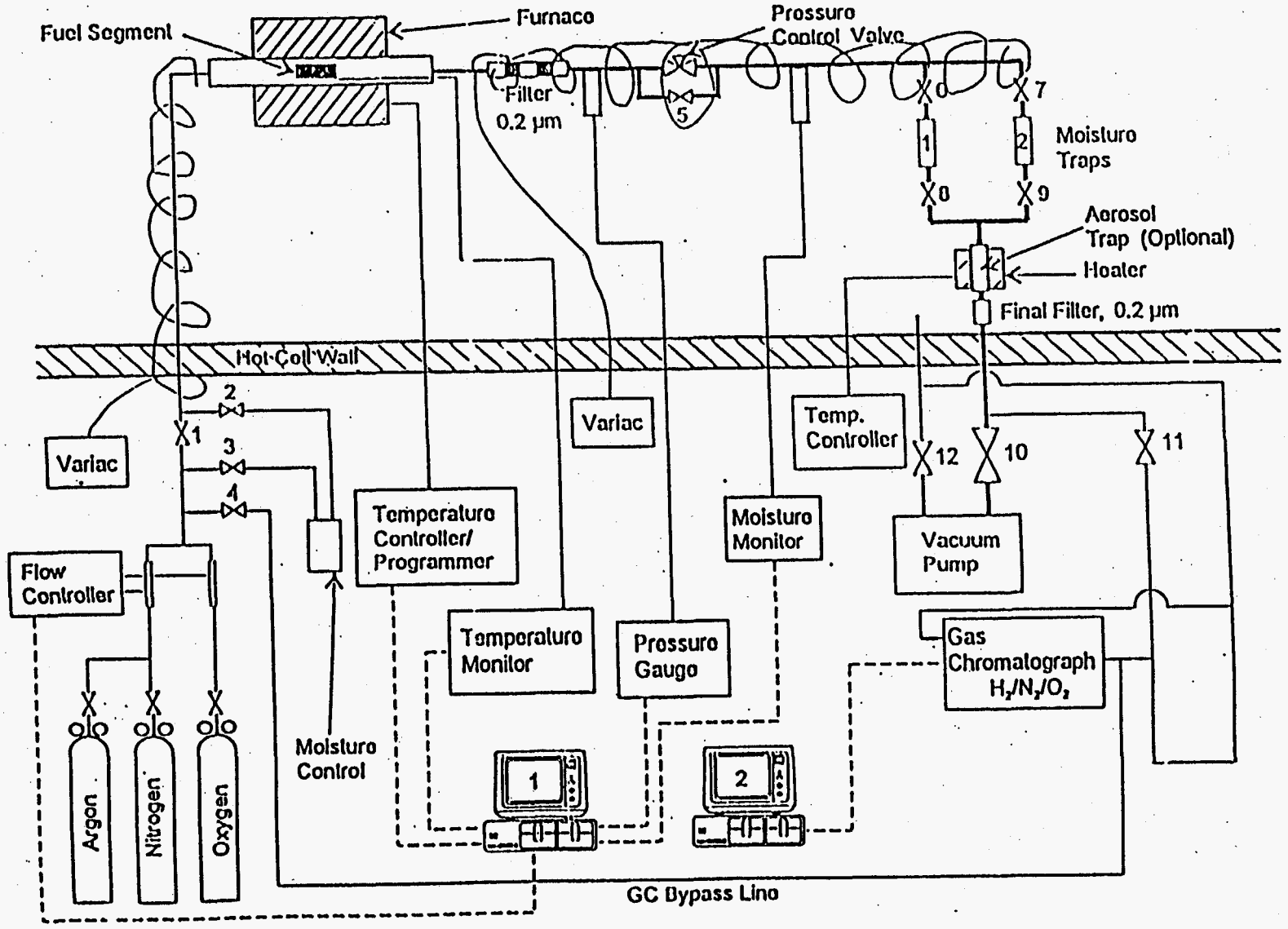
2.7 The operator shall continuously monitor the specimen temperature above 250°C. If the specimen temperature shows signs of rapid change (see Attachment 3) then switch the gas supply from dry air to ultra high purity argon (flow rate of about 1 liter/min) to quench the reaction. Shut down the furnace manually with argon flowing through the system.

2.8 Remove the moisture trap that was used and weigh it on a QA category one balance with 10 mg sensitivity. Repeat the weight measurement and record the weights in blocks 29 & 31 on the data sheet of Attachment 2.

2.9 Open the furnace and carefully remove the specimen. Weigh the specimen twice using a QA Category 1 balance with 0.1 mg sensitivity. Record the weights in blocks 11 & 20 on the data sheet of Attachment 2. Record other observations in the PTL log book for inclusion in the project Laboratory Record Book (LRB).

2.10 Perform the test shut down steps that leave the furnace system in standby condition, that is, at ambient temperature with argon flow rate of about 100 cc/min.

Schematic Of The Furnace System



Attachment 2

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
4 - Moisture Trap Balance Description	5 - Moisture Trap Balance Calibration Number	6 - Calibration Expiration Date
	7 - Ruler Calibration Number	8 - Ruler Calibration Date

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S2-H								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	10	11	12	13	14	15	16	17	18
2	19	20	21	22	23	24	25	26	27

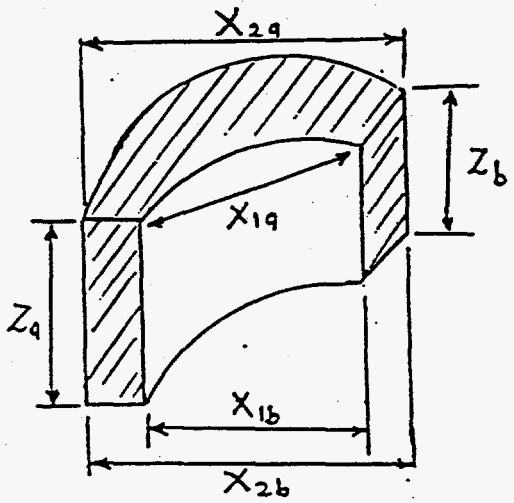
Measurement Number	Moisture Trap #1	
	Weight Before Test (g)	Weight After Test (g)
1	28	29
2	30	31

SIGNATURE/VERIFICATION BLOCKS

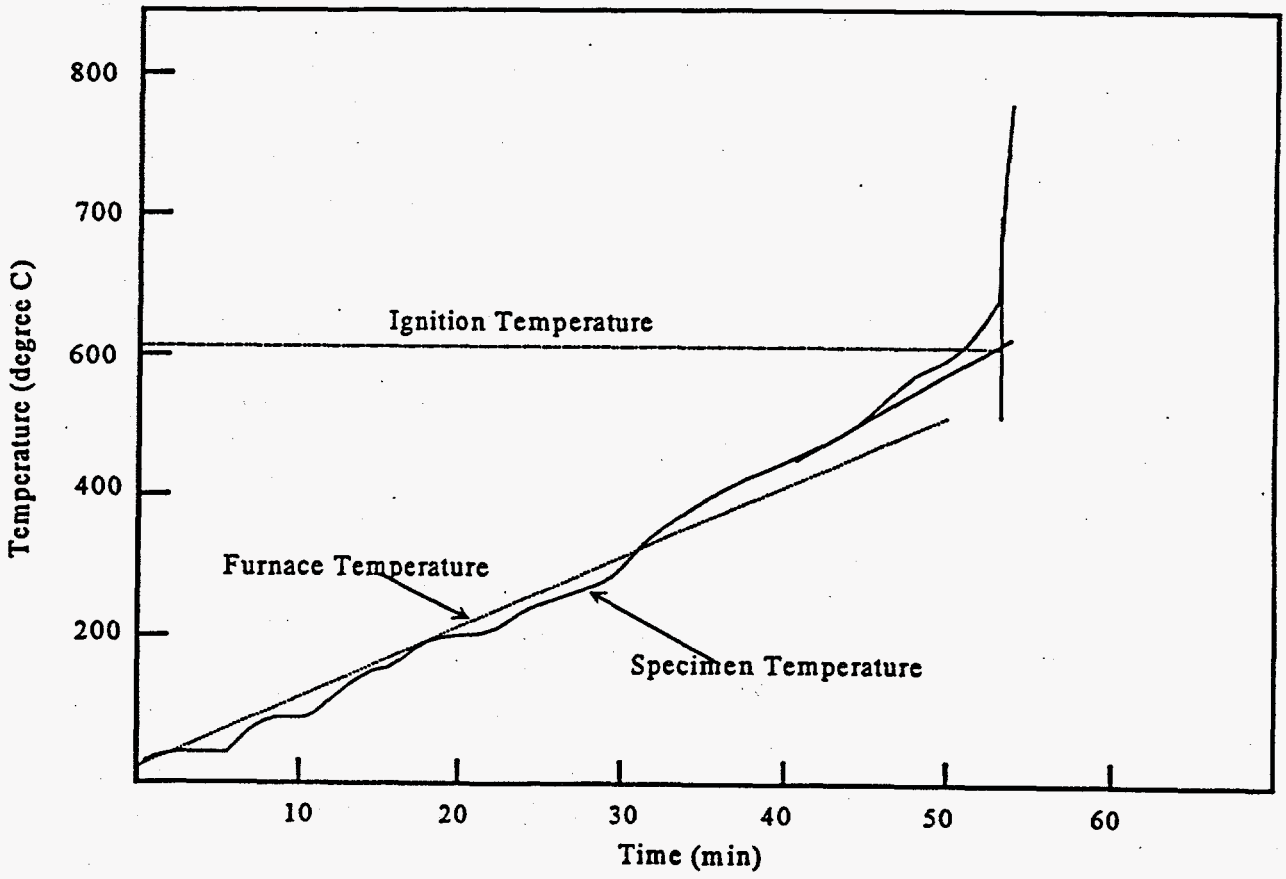
32

Attachment 3

Specimen Dimensions



Ignition Behavior of Metallic Uranium in Oxygen at Constant Heating Rate



CONTROLLED
COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 8
Title: Ignition testing of Specimen SFEC5,4378-S2-D		
Work Location: 327 (Building/Room or "General")	**TI No.:** SNF-CT-018	
Author: John Abrefah	**Effective Date:** 11/07/95 **Supersedes Date:**	

Signatures:

Author _____ (Signature) 11-06-95 (Date)

Technical Reviewer _____ (Signature) 11-7-95 (Date)

Concurrences:

PTL Manager _____ (James M. Seay) 11/7/95 (Date)

HSNFP Charact. Manager _____ (Ronald P. Omberg) 11/8/95 (Date)

Purpose/Scope The purpose of this test instruction is to direct the ignition testing (ignition test # 4) of post-conditioned Spent Nuclear Fuel (SNF) specimen SFEC5,4378-S2-D in a controlled temperature and atmosphere furnace. The specimen was sectioned from SNF element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF specimen SFEC5,4378-S2-D taken from the mid-length of the element SFEC5,4378 and conditioned in a controlled atmosphere furnace (TI #: SNF-CT-007).

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) shall read and follow the steps in the procedure PTL-151: *Operation of Conditioning Furnace System.*

Equipment Reconfiguration Use the alumina tube for the ignition test. Place a spring in the specimen boat to keep the specimen in contact with the thermocouple.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

JA. GLK 11/7/95

SNF specimen SFEC5,4378-S2-D will be loaded into the resistively heated furnace shown schematically in Attachment 1. The ~~silicon carbide~~ alumina furnace tube is preferred for this testing. If the furnace system is in standby condition, then skip steps 1.1 through 1.3 (if steps 1.1 through 1.3 are skipped, enter "N/A" in the appropriate boxes on the check list).

1.1 Perform the *Equipment Readiness/Start Up* steps of procedure PTL-151: *Operation of Conditioning Furnace System.* Record completion of each step with the operator's initials, time and date in the PTL log book.

1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 10 and 12 and opening valves 1, 5, 6, 7, 8, 9 and 11.

Before proceeding further in procedure PTL-151 perform the following steps:

1.3 Dry the furnace system before loading the test specimen using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective (i.e. moisture monitor reads > 5 ppm), then try the step 1.3.2 after discussions with the cognizant scientist. Otherwise, disregard step 1.3.2.

