NASA/TM—2020-220458

Observation of Hydrogen Isotope Diffusion Through Thin Palladium-Silver Samples

Phillip J. Smith
Glenn Research Center, Cleveland, Ohio

Anthony J. Colozza
Vantage Partners, LLC, Brook Park, Ohio

Ian J. Jakupca and Gustave C. Fralick
Glenn Research Center, Cleveland, Ohio

June 2020

NASA STI Program . . . in Profile

Since its founding, NASA has been dedicated to the advancement of aeronautics and space science. The NASA Scientific and Technical Information (STI) Program plays a key part in helping NASA maintain this important role.

The NASA STI Program operates under the auspices of the Agency Chief Information Officer. It collects, organizes, provides for archiving, and disseminates NASA's STI. The NASA STI Program provides access to the NASA Technical Report Server—Registered (NTRS Reg) and NASA Technical Report Server—Public (NTRS) thus providing one of the largest collections of aeronautical and space science STI in the world. Results are published in both non-NASA channels and by NASA in the NASA STI Report Series, which includes the following report types:

- TECHNICAL PUBLICATION. Reports of completed research or a major significant phase of research that present the results of NASA programs and include extensive data or theoretical analysis. Includes compilations of significant scientific and technical data and information deemed to be of continuing reference value. NASA counter-part of peer-reviewed formal professional papers, but has less stringent limitations on manuscript length and extent of graphic presentations.

- TECHNICAL MEMORANDUM. Scientific and technical findings that are preliminary or of specialized interest, e.g., "quick-release" reports, working papers, and bibliographies that contain minimal annotation. Does not contain extensive analysis.

- CONTRACTOR REPORT. Scientific and technical findings by NASA-sponsored contractors and grantees.

- CONFERENCE PUBLICATION. Collected papers from scientific and technical conferences, symposia, seminars, or other meetings sponsored or co-sponsored by NASA.

- SPECIAL PUBLICATION. Scientific, technical, or historical information from NASA programs, projects, and missions, often concerned with subjects having substantial public interest.

- TECHNICAL TRANSLATION. English-language translations of foreign scientific and technical material pertinent to NASA's mission.

For more information about the NASA STI program, see the following:

- Access the NASA STI program home page at http://www.sti.nasa.gov

- E-mail your question to help@sti.nasa.gov

- Fax your question to the NASA STI Information Desk at 757-864-6500

- Telephone the NASA STI Information Desk at 757-864-9658

- Write to:
  NASA STI Program
  Mail Stop 148
  NASA Langley Research Center
  Hampton, VA 23681-2199

NASA/TM—2020-220458

Observation of Hydrogen Isotope Diffusion Through Thin Palladium-Silver Samples

Phillip J. Smith
Glenn Research Center, Cleveland, Ohio

Anthony J. Colozza
Vantage Partners, LLC, Brook Park, Ohio

Ian J. Jakupca and Gustave C. Fralick
Glenn Research Center, Cleveland, Ohio

National Aeronautics and
Space Administration

Glenn Research Center
Cleveland, Ohio 44135

June 2020

Acknowledgments

The authors would like to thank Bruce Steinetz for his leadership and research guidance on this project. In addition, Richard Martin and Nicholas Penney were exceptionally helpful in setting up and operating the x-ray test series.

Level of Review: This material has been technically reviewed by technical management.

Available from

NASA STI Program
Mail Stop 148
NASA Langley Research Center
Hampton, VA 23681-2199

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
703-605-6000

This report is available in electronic form at http://www.sti.nasa.gov/ and http://ntrs.nasa.gov/

Contents

Observation of Hydrogen Isotope Diffusion Through Thin Palladium-Silver Samples

Phillip J. Smith
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Anthony J. Colozza
Vantage Partners, LLC
Brook Park, Ohio 44142

Ian J. Jakupca and Gustave C. Fralick
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Summary

Observations from several experimental scenarios by various research groups found that releasing deuterium gas from hydrided palladium (Pd) metal can result in unexplained temperature increases. This heating is possible evidence of heat generation in what is ostensibly an endothermic process. Creating a method for identifying and repeating this unusual scenario is beneficial for understanding the occurrence of such temperature increases. It is suspected that x-ray radiation and significant deuterium concentrations may contribute to such reactions. The work, described here, attempted to determine if x-ray stimulation, combined with diffusion of hydrogen or deuterium gas, can cause anomalous nuclear behavior in thin Pd-silver (Ag) and titanium (Ti)-carbide coated Pd-Ag sample disks.

The disks were placed in a chamber called a diaphragm reactor. Samples were continuously monitored by multiple instruments during testing to capture evidence of any reactions. The monitoring included recording disk temperature with an infrared (IR) camera and thermocouples on the disk surface, analyzing product gas composition and pressure changes throughout test operation, and quantifying radioactivity before, during, and after testing. Test variables consisted of disk temperatures ranging from 20 to 400 °C, hydrogen or deuterium gas, reactor gas pressures ranging from 0 to 50 psig, x-ray beam power and exposure orientation, and test durations up to several hours. No evidence was found in the test data for the occurrence of nuclear reactions or anomalous heating in the selected test conditions.

To offer the best opportunity to identify heat production, future iterations of this experiment would ideally involve a calorimeter so that heat input and output are quantified. Thermocouples and IR cameras are neither sensitive nor precise enough to identify temperature differentials over time as evidence of low-energy nuclear-scale-based heat production. Additionally, more responsive real-time nuclear emissions detection may significantly improve the likelihood of reaction detection. It remains unclear whether any feasible combination of pressure and temperature can create a high enough hydrogen concentration within the Pd metal for any potential fusion mechanism to proceed.

Nomenclature

AC	alternating current
AEC	Advanced Energy Conversion
Ag	silver
C	carbon
COTS	commercial-off-the-shelf
D	deuterium
GL	gas loaded
HFR	hydrogen flow restrictor
HMV	hydrogen manual valve
HPG	hydrogen pressure gauge
HPR	hydrogen pressure regulator
HPS	external temperature controller
HPT	hydrogen pressure transducer
HR	reactor heater
HRV	hydrogen relief valve
HS	gas supply tank
HV	voltage sensor
HVAC	heating, ventilation, and air conditioning
I	AC amperage
ID	inner diameter
IR	infrared
NL	not loaded
OD	outer diameter
P	gas pressure
Pd	palladium
PPE	personal protective equipment
PT	pressure transducer
RGA	residual gas analyzer
SI	current sensor
SOC HD	socket head
T	temperature
TC	thermocouples
Ti	titanium
UNF	unified fine pitch thread
USB	universal serial bus
V_L	voltage measured by AC root-mean-square voltage transducer
V_{PT}	voltage measured by pressure transducer
VFM	RGA
VCR	vacuum coupling radiation
VMV	vacuum manual valve
VP	vacuum pump
VPG	vacuum pressure gauge
VPT	vacuum pressure transducer
VRV	vacuum relief valve

VS sample cylinder
Z atomic number

Background and Purpose

Several research groups have witnessed unexpected heat production and anomalous element generation when exposing palladium (Pd) metal to deuterium gas (Refs. 1 to 5). Attempts to quantify this heat production have shown up to a 25 percent excess of thermal power produced in the course of removing the gas from a 15-g metal sample (Ref. 6). There is no satisfactory explanation yet for observing these exothermal events during an endothermic process. Development of an explanatory theory is complicated by a lack of consistency when attempting to replicate experimental results.

To aid in identification of this reaction, it is desirable to evaluate by multiple methods the thermal response of a Pd sample. In this case, both impinging thermocouples and an infrared (IR) camera monitored the disk surface temperature. The gas purity and pressure was continuously monitored in situ by a residual gas analyzer (RGA) on the low-pressure side of the Pd disk. Therories exist that there may be nonmechanical and nonchemical explanations for this exothermic phenomena. To evaluate a possible source, the local ionizing radiation environment was monitored and recorded before, during, and after testing. These evaluations are likely to be most beneficial if there is also identification of an operating condition that reliably produces aberrant results.

The purpose of this study was to determine if x-ray stimulation, combined with high concentration gradients resulting from hydrogen or deuterium gas diffusion, can cause anomalous nuclear behavior in Pd-silver (Ag) and titanium (Ti)-carbide-coated Pd-Ag disks. Test variables consist of diaphragm temperature, hydrogen gas isotope, supply gas pressure, x-ray beam power and exposure orientation, and test duration.

Test Material, Equipment, and Procedures

A process flow diagram is shown in Figure 1 to illustrate the experimental test system. A single supply lecture bottle, of at least 99.999 percent pure hydrogen, deuterium, or nitrogen, provides gas for the high-pressure side of the system when the hydrogen manual valve HMV03 is opened. The gas vendor supplied batch certifications for the supply gas species when shipped. It must be noted that the use of "hydrogen" in the component name represents the location of that item on the high-pressure gas supply section of the system. The same component is in place even if nitrogen or deuterium cylinders are installed instead of a hydrogen cylinder. The hydrogen flow restrictor HFR01 is in place to reduce the required hydrogen relief valve HRV01 capacity. That relief valve prevents an overpressurization event from damaging any pressure-limited components.

Hydrogen pressure gauge, HPG01, displays the supply bottle pressure while a second gauge HPG02 shows the regulated gas pressure downstream of hydrogen pressure regulator HPR01. Manual system venting and purging is accomplished by opening the hydrogen dump valve HMV01 and gas is directed towards a heated reactor through manual valve HMV02. Gas pressure downstream of HMV02 is confirmed by viewing pressure gauge HPG03. The hydrogen pressure transducer HPT01 allows for recording, by a Graphtec data logger (Graphtec America, Inc.), of the regulated supply gas pressure near the reactor.

On the opposite side of the reactor section, there is an RGA (VFM01) that requires a vacuum subsystem to maintain a constant low pressure. In order to function, the RGA requires a level of vacuum less than 10^{-3} torr.

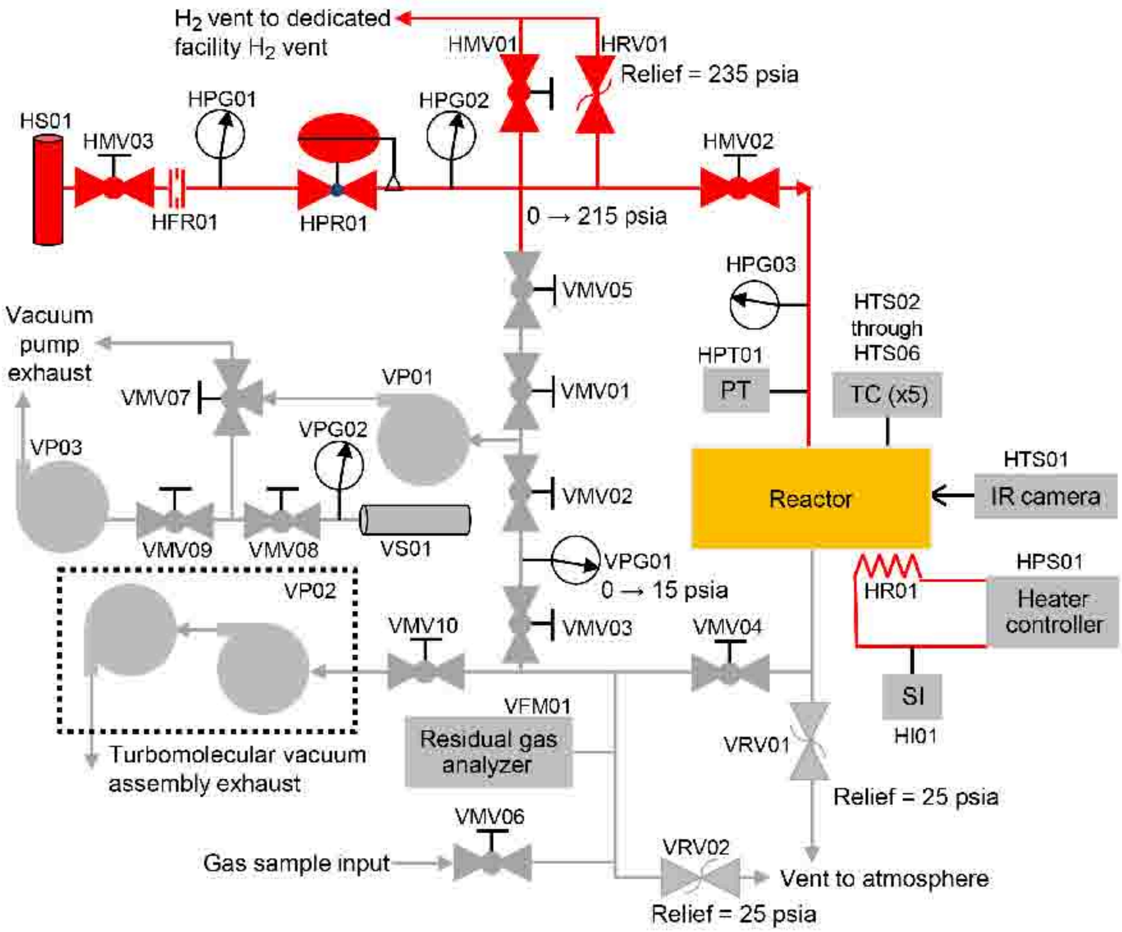

Figure 1.—Diaphragm reactor test system. IR is infrared. PT is pressure transducer. SI is current sensor. TC is thermocouple.

The vacuum is initially created by the roughing pump VP01 to remove gas and water vapor. The turbomolecular pump assembly VP02 pumps the system to higher vacuum levels. Vacuum manual valves (VMV02, VMV03, VMV04, VMV10) are present to isolate the pumps from any pressure sources. To protect the pumps from sudden overpressurization, two relief mechanisms are present: the vacuum relief valve VRV01 is nearest to the reactor and the burst disc VRV02 is located close to the vacuum pump VP02. Prior to test startup, the high-pressure side of the system may be evacuated by opening vacuum manual valves VMV01 and VMV05 to ensure high gas purity is maintained.

In certain circumstances, the low-pressure gas that diffuses through the reactor may be pumped into a sample cylinder, VS01, for later analysis. The auxiliary pump VP03 is connected to cylinder VS01 through VMV09 and VMV08 to evacuate the cylinder prior to filling. When collection is desired, VMV09 and VMV10 are closed, VMV08 is opened, VMV07 is turned away from the exhaust and towards VS01, and cylinder pressure is monitored on vacuum pressure gauge VPG02. The cylinder may later be connected to VMV06 so that gas is supplied to the RGA. For further reference, a bill of materials is presented in Appendix D.

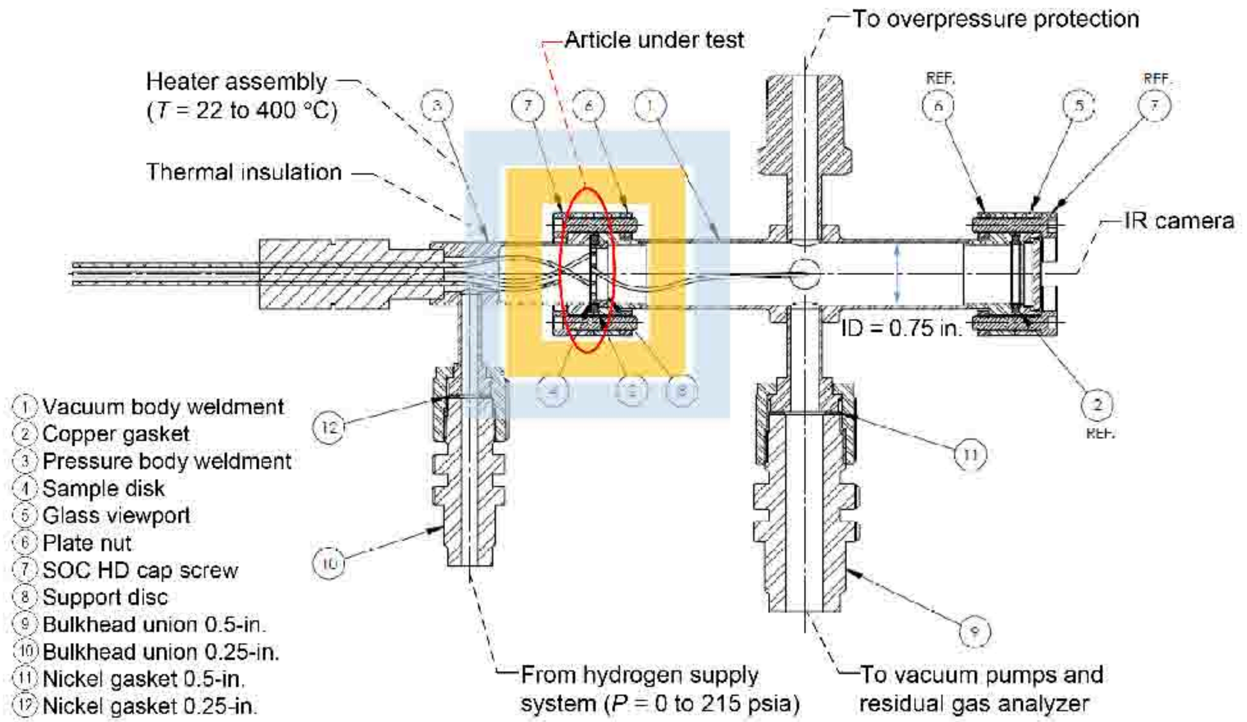

Figure 2.—Diaphragm reactor assembly. ID is inner diameter. IR is infrared. *P* is pressure. SOC HD is socket head. *T* is temperature.

The diaphragm reactor assembly is detailed in Figure 2. This reactor enables control over the rate that hydrogen isotopes transverse a thin disk material. Initially, the disk material is 75 wt% Pd and 25 wt% Ag with a total thickness of 0.004 in. Onto this sample disk was deposited a 2-nm Ti-carbide coating followed by a 40-nm Pd-Ag layer. The disk, identified within the "Article under test" notation in Figure 2, is restrained within a fixture comprising slightly modified commercial-off-the-shelf (COTS) flanges and a restraining support disk, identified as item number 8. The disk temperature and differential pressure across the disk are varied according to the test plan. The disk is instrumented with thermocouples (HTS02 to HTS06) and is video monitored by a FLIR® E40 IR camera aimed through a sapphire window in line with the vacuum side of the sample disk.

This diaphragm reactor assembly is shown partially disassembled in Figure 3. It is heated by means of an external band heater strapped to the outer diameter (OD) of the customized COTS flanges. An external temperature controller HPS01 regulates the temperature of the flange OD surface by pulse width modulating the alternating current (AC) power to the resistive heating element within the band heater. To minimize the necessary power output of the heating element and protect personnel in the area, the heater is insulated to reduce the temperature of the exposed surfaces.

For each test, the Pd-Ag disk was installed between the customized COTS flanges in the diaphragm reactor assembly. An example Pd-Ag sample disk image is presented in Figure 4. More sample and system images are available in Appendix C. Samples were named in the format Project Name-Sample Bulk Material-Test Exposure-Prior Exposure-Date in Year, Month, Day format-Sample Number. Once the Graphtec data acquisition system was powered on, VP01 was started and VMV01 was opened. The RGA interface computer was powered on to initially monitor the vacuum system pressure using the Pirani and ion gauges internal to the RGA. When the Pirani gauge displayed less than 0.1 torr, the second roughing pump, contained within VP02, was manually activated. The turbomolecular pump automatically started once the vacuum pressure was sufficiently low.

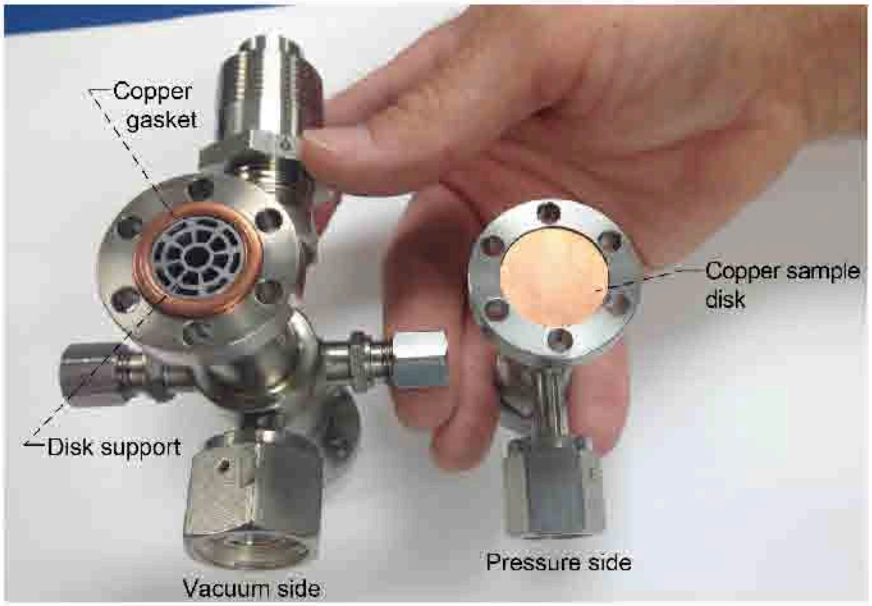

Figure 3.—Disassembled diaphragm reactor assembly with sample disk and seal in place.


Figure 4.—Palladium-silver sample disk AEC–Pd–GL–NL–20140916–111.

Simultaneous with the evacuation process, HPR01 was fully closed. The COTS bottle valve was opened and the supply pressure was recorded from HPG01. If this bottle pressure was insufficient to support testing as outlined in the test plan, the bottle was replaced. Once a bottle with sufficient pressure was installed, HMV01 was verified to be closed and HPR01 was increased to 50±10 psig as shown on HPG02. Next, HMV01 was closed and HMV03 was left open. As required to complete the test plan, the reactor temperature setpoint was adjusted on HPS01 and the supply pressure was set on HPR01. During steady-state operation, VP01 was powered off and VMV02 was closed.

If the test plan specified using an x-ray generator, the beam was directed radially downwards from the top or into the side of the diaphragm reactor. The standard x-ray head beam settings were 150 kV and 10 mA. The beam on duration was increased every hour of test operation.

At the conclusion of testing, the heater power supply was disconnected and HMV03 was closed. To slowly depressurize the system to atmospheric levels, HMV01 was opened. The turbomolecular vacuum pump assembly was powered off and the test data was saved on the Graphtec data logger. A complete test procedure check sheet is provided in Appendix E.

Data Analysis

It was necessary to apply conversion factors to convert two sensor voltage measurements into pressure and current outputs. Gas pressure, *P*, supplied to the sample disk was calculated using

$$P = 100V_{PT} \tag{1}$$

where V_{PT} is the voltage measured by the pressure transducer. The AC amperage *I* was determined from

$$I = 2V_L \tag{2}$$

where V_L is the voltage measured by the AC root-mean-square voltage transducer.

Results and Discussion

By all monitored means, all tests proceeded similarly regardless of the hydrogen-isotope test gas. No evidence characterized anomalous heat production or uneven heating on the disk surface. It remains possible that such exothermal phenomena can occur, however, the set of conditions described herein did not produce measurable results. Test samples analyzed by a Mirion Technologies (Canberra), Inc., Series 5 XLB™ counting system for alpha and beta emissions returned no evidence of continuing nuclear activity. Furthermore, the continued lack of an identifiable positive test condition prevented further development of any explanatory theory.

At baseline conditions with no gas supply connected, the RGA tended to show small peaks at AMU 14 to 19. These represent the presence of small initial amounts of water and air within the vacuum system. Once steady-state temperature and pressure test conditions were attained, the vacuum pumping rate could not typically exceed the hydrogen diffusion rate through a sample disk. Thus, the RGA frequently displayed pressure measurements on the high-pressure end of the sensor range. This elevated pressure showed the hydrogen gas species in the trivalent form as a mass spike at AMU 6. As both the trivalent form of deuterium and tritium appear at AMU 6, as implemented, this method is insufficient to distinguish between the hydrogen isotopes. For further reference, full test results are arranged in Appendix A, presenting temperature, pressure, and RGA data from each test day. Table I provides a summary of these test conditions.

Test performance was also evaluated through several materials analysis methods. Using time-of-flight secondary ion mass spectrometry, a depth profile elemental analysis was performed on several sample disks, both before and after testing, to both review coating effectiveness and examine for the presence of unexpected elements. No elemental anomalies were identified and the coating was generally uniform relative to the total material thickness. An example results plot is provided in Figure 5. The Ti and carbon (C) profile curvatures make it appear that Ti and C may be diffusing into the Pd-Ag.

Initial and post-test materials were scanned with a Series 5 XLB™ automatic low background alpha and beta counting system. This gas proportional counter was used for 10 min alpha and beta emission counts. The results were well within the background uncertainty range for all samples. Additionally, a Geiger counter, placed in various locations near the reactor section throughout all testing, never indicated any radiation levels above the laboratory background.

TABLE I.—TEST DAYS AND CONDITIONS

Test date	Sample disk	Gas species	Set pressure, psig	Set temperature, °C	Related Appendix A figures
Aug. 14, 2014	AEC–Pd–GL–NL–20140814–1	Nitrogen	125	400	A.1 to A.3
Aug. 14, 2014	AEC–Pd–GL–NL–20140814–1	Deuterium	45	375	A.4 to A.6
Aug. 22, 2014	AEC–Pd–GL–NL–20140814–1	Deuterium	5	350	A.7 to A.10
Aug. 27, 2014	AEC–Pd–GL–NL–20140814–1	Deuterium	8	325	A.11 to A.15
Sept. 9, 2014	AEC–Pd–GL–NL–20140903–101	Hydrogen	8	300	A.16 to A.18
Sept. 11, 2014	AEC–Pd–GL–NL–20140903–101	Deuterium	8	325	A.19 to A.21
Sept. 12, 2014	AEC–Pd–GL–NL–20140903–101	Deuterium	8	325	A.22 to A.24
Sept. 15, 2014	AEC–Pd–GL–NL–20140903–101	Deuterium	8	325	A.25 to A.27
Feb. 18, 2015	AEC–Pd–GL–NL–20140903–102	Deuterium	8	275	A.28 to A.30
Mar. 2, 2015	AEC–Pd–GL–NL–20140903–103	Deuterium	8	375	A.31 to A.32
Mar. 9, 2015	AEC–Pd–GL–NL–20140903–104	Deuterium	8	375	A.33 to A.36
Jan. 28, 2015	AEC–Pd–GL–NL–20140916–106	Deuterium	18	375	A.37 to A.39
Feb. 3, 2015	AEC–Pd–GL–NL–20140916–107	Deuterium	0	375	A.40 to A.42
Sept. 19, 2014	AEC–Pd–GL–NL–20140916–111	Deuterium	8	400	A.43 to A.45
Sept. 25, 2014	AEC–Pd–GL–NL–20140916–112	Deuterium	8	400	A.46 to A.48
Oct. 1, 2014	AEC–Pd–GL–NL–20140916–113	Nitrogen	45	400	A.49 to A.51
Oct. 2, 2014	AEC–Pd–GL–NL–20140916–113	Deuterium	8	375	A.52 to A.54
Oct. 7, 2014	AEC–Pd–GL–NL–20140916–113	Deuterium	8	400	A.55 to A.57
Oct. 8, 2014	AEC–Pd–GL–NL–20140916–113	Deuterium	8	400	A.58 to A.60
Jan. 20, 2015	AEC–Pd–GL–NL–20140916–116	Deuterium	8	375	A.61 to A.63
Jan. 22, 2015	AEC–Pd–GL–NL–20140916–117	Deuterium	9	375	A.64 to A.66

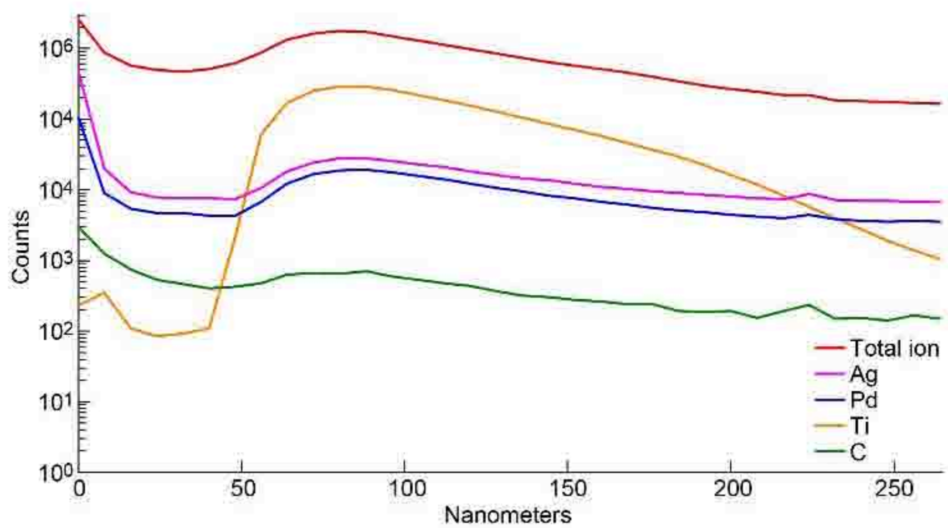

Figure 5.—Energy-dispersive x-ray spectroscopy elemental analysis depth profile of sample AEC–Pd–GL–NL–20140916–118. Ag is silver. C is carbon. Pd is palladium. Ti is titanium.