1.3.1 Dry the furnace system by flowing ultra high purity gas (e.g. 99.999% Ar) through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer. When a constant concentration is reached, stop the drying process.

Hold Point: Consult with the cognizant scientist before performing the alternate drying method in step 1.3.2 below and/or before proceeding with the remaining steps of this test instruction.

1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

1.4 Use a calibrated ruler (record the calibration information in blocks **4 & 5** on the data sheet of Attachment 2) with an accuracy of ± 1mm to measure and record in blocks **8** through **14** and **17** through **23** on the data sheet of Attachment 2 the specimen dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp "burrs" that interfere with the measurements, sand them off with silicon carbide abrasive paper. Do not use a lubricant.

1.5 Weigh the specimen twice on a QA Category 1 balance (record the balance calibration information in blocks **1, 2 & 3** on the data sheet of Attachment 2) with 0.1 mg sensitivity. Record the specimen weight in blocks **6 & 15** on the data sheet of Attachment 2.

Note: If the subsequent steps are performed immediately following step 1.3, then make sure that the furnace system has cooled to the ambient hot cell temperature before loading the specimens. Minimize the time the furnace tube and gas lines are open to air in the hot cell.

1.6 Mount the specimen in the sample holder such that the specimen is in the center of the uniform heated zone when loaded into the furnace. Ensure that the specimen is in contact with the specimen thermocouple.

1.7 Load the sample holder into the furnace tube with extreme care, and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

1.8 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the PTL log book.

2.0 Ignition Test

The specimen will be heated to the point of ignition in a flowing dry air atmosphere and then quenched by purging the system with high purity argon. The moisture content of the off-gas will be detected by the moisture monitor and the hydrogen in the gas stream will be monitored by the gas chromatograph.

2.1 Open the dry air cylinder and configure the valves so that the dry air flows through the system. Establish a dry air flow of 500 cc/min.

2.2 Configure valves 6 to 9 (i.e., either valves 6 and 8 opened with valves 7 and 9 closed or valves 6 and 8 closed with valves 7 and 9 opened) so that gas flows through only one of the moisture traps during the testing.

2.3 Verify that all the analytical instruments (e.g., moisture monitor and pressure gauge) are responding properly by looking at the readings on computer #1. Notify the cognizant scientist of any anomalous readings.

2.4 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the testing. The heating tape upstream from the furnace can be left turned off.

2.5 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual):

2.5.1 to heat at a constant rate of 15°C/min to 700±5°C and hold the 700±5°C for 15 minutes.

2.5.2 then allow the furnace to cool to ambient temperature.

2.6 Record in block 24 on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that computer #1 is monitoring and recording the:

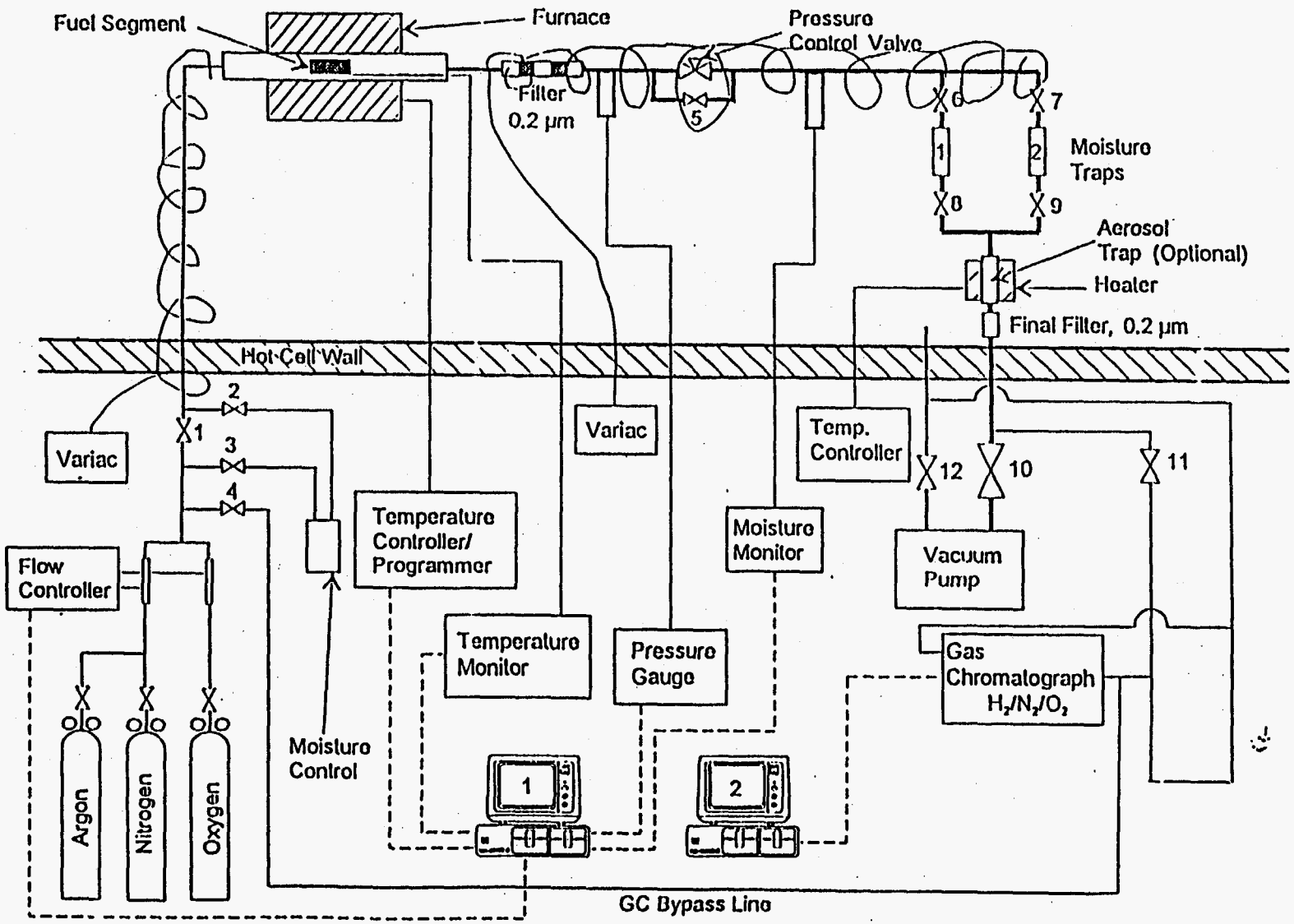
2.6.1 Water vapor concentration with the moisture monitor
2.6.2 Pressure with the pressure gauge
2.6.3 Furnace and specimen temperatures

2.7 The operator shall continuously monitor the specimen temperature above 250°C. If the specimen temperature shows signs of rapid change (see Attachment 3) then switch the gas supply from dry air to ultra high purity argon (flow rate of about 1 liter/min) to quench the reaction. Shut down the furnace manually with argon flowing through the system.

2.8 Open the furnace and carefully remove the specimen. Weigh the specimen twice using a QA Category 1 balance with 0.1 mg sensitivity. Record the weights in blocks 7 & 16 on the data sheet of Attachment 2. Record other observations in the PTL log book for inclusion in the project Laboratory Record Book (LRB).

2.9 Perform the test shut down steps that leave the furnace system in standby condition, that is, at ambient temperature with argon flow rate of about 100 cc/min.

Schematic Of The Furnace System



Attachment 2

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
	4 - Ruler Calibration Number	5 - Ruler Calibration Date

SAMPLE MEASUREMENTS

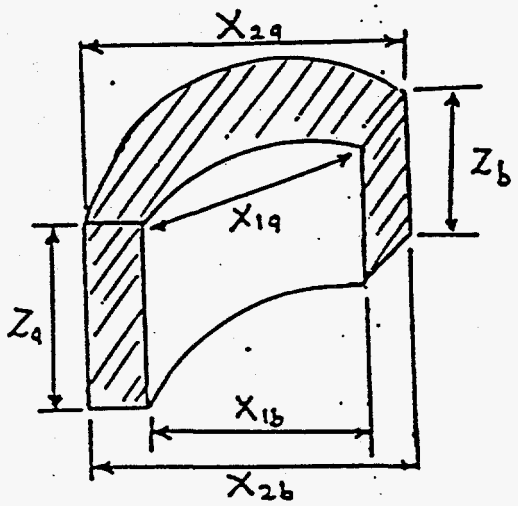
Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S2-D								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	6	7	8	9	10	11	12	13	14
2	15	16	17	18	19	20	21	22	23

SIGNATURE/VERIFICATION BLOCK

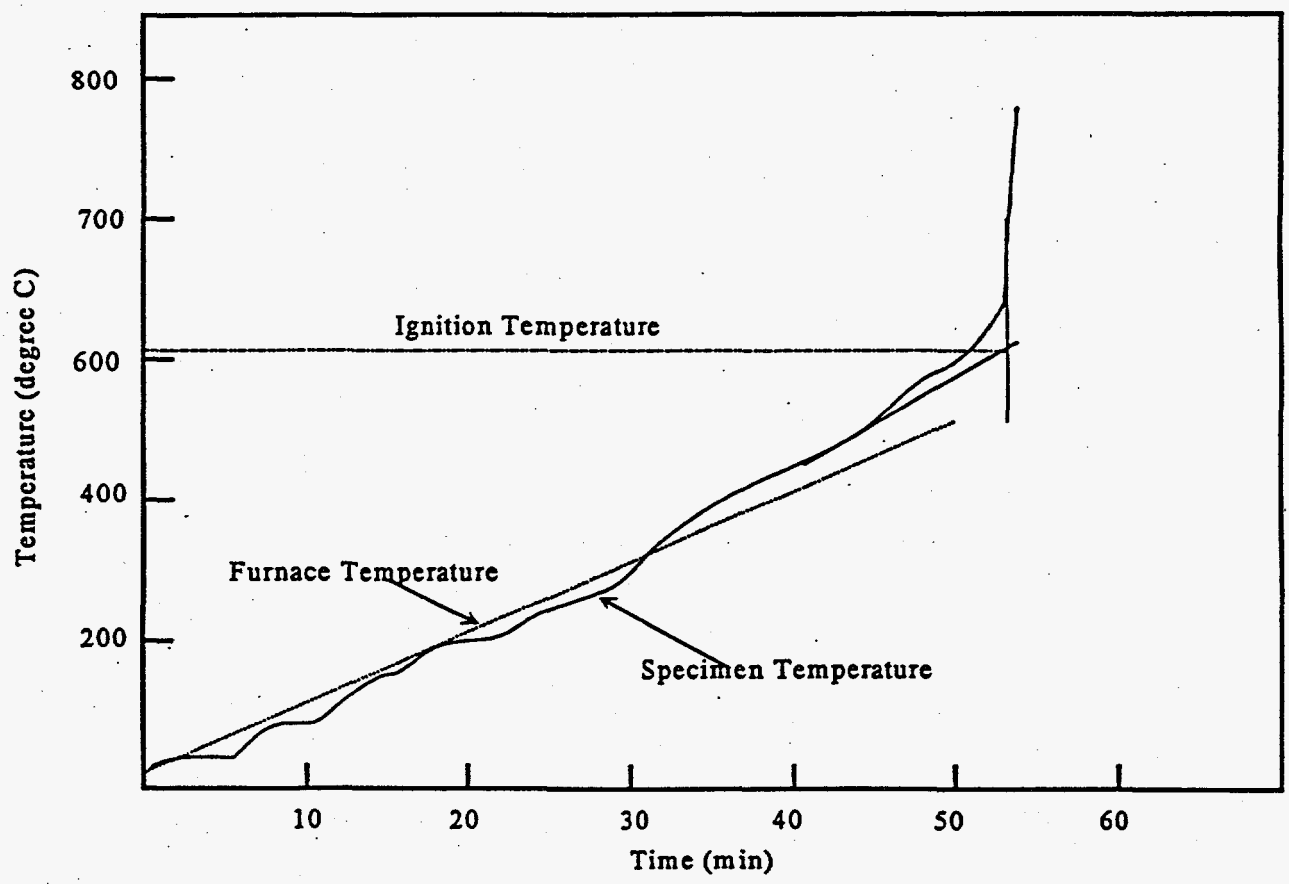
24

Attachment 3

Specimen Dimensions



Ignition Behavior of Metallic Uranium in Oxygen at Constant Heating Rate



CONTROLLED
COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 8
Title: Ignition testing of Specimen SFEC5,4378-S2-I		
Work Location: 327 (Building/Room or "General")	**TI No.: SNF-CT-019**	
Author: John Abrefah	**Effective Date:** 11/16/95 **Supersedes Date:**	

Signatures:

Author _____ 11-14-95
(Signature) (Date)

Technical Reviewer _____ 11-14-95
(Signature) (Date)

Concurrences:

PTL Manager _____ 11/15/95
(James M. Seay) (Date)

HSNFP Charact. Manager _____ 11/16/95
(Ronald P. Omberg) (Date)

Purpose/Scope The purpose of this test instruction is to direct the ignition testing (ignition test # 3R) of post-conditioned Spent Nuclear Fuel (SNF) specimen SFEC5,4378-S2-I in a controlled temperature and atmosphere furnace. The specimen was sectioned from SNF element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF specimen SFEC5,4378-S2-I taken from the mid-length of the element SFEC5,4378 and conditioned in a controlled atmosphere furnace (TI #: SNF-CT-005).

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) shall read and follow the steps in the procedure PTL-151: *Operation o Conditioning Furnace System.*

Equipment Reconfiguration Use the alumina tube for the ignition test. Place a spring in the specimen boat to keep the specimen in contact with the thermocouple.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

SNF specimen SFEC5,4378-S2-I will be loaded into the resistively heated furnace shown schematically in Attachment 1. The alumina furnace tube is preferred for this testing. If the furnace system is in standby condition, then skip steps 1.1 through 1.3 (if steps 1.1 through 1.3 are skipped, enter "N/A" in the appropriate boxes on the check list).

1.1 Perform the *Equipment Readiness/Start Up* steps of procedure PTL-151: *Operation o Conditioning Furnace System.* Record completion of each step with the operator's initials, time and date in the PTL log book.

1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 10 and 12 and opening valves 1, 5, 6, 7, 8, 9 and 11.

Before proceeding further in procedure PTL-151 perform the following steps:

1.3 Dry the furnace system before loading the test specimen using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective (i.e. moisture monitor reads > 5 ppm), then try the step 1.3.2 after discussions with the cognizant scientist. Otherwise, disregard step 1.3.2.

1.3.1 Dry the furnace system by flowing ultra high purity gas (e.g. 99.999% Ar) through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer. When a constant concentration is reached, stop the drying process.

Hold Point: Consult with the cognizant scientist before performing the alternate drying method in step 1.3.2 below and/or before proceeding with the remaining steps of this test instruction.

1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

1.4 Use a calibrated ruler (record the calibration information in blocks 4 & 5 on the data sheet of Attachment 2) with an accuracy of ± 1mm to measure and record in blocks 8 through 14 and 17 through 23 on the data sheet of Attachment 2 the specimen dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp "burrs" that interfere with the measurements, sand them off with silicon carbide abrasive paper. Do not use a lubricant.

1.5 Weigh the specimen twice on a QA Category 1 balance (record the balance calibration information in blocks 1, 2 & 3 on the data sheet of Attachment 2) with 0.1 mg sensitivity. Record the specimen weight in blocks 6 & 15 on the data sheet of Attachment 2.

Note: If the subsequent steps are performed immediately following step 1.3, then make sure that the furnace system has cooled to the ambient hot cell temperature before loading the specimens. Minimize the time the furnace tube and gas lines are open to air in the hot cell.

1.6 Mount the specimen in the sample holder such that the specimen is in the center of the uniform heated zone when loaded into the furnace. Ensure that the specimen is in contact with the specimen thermocouple.

1.7 Load the sample holder into the furnace tube with extreme care, and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

1.8 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the PTL log book.

2.0 Ignition Test

The specimen will be heated to the point of ignition in an atmosphere of flowing dry air and then quenched by purging the system with high purity argon. The moisture content of the off-gas will be detected by the moisture monitor. Hydrogen in the off gas stream will be monitored by the gas chromatograph.

2.1 Open the dry air cylinder and configure the valves so that the dry air flows through the system. Establish a dry air flow of 500 cc/min.

2.2 Configure valves 6 to 9 (i.e., either valves 6 and 8 opened with valves 7 and 9 closed or valves 6 and 8 closed with valves 7 and 9 opened) so that gas flows through only one of the moisture traps during the testing.

2.3 Verify that all the analytical instruments (e.g., moisture monitor and pressure gauge) are responding properly by looking at the readings on computer #1. Notify the cognizant scientist of any anomalous readings.

2.4 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the testing. The heating tape upstream from the furnace can be left turned off.

2.5 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual):

2.5.1 J-K 800 to heat at a constant rate of 15°C/min to ~~700~~±5°C and hold the 800 ~~700~~±5°C for 15 minutes.

2.5.2 then allow the furnace to cool to ambient temperature.

2.6 Record in block **24** on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that computer #1 is monitoring and recording the:

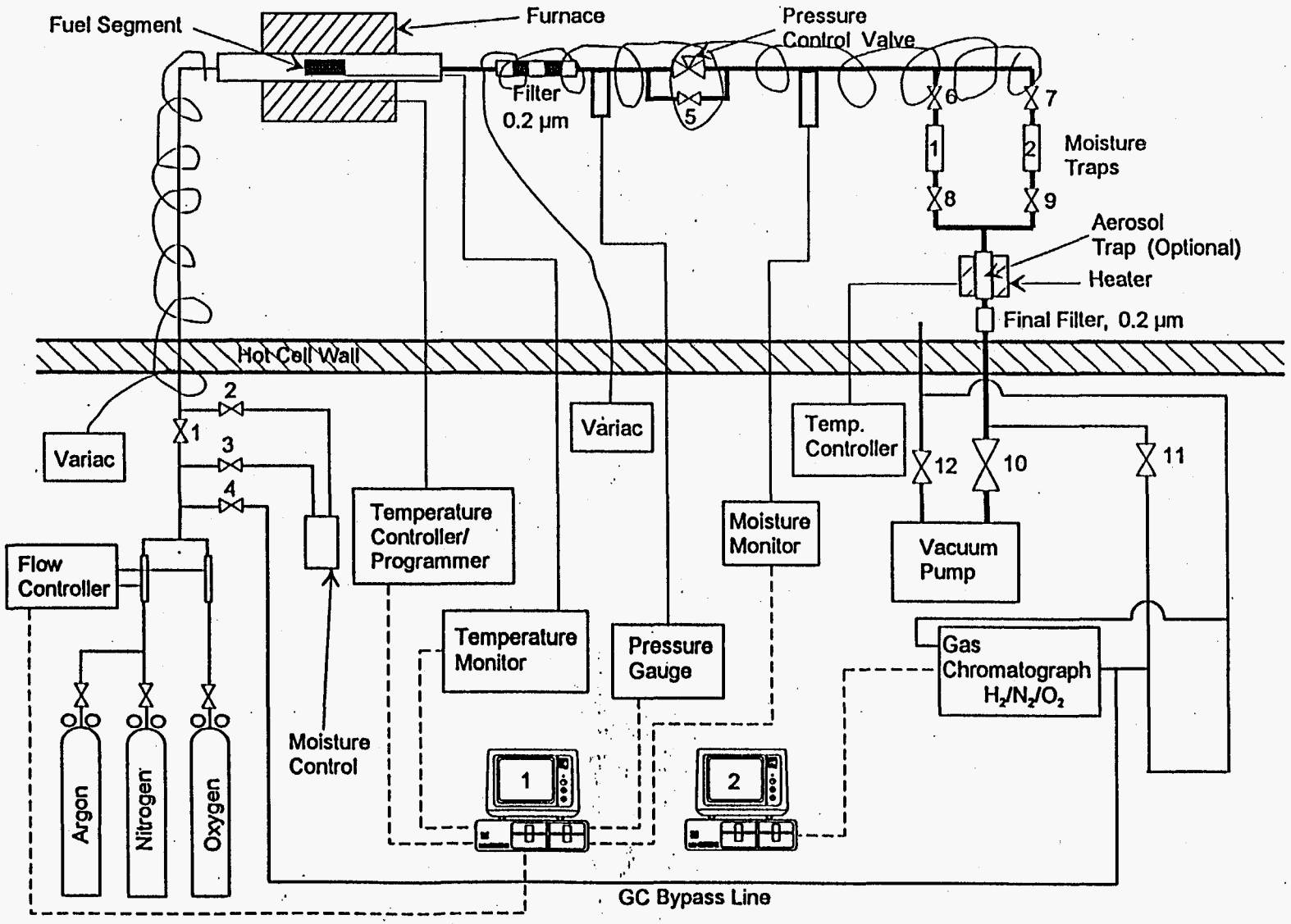
2.6.1 Water vapor concentration with the moisture monitor
2.6.2 Pressure with the pressure gauge
2.6.3 Furnace and specimen temperatures

2.7 The operator shall continuously monitor the specimen temperature above 250°C. If the specimen temperature shows signs of rapid change (see Attachment 3) then switch the gas supply from dry air to ultra high purity argon (flow rate of about 1 liter/min) to quench the reaction. Shut down the furnace manually with argon flowing through the system.

2.8 Open the furnace and carefully remove the specimen. Weigh the specimen twice using a QA Category 1 balance with 0.1 mg sensitivity. Record the weights in blocks **7 & 16** on the data sheet of Attachment 2. Record other observations in the PTL log book for inclusion in the project Laboratory Record Book (LRB).

2.9 Perform the test shut down steps that leave the furnace system in standby condition, that is, at ambient temperature with argon flow rate of about 100 cc/min.

Schematic Of The Furnace System



Attachment 2

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
	4 - Ruler Calibration Number	5 - Ruler Calibration Date

SAMPLE MEASUREMENTS

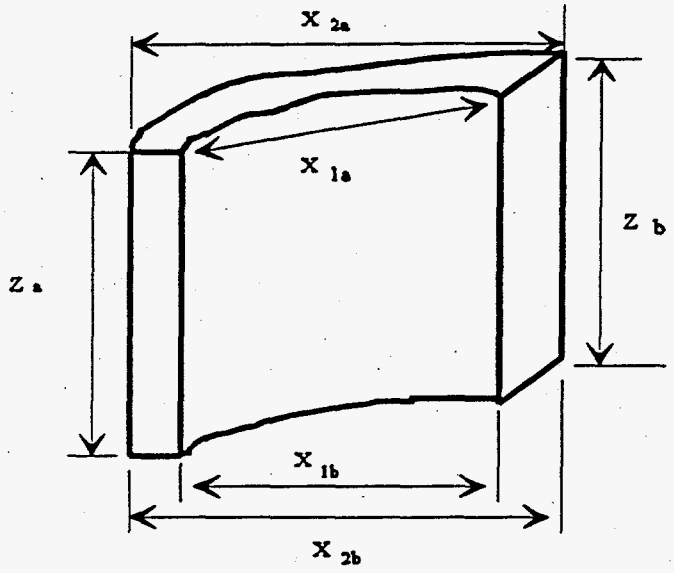
Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S2-I								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	6	7	8	9	10	11	12	13	14
2	15	16	17	18	19	20	21	22	23