Conclusions and Recommendations

To offer the best opportunity to identify heat production, future iterations of this experiment should involve development of a calorimeter. It seems unlikely that thermocouples and infrared cameras are sensitive and precise enough to make concrete declarations regarding nuclear-scale-based heat production. A real-time nuclear emission detector with increased sensitivity may significantly improve the likelihood of reaction detection, but it is still not clear that any viable combination of pressure and temperature can create a high enough hydrogen concentration within the palladium metal for any potential fusion mechanism to proceed.

Pure titanium samples are a desirable additional test material due to the capacity of the material to stably contain hydrogen. Much higher pressure and temperature levels, beyond the limits of this test apparatus, are required for initial loading of hydrogen into that material. Assuming the nonmechanical and nonchemical exothermal phenomena are real, larger volume samples will present a potential for greater heat output that would be easier to identify. In this set of experiments, the samples were intentionally thin and minimally massive.

Appendix A.—Raw Data Charts and Tables

The following information contains the raw data charts and tables for the various palladium (Pd)-silver (Ag) samples. Testing dates from 2014 are found in Figure A.1 to Figure A.27 and Figure A.43 to Figure A.60. Figure A.28 to Figure A.42 and Figure A.61 to Figure A.66 contain the 2015 testing dates. See Table I for more details.

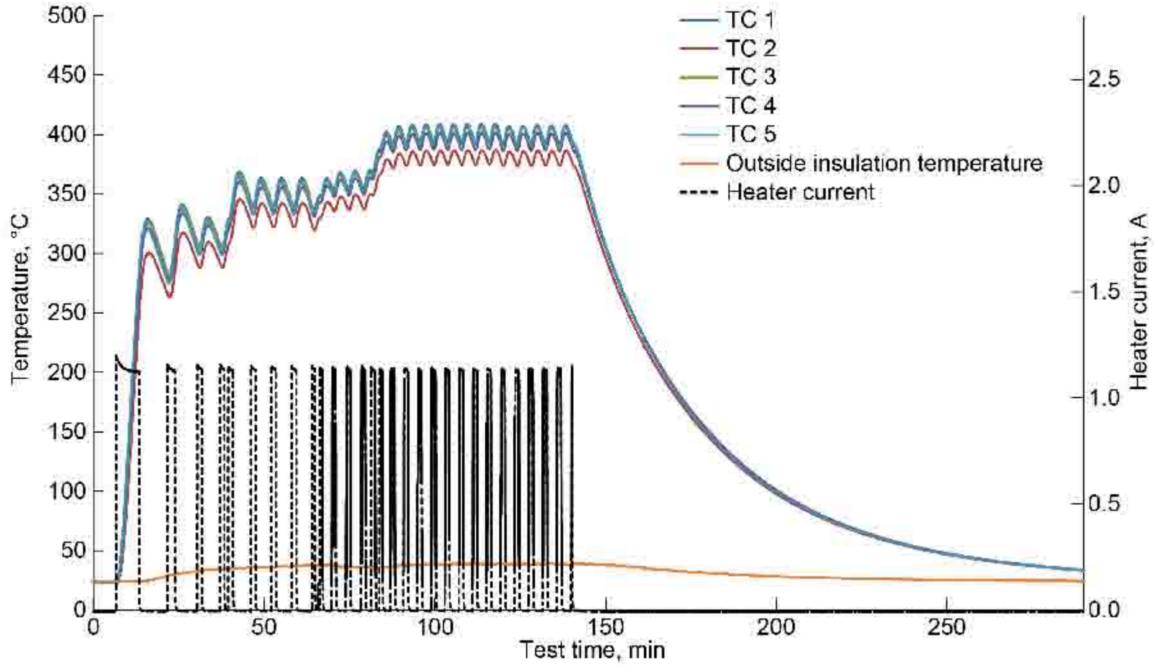

Figure A.1.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140814–1 tested on August 14, 2014, with nitrogen gas.

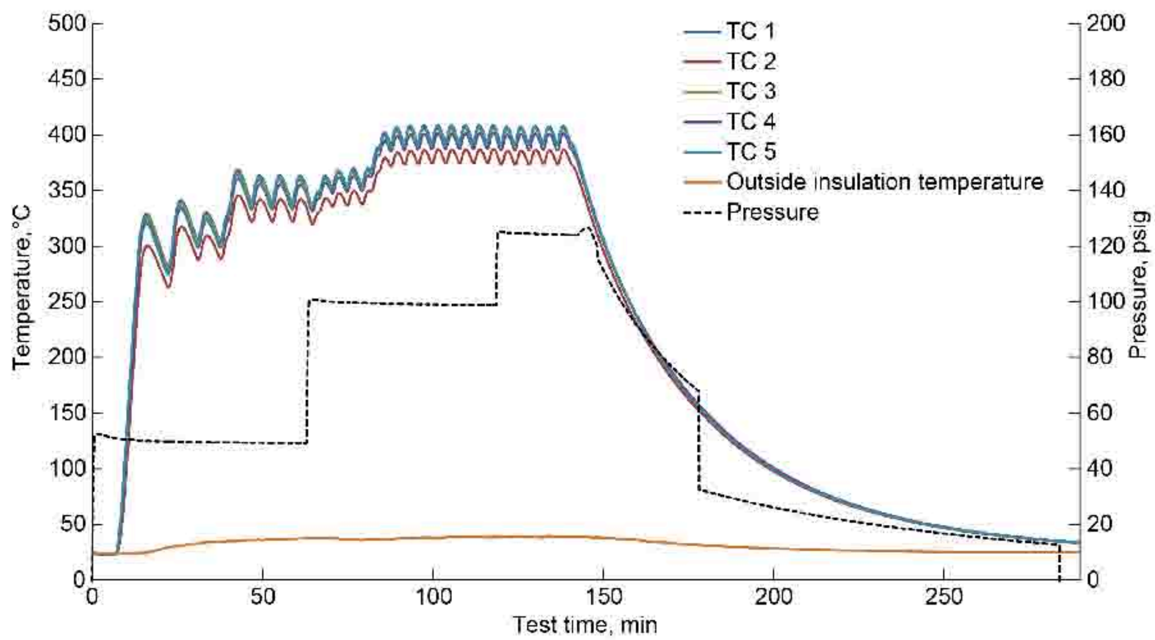

Figure A.2.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140814–1 tested on August 14, 2014, with nitrogen gas.

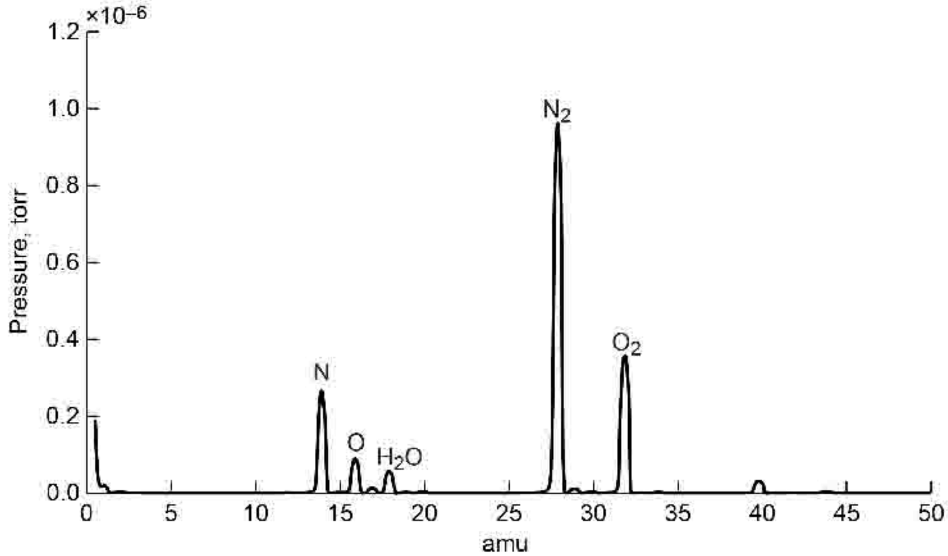

Figure A.3.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140814–1 tested on August 14, 2014, with nitrogen gas at 125 psig and 400 °C.

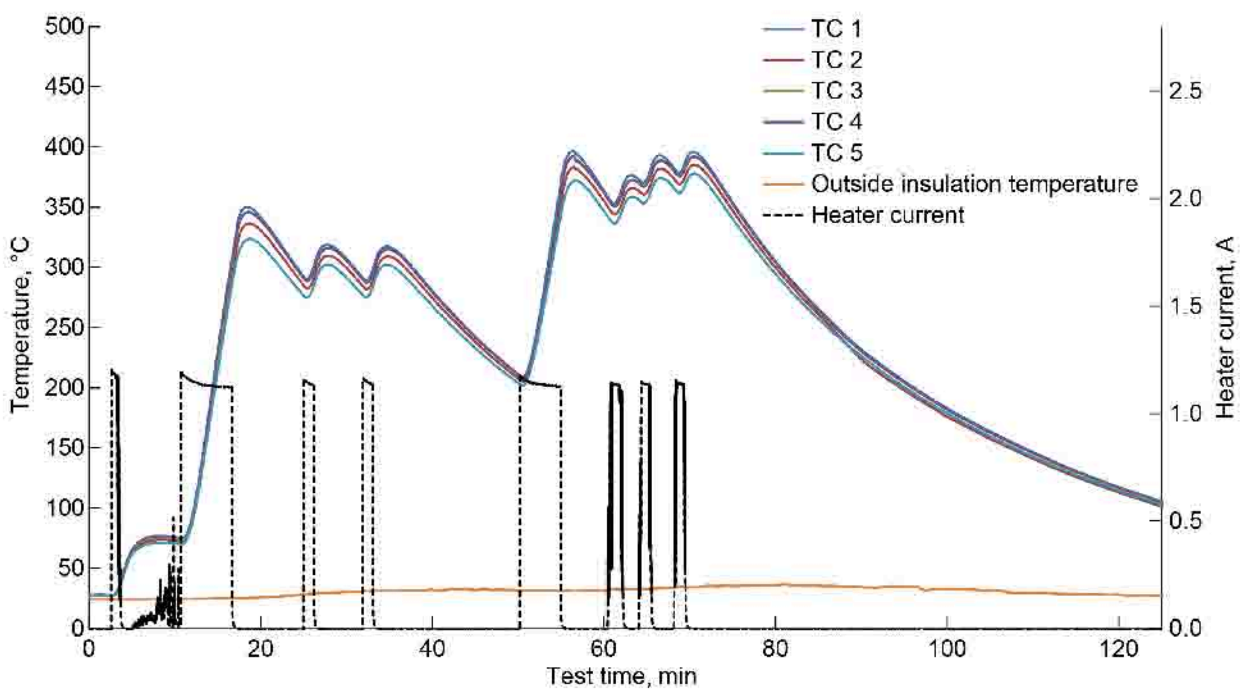

Figure A.4.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140814–1 tested on August 14, 2014, with deuterium gas.

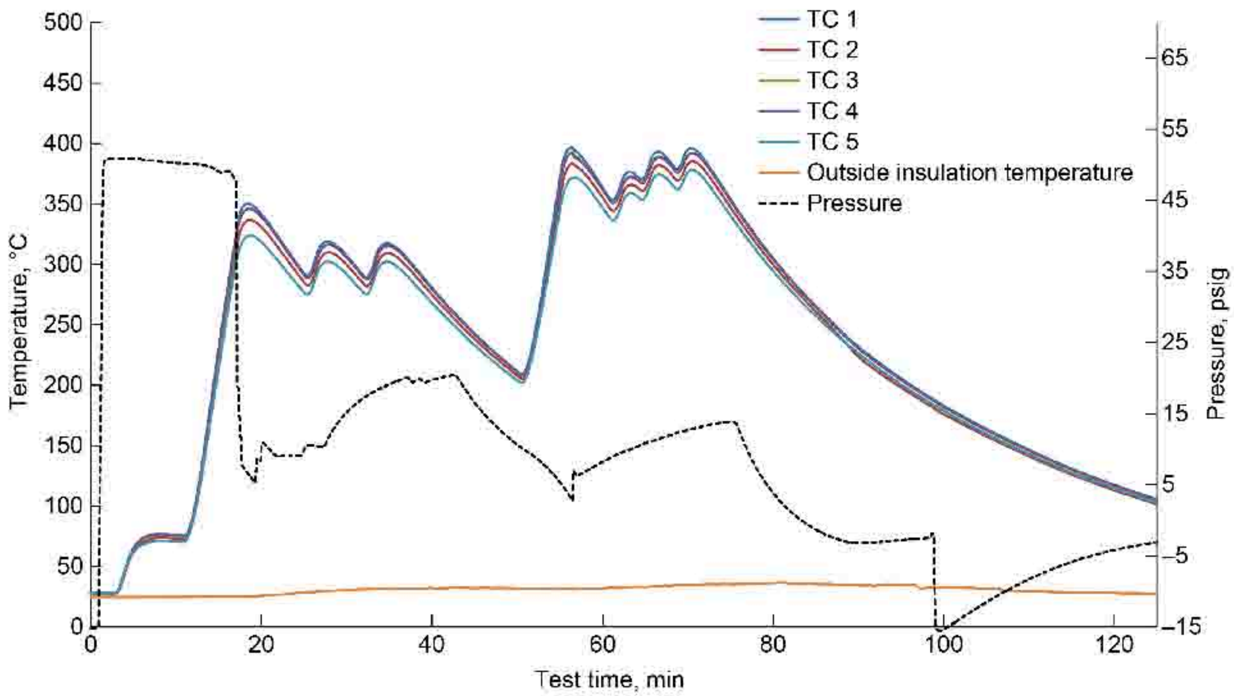

Figure A.5.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140814–1 tested on August 14, 2014, with deuterium gas.

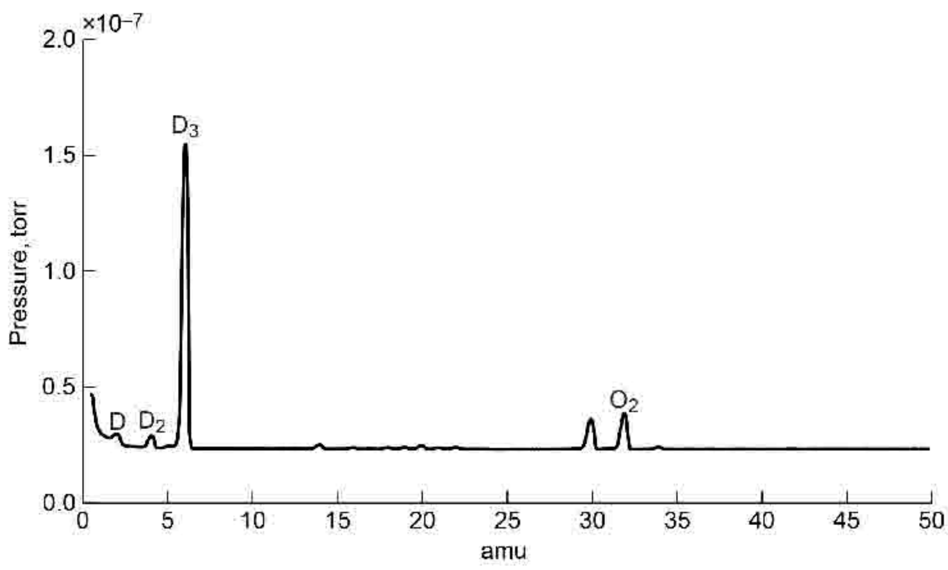

Figure A.6.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140814–1 tested on August 14, 2014, with deuterium gas at 8 psig and 375 °C.

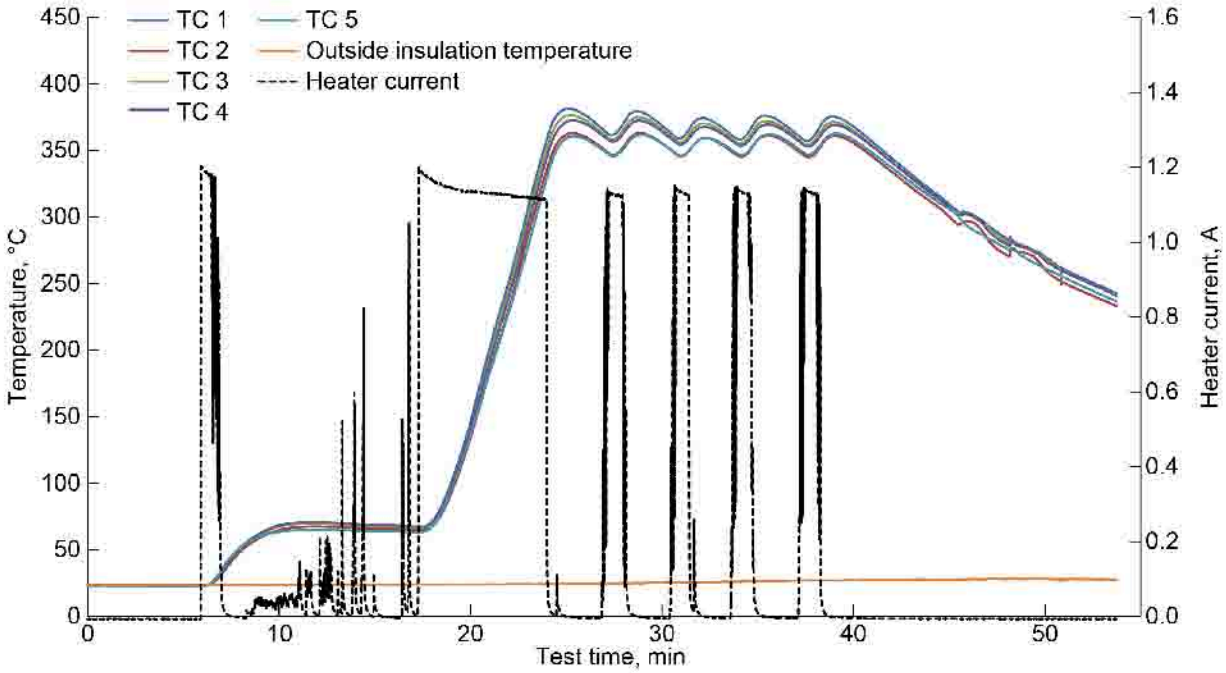

Figure A.7.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140814–1 tested on August 22, 2014, with deuterium gas.

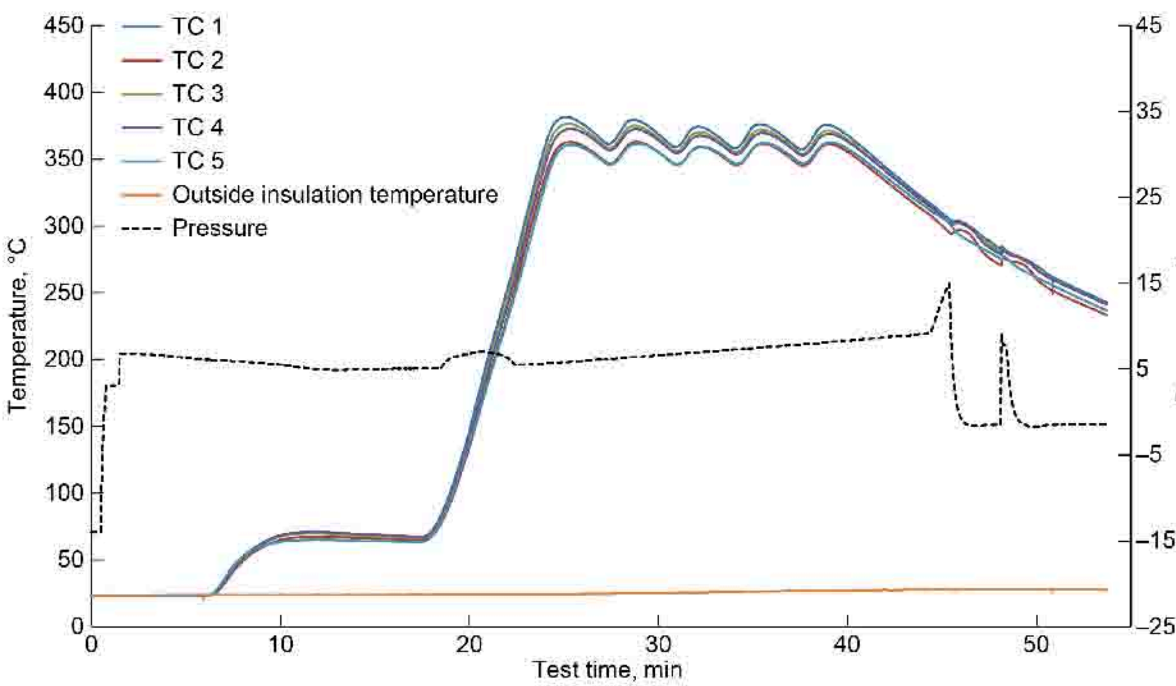

Figure A.8.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140814–1 tested on August 22, 2014, with deuterium gas.

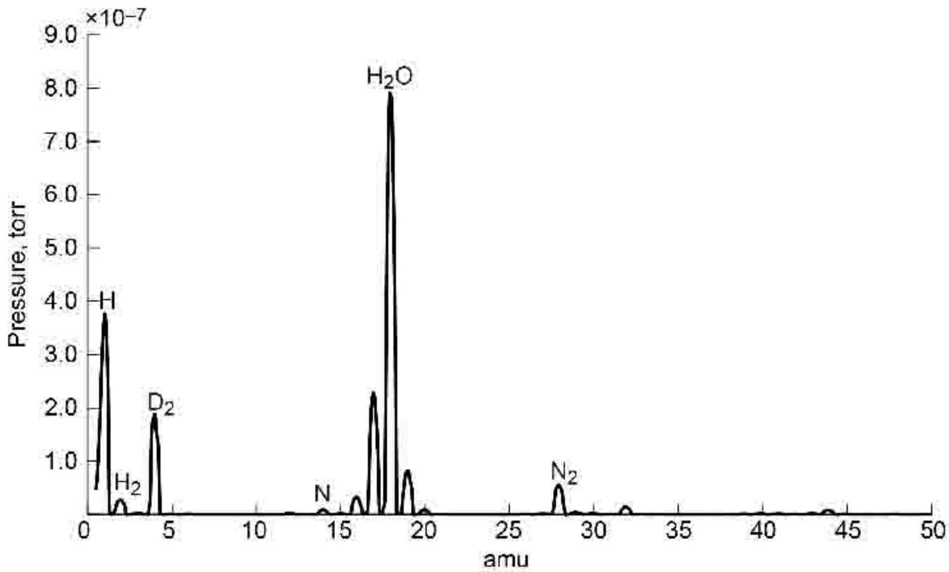

Figure A.9.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140814–1 tested on August 22, 2014, with deuterium gas at 7 psig and 23 °C.

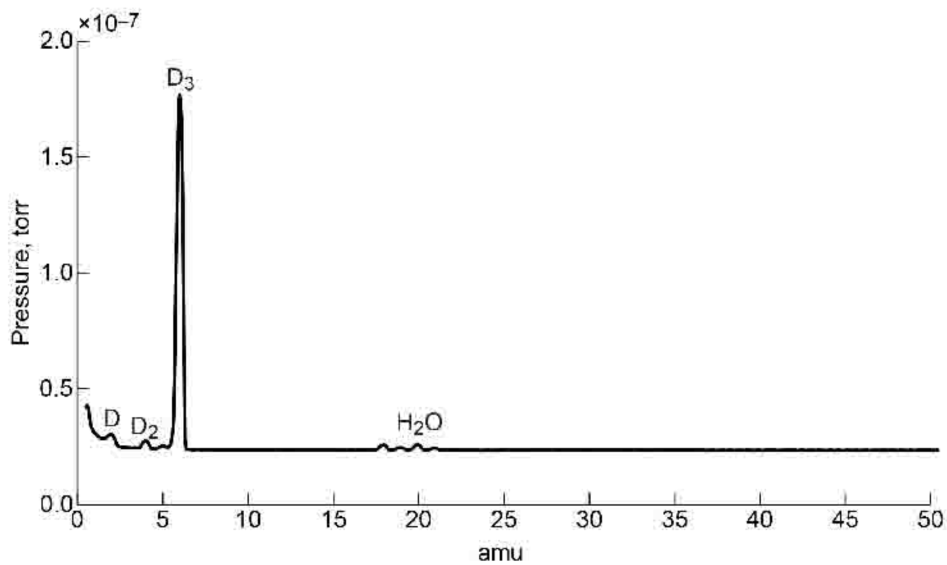

Figure A.10.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140814–1 tested on August 22, 2014, with nitrogen gas at 8 psig and 350 °C.