SIGNATURE/VERIFICATION BLOCK

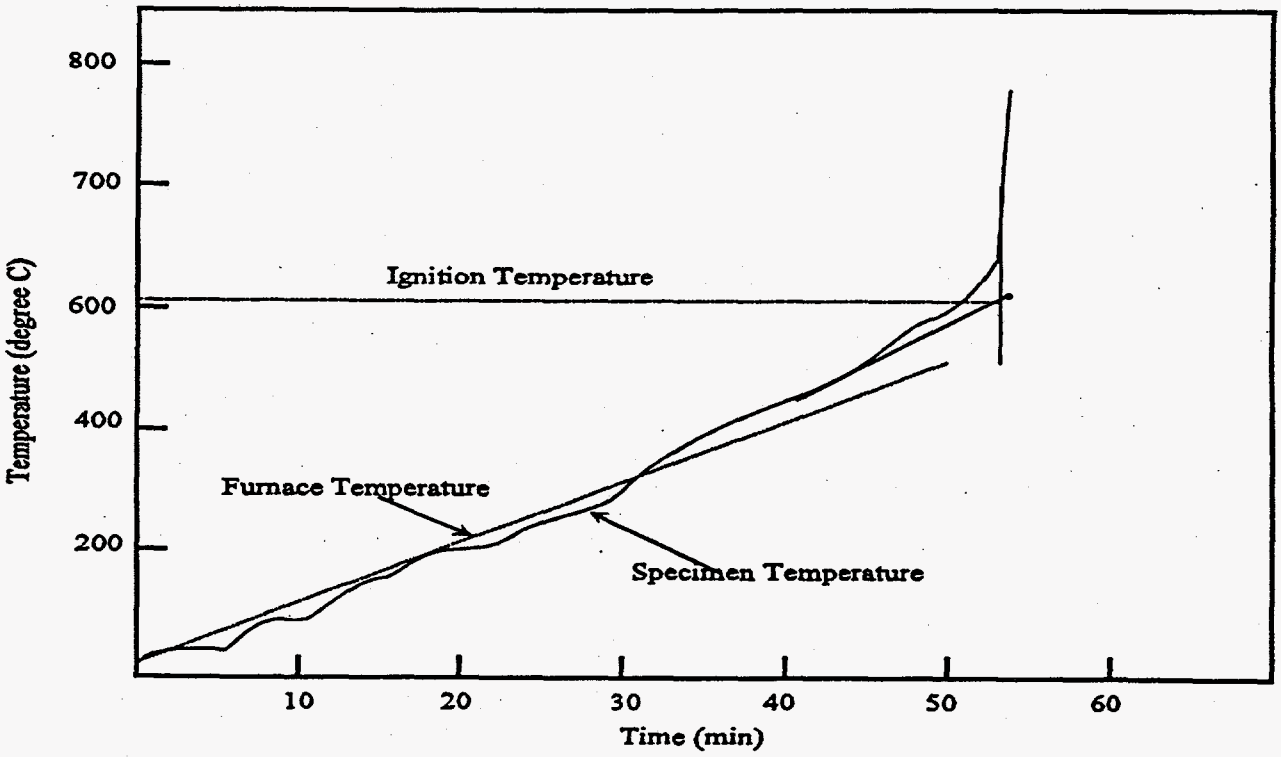
24

Attachment 3

Specimen Dimensions



Ignition Behavior of Metallic Uranium in Oxygen at Constant Heating Rate



COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 8
Title: Ignition testing of Specimen SFEC5,4378-S1A-D1		
Work Location: 327 (Building/Room or "General")	**TI No.: SNF-CT-021**	
Author: John Abrefah	**Effective Date:** 11/30/95 **Supersedes Date:**	

Signatures:

Author _____ 11-28-95
(Signature) (Date)

Technical Reviewer _____ 11-28-95
(Signature) (Date)

Concurrences:

PTL Manager _____ 11/30/95
(James M. Seay) (Date)

HSNFP Charact. Manager _____ 12/1/95
(Ronald P. Omberg) (Date)

Purpose/Scope The purpose of this test instruction is to direct the ignition testing (ignition test # 5) of as received (i.e., pre-conditioned) Spent Nuclear Fuel (SNF) specimen SFEC5,4378-S1A-D1 in a controlled temperature and atmosphere furnace. The specimen was sectioned from the damaged end of SNF element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF specimen SFEC5,4378-S1A-D1 taken from the damaged end of the element SFEC5,4378.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) shall read and follow the steps in the procedure PTL-151: *Operation of Conditioning Furnace System.*

Equipment Reconfiguration Use the alumina tube for the ignition test. Place a spring in the specimen boat to keep the specimen in contact with the thermocouple.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

SNF specimen SFEC5,4378-S1A-D1 will be loaded into the resistively heated furnace shown schematically in Attachment 1. The alumina furnace tube is preferred for this testing. If the furnace system is in standby condition, then skip steps 1.1 through 1.3 (if steps 1.1 through 1.3 are skipped, enter "N/A" in the appropriate boxes on the check list).

1.1 Perform the *Equipment Readiness/Start Up* steps of procedure PTL-151: *Operation of Conditioning Furnace System.* Record completion of each step with the operator's initials, time and date in the PTL log book.

1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 10 and 12 and opening valves 1, 5, 6, 7, 8, 9 and 11.

Before proceeding further in procedure PTL-151 perform the following steps:

1.3 Dry the furnace system before loading the test specimen using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective (i.e. moisture monitor reads > 5 ppm), then try the step 1.3.2 after discussions with the cognizant scientist. Otherwise, disregard step 1.3.2.

1.3.1 Dry the furnace system by flowing ultra high purity gas (e.g. 99.999% Ar) through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer. When a constant concentration is reached, stop the drying process.

Hold Point: Consult with the cognizant scientist before performing the alternate drying method in step 1.3.2 below and/or before proceeding with the remaining steps of this test instruction.

1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

1.4 Use a calibrated ruler (record the calibration information in blocks **4 & 5** on the data sheet of Attachment 2) with an accuracy of ± 1mm to measure and record in blocks **8** through **14** and **17** through **23** on the data sheet of Attachment 2 the specimen dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp "burrs" that interfere with the measurements, sand them off with silicon carbide abrasive paper. Do not use a lubricant.

1.5 Weigh the specimen twice on a QA Category 1 balance (record the balance calibration information in blocks **1, 2 & 3** on the data sheet of Attachment 2) with 0.1 mg sensitivity. Record the specimen weight in blocks **6 & 15** on the data sheet of Attachment 2.

Note: If the subsequent steps are performed immediately following step 1.3, then make sure that the furnace system has cooled to the ambient hot cell temperature before loading the specimens. Minimize the time the furnace tube and gas lines are open to air in the hot cell.

1.6 Mount the specimen in the sample holder such that the specimen is in the center of the uniform heated zone when loaded into the furnace. Ensure that the specimen is in contact with the specimen thermocouple.

1.7 Load the sample holder into the furnace tube with extreme care, and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

1.8 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the PTL log book.

2.0 Ignition Test

The specimen will be heated to the point of ignition in an atmosphere of flowing dry air and then quenched by purging the system with high purity argon. The moisture content of the off-gas will be detected by the moisture monitor. Hydrogen in the off gas stream will be monitored by the gas chromatograph.

2.1 Open the dry air cylinder and configure the valves so that the dry air flows through the system. Establish a dry air flow of 500 cc/min.

2.2 Configure valves 6 to 9 (i.e., either valves 6 and 8 opened with valves 7 and 9 closed or valves 6 and 8 closed with valves 7 and 9 opened) so that gas flows through only one of the moisture traps during the testing.

2.3 Verify that all the analytical instruments (e.g., moisture monitor and pressure gauge) are responding properly by looking at the readings on computer #1. Notify the cognizant scientist of any anomalous readings.

2.4 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the testing. The heating tape upstream from the furnace can be left turned off.

2.5 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual):

2.5.1 to heat at a constant rate of 15°C/min to 700±5°C and hold the 700±5°C for 15 minutes.

2.5.2 then allow the furnace to cool to ambient temperature.

2.6 Record in block 24 on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that computer #1 is monitoring and recording the:

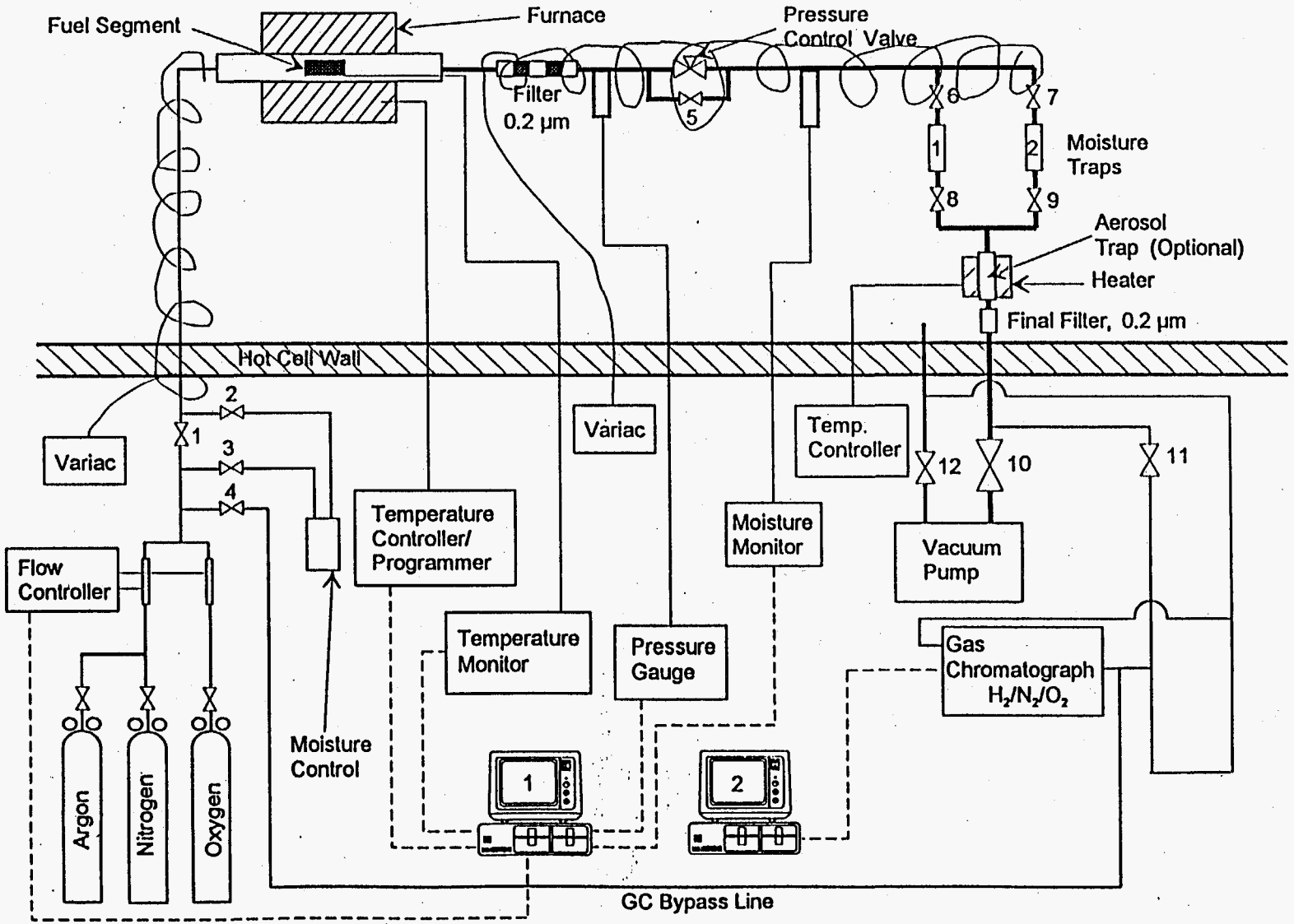
2.6.1 Water vapor concentration with the moisture monitor
2.6.2 Pressure with the pressure gauge
2.6.3 Furnace and specimen temperatures

2.7 The operator shall continuously monitor the specimen temperature above 250°C. If the specimen temperature shows signs of rapid change (see Attachment 3) then switch the gas supply from dry air to ultra high purity argon (flow rate of about 1 liter/min) to quench the reaction. Shut down the furnace manually with argon flowing through the system.

2.8 Open the furnace and carefully remove the specimen. Weigh the specimen twice using a QA Category 1 balance with 0.1 mg sensitivity. Record the weights in blocks 7 & 16 on the data sheet of Attachment 2. Record other observations in the PTL log book for inclusion in the project Laboratory Record Book (LRB).

2.9 Perform the test shut down steps that leave the furnace system in standby condition, that is, at ambient temperature with argon flow rate of about 100 cc/min.

Schematic Of The Furnace System



Attachment 2

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
	4 - Ruler Calibration Number	5 - Ruler Calibration Date

SAMPLE MEASUREMENTS

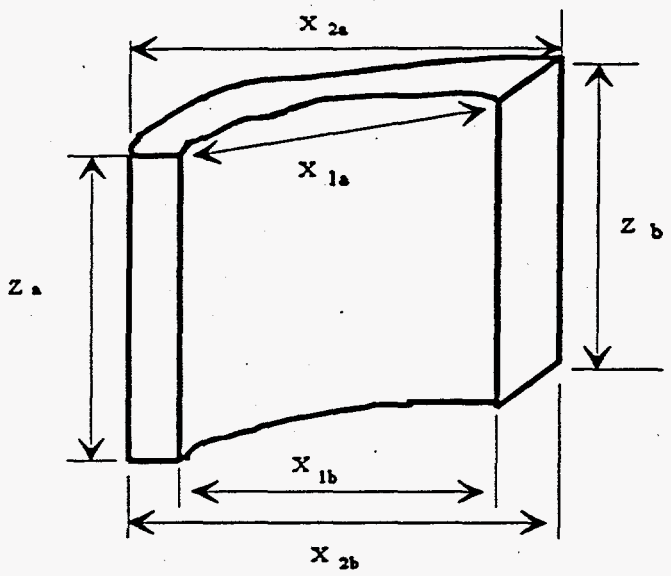
Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S1A-D1								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	6	7	8	9	10	11	12	13	14
2	15	16	17	18	19	20	21	22	23

SIGNATURE/VERIFICATION BLOCK

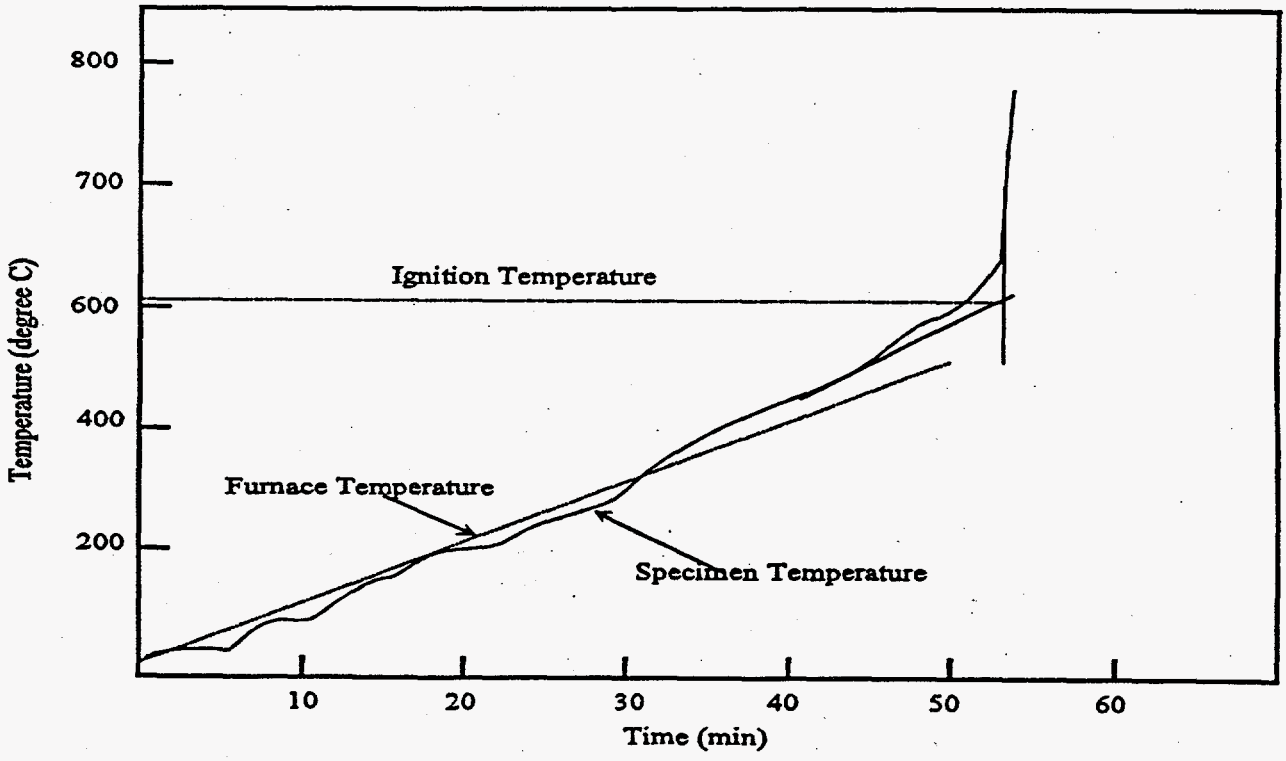
24

Attachment 3

Specimen Dimensions



Ignition Behavior of Metallic Uranium in Oxygen at Constant Heating Rate



CONTROLLED
COPY #1

Test Instruction	Rev. No.: 0	Page 1 of 9
Title: Vacuum Drying and Conditioning Testing of Wet SNF Specimen SFEC5,4378-S2-F4		
Work Location: 327 (Building/Room or "General")	**TI No.:** SNF-CT-025	
Author: John Abrefah	**Effective Date:** 12/15/95 **Supersedes Date:** New	

Signatures:

Author _____ 12-15-95
(Signature) (Date)

Technical Reviewer Steve McGuire 12-18-95
(Signature) (Date)

Concurrences:

PTL Manager _____ 12/20/95
(James M. Seay) (Date)

HSNFP Charact. Manager Brian Mahaffey for 12/21/95
(Ronald P. Omberg) (Date)

Purpose/Scope The purpose of this test instruction is to direct the drying and conditioning testing of Spent Nuclear Fuel (SNF) specimen in a controlled temperature and atmosphere furnace. This test is similar to furnace tests 4 through 6, except that there has been changes in the system configuration to enable hydrogen concentration in the off gas stream to be detected by the gas chromatograph during the vacuum drying cycle.

Applicability This test instruction is applicable to SNF specimen SFEC5,4378-S2-F4 taken from the mid-length of SNF element SFEC5,4378.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) should read and follow the steps in the procedure PTL-151: *Operation of Conditioning Furnace System.* Any deviation from this TI must be recorded on the work place copy with operator initials, time and date, and must include cognizant scientist and PTL cognizant engineer written concurence before proceeding.

Equipment Reconfiguration The bellows pump will be used to evacuate the system.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

SNF specimen SFEC5,4378-S2-F4 will be loaded into the resistively heated furnace shown schematically in Attachment 1. If the furnace system is in standby condition, then skip steps 1.1 through 1.3 (if steps 1.1 through 1.3 are skipped, enter "N/A" in the appropriate boxes on the check list).

1.1 Perform the *Equipment Readiness/Start Up* steps of procedure PTL-151: *Operation of Conditioning Furnace System.* Record completion of each step with the operator's initials, time and date in the PTL log book.

1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 10 and 12 and opening valves 1, 5, 6, 7, 8, 9 and 11.

Before proceeding further in the procedure PTL-151 perform the following steps:

1.3 Dry the furnace system before loading the test specimen using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective then try the step 1.3.2 after discussions with the cognizant scientist otherwise disregard step 1.3.2.

1.3.1 Dry the furnace system by flowing high purity gas (e.g. 99.999% Ar) through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

Hold Point: At the written direction of the cognizant scientist perform the alternate drying method in step 1.3.2 below and proceed to the remaining steps of this test instruction.

1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

1.4 Use a calibrated ruler (record the calibration information in blocks 7 & 8 on the data sheet of Attachment 2) with an accuracy of ± 1mm to measure and record in blocks 12 through 18 and 21 through 27 on the data sheet of Attachment 2 the specimens dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp 'lips' that interfere with the measurements, sand them off with silicon carbide abrasive paper. Do not use a lubricant.

1.5 Weigh the specimen twice on a QA category one balance (record the balance calibration information in blocks 1, 2 & 3 on the data sheet of Attachment 2) with 0.1 mg sensitivity. Record the specimens weights in blocks 10 & 19 on the data sheet of Attachment 2.

Caution: Make sure the furnace system has cooled to the ambient hotcell temperature before loading the specimens. Minimize the time the furnace tube and gas lines will be open to air in the cell.

1.6 Mount the specimen in the sample holder such that it is in the center of the uniform heated zone when loaded into the furnace. Ensure that the specimen is in contact with the specimen thermocouple.

1.7 Fill the sample holder with measured volume (between 2.0 to 5.0±0.2 ml) of de-ionized water.

1.8 Load the sample holder into the furnace tube with extreme care to avoid loss of water and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

1.9 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the attached data sheet (**Block 36**).

2.0 Vacuum Drying Test

The specimen will be initially tested to determine the water content by driving off free and bound water first at low temperature vacuum followed by relatively high temperature vacuum. The moisture content of the off-gas will be detected by the moisture monitor, and the cumulative water will be trapped in the moisture traps. Before and after weights of the traps will be a measure of the water that came off. The hydrogen in the off gas stream will be measured by the gas chromatograph (GC).

2.1 Prior to beginning the drying test of the specimen, determine the weight of the moisture traps on a QA category 1 balance (record the balance calibration information in blocks **4, 5 & 6** on the data sheet of Attachment 2) with 10 mg sensitivity. Repeat each measurement and record their weights in blocks **28 & 32** and **30 & 34** on the data sheet of Attachment 2.

Note: Due to the swapping of moisture traps after the first low temperature vacuum drying cycle, the test must be scheduled such that the first cycle ends during regular working hours, otherwise arrange for that step to be performed during off hours.

2.2 Configure the valves such that the bellows pump is used to evacuate the system.

2.3 Pump the system down and control the system pressure to within the range of 40 to 70 Torr after closing valves 1, 2 and 11 and opening valves 5, 6, 7, 8, 9 and 10.

2.4 Configure valves 6 to 9 of Attachment 1 so that the pumping is through the two moisture traps (i.e., valves 6 to 9 opened).

2.5 Verify that all the analytical instruments (e.g., moisture monitor, the GC and pressure gauge) are responding properly by looking at the readings on the computers #1 and #2. Notify the cognizant scientist of any anomalous readings.

2.6 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the drying cycles. The heating tape upstream from the furnace will be turned off or on at the direction of the cognizant scientist.

2.7 Close valves 7 and 9 of Attachment 1 so that only moisture trap #1 is used in the low temperature vacuum drying cycle.

2.8 Turn on the furnace and program it to perform the following (refer to the Omega Temperature Controller/Programmer manual):

2.8.1 to heat at a constant rate (e.g., between 10 and 15°C/min) to 50±2°C and hold the 50±2°C for 10 hours.

2.8.2 resume the constant heating rate (i.e., at the same rate as in Step 2.8.1) to 300±2°C and hold the 300±2°C for 24 more hours.

2.8.3 then allow the furnace to cool to ambient temperature.

2.9 Record in block 37 on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that computer #1 is monitoring and recording the:

2.9.1 Water vapor concentration with the moisture monitor.
2.9.2 Pressure with the pressure gauge.
2.9.3 Furnace and specimen temperatures

2.10 Getting to the end of cold vacuum dry cycle of step 2.8.1, isolate trap #1 by closing valves 6 and 8 (Attachment 1) and connect moisture trap #2 to the system by opening valves 7 and 9 (Attachment 1). Perform this switch over of traps during the last 30 minutes of the cold vacuum drying cycle.

2.11 Remove the moisture trap #1 during the second test cycle (i.e. the hot vacuum drying cycle). Weigh the trap #1 on a QA category 1 balance with 10 mg sensitivity. Repeat the weight measurement and record the weights in blocks 29 & 33 on the data sheet of Attachment 2. Reconnect trap # 1 to the system.

2.12 After the second drying cycle, and with the furnace and specimen at ambient temperature, isolate trap # 2 by closing valves 7 & 9, remove moisture trap #2 and weigh it twice on a QA category one balance with 10 mg sensitivity. Record the weights in blocks 31 & 35 on the data sheet of Attachment 2. Reconnect trap # 2 to the system.

3.0 Conditioning Testing

The SNF passivation step in the furnace testing will involve exposing the test specimens to a gas mixture of 98% argon and 2% oxygen by volume at a flow rate of 100±2 cm^3/min.

3.1 Configure valves 6 to 9 (i.e. either valves 6 and 8 opened with valves 7 and 9 closed or valves 6 and 8 closed with valves 7 and 9 opened) so that gas flows through only one of the moisture traps. Record in the PTL log book the trap that was used.