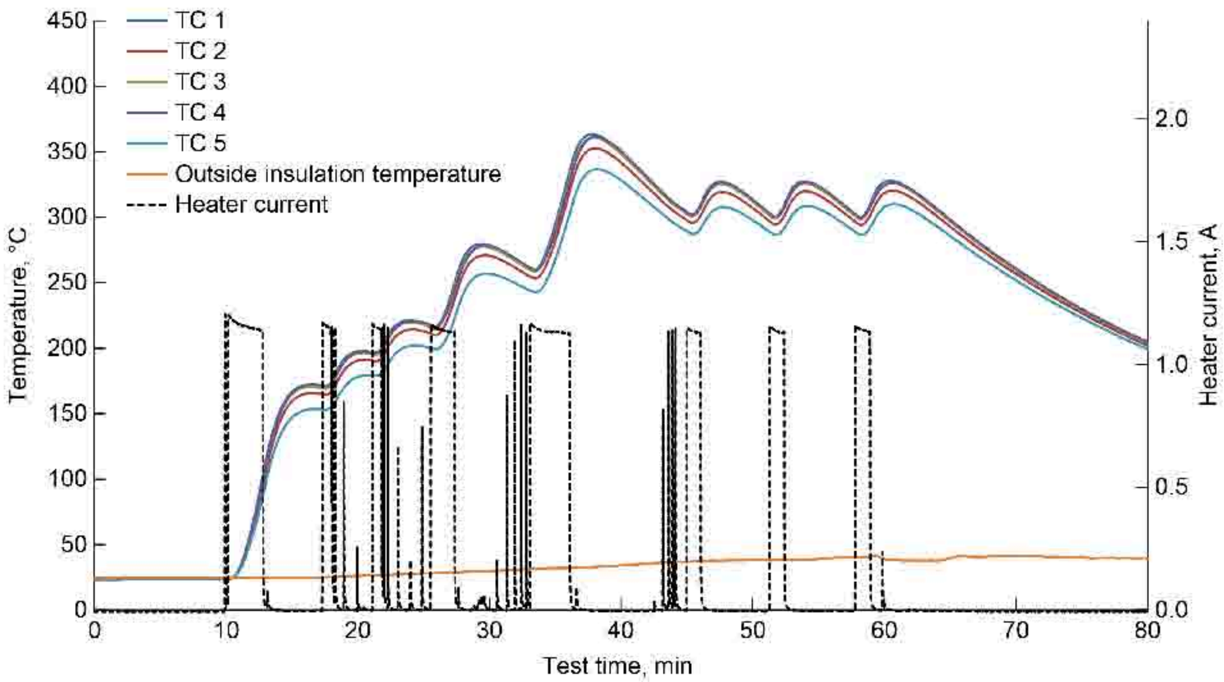

Figure A.11.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140814–1 tested on August 27, 2014, with deuterium gas.

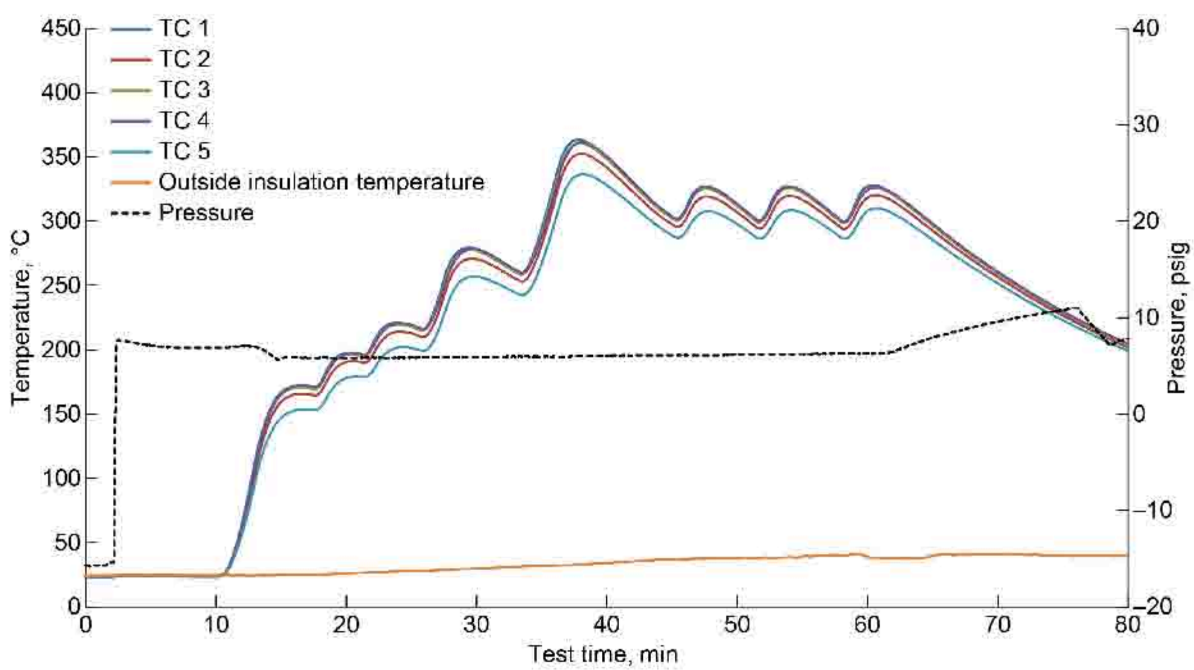

Figure A.12.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140814–1 tested on August 27, 2014, with deuterium gas.

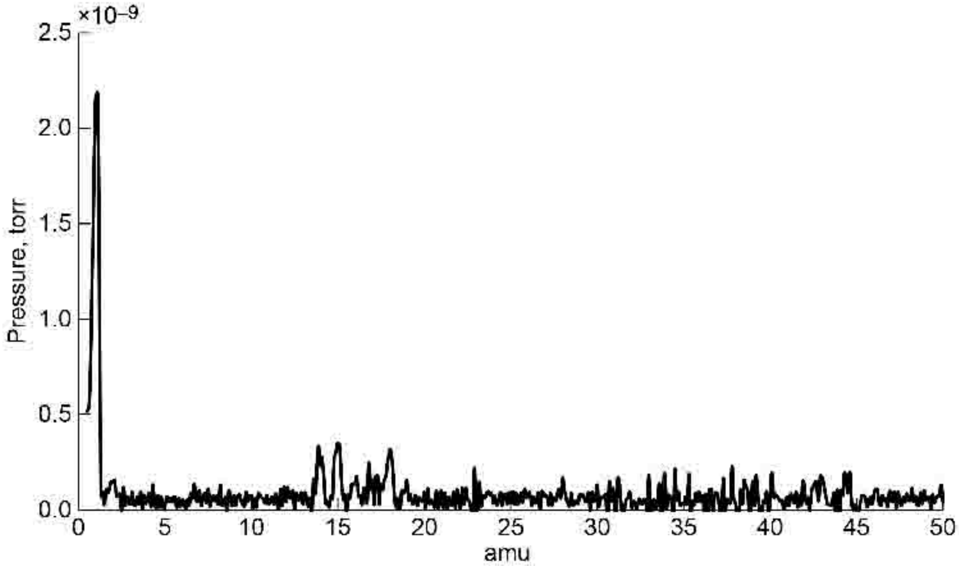

Figure A.13.—Baseline residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140814–1 tested on August 27, 2014, with no gas supplied and 20 °C. No identifiable peaks.

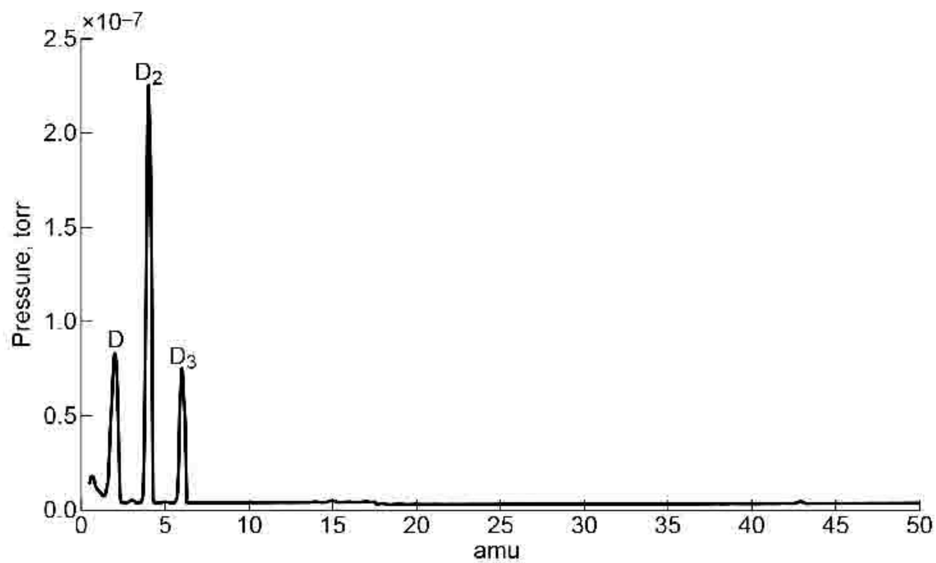

Figure A.14.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140814–1 tested on August 27, 2014, with deuterium gas at 7 psig and 60 °C.

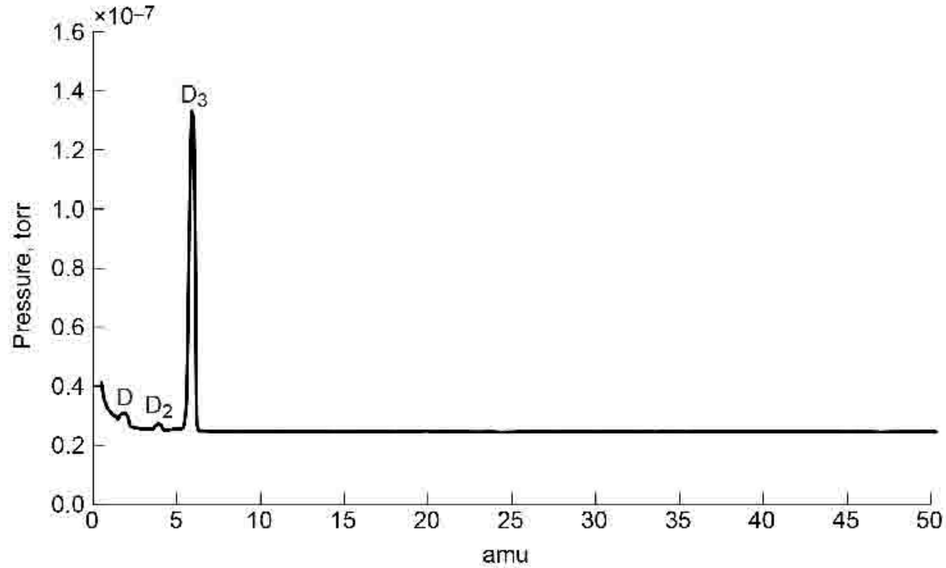

Figure A.15.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140814–1 tested on August 27, 2014, with deuterium gas at 6 psig and 350 °C.

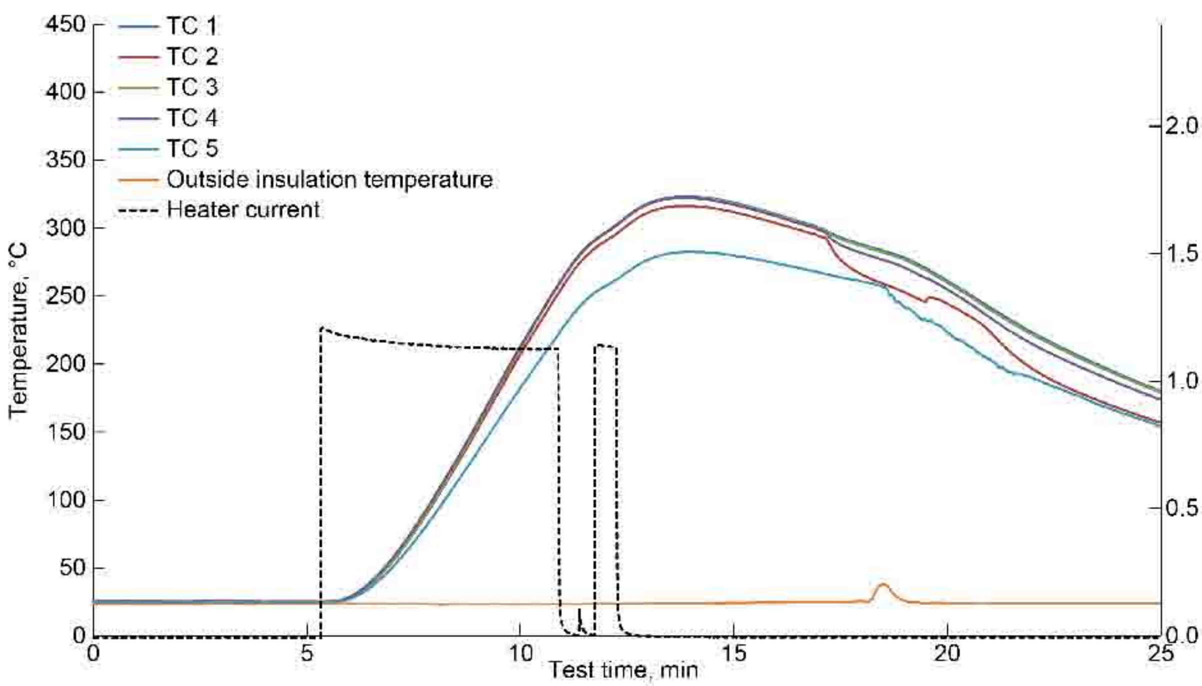

Figure A.16.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140903–101 tested on September 9, 2014, with hydrogen gas.

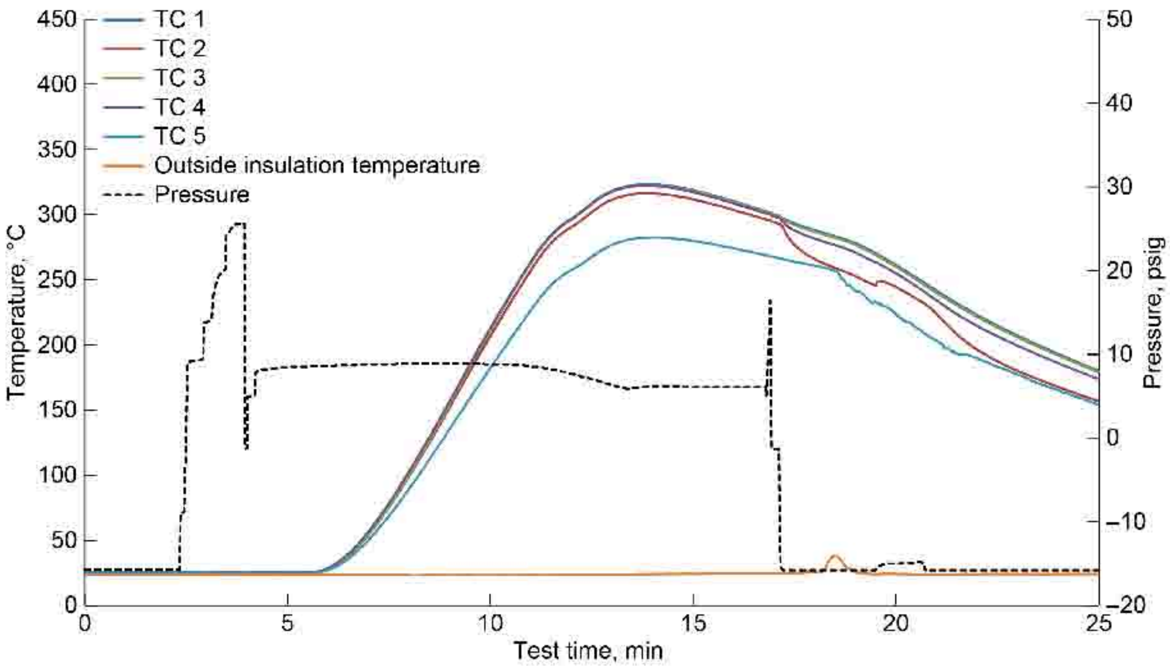

Figure A.17.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140903–101 tested on September 9, 2014, with hydrogen gas.

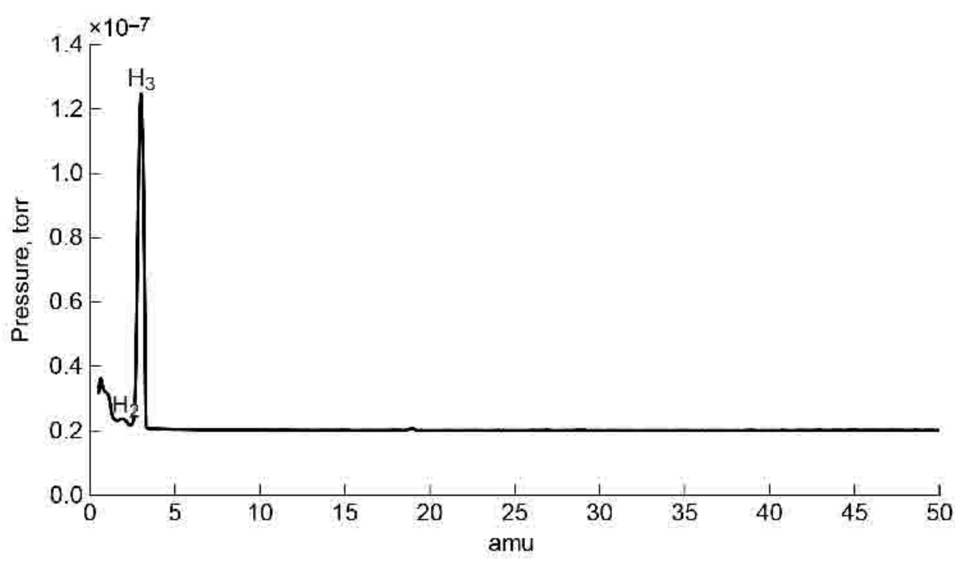

Figure A.18.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140903–101 tested on September 9, 2014, with hydrogen gas at 8 psig and 300 °C.

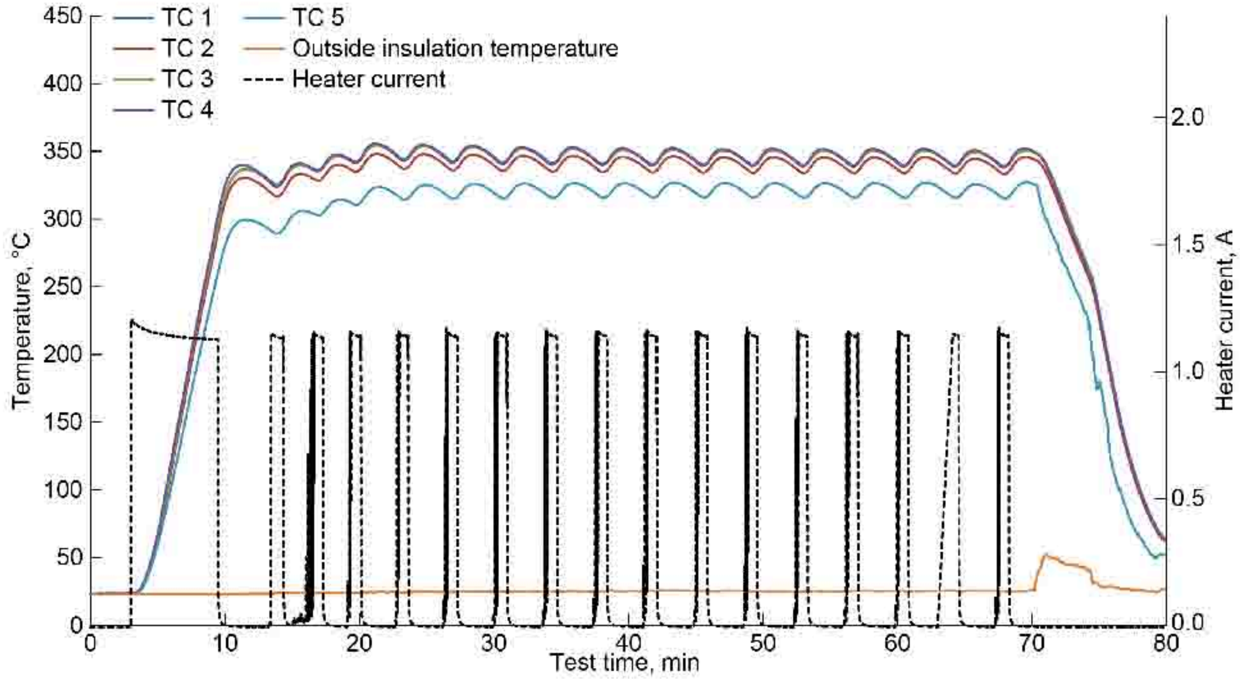

Figure A.19.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140903–101 tested on September 11, 2014, with deuterium gas.

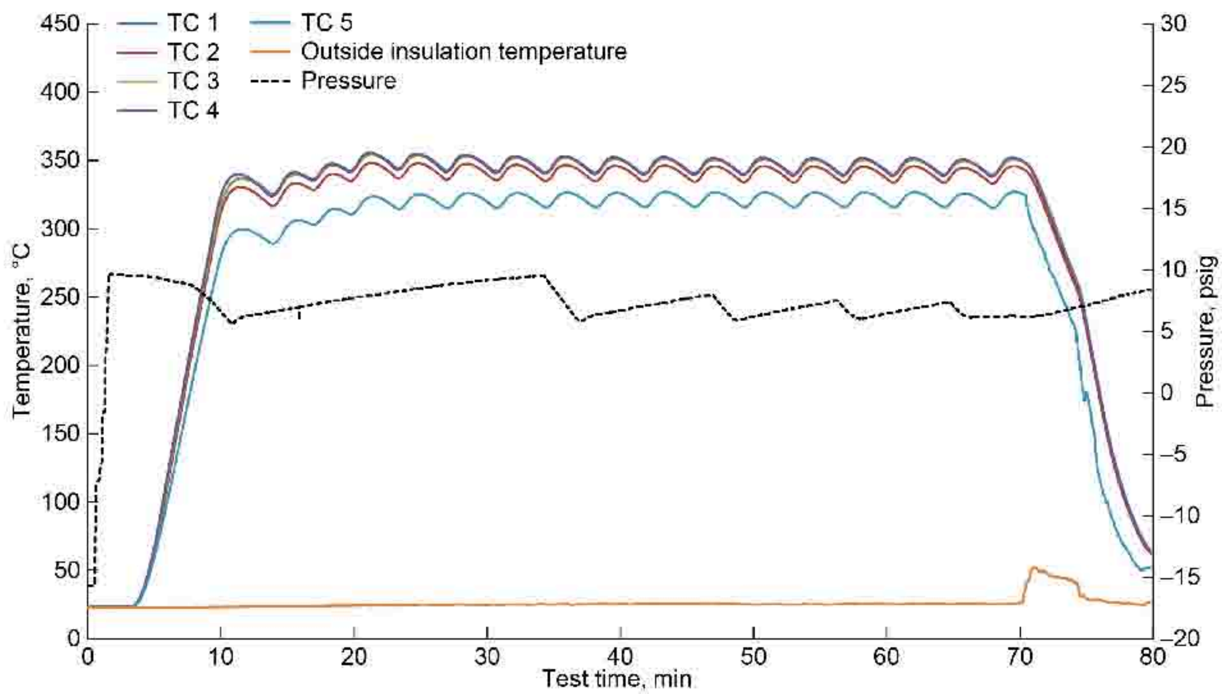

Figure A.20.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140903–101 tested on September 11, 2014, with deuterium gas.

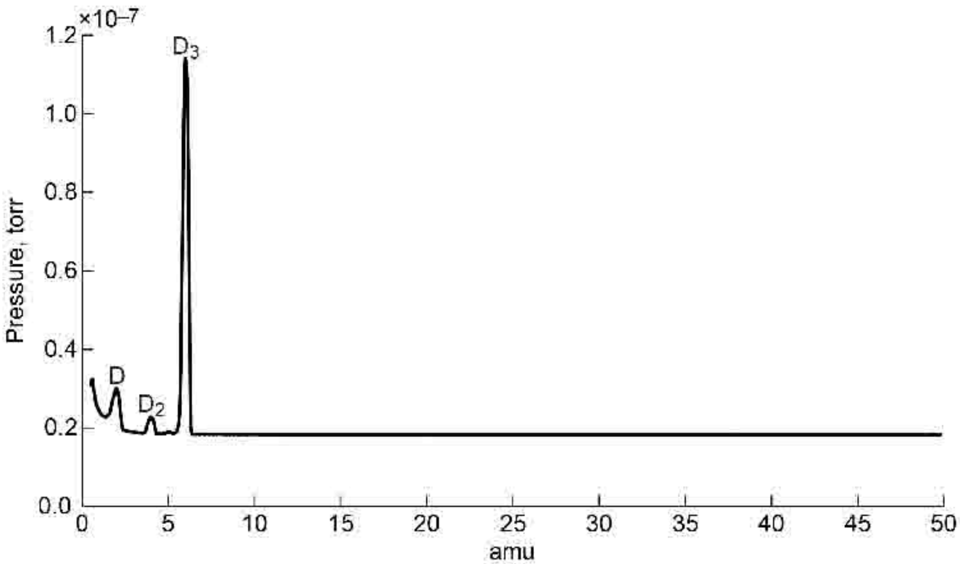

Figure A.21.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140903–101 tested on September 11, 2014, with deuterium gas at 9 psig and 345 °C.

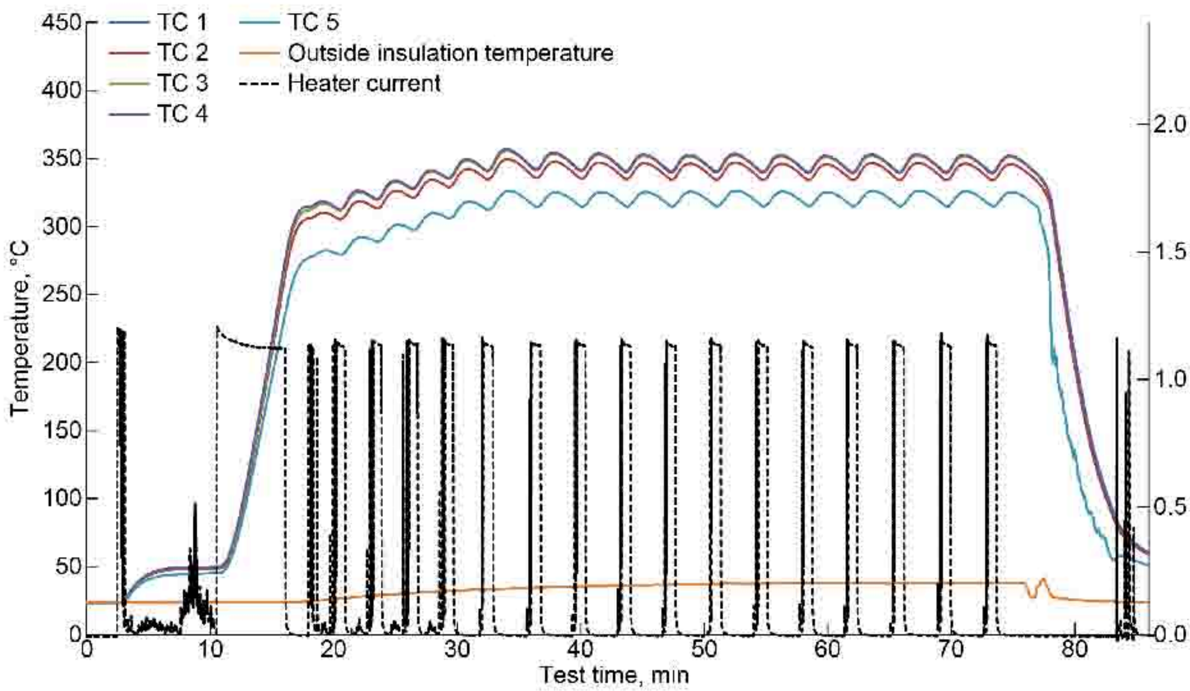

Figure A.22.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140903–101 tested on September 12, 2014, with deuterium gas.