3.2 Adjust the heating tape (downstream from the furnace) temperature to a constant value in the range of 50 to 100°C or the value specified by the cognizant scientist.

3.3 Purge the system with pure argon flow of 100±2 cm^3/min (i.e., by opening valves 1, 5, 6 & 8 or 7 & 9, and 11, but with valves 2,3,4, and 10 closed) for about 45 minutes and monitor the moisture content in the system. If the moisture monitor indicates high concentration (i.e., greater than -50°C dew point) then consult with the cognizant scientist before proceeding to the next step.

3.4 Set the argon and oxygen flow controllers to give the flows indicated above (i.e., 98% argon, 2% oxygen at a flow rate of 100±2 cm^3/min).

3.5 Switch the gas flow to the GC through the bypass line (i.e., opening valve 4 and closing valves 1, 2, 3, and 11 of Attachment 1). Record in block 38 on the data sheet of Attachment 2 the switch over time. Program computer #2 to monitor and record the oxygen concentration in the gas stream every 2 minutes for a period of at least 15 minutes after equilibration to establish the baseline.

3.6 Switch the gas flow to the furnace by opening valve 1, 5, and 11, and by closing valves 2, 3, 4 and 10. Record in block 39 on the data sheet of Attachment 2. Program computer #2 to monitor and record the oxygen concentration in the gas stream measured by the GC every 5 minutes until a constant oxygen concentration is established. At that constant concentration, monitor the gas stream for at least 30 minutes to establish the baseline concentration.

3.7 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual) to heat at a constant rate (e.g., between 12 to 15°C/min) to 200±2°C and hold at that temperature indefinitely. (Note that furnace cooling will be initiated manually after about 10 hours as described in Step 3.8.) Verify that the computers are monitoring and recording the:

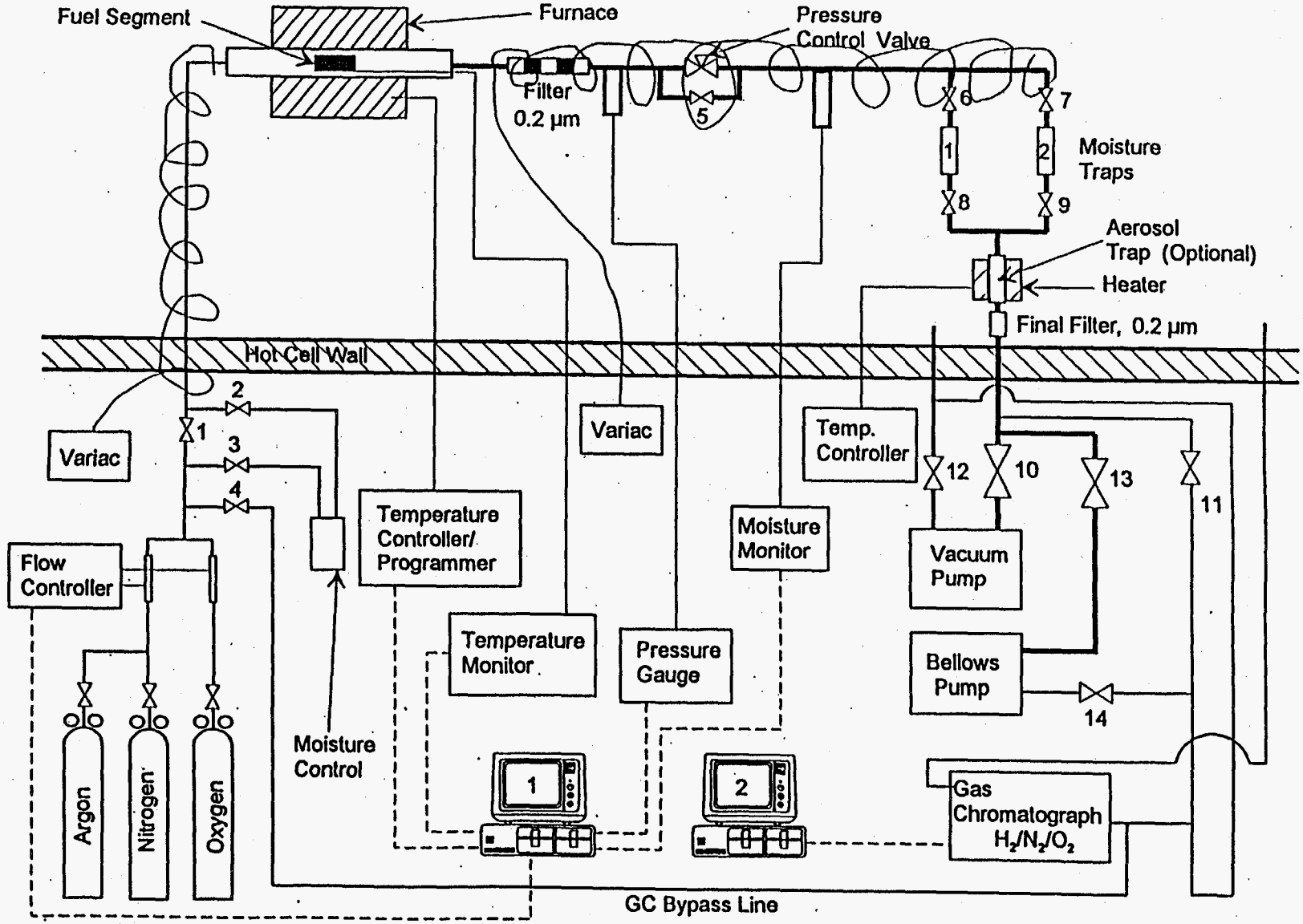
- 3.7.1 Moisture concentration using the moisture monitor for every 5 minutes.
- 3.7.2 Oxygen and hydrogen concentrations using the gas chromatograph for every 5 minutes.
- 3.7.3 Furnace and specimen temperatures every 5 minutes.

3.8 After 10 hours of conditioning of the specimens, turn off the oxygen flow and the furnace, and allow the specimen to cool to ambient temperature in flowing argon. Record in block 40 on the data sheet of Attachment 2 the time oxygen gas is turned off.

3.9 Open the furnace and carefully remove the specimen. Weigh the specimen twice using a QA Category 1 balance with 0.1 mg sensitivity. Record the weights in blocks 11 & 20 on the data sheet of Attachment 2. Record other observations in the PTL log book for inclusion in the project Laboratory Record Book (LRB).

3.10 Perform the test shut down steps per PTL-151 Section 3.0. Leave the furnace system in standby condition, that is, at ambient temperature with argon flow rate of about 100 cc/min.

Schematic Of The Furnace System



Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
4 - Moisture Trap Balance Description	5 - Moisture Trap Balance Calibration Number	6 - Calibration Expiration Date
	7 - Ruler Calibration Number	8 - Ruler Calibration Date

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code SFEC5,4378-S2-F4								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	10	11	12	13	14	15	16	17	18
2	19	20	21	22	23	24	25	26	27

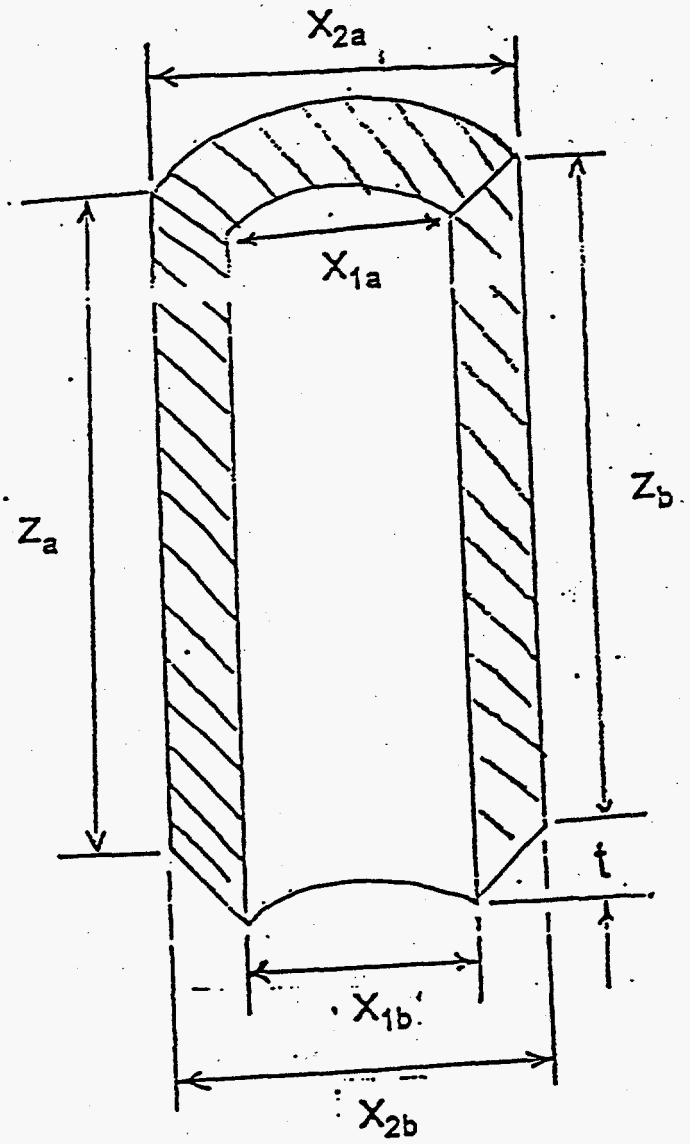
Measurement Number	Moisture Trap #1		Moisture Trap #2	
	Weight Before Test (g)	Weight After Test (g)	Weight Before Test (g)	Weight After Test (g)
1	28	29	30	31
2	32	33	34	35

SYSTEM STATUS

36 - Time/Date	37 - Time/Date	38 - Time/Date	39 - Time/Date	40 - Time/Date

Attachment 3

Specimen Dimensions



CONTROLLED DOCUMENT #1

Test Instruction	Rev. No.: 0	Page 1 of 9
Title: Vacuum Drying and Conditioning Testing of Wet SNF Specimen SFEC5,4378-S1A-C		
Work Location: 327 (Building/Room or "General")	**TI No.:** SNF-CT-026	
Author: John Abrefah	**Effective Date:** 01/10/96 **Supersedes Date:** New	

Signatures:

Author _____ 01-09-96
(Signature) (Date)

Technical Reviewer _____ 1-9-96
(Signature) (Date)

Concurrences:

PTL Manager _____ 1/9/96
(James M. Seay) (Date)

HSNFP Charact. Manager _____ 1/10/96
(Ronald P. Omberg) (Date)

Purpose/Scope The purpose of this test instruction is to direct the cold and hot vacuum drying followed by conditioning testing of Spent Nuclear Fuel (SNF) specimen in a controlled temperature and atmosphere furnace. This test is similar to furnace tests 16, except that the specimen was cut from the damaged end of SNF element SFEC5,4378.

Applicability This test instruction is applicable to SNF specimen SFEC5,4378-S1A-C taken from the damaged end of SNF element SFEC5,4378.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) should read and follow the steps in the procedure PTL-151: *Operation of Conditioning Furnace System.* Any deviation from this TI must be recorded on the work place copy with operator initials, time and date, and must include cognizant scientist and PTL cognizant engineer written concurence before proceeding.

Equipment Reconfiguration The moisture traps will not be used to determine cumulative moisture content during the testing.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

SNF specimen SFEC5,4378-S1A-C will be loaded into the resistively heated furnace shown schematically in Attachment 1. If the furnace system is in standby condition, then skip steps 1.1 through 1.3 (if steps 1.1 through 1.3 are skipped, enter "N/A" in the appropriate boxes on the check list).

1.1 Perform the *Equipment Readiness/Start Up* steps of procedure PTL-151: *Operation of Conditioning Furnace System.* Record completion of each step with the operator's initials, time and date in the PTL log book.

1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 10 and 12 and opening valves 1, 5, 6, 7, 8, 9 and 11.

Before proceeding further in the procedure PTL-151 perform the following steps:

1.3 Dry the furnace system before loading the test specimen using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective then try the step 1.3.2 after discussions with the cognizant scientist otherwise disregard step 1.3.2.

1.3.1 Dry the furnace system by flowing high purity gas (e.g. 99.999% Ar) through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

Hold Point: At the written direction of the cognizant scientist perform the alternate drying method in step 1.3.2 below and proceed to the remaining steps of this test instruction.

1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

1.4 Attach a calibrated ruler (record the calibration information in blocks 4 & 5 on the data sheet of Attachment 2) with an accuracy of ± 1mm to the specimen and take photographs of the exposed and cut fuel surfaces. Use the photographs to measure and record in blocks 9 through 15 and 18 through 24 on the data sheet of Attachment 2 the specimens dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp 'lips' at the cut surfaces that interfere with the measurements, sand them off with silicon carbide abrasive paper. Do not use a lubricant.

1.5 Weigh the specimen twice on a QA category one balance (record the balance calibration information in blocks 1, 2 & 3 on the data sheet of Attachment 2) with 10 mg or better sensitivity. Record the specimens weights in blocks 7 & 16 on the data sheet of Attachment 2.

Caution: Make sure the furnace system has cooled to the ambient hotcell temperature before loading the specimens. Minimize the time the furnace tube and gas lines will be open to air in the cell.

1.6 Mount the specimen in the sample holder such that it is in the center of the uniform heated zone when loaded into the furnace. Ensure that the specimen is in contact with the specimen thermocouple.

1.7 Fill the sample holder with measured volume (between 2.0 to 5.0±0.2 ml) of de-ionized water. Record the volume of the water in the PTL Log Book.

1.8 Load the sample holder into the furnace tube with extreme care to avoid loss of water and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

1.9 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the attached data sheet (**Block 25**).

2.0 Vacuum Drying Test

The specimen will be initially tested to determine the water content by driving off free and bound water first at low temperature vacuum followed by relatively high temperature vacuum. The moisture content of the off-gas will be detected by the moisture monitor. The hydrogen in the off gas stream will be measured by the gas chromatograph (GC).

2.1 Configure the valves such that the bellows pump is used to evacuate the system.

2.2 Pump the system down and control the system pressure to within the range of 30 to 70 Torr after closing valves 1, 2 and 11 and opening valves 5, 6, 7, 8, 9 and 10.

2.3 Configure valves 6 to 9 of Attachment 1 so that the pumping is through the moisture trap that has the least leakage at its joints. Use this configuration throughout the test. Record in the PTL Log Book the trap that was used.

2.4 Verify that all the analytical instruments (e.g., moisture monitor, the GC and pressure gauge) are responding properly by looking at the readings on the computers #1 and #2. Notify the cognizant scientist of any anomalous readings.

2.5 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the drying cycles. The heating tape upstream from the furnace will be turned off or on at the direction of the cognizant scientist.

2.6 Turn on the furnace and program it to perform the following (refer to the Omega Temperature Controller/Programmer manual):

2.6.1 to heat at a constant rate (e.g., between 10 and 15°C/min) to 50±2°C and hold the 50±2°C for 10 hours.

2.6.2 resume the constant heating rate (i.e., at the same rate as in Step 2.6.1) to 300±2°C and hold the 300±2°C for 24 more hours.

2.6.3 then allow the furnace to cool to ambient temperature.

2.7 Record in block 26 on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that computer #1 is monitoring and recording the:

2.7.1 Water vapor concentration with the moisture monitor.
2.7.2 Pressure with the pressure gauge.
2.7.3 Furnace and specimen temperatures

3.0 Conditioning Testing

The SNF passivation step in the furnace testing will involve exposing the test specimens to a gas mixture of 98% argon and 2% oxygen by volume at a flow rate of 100±2 cm^3/min.

3.1 Adjust the heating tape (downstream from the furnace) temperature to a constant value in the range of 50 to 100°C or the value specified by the cognizant scientist.

3.2 Purge the system with pure argon flow of 100±2 cm^3/min (i.e., by opening valves 1, 5, 6 & 8 or 7 & 9, and 11, but with valves 2,3,4, and 10 closed) for about 45 minutes and monitor the moisture content in the system. If the moisture monitor indicates high concentration (i.e., greater than -50°C dew point) then consult with the cognizant scientist before proceeding to the next step.

3.3 Set the argon and oxygen flow controllers to give the flows indicated above (i.e., 98% argon, 2% oxygen at a flow rate of 100±2 cm^3/min).

3.4 Switch the gas flow to the GC through the bypass line (i.e., opening valve 4 and closing valves 1, 2, 3, and 11 of Attachment 1). Record in block 27 on the data sheet of Attachment 2 the switch over time. Program computer #2 to monitor and record the oxygen concentration in the gas stream every 2 minutes for a period of at least 15 minutes after equilibration to establish the baseline.

3.5 Switch the gas flow to the furnace by opening valve 1, 5, and 11, and by closing valves 2, 3, 4 and 10. Record in block 28 on the data sheet of Attachment 2. Program computer #2 to monitor and record the oxygen concentration in the gas stream measured by the GC every 5 minutes until a constant oxygen concentration is established. At that constant concentration, monitor the gas stream for at least 30 minutes to establish the baseline concentration.

3.6 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual) to heat at a constant rate (e.g., between 12 to 15°C/min) to 250±2°C and hold at that temperature indefinitely. (Note that furnace cooling will be initiated manually after about 10 hours as described in Step 3.8.) Verify that the computers are monitoring and recording the:

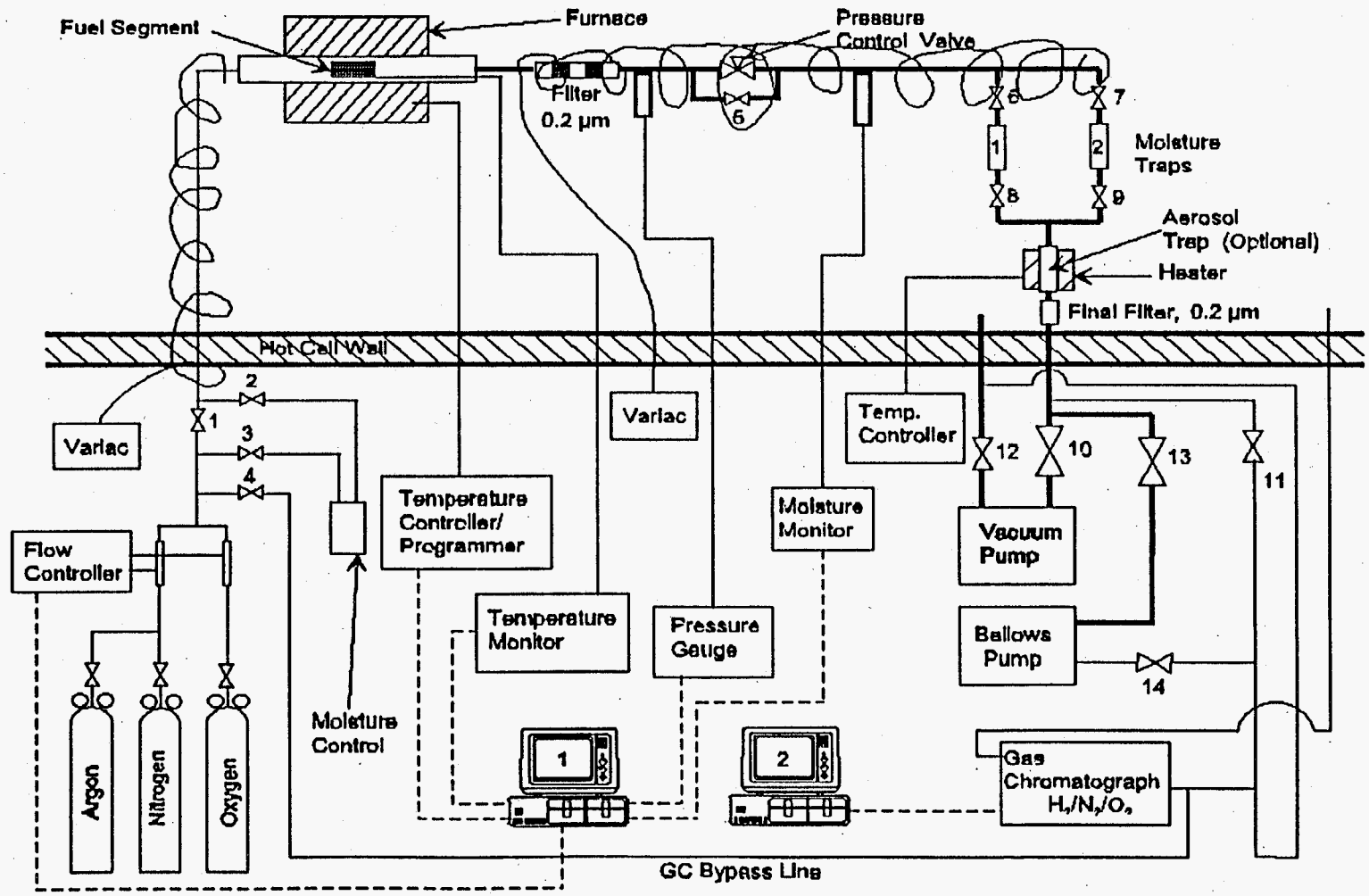
3.6.1 Moisture concentration using the moisture monitor for every 5 minutes.
3.6.2 Oxygen and hydrogen concentrations using the gas chromatograph for every 5 minutes.
3.6.3 Furnace and specimen temperatures every 5 minutes.

3.7 After 10 hours of conditioning of the specimens, turn off the oxygen flow and the furnace, and allow the specimen to cool to ambient temperature in flowing argon. Record in block 29 on the data sheet of Attachment 2 the time oxygen gas is turned off.

3.8 Open the furnace and carefully remove the specimen. Weigh the specimen twice using a QA Category 1 balance with 10 mg or better sensitivity. Record the weights in blocks 8 & 17 on the data sheet of Attachment 2. Record other observations in the PTL log book for inclusion in the project Laboratory Record Book (LRB).

3.10 Perform the test shut down steps per PTL-151 Section 3.0. Leave the furnace system in standby condition, that is, at ambient temperature with argon flow rate of about 100 cc/min.

Schematic Of The Furnace System



Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
	4 - Ruler Calibration Number	5 - Ruler Calibration Date

SAMPLE MEASUREMENTS

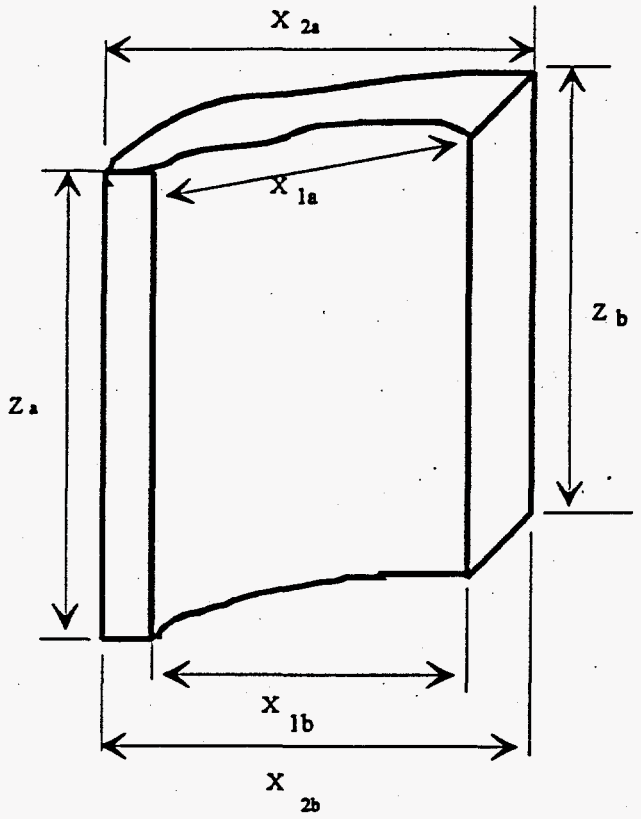
Measurement Number	6 - Specimen Identification Code SFEC5,4378-S1A-C								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	7	8	9	10	11	12	13	14	15
2	16	17	18	19	20	21	22	23	24

SYSTEM STATUS

25 - Time/Date	26 - Time/Date	27 - Time/Date	28 - Time/Date	29 - Time/Date

Attachment 3

Specimen Dimensions



CONTROLLED DOCUMENT

Test Instruction	Rev. No.: 0	Page 1 of 7
Title: Ignition testing of Specimen SFEC5,4378-S1A-H	**Furnace Run #: 019**	
Work Location: 327 (Building/Room or "General")	**TI No.: SNF-CT-029**	
Author: John Abrefah	**Effective Date:** 1/18/96 **Supersedes Date: New**	

Signatures:

Author _____ (Signature) 1-18-96 (Date)

Technical Reviewer Steve Mosley (Signature) 1-18-96 (Date)

Concurrences:

PTL Manager _____ (James M. Seay) 1/22/96 (Date)

HSNFP Charact. Manager _____ (Ronald P. Omberg) 1/22/96 (Date)

Purpose/Scope The purpose of this test instruction is to direct the ignition testing (Furnace run #19) of as received (i.e., unconditioned) Spent Nuclear Fuel (SNF) specimen SFEC5,4378-S1A-H in a controlled temperature and atmosphere furnace. The specimen was sectioned from the damaged end of SNF element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF specimen SFEC5,4378-S1A-H taken from the damaged end of the element SFEC5,4378.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) shall read and follow the steps in the procedure PTL-151: *Operation of Conditioning Furnace System.*

Equipment Reconfiguration The moisture traps will not be used to determine cumulative moisture content during the testing. Place a spring in the specimen boat to keep the specimen in contact with the thermocouple.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

SNF specimen SFEC5,4378-S1A-H will be loaded into the resistively heated furnace shown schematically in Attachment 1. The alumina furnace tube will be used for this testing.