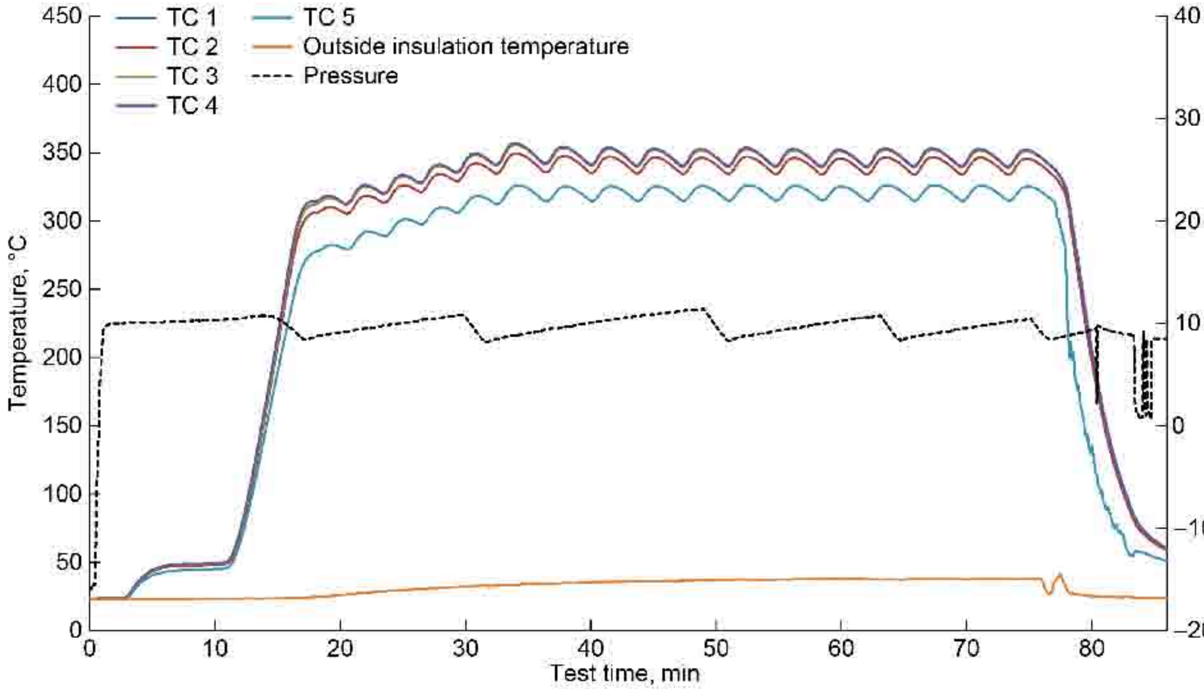

Figure A.23.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140903–101 tested on September 12, 2014, with deuterium gas.

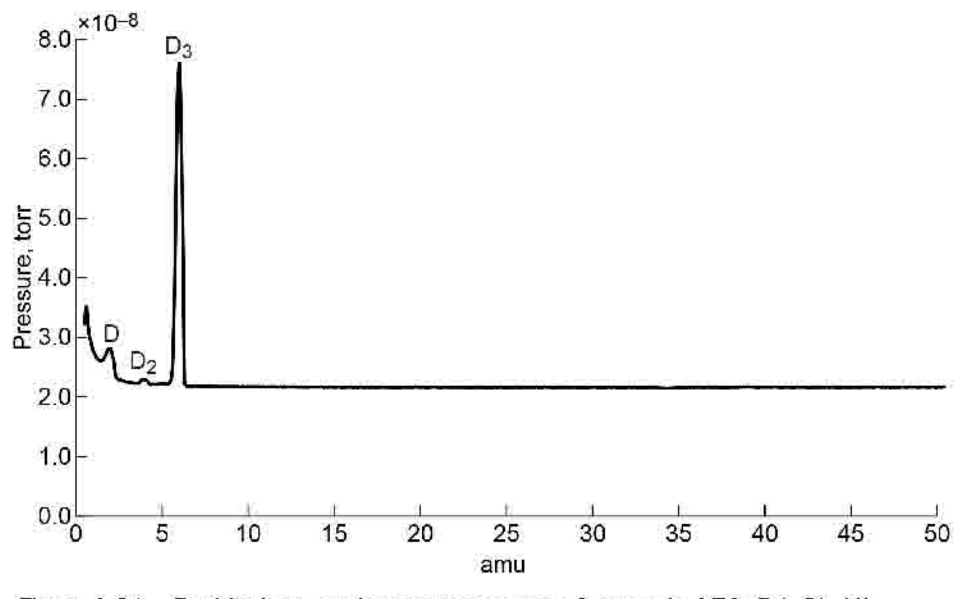

Figure A.24.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140903–101 tested on September 12, 2014, with deuterium gas at 10 psig and 340 °C.

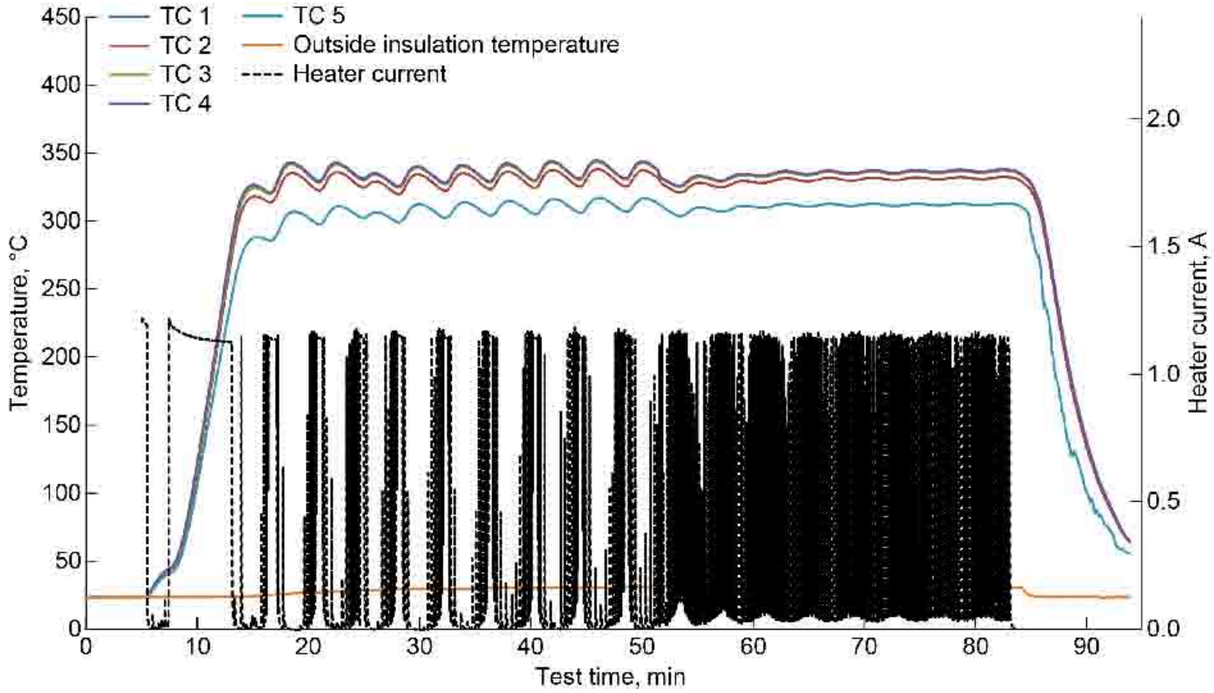

Figure A.25.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140903–101 tested on September 15, 2014, with deuterium gas.

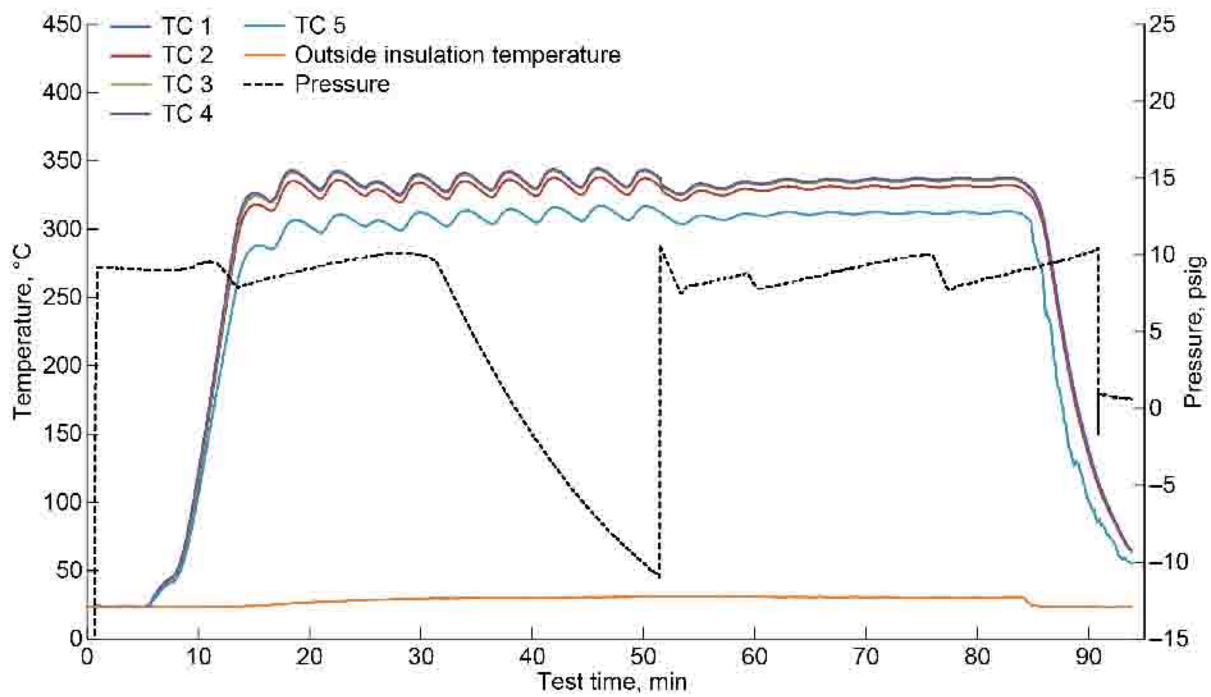

Figure A.26.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140903–101 tested on September 15, 2014, with deuterium gas.

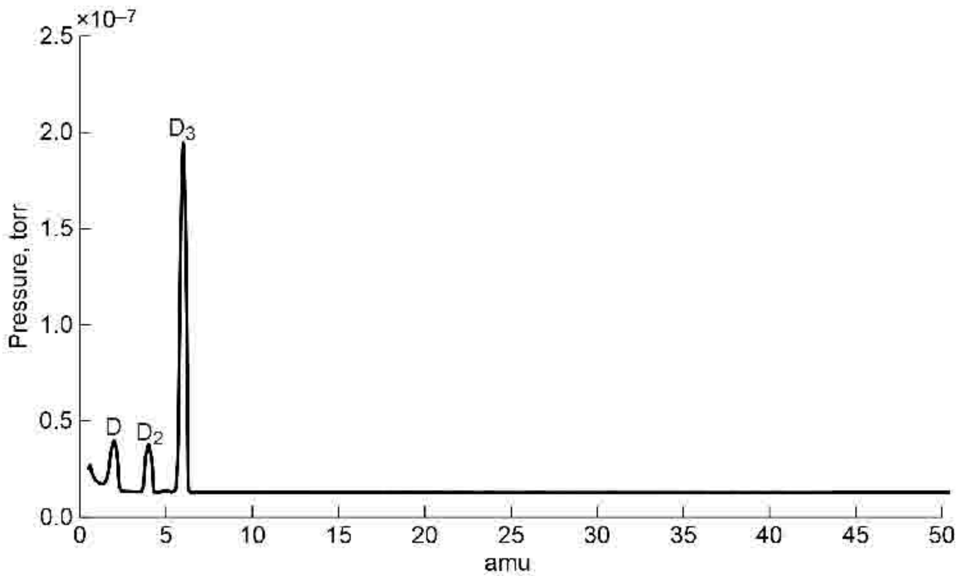

Figure A.27.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140903–101 tested on September 15, 2014, with deuterium gas at 8 psig and 340 °C.

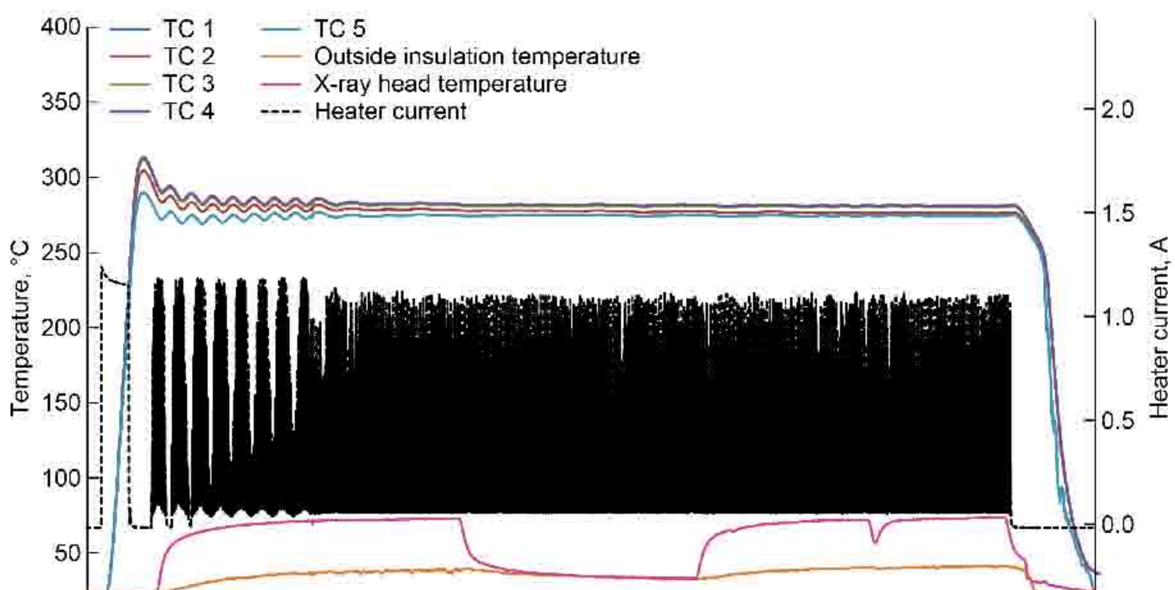

Figure A.28.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140903–102 tested on February 18, 2015, with deuterium gas.

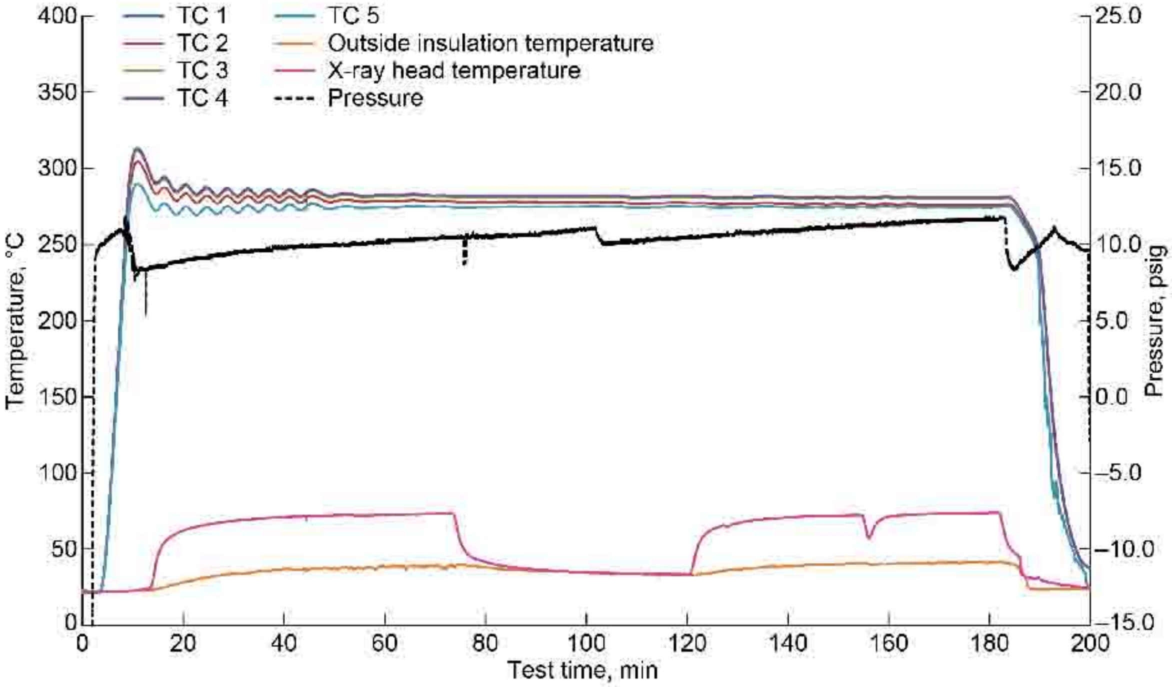

Figure A.29.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140903–102 tested on February 18, 2015, with deuterium gas.

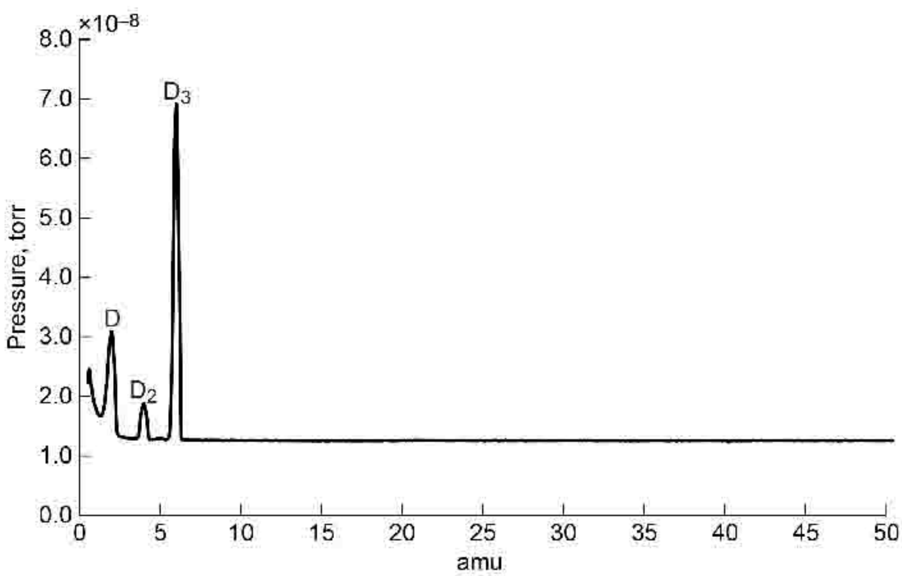

Figure A.30.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140903–102 tested on February 18, 2015, with deuterium gas at 9 psig and 285 °C with the x-ray directed at the reactor.

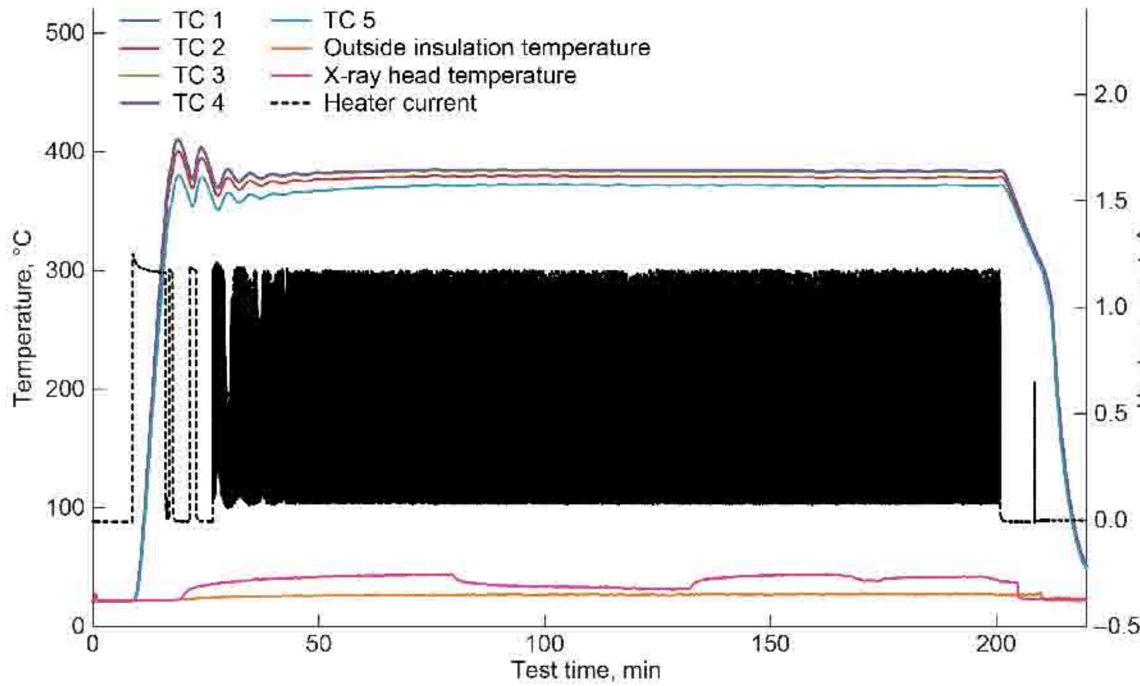

Figure A.31.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140903–103 tested on March 2, 2015, with deuterium gas.

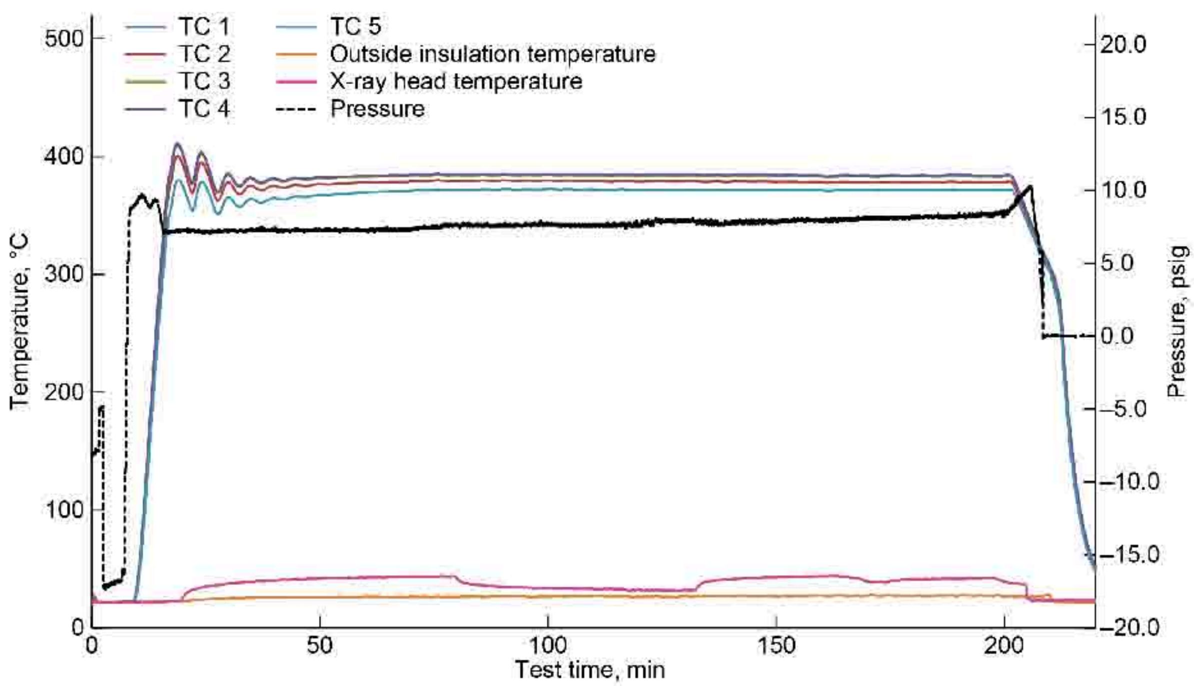

Figure A.32.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140903–103 tested on March 2, 2015, with deuterium gas.

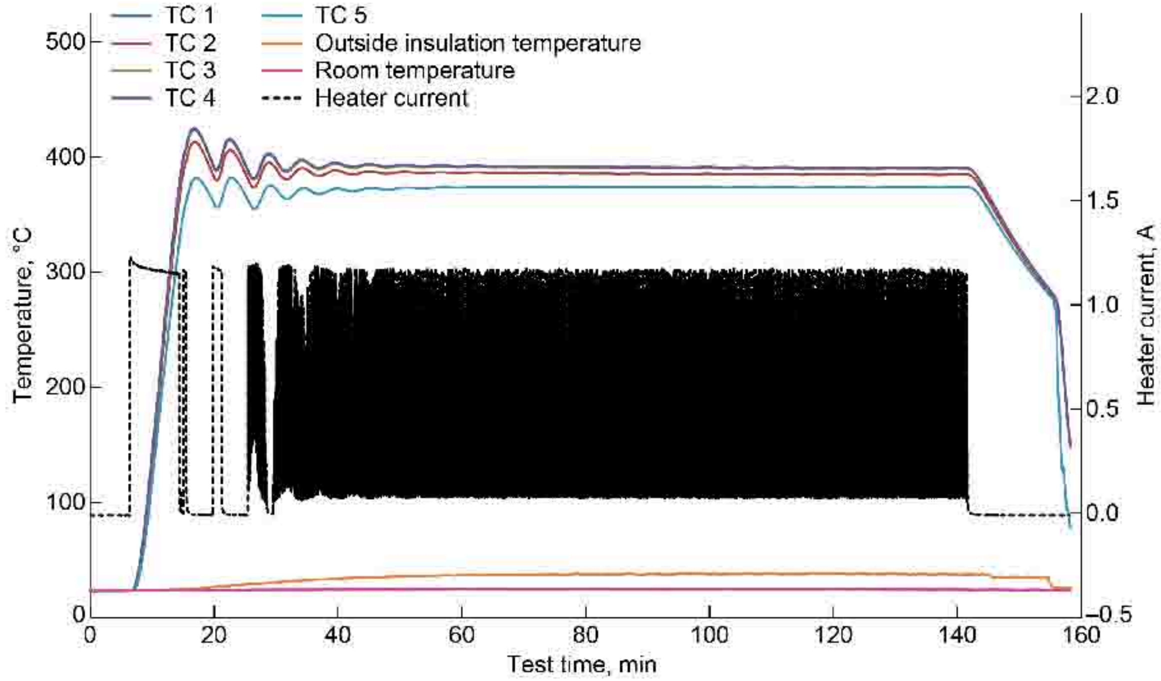

Figure A.33.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140903–104 tested on March 9, 2015, with deuterium gas.