1.1 Verify that the furnace is in standby condition with argon flowing at 20 to 200 cc/min with valves 2, 3, 4, 6, 8, 10, 12, 13 and 14 closed and valves 1, 5, 7, 9 and 11 open (note moisture trap #1 is out of service). If the furnace is not in standby condition, discontinue activity associated with this TI and notify the PTL cognizant engineer and the cognizant scientist.

1.2 Attach a calibrated ruler (record the calibration information in blocks 4 & 5 on the data sheet of Attachment 2) with an accuracy of ± 1mm to the specimen and take photographs of the exposed and cut fuel surfaces. Use the photographs to measure and record in blocks 8 through 14 and 17 through 23 on the data sheet of Attachment 2 the specimen dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp "burrs" at the cut surfaces that interfere with the measurements, sand them off with silicon carbide abrasive paper. Do not use a lubricant.

1.3 Weigh the specimen twice on a QA Category 1 balance (record the balance calibration information in blocks 1, 2 & 3 on the data sheet of Attachment 2) with 50 mg or better sensitivity. Record the specimen weight in blocks 6 & 15 on the data sheet of Attachment 2.

1.4 Make sure the 0 to 800 cc/min air flow controller is installed. Also ensure that the flow controller and channel 6 of the Aimax Data Acquisition System (DAS) on Computer #1 are both connected to position 3 of the Multichannel Dynablender.

1.5 Verify that channel 6 of the Aimax DAS on computer #1 has a proper calibration factor for the 0 to 800 cc/min flow controller. Perform the check by setting the flow controller to 50% and making sure that channel 6 on computer #1 reads 400 cc/min.

1.6 Verify that the oxygen/air cylinder in Attachment 1 (Schematic of the Furnace System) is connected to the dry air cylinder.

1.7 If the moisture monitor reads greater than 3 ppm (-70°C dew point), notify the PTL cognizant engineer and the cognizant scientist. Do not proceed without written direction for methods to dry out the system. Record the final moisture level prior to loading the specimen(s) into the furnace in the PTL Log Book.

Caution: Make sure the furnace system has cooled to the ambient hotcell temperature before loading the specimens. Minimize the time the furnace tube and gas lines will be open to air in the cell.

1.8 Mount the specimen in the sample holder such that the specimen is in the center of the uniform heated zone when loaded into the furnace. Ensure that the specimen is in contact with the specimen thermocouple. Record the method used to ensure contact in the PTL Log Book.

1.9 Load the sample holder into the furnace tube with extreme care, and assemble the rest of the system, i.e., the gas supply components and the effluent gas analytical components.

1.10 Prior to the start of the test, check for leakage in the system by pressurizing it and monitoring the pressure decay. Record leak check information (method and results) in the PTL log book. If the system leaks at a rate > 60 Torr/min notify the cogniznat scientist.

2.0 Ignition Test

The specimen will be heated to the point of ignition in an atmosphere of flowing dry air and then quenched by purging the system with high purity argon. The moisture content of the off-gas will be detected by the moisture monitor. Hydrogen in the off gas stream will be monitored by the gas chromatograph.

2.1 Open the dry air cylinder and ensure that the dry air flows through the system with valves 1, 5, 7, 9, and 11 open and valves 2, 3, 4, 6, 8, 10, 12, 13 and 14 closed. Establish a dry air flow rate of 500 cc/min.

2.2 Verify that all the moisture monitor is reading the system moisture content, the pressure gauge is reading close to cell's pressure by looking at the readings on computer #1. Notify the cognizant scientist of any anomalous readings.

2.3 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the testing. The heating tape upstream from the furnace will be left off.

2.4 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual):

2.4.1 to heat at a constant rate of 15°C/min to 700±5°C and hold the 700±5°C for 15 minutes.

2.4.2 then allow the furnace to cool to ambient temperature.

2.4.3 over-temperature protection to 50°C above furnace set-point.

2.5 Record in block 24 on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that computer #1 is monitoring and recording the:

2.5.1 Water vapor concentration with the moisture monitor

2.5.2 Pressure with the pressure gauge

2.5.3 Furnace and specimen temperatures.

2.6 The operator, RCT and PTL Technician shall continuously monitor the specimen temperature above 200°C. If the specimen temperature shows signs of very rapid change (see Attachment 3) then switch the gas supply from dry air to ultra high purity argon (flow rate of about 1 liter/min) to quench the reaction. Shut down the furnace manually with argon flowing through the system.

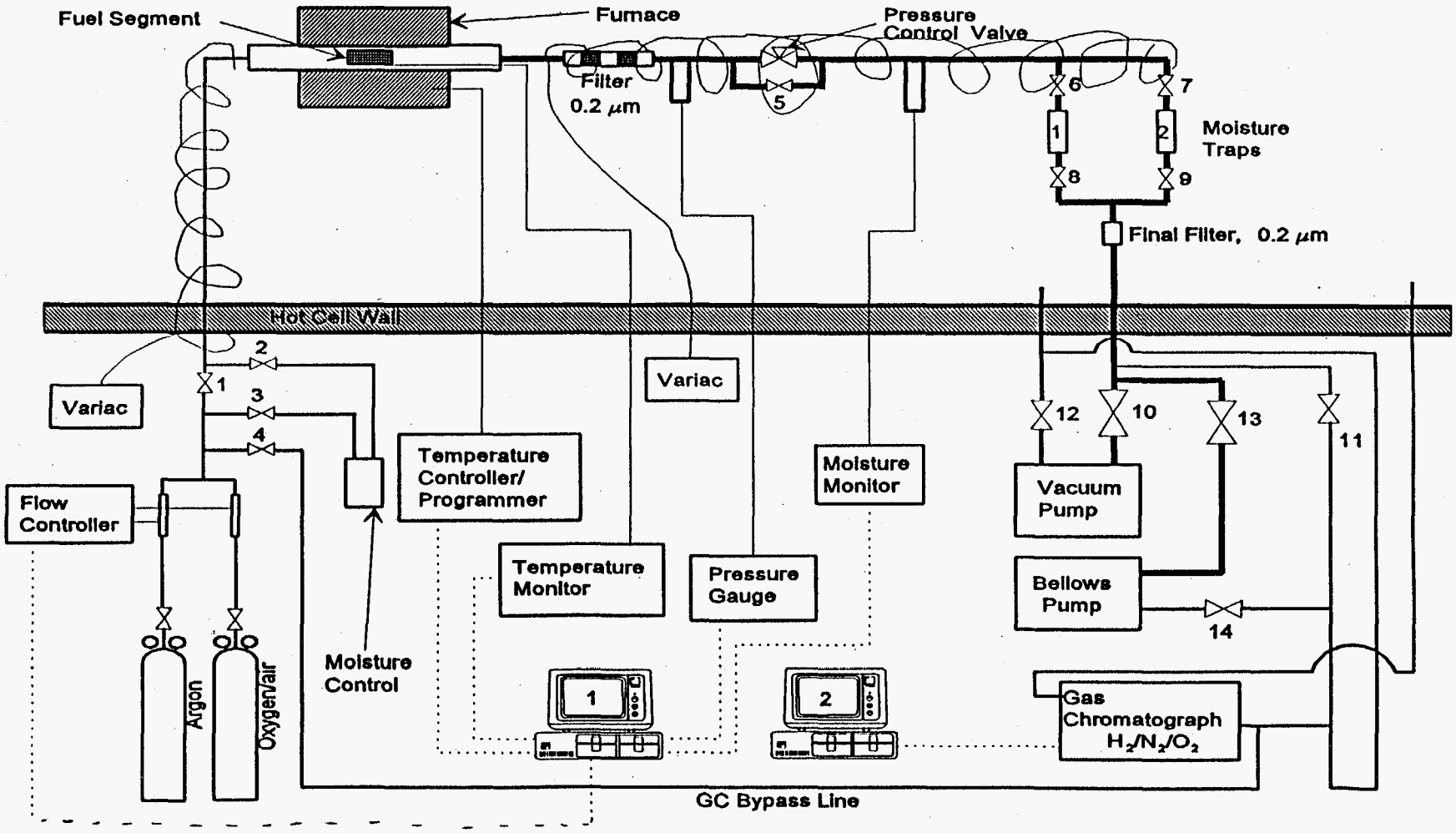
2.7 Ensure the furnace has cooled down to the ambient temperature.

2.8 Open the furnace and carefully remove the specimen. Weigh the specimen twice using a QA Category 1 balance with 50 mg or better sensitivity. Record the weights in blocks 7 & 16 on the data sheet of Attachment 2. Record other observations in the PTL log book for inclusion in the project Laboratory Record Book (LRB).

2.9 Visually inspect the specimen and take photograph per request of the cognizant scientist.

2.10 Perform the test shut down steps that leave the furnace system in standby condition, that is, at ambient temperature with argon flow rate of about 100 cc/min.

Schematic Of The Furnace System



Attachment 2

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
	4 - Ruler Calibration Number	5 - Ruler Calibration Date

SAMPLE MEASUREMENTS

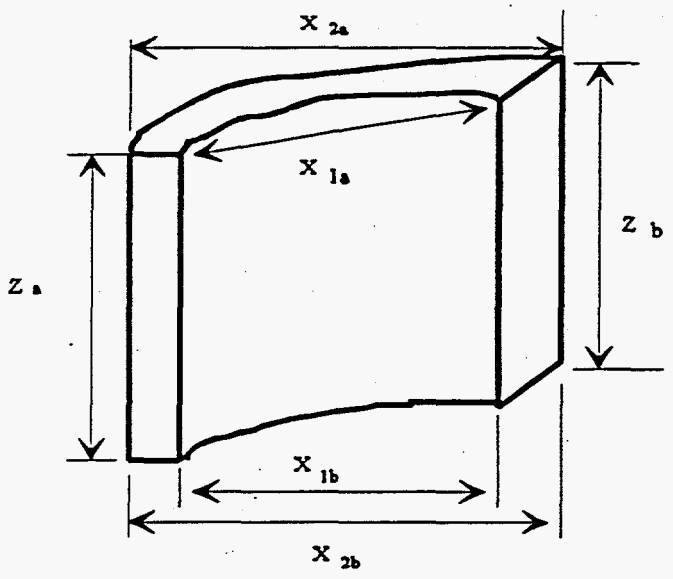
Measurement Number	9 - Specimen Identification Code: SFEC5,4378-S1A-H								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z_a	Z_b		X_{1a}	X_{1b}	X_{2a}	X_{2b}
1	6	7	8	9	10	11	12	13	14
2	15	16	17	18	19	20	21	22	23

SIGNATURE/VERIFICATION BLOCK

24

Attachment 3

Specimen Dimensions



Ignition Behavior of Metallic Uranium in Oxygen at Constant Heating Rate