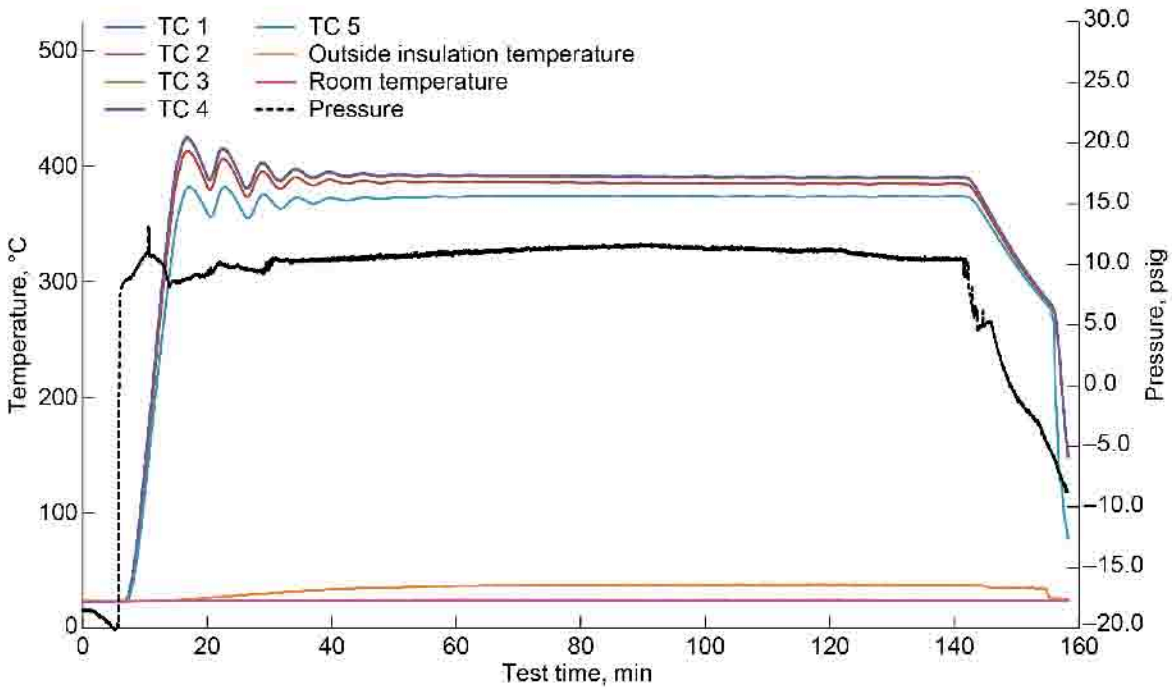

Figure A.34.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140903–104 tested on March 9, 2015, with deuterium gas.

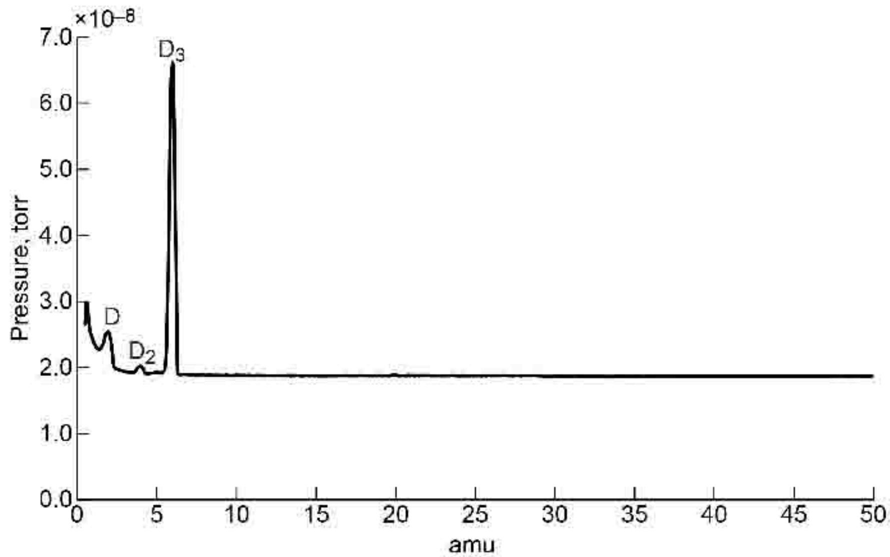

Figure A.35.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140903–104 tested on March 9, 2015, with deuterium gas at 9 psig and 400 °C.

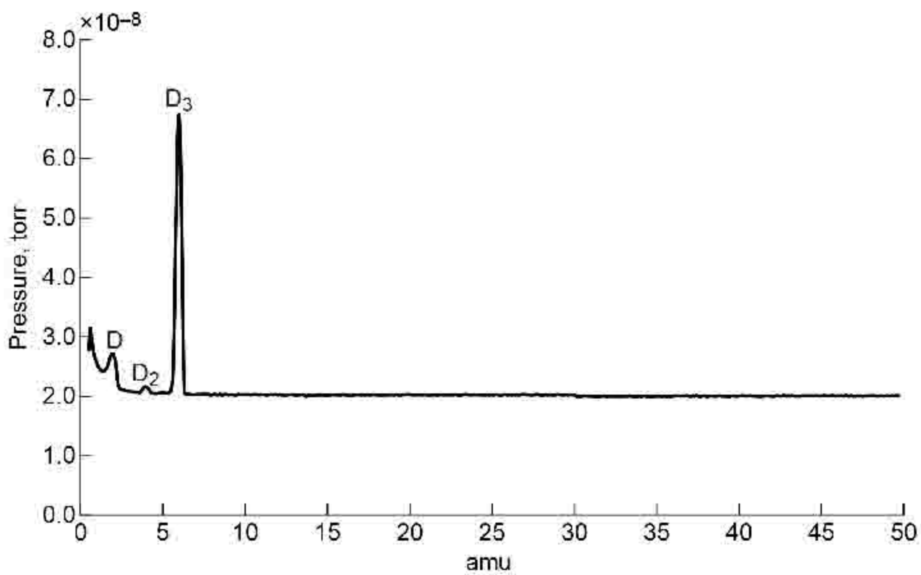

Figure A.36.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140903–104 tested on March 9, 2015, with deuterium gas at 9 psig and 390 °C with the x-ray directed at the reactor.

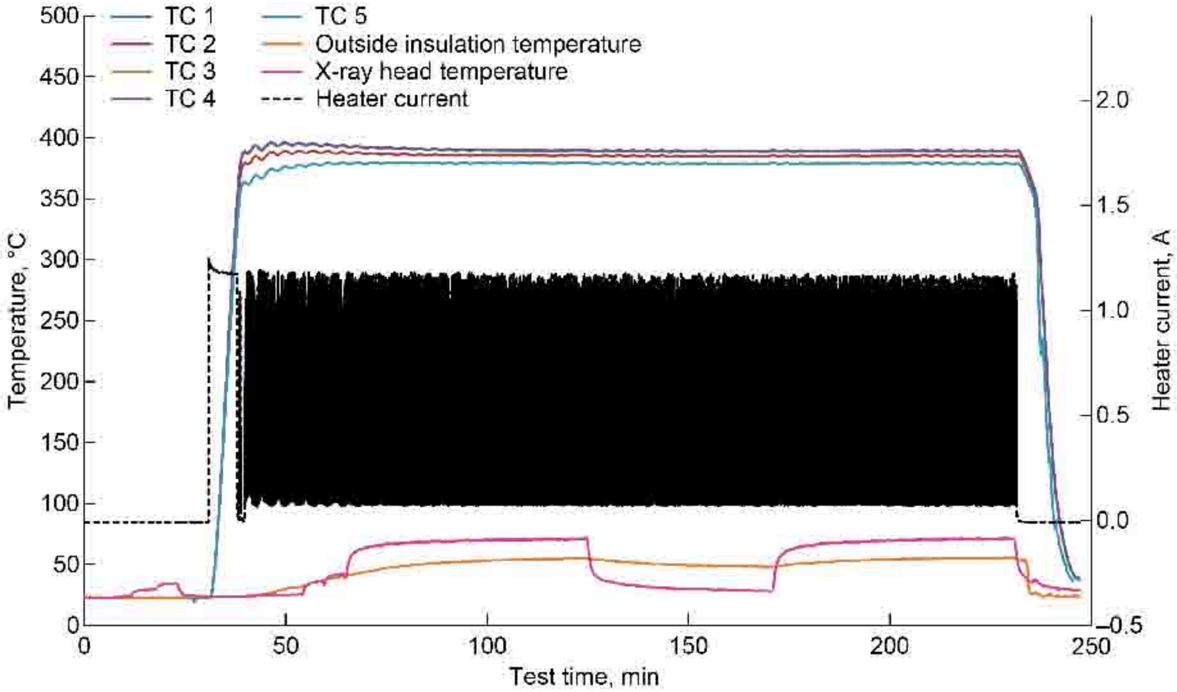

Figure A.37.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140916–106 tested on January 28, 2015, with deuterium gas.

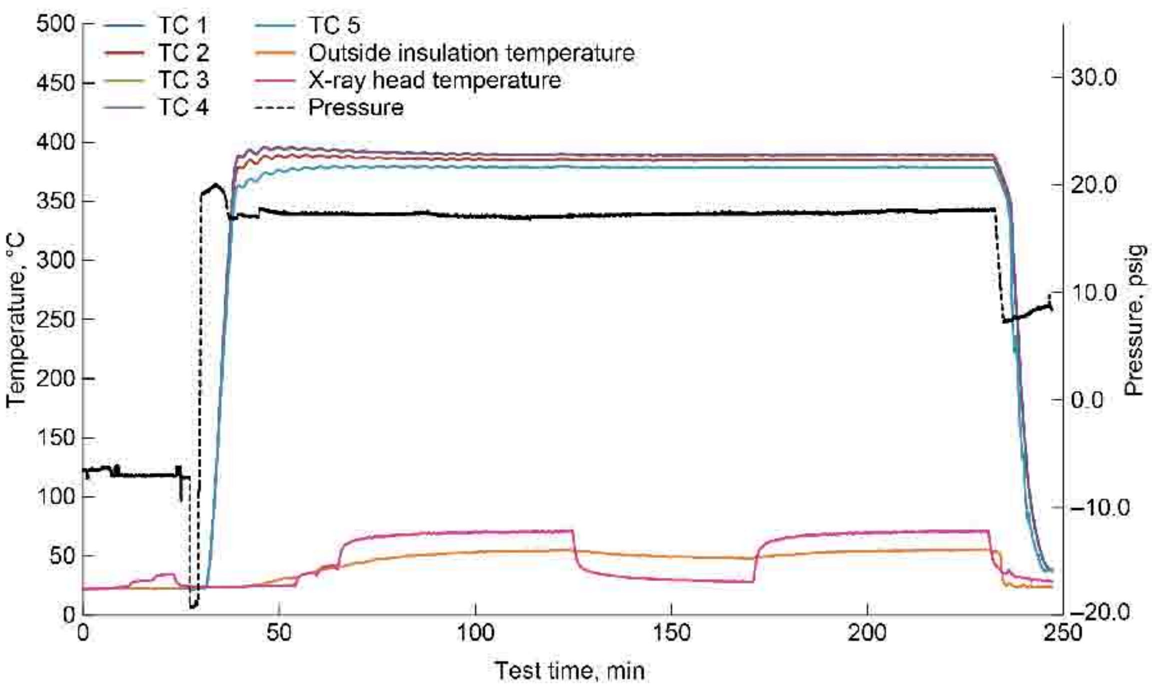

Figure A.38.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140916–106 tested on January 28, 2015, with deuterium gas.

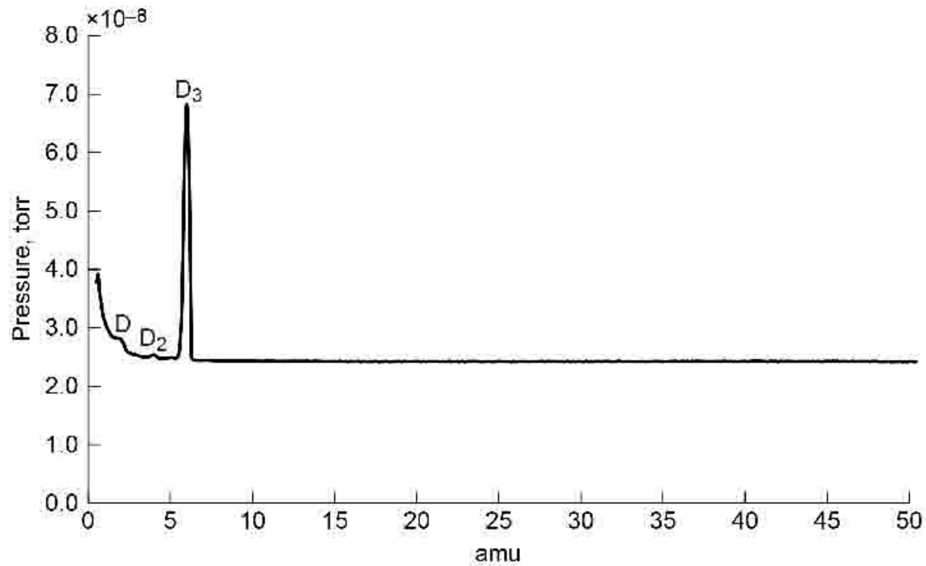

Figure A.39.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140916–106 tested on January 28, 2015, with deuterium gas at 17 psig and 390 °C.

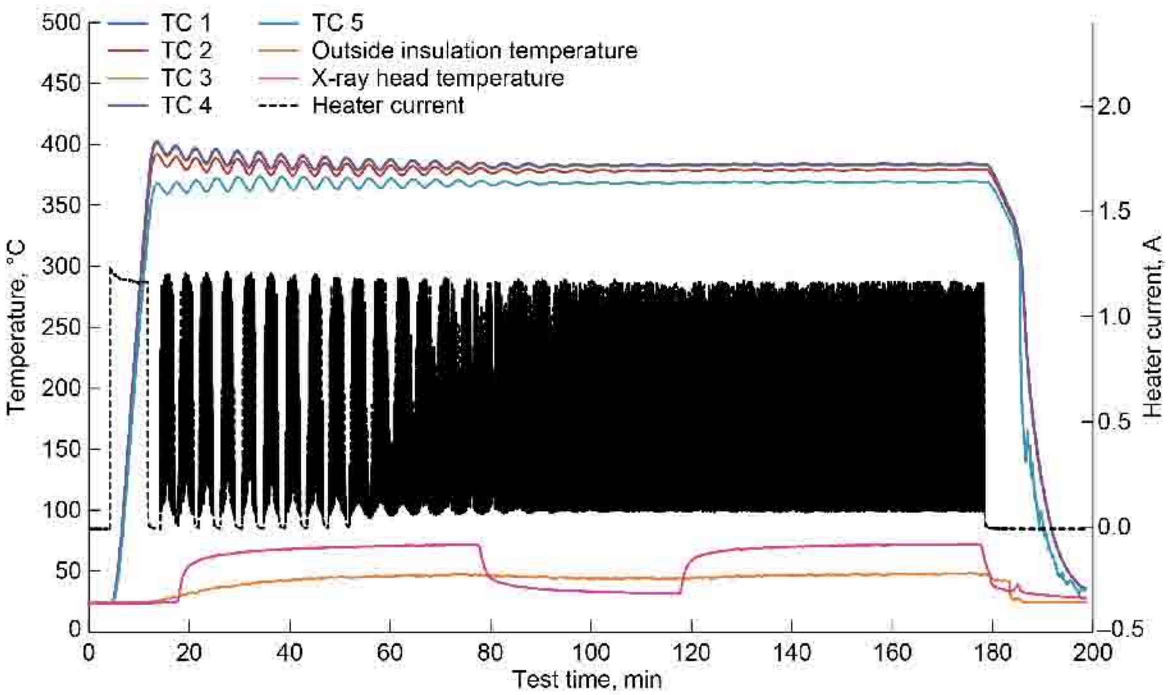

Figure A.40.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140916–107 tested on February 3, 2015, with deuterium gas.

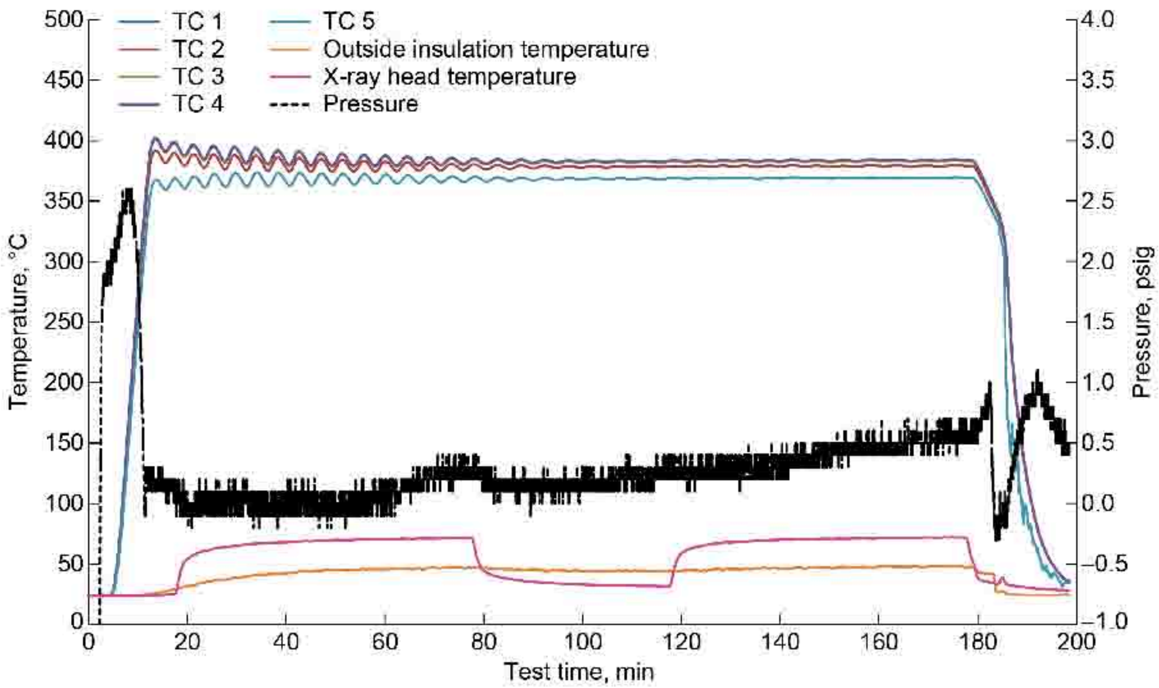

Figure A.41.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140916–107 tested on February 3, 2015, with deuterium gas.

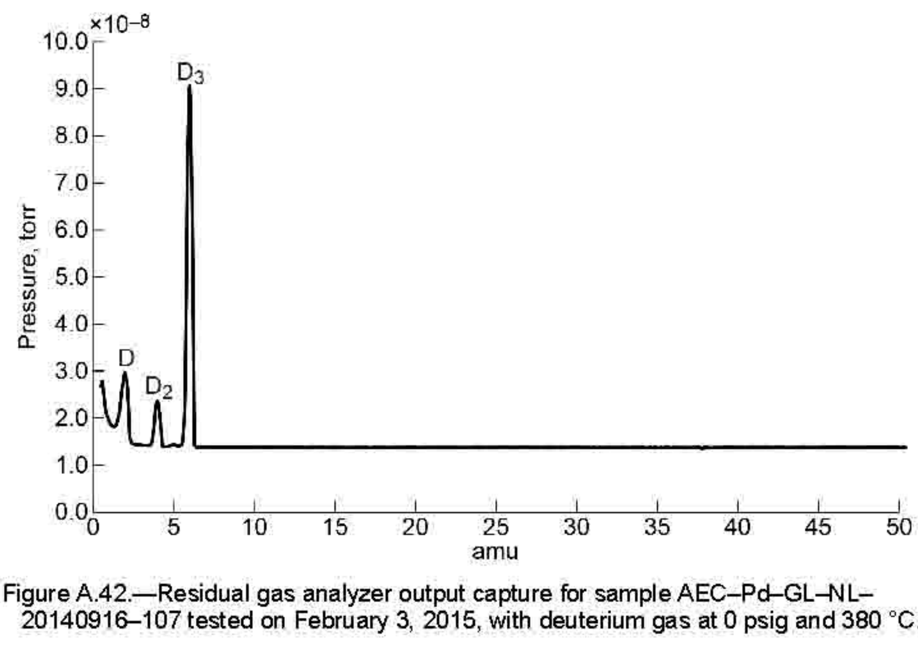

Figure A.42.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140916–107 tested on February 3, 2015, with deuterium gas at 0 psig and 380 °C.

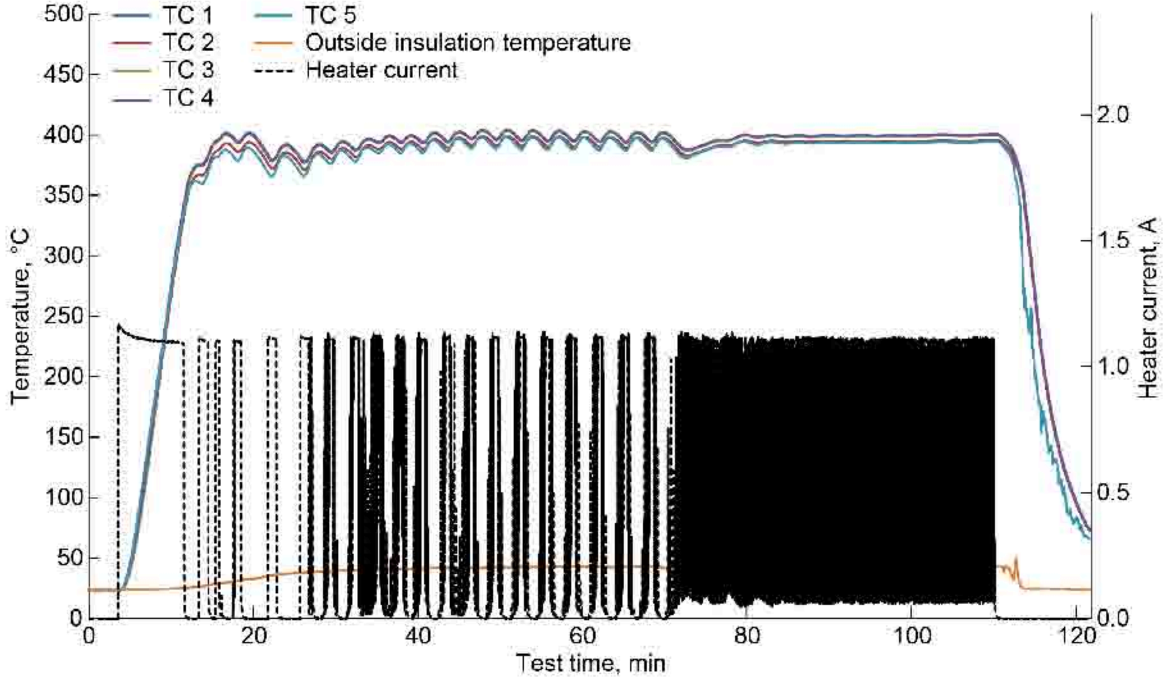

Figure A.43.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140916–111 tested on September 19, 2014, with deuterium gas.

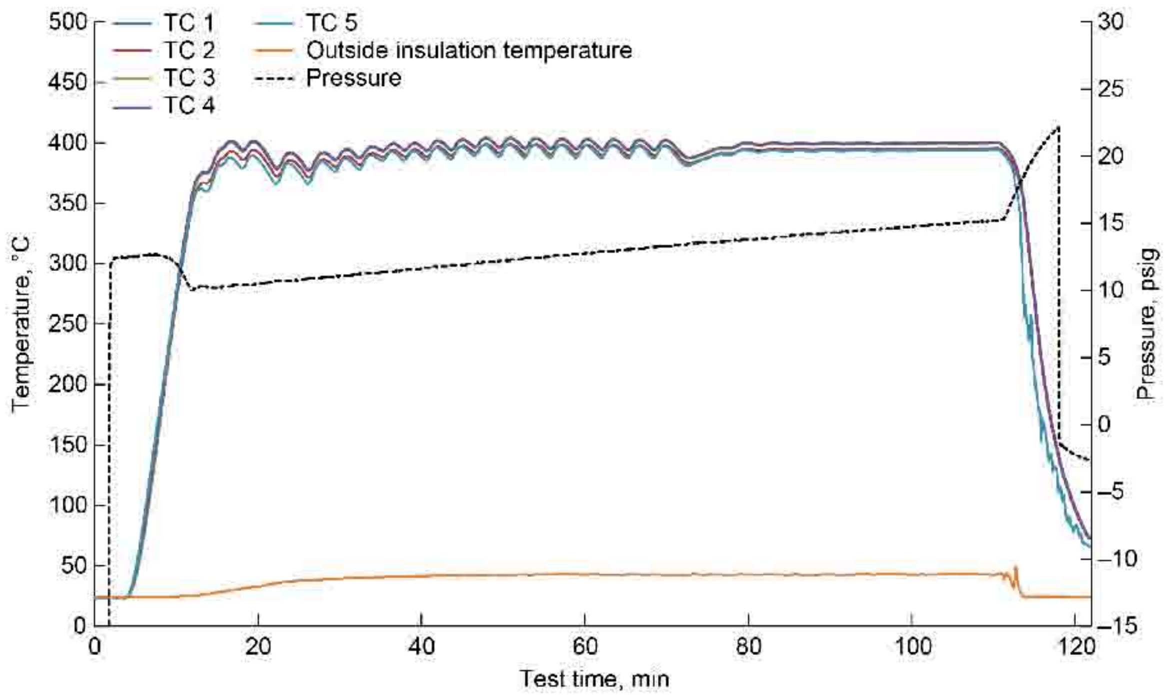

Figure A.44.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140916–111 tested on September 19, 2014, with deuterium gas.

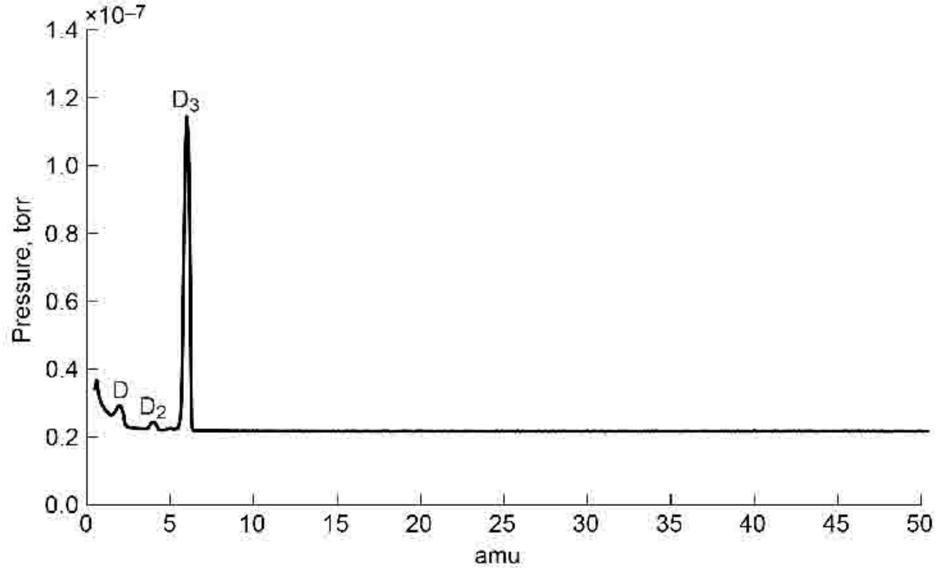

Figure A.45.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140916–111 tested on September 19, 2014, with deuterium gas at 14 psig and 400 °C.

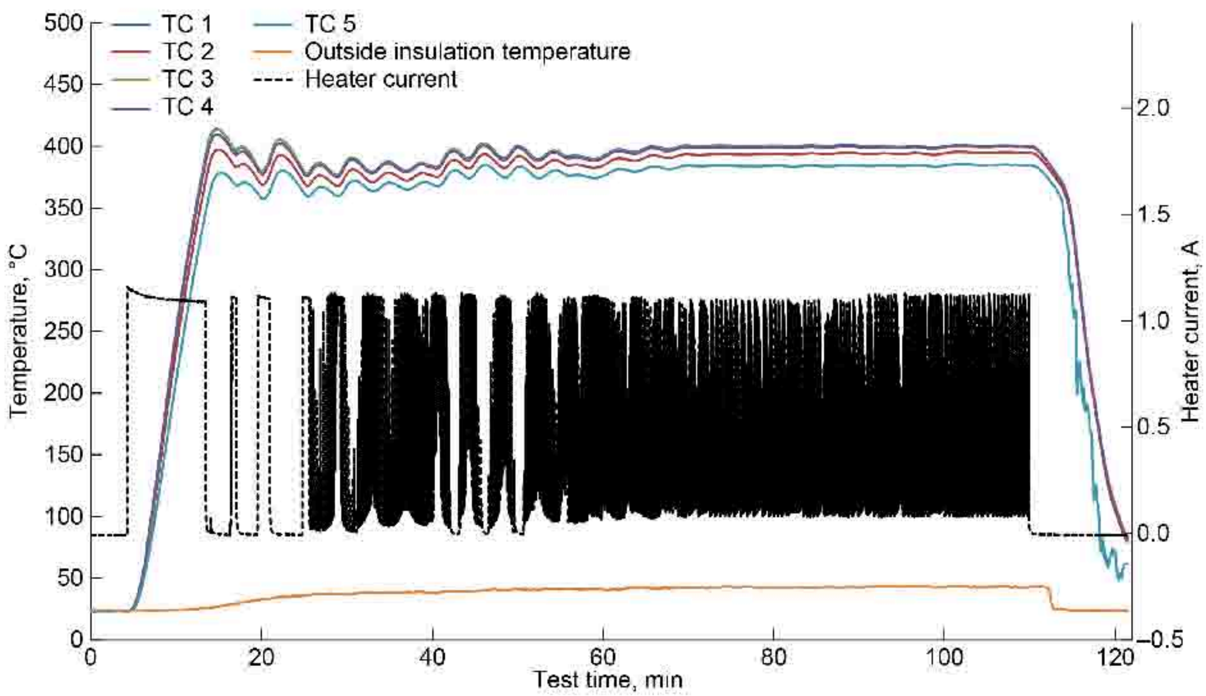

Figure A.46.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140916–112 tested on September 25, 2014, with deuterium gas.

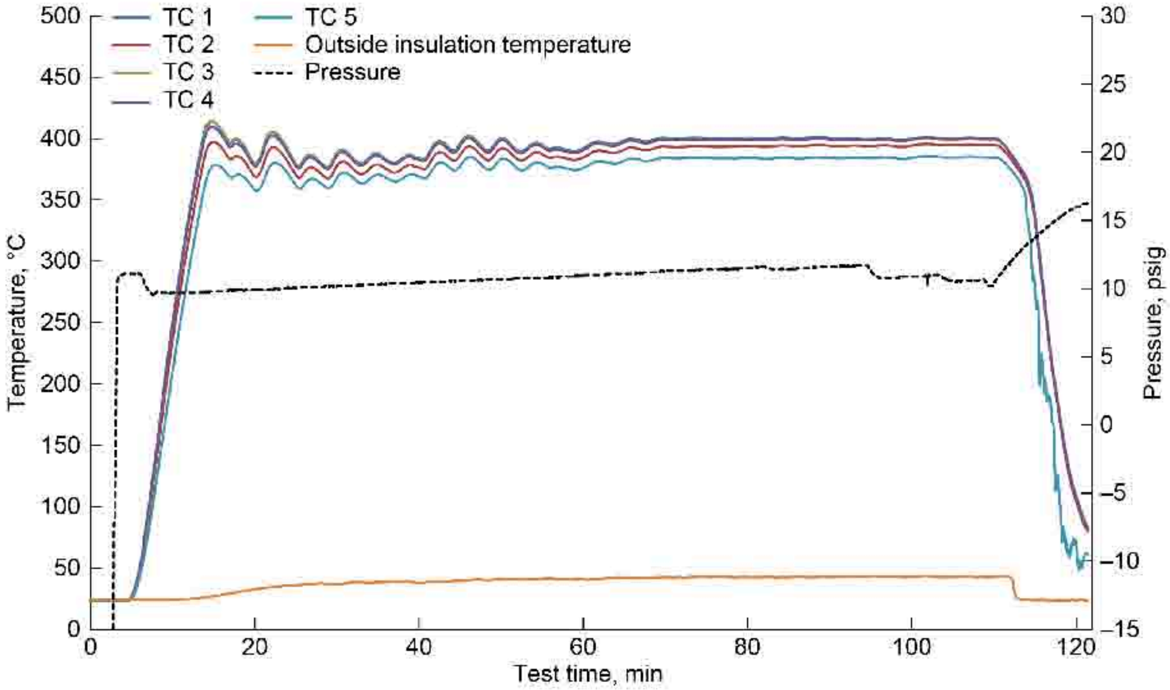

Figure A.47.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140916–112 tested on September 25, 2014, with deuterium gas.

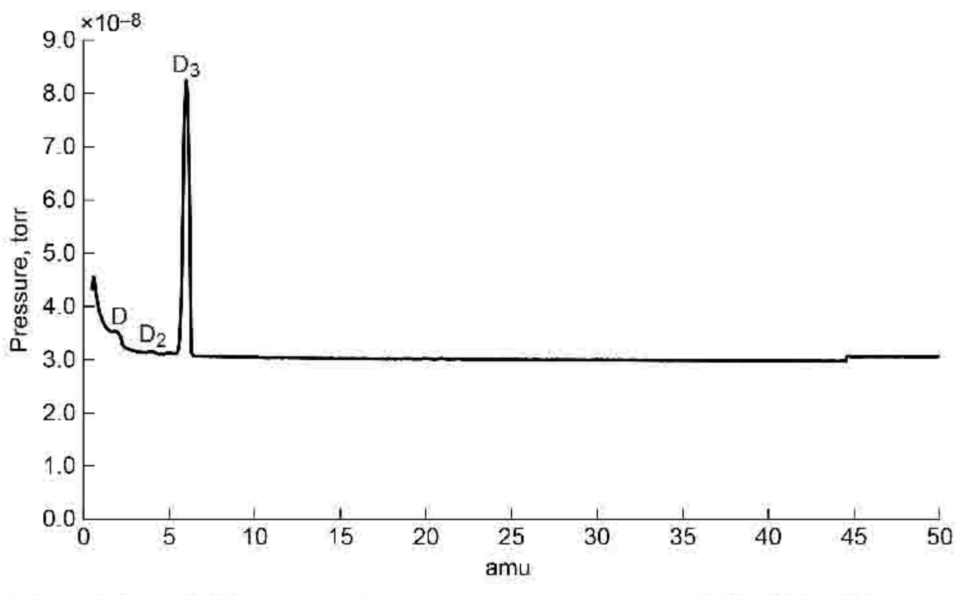

Figure A.48.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140916–112 tested on September 25, 2014, with deuterium gas at 12 psig and 400 °C.

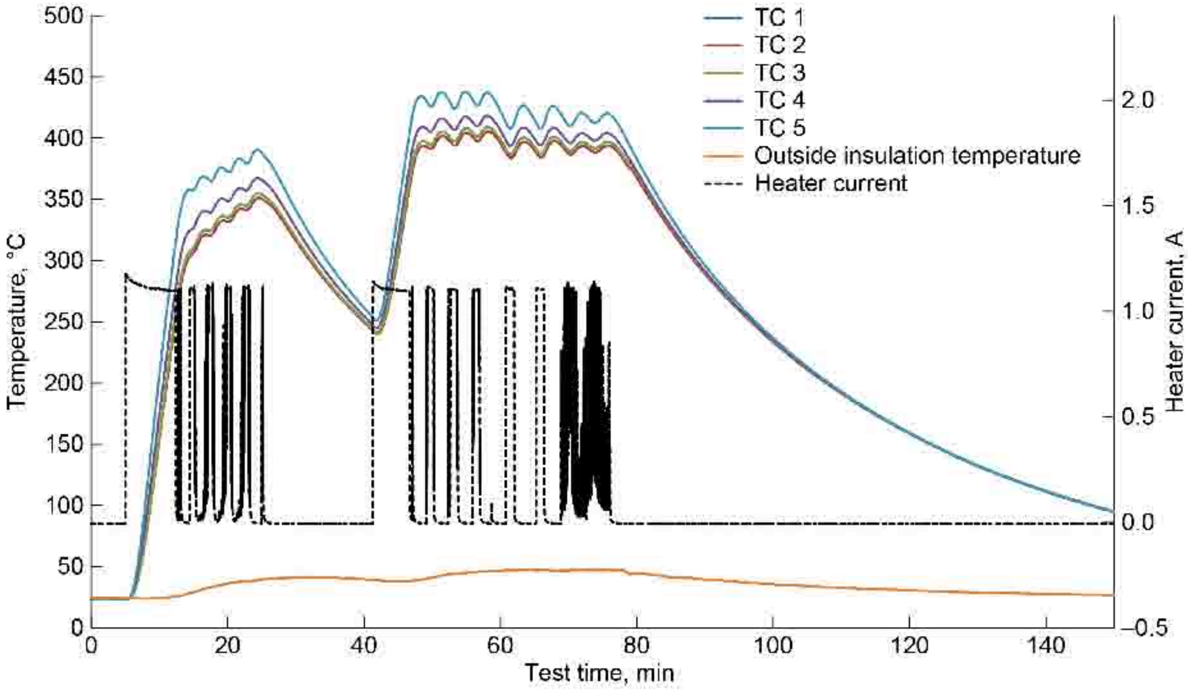

Figure A.49.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140916–113 tested on October 1, 2014, with nitrogen gas.

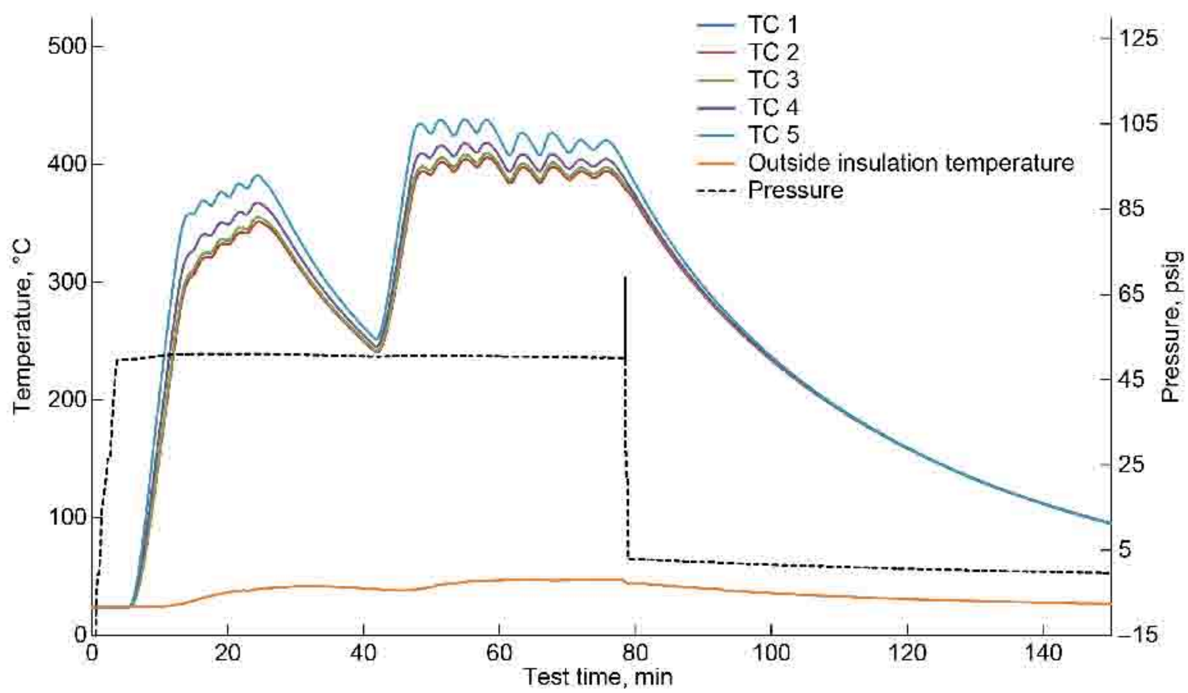

Figure A.50.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140916–113 tested on October 1, 2014, with nitrogen gas.

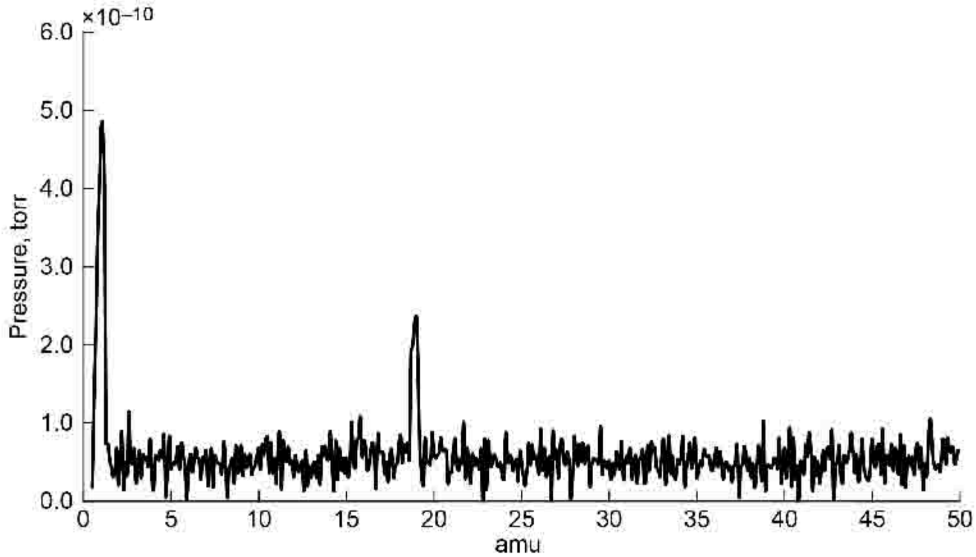

Figure A.51.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140916–113 tested on October 1, 2014, with nitrogen gas at 50 psig and 390 °C. No identifiable peaks.

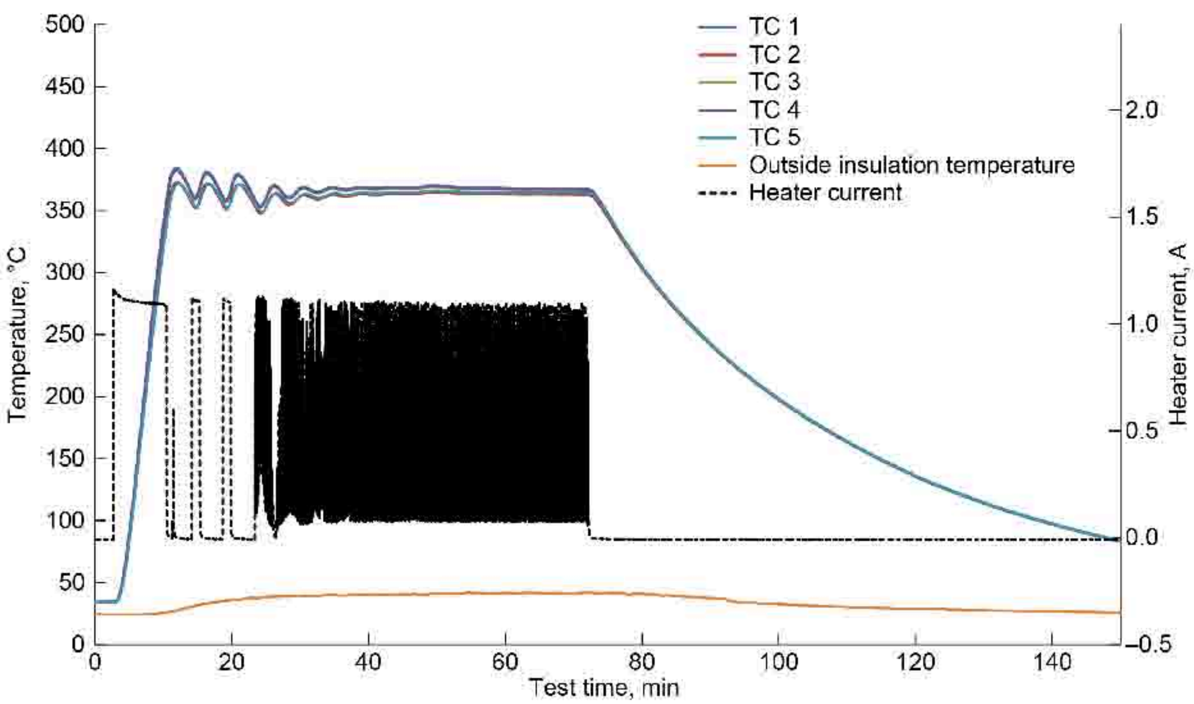

Figure A.52.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140916–113 tested on October 2, 2014, with deuterium gas.

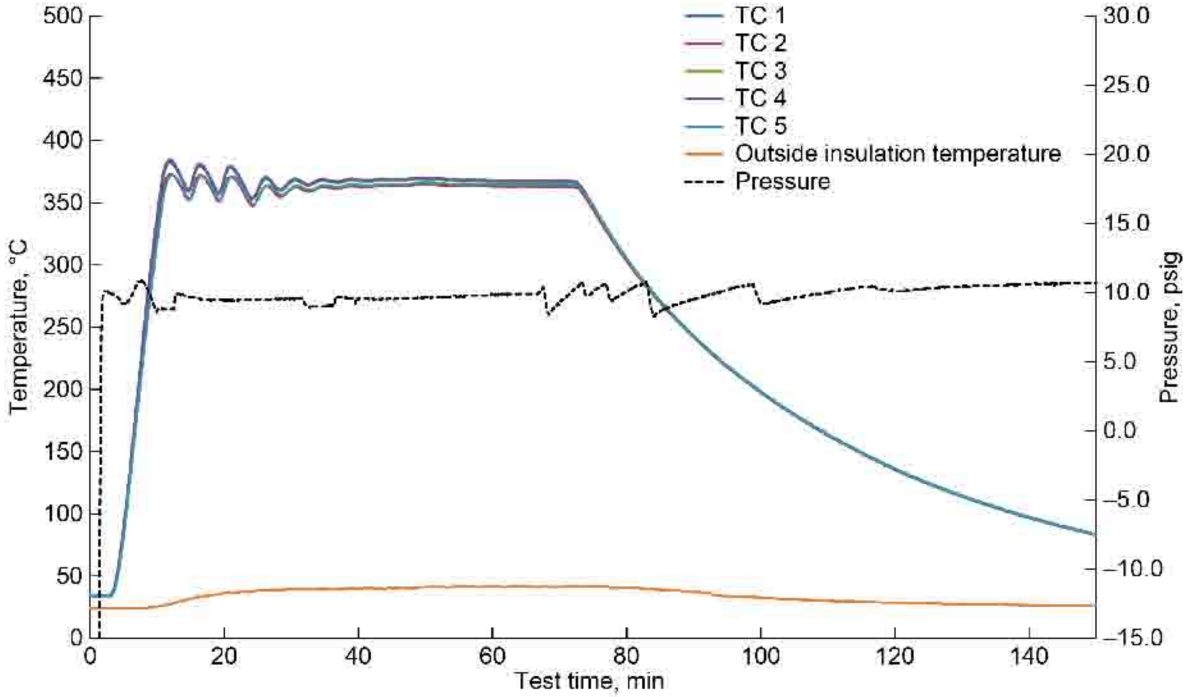

Figure A.53.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140916–113 tested on October 2, 2014, with deuterium gas.

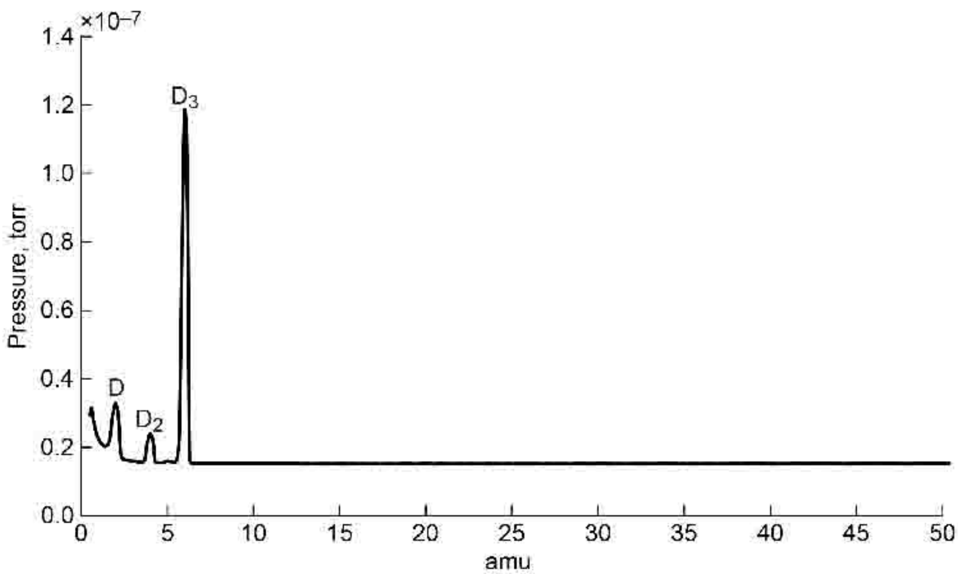

Figure A.54.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140916–113 tested on October 2, 2014, with deuterium gas at 9 psig and 380 °C.

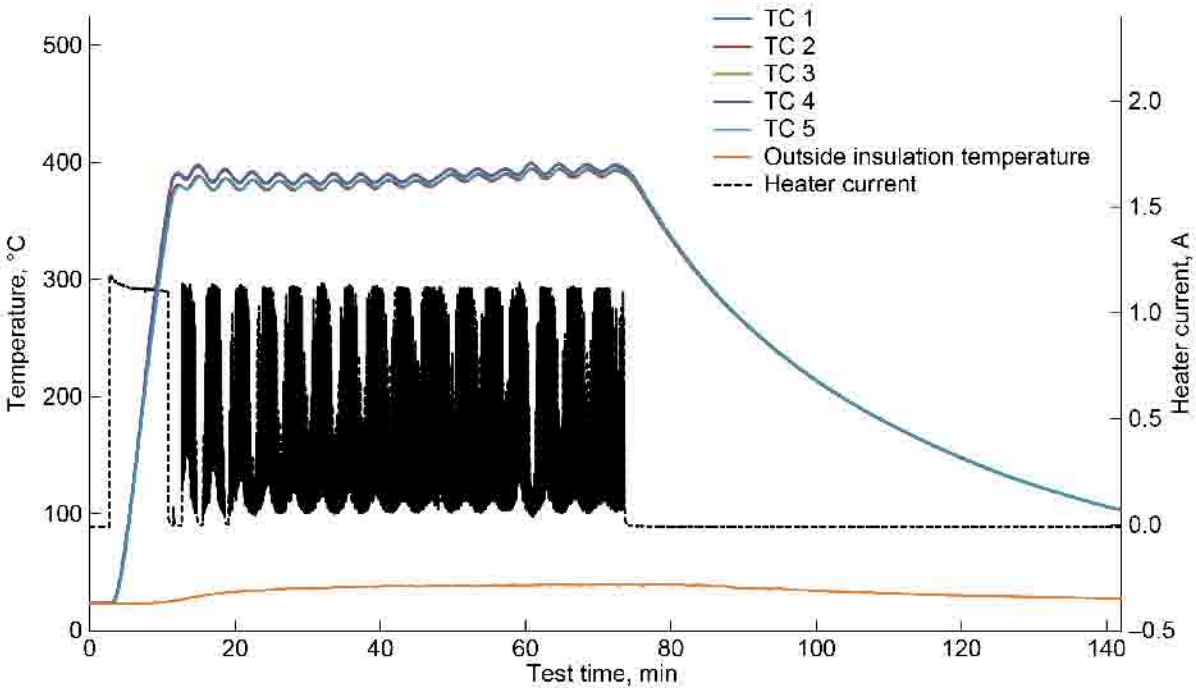

Figure A.55.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140916–113 tested on October 7, 2014, with deuterium gas.

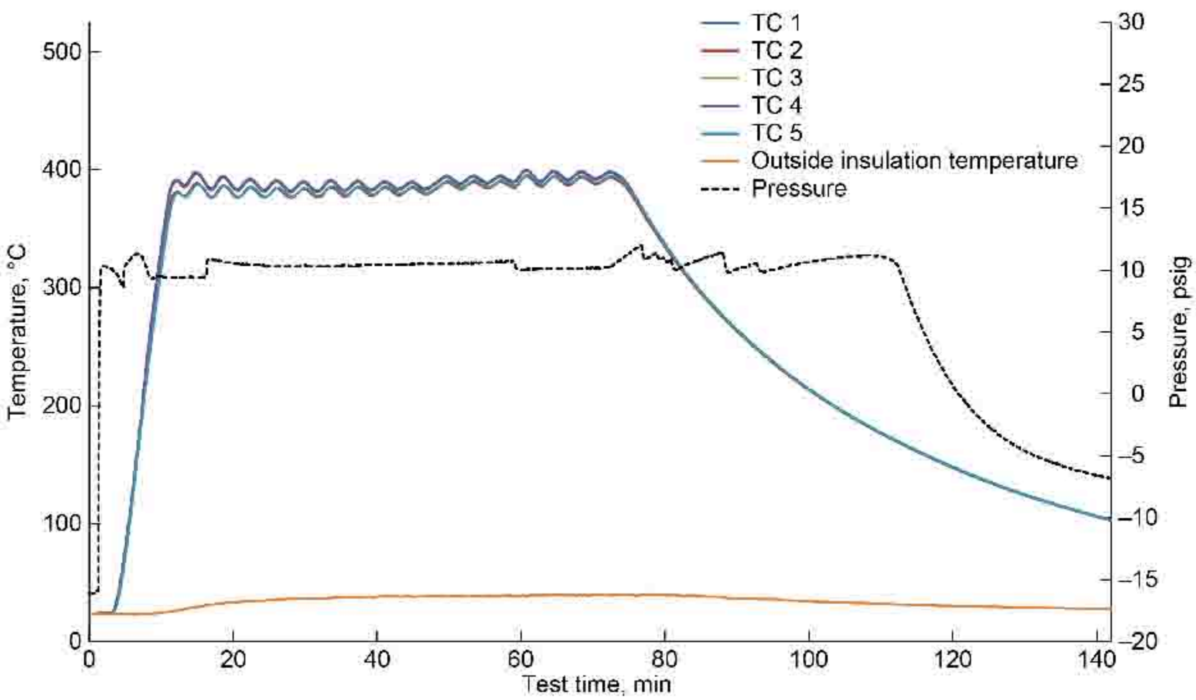

Figure A.56.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140916–113 tested on October 7, 2014, with deuterium gas.

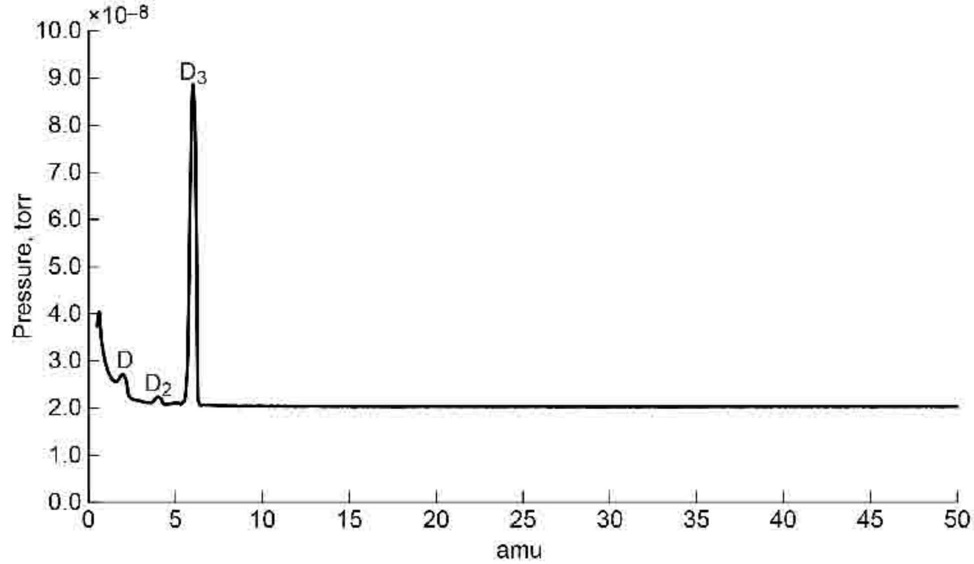

Figure A.57.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140916–113 tested on October 7, 2014, with deuterium gas at 10 psig and 385 °C.

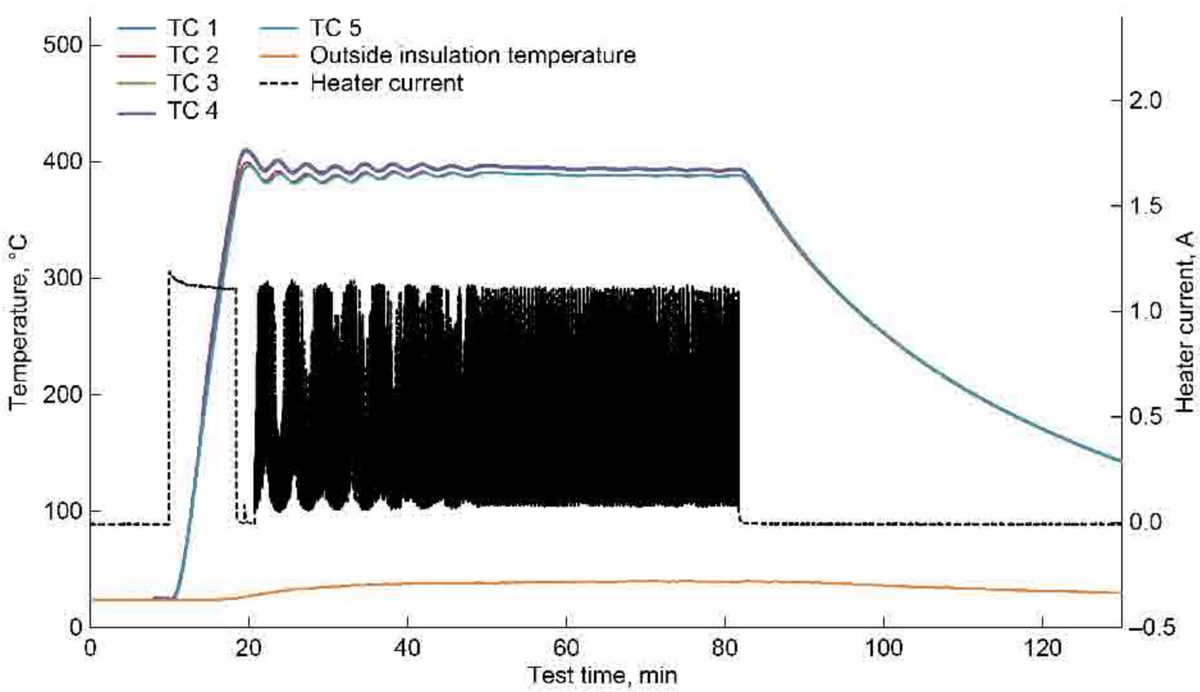

Figure A.58.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140916–113 tested on October 8, 2014, with deuterium gas.

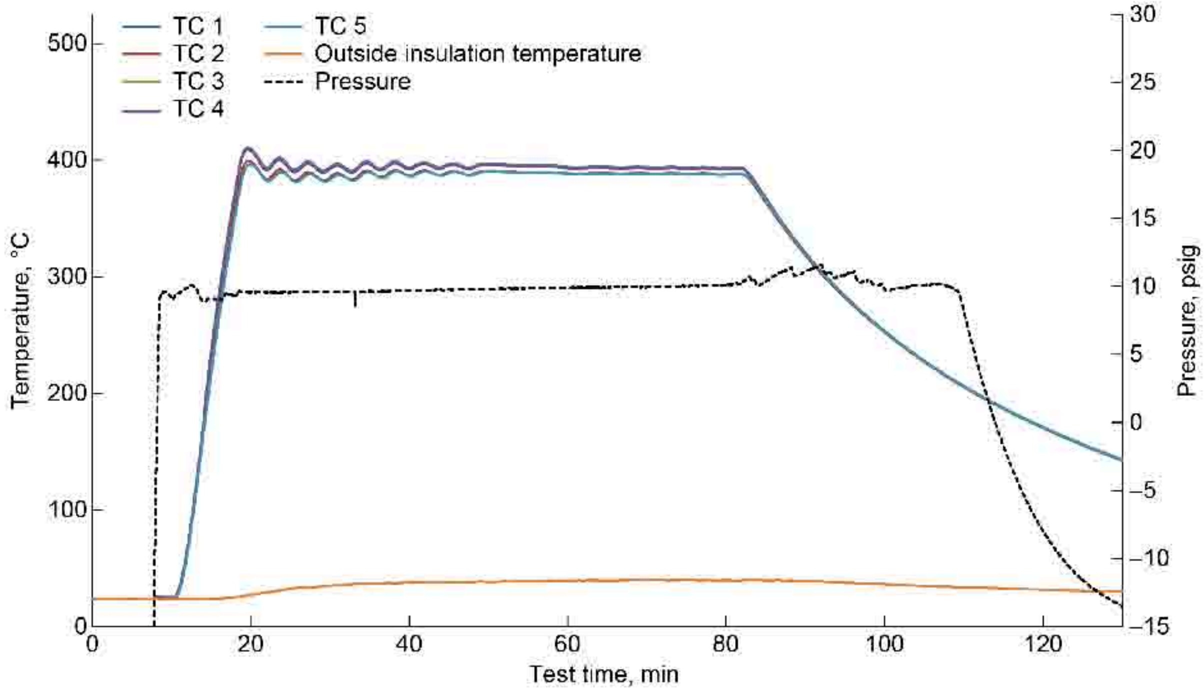

Figure A.59.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140916–113 tested on October 8, 2014, with deuterium gas.

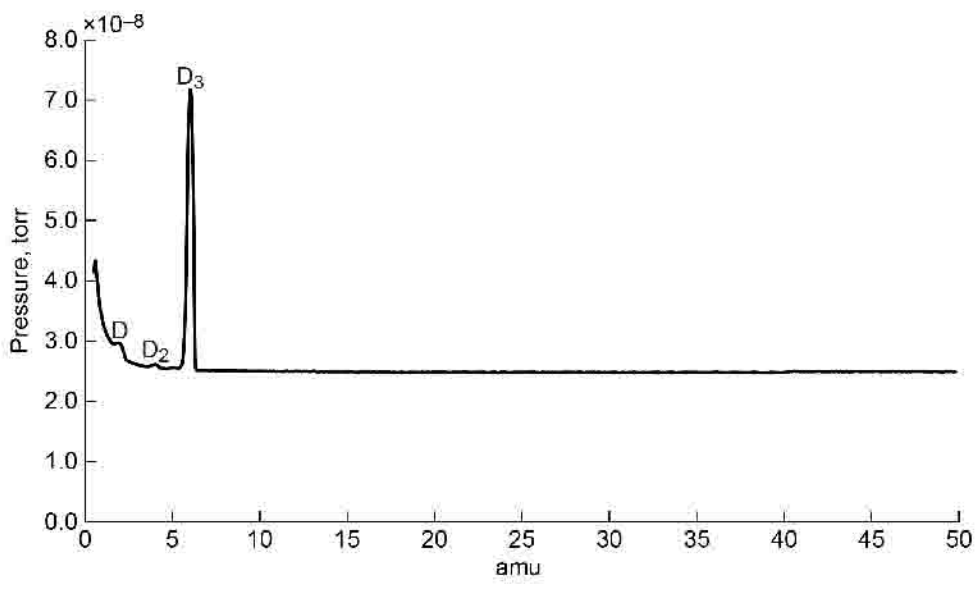

Figure A.60.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140916–113 tested on October 8, 2014, with deuterium gas at 10 psig and 403 °C.

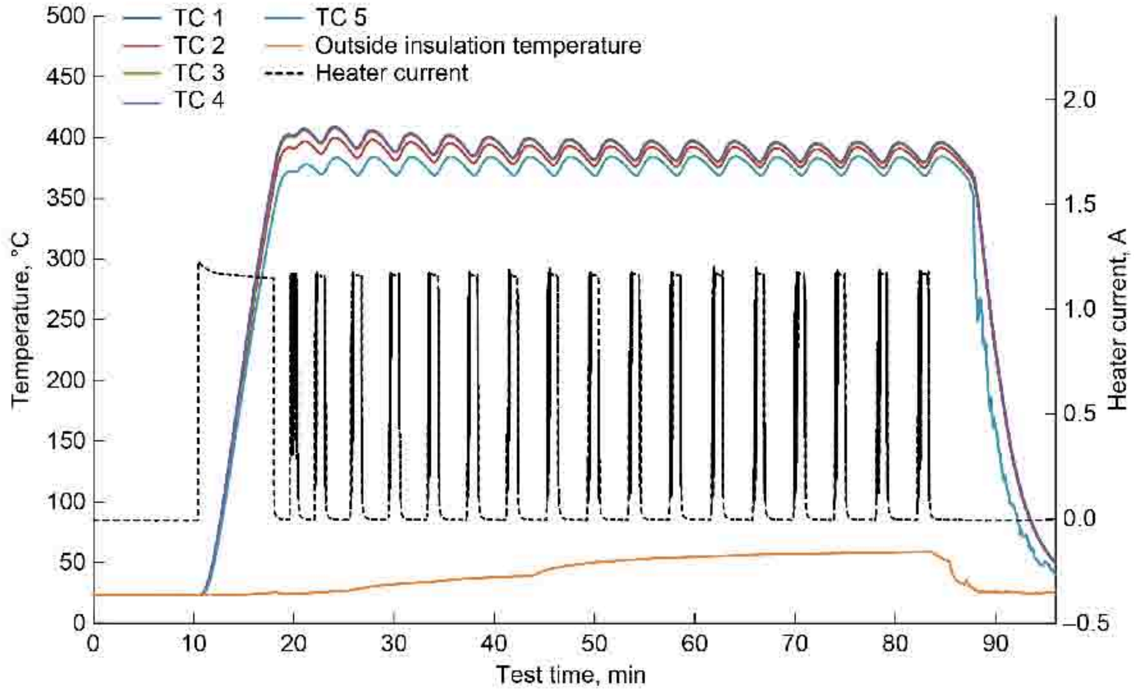

Figure A.61.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140916–116 tested on January 20, 2015, with deuterium gas.

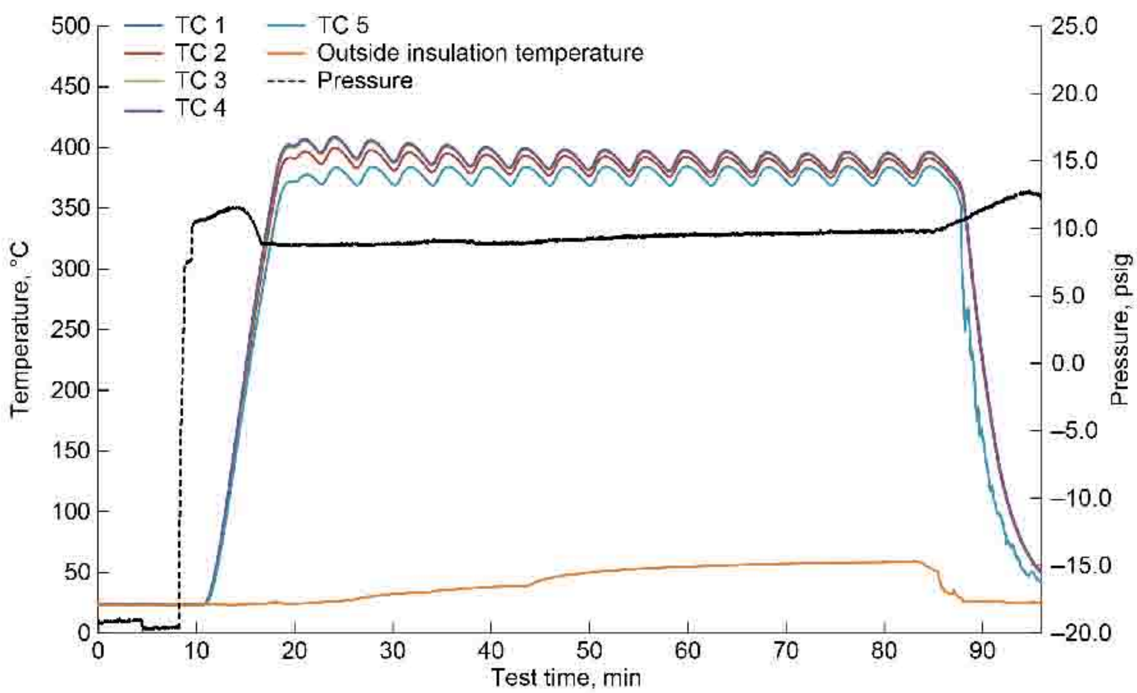

Figure A.62.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140916–116 tested on January 20, 2015, with deuterium gas.

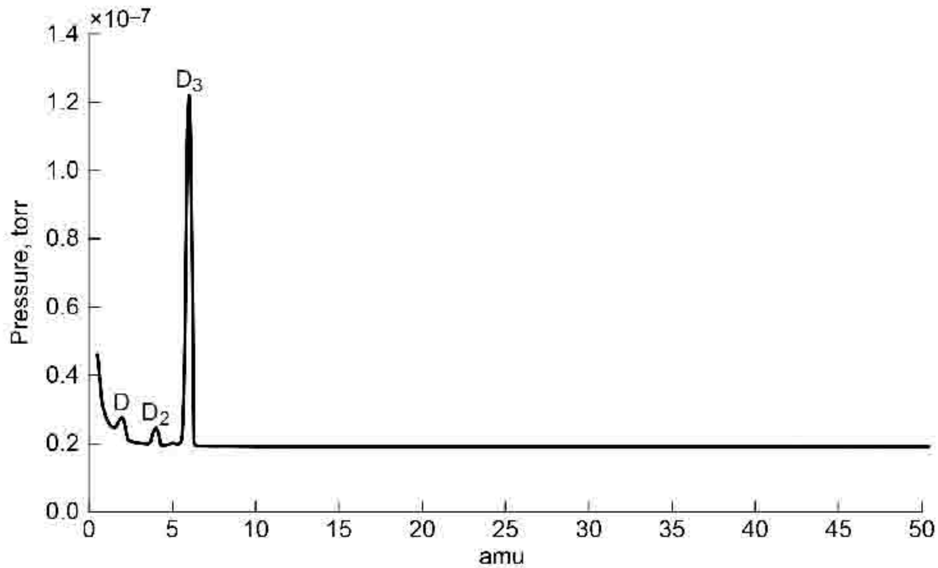

Figure A.63.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140916–116 tested on January 20, 2015, with deuterium gas at 10 psig and 400 °C.

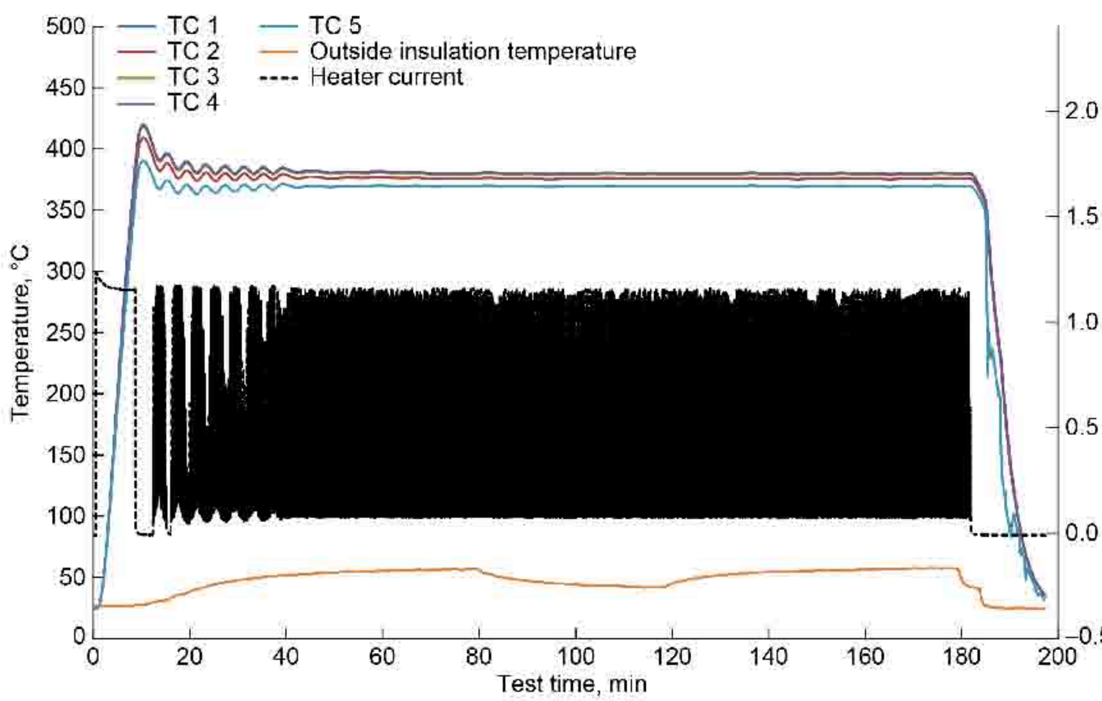

Figure A.64.—Temperature and heater current data for sample AEC–Pd–GL–NL–20140916–117 tested on January 22, 2015, with deuterium gas.

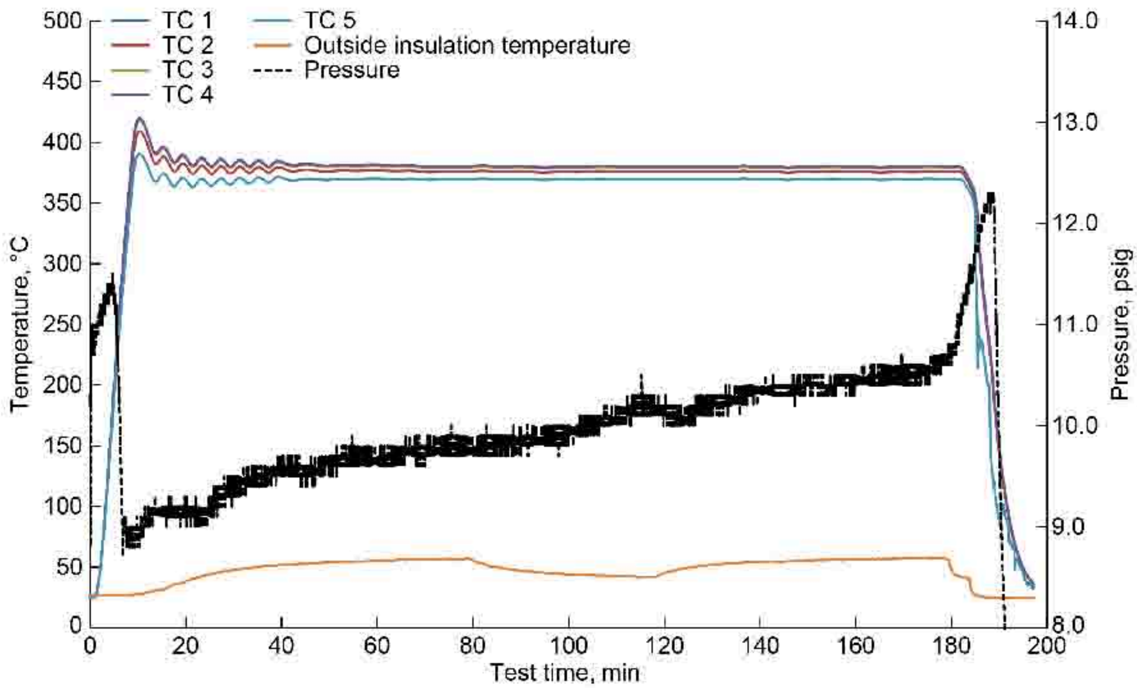

Figure A.65.—Temperature and pressure data for sample AEC–Pd–GL–NL–20140916–117 tested on January 22, 2015, with deuterium gas.

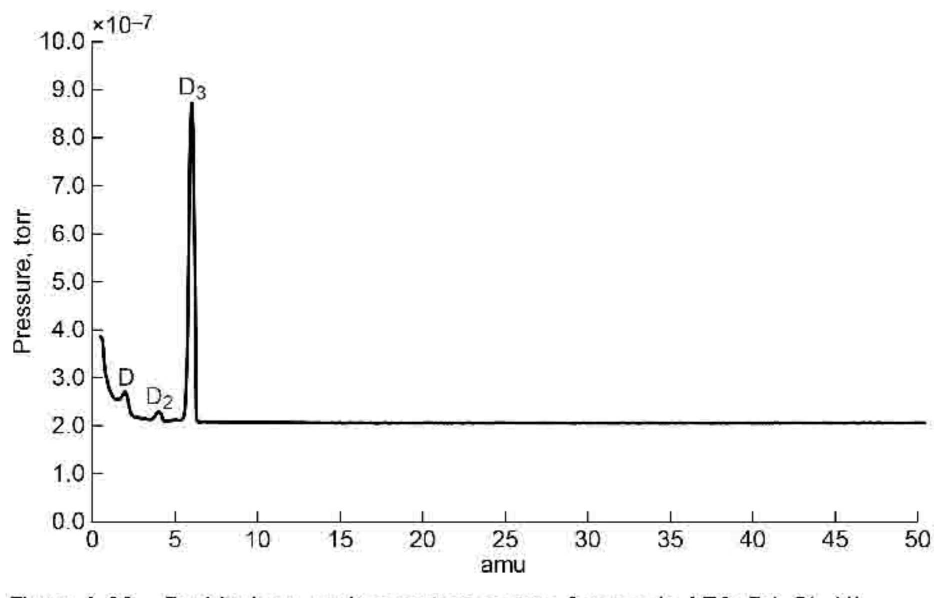

Figure A.66.—Residual gas analyzer output capture for sample AEC–Pd–GL–NL–20140916–117 tested on January 22, 2015, with deuterium gas at 9 psig and 380 °C.

Appendix B.—Formal System Schematics

The diaphragm reactor system overview and piping and instrumentation diagram are found in Figure B.1 and Figure B.2, respectively.

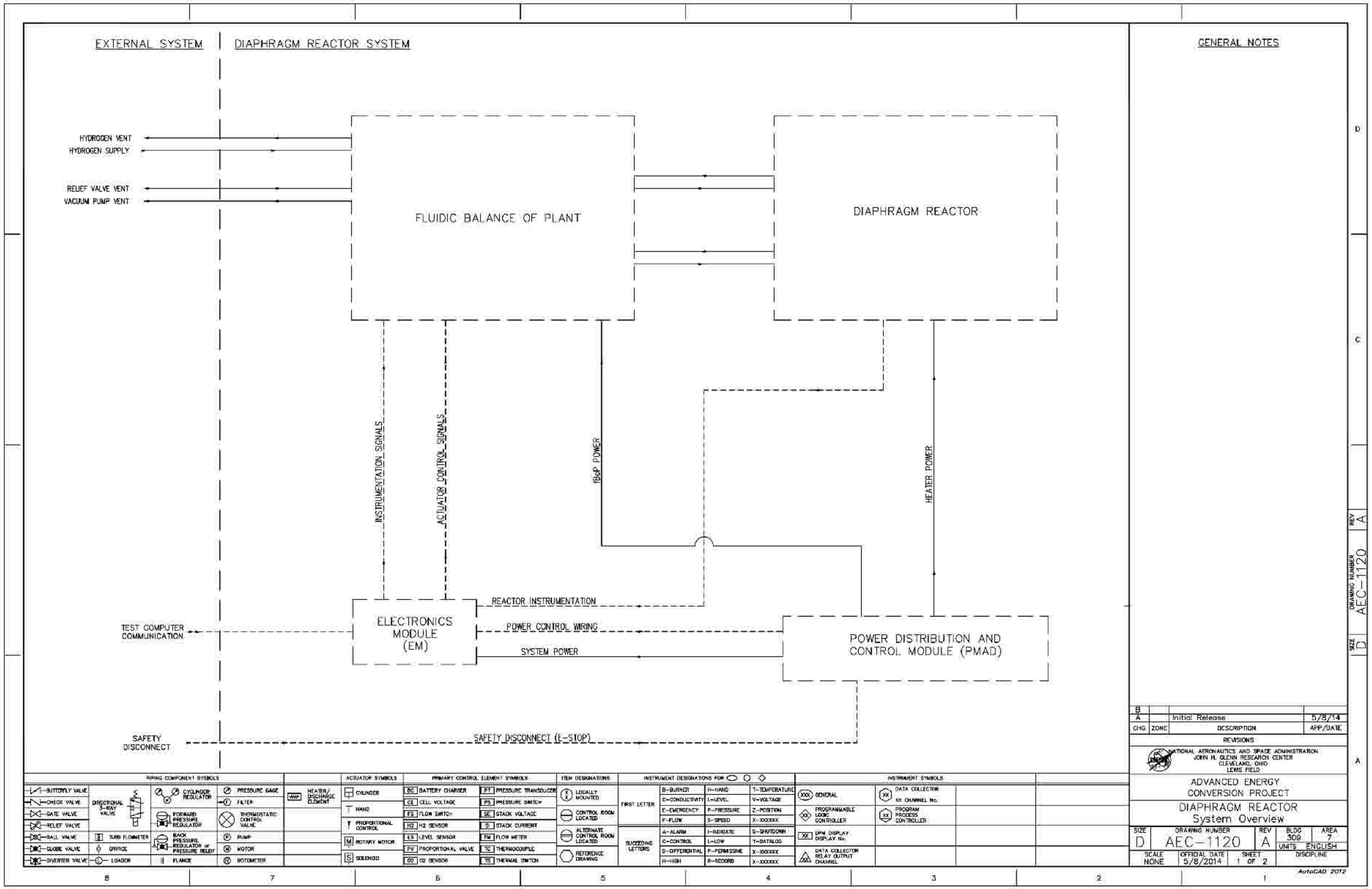

Figure B.1.—System overview.

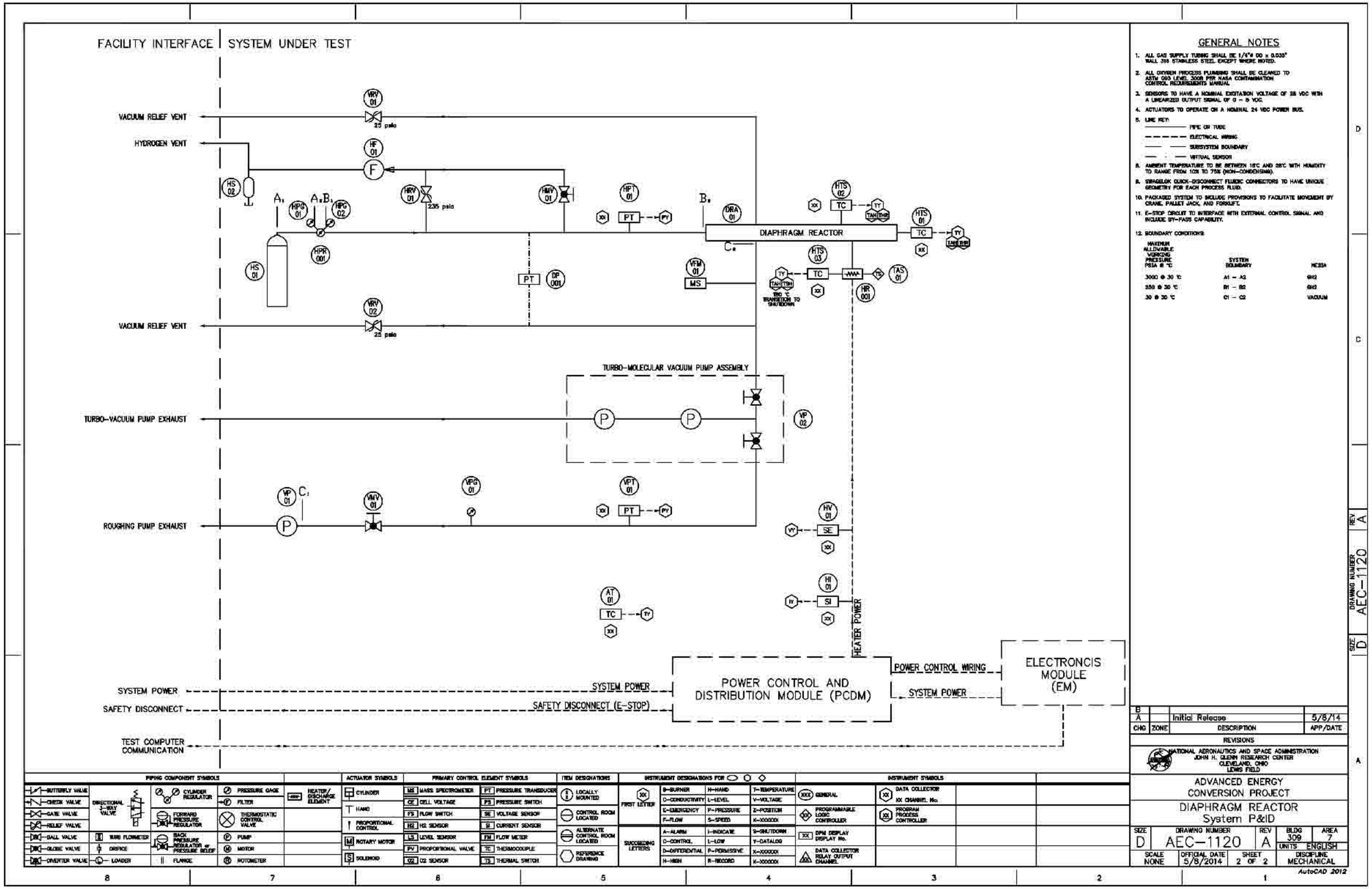

Figure B.2.—System piping and instrumentation diagram (P&ID).

Appendix C.—Test Apparatus Images

Figure C.1 and Figure C.2 show the complete test and vacuum systems, respectively. The diaphragm reactor components and test samples can be seen in various states of assembly in Figure C.3 to Figure C.8, leading to the insulated reactor images in Figure C.9 to Figure C.11. Figure C12 to Figure C.18 show different samples upon reactor disassembly. The x-ray system setup can be seen in Figure C.19.

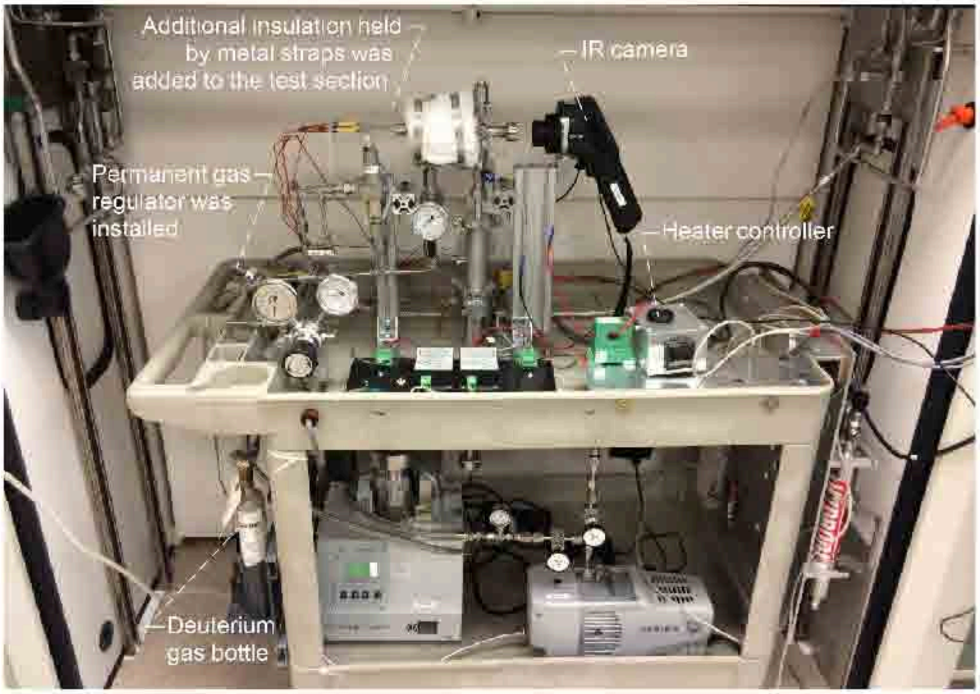

Figure C.1.—Complete assembled test system. IR is infrared.

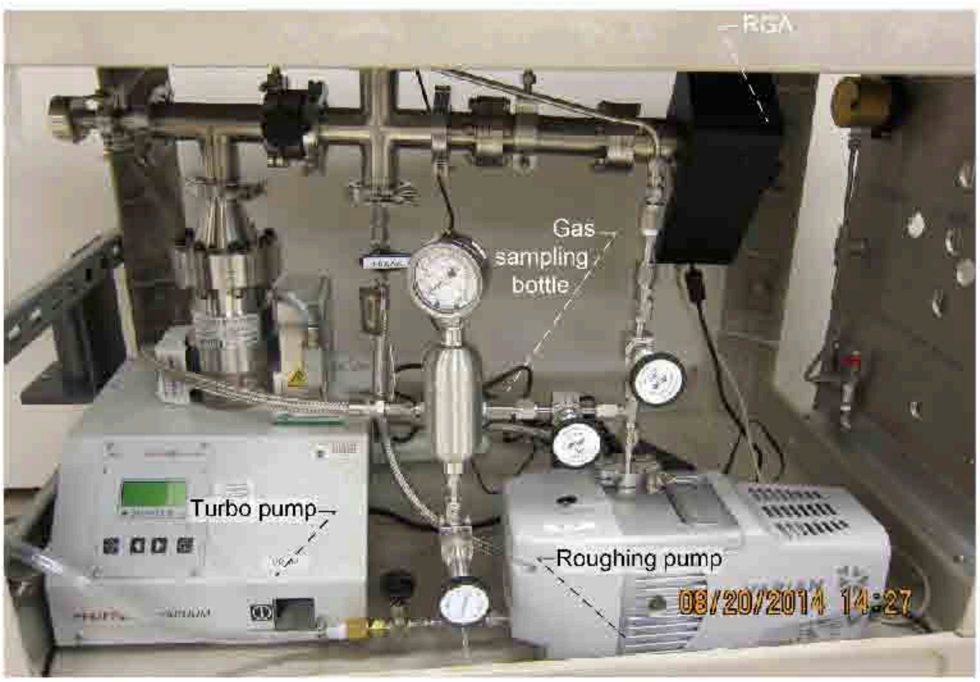

Figure C.2.—Vacuum subsystem. RGA is residual gas analyzer.

Figure C.3.—Disassembled diaphragm reaction chamber.

Figure C.4.—Vacuum side of diaphragm reaction chamber showing support disk.

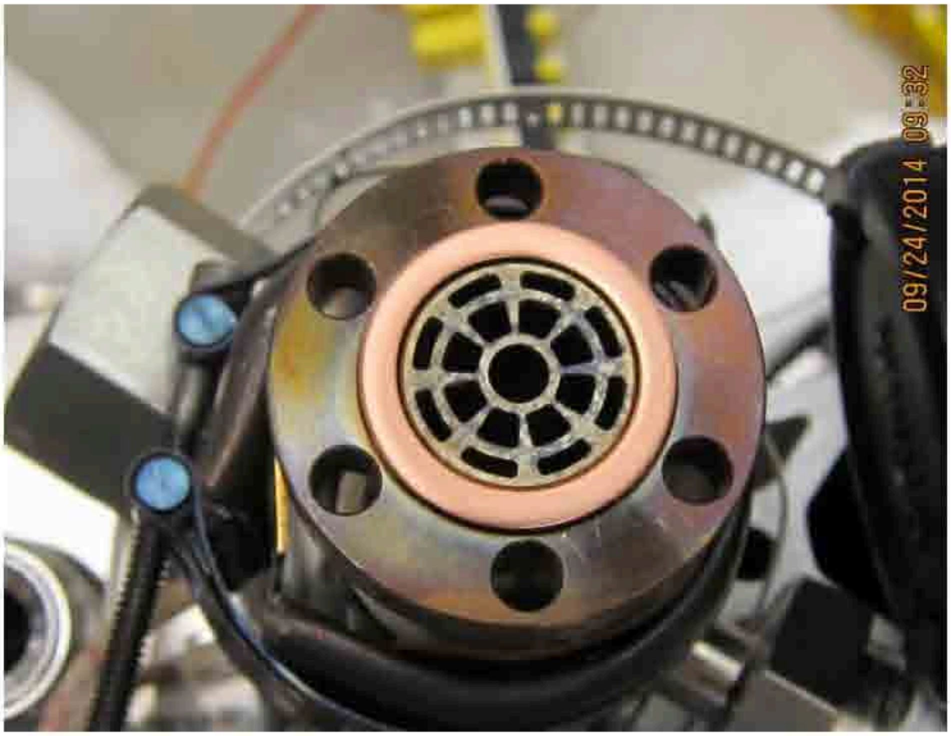

Figure C.5.—Metal frame support for sample disk on vacuum side of test fixture.

Figure C.6.—Sample AEC–Pd–GL–NL–20140903–101 prior to installation into test fixture.

Figure C.7.—Sample AEC–Pd–GL–NL–20140916–111, appearing as highly reflective circle near top of image, situated on frame support prior to sealing reactor.

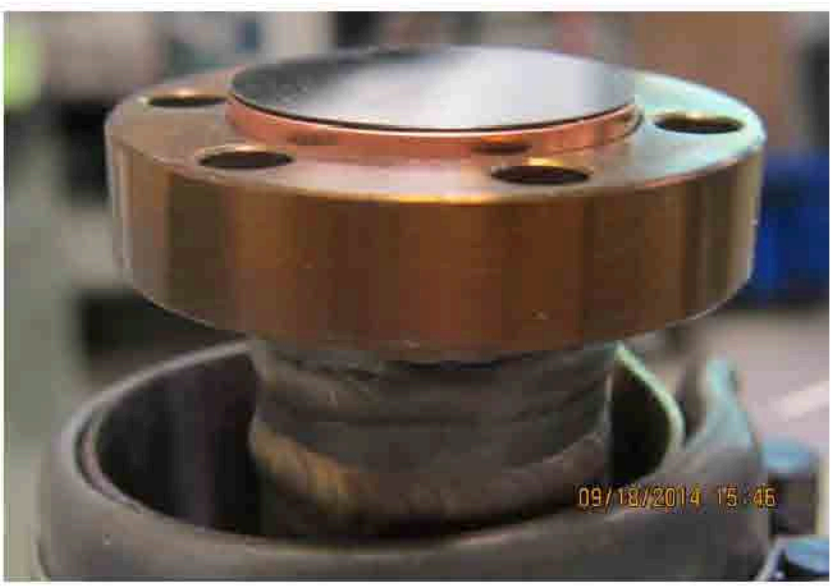

Figure C.8.—Closeup of sample AEC–Pd–GL–NL–20140916–111 situated on frame support prior to sealing reactor with band heater visible at bottom of image.

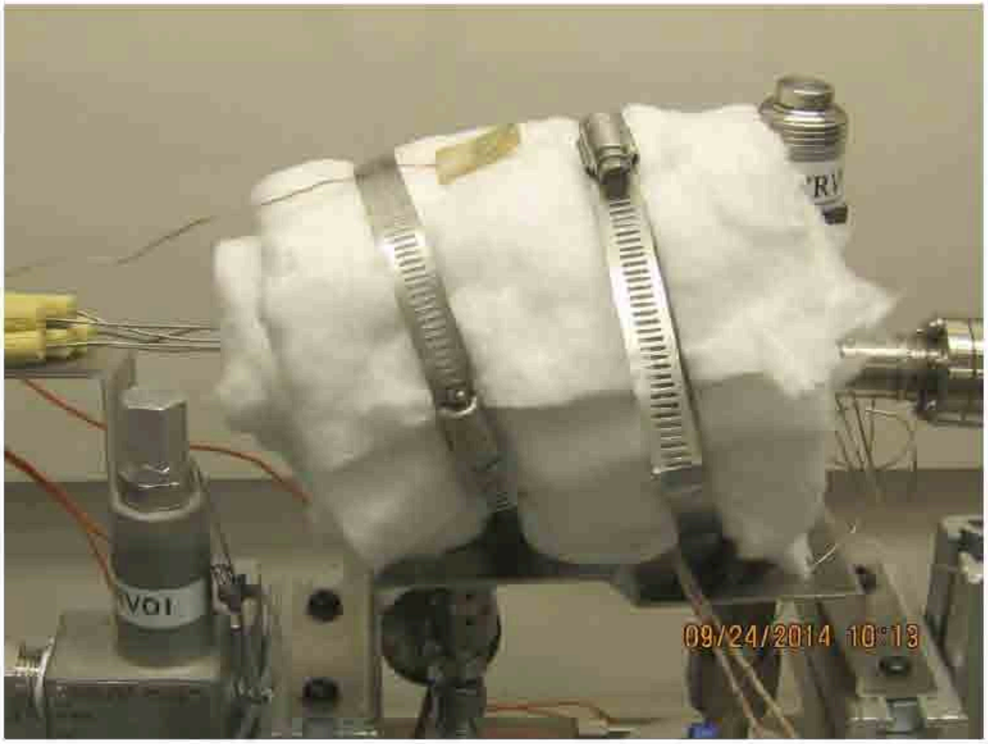

Figure C.9.—Insulated exterior of reactor section.

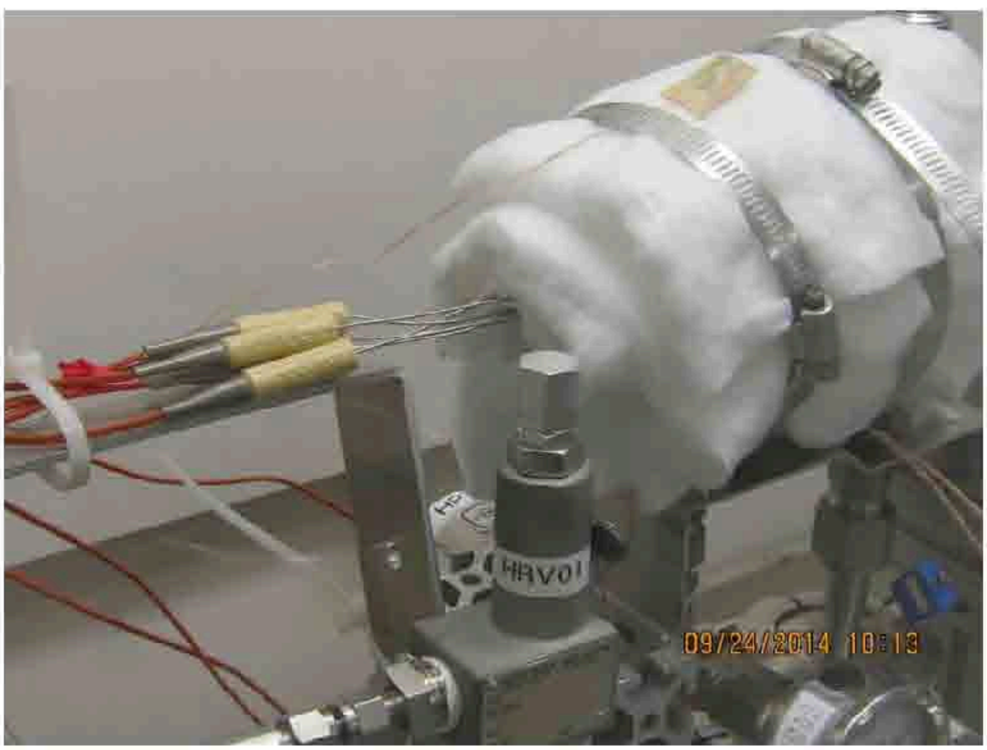

Figure C.10.—Thermocouple probe placement on gas supply side of reactor section.

Figure C.11.—Infrared camera placement on vacuum side of reactor section.

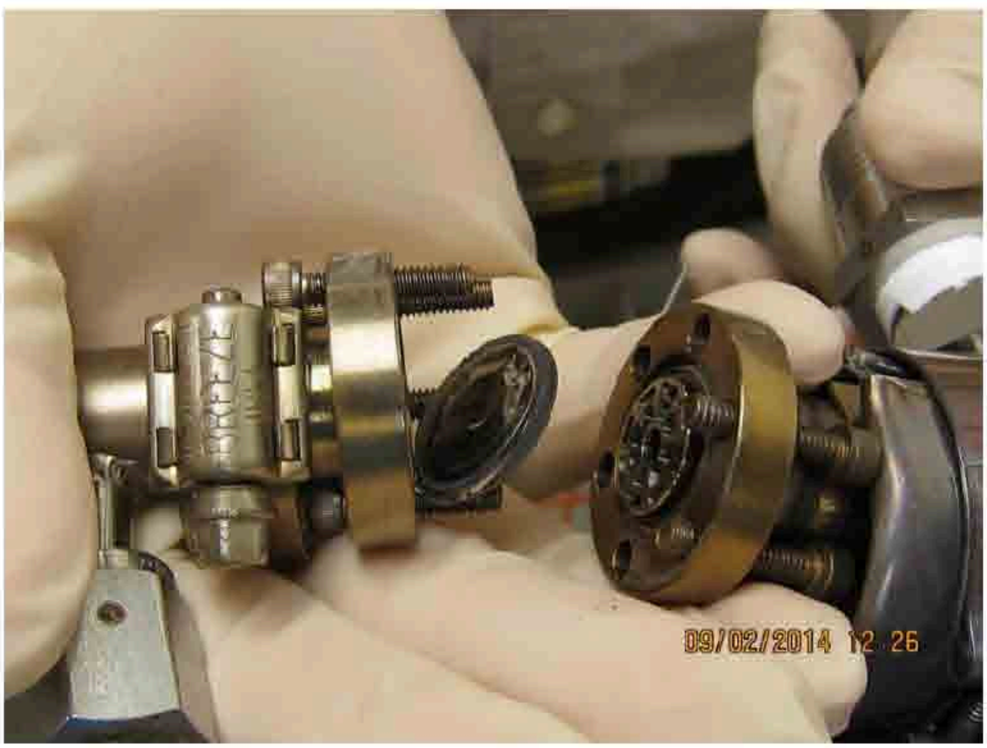

Figure C.12.—Sample AEC–Pd–GL–NL–20140814–1 during reactor disassembly.

Figure C.13.—Gas supply facing side of sample AEC–Pd–GL–NL–20140903–101 situated on frame support during reactor disassembly.

Figure C.14.—Angled view of sample AEC–Pd–GL–NL–20140903–101 on gas supply facing side situated over frame support during reactor disassembly.

Figure C.15.—Gas supply facing side of sample AEC–Pd–GL–NL–20140814–1 following removal from test fixture.

Figure C.16.—Vacuum facing side of sample AEC–Pd–GL–NL–20140814–1 with copper sealing ring still in place.

Figure C.17.—Angled view of vacuum facing side of sample AEC–Pd–GL–NL–20140814–1 with copper sealing ring still in place.

Figure C.18.—Vacuum facing side of sample AEC–Pd–GL–NL–20140903–101 with copper sealing ring still in place.

Figure C.19.—Placement of x-ray unit over test article.

Appendix D.—Bill of Materials

This section includes the bill of materials for the diaphragm reactor system in Table D.1.

TABLE D.1.—DIAPHRAGM REACTOR SYSTEM BILL OF MATERIALS

Tag	Description	Maximum allowable working pressure, psig	Setpoint, psig	Maximum allowable working temperature, °C	Set point, °C	Vendor	Part number
HI01	Heater power bus current sensor	N/A	N/A	--------	-----	McMaster-Carr®	6733T11
HMV01	Hydrogen manual dump valve	3,000	N/A	65	22	Swagelok®	42G SS–42GS4
HPG01	Hydrogen bottle pressure gauge	3,000	N/A	100	25	Swagelok®	PGI–63B–PG3000–BAOX
HPG02	Regulated hydrogen pressure gauge	3,000	N/A	100	25	Swagelok®	PGI–63B–PG3000–BAOX
HPR01	Hydrogen forward pressure regulator	10,000	1,500	74	22	Tescom	44–1114–CFGN20–834
HPS01	Heater power controller	N/A	N/A	--------	-----	McMaster-Carr®	38615K71
HPT01	Regulated hydrogen pressure transducer	2,985	N/A	185	25	NOSHOK, Inc.	615–5000–1–1–2–36
HR01	Reactor heater	N/A	N/A	649	450	McMaster-Carr®	3594K121
HRV01	Regulated hydrogen relief valve	6,000	2,200	200	22	Swagelok®	SS–4R3A5–D
HS001	Deuterium supply tank	2,000	1,775	50	21	Matheson Tri-Gas, Inc.	G2671584
HTS01	Infrared camera	N/A	N/A	40	22	FLIR®	E40
HTS02	Surface thermal couple	N/A	N/A	980	450	Omega™	XCIB–K–1–6–3
HTS03	Surface thermal couple	N/A	N/A	980	450	Omega™	XCIB–K–1–6–3
HV01	Heater power bus voltage sensor	N/A	N/A	N/A	N/A	--------------------	----------------------
VFM01	Residual Gas Analyzer	25	0	70	22	Extorr Inc.	XT200
VMV01	Roughing pump manual isolation valve	3,000	N/A	93	22	Swagelok®	SS–4BK–V51
VP01	Roughing vacuum pump	6.5	0.25	40	25	Varian	IDP3B01
VP02	Turbomolecular pump	0	<0	80	25	Pfeiffer	PMP02801G
VPG01	Vacuum pressure gauge	50	N/A	100	25	Swagelok®	PGI–63B–PC30–BAQX
VPT01	Vacuum pressure transducer	50	N/A	--------	-----	NOSHOK	621-30/30-1-1-8-6
VRV01	Reactor vacuum assembly relief valve	10	10	150	25	MDC Vacuum Products LLC	420034
VRV02	High vacuum relief valve	10	10	--------	25	Pfeiffer/Varian	A7003665-1
--------	Reactor	3,000	N/A	500	400	NASA	----------------------
--------	Insulation	N/A	N/A	1,090	400	McMaster-Carr®	93315K51

Appendix E.—Test Procedures

This section covers the operation of the diaphragm reactor and x-ray systems. The diaphragm reactor system shown in Figure 1 is repeated here.

E.1 Diaphragm Reactor System Operation Check Sheet

NAME OF TEST: ______________________________ Diaphragm ID: ____________________

TEST DIRECTOR: __________________________________ DATE: _____/_____/_______

TEST ENGINEER: __________________________________ TIME: ____________am / pm

GAS SPECIES: ___________ GAS BOTTLE LOT # & FILL DATE: _______________________

TANK PRESSURE: Initial _________ psig Final _________ psig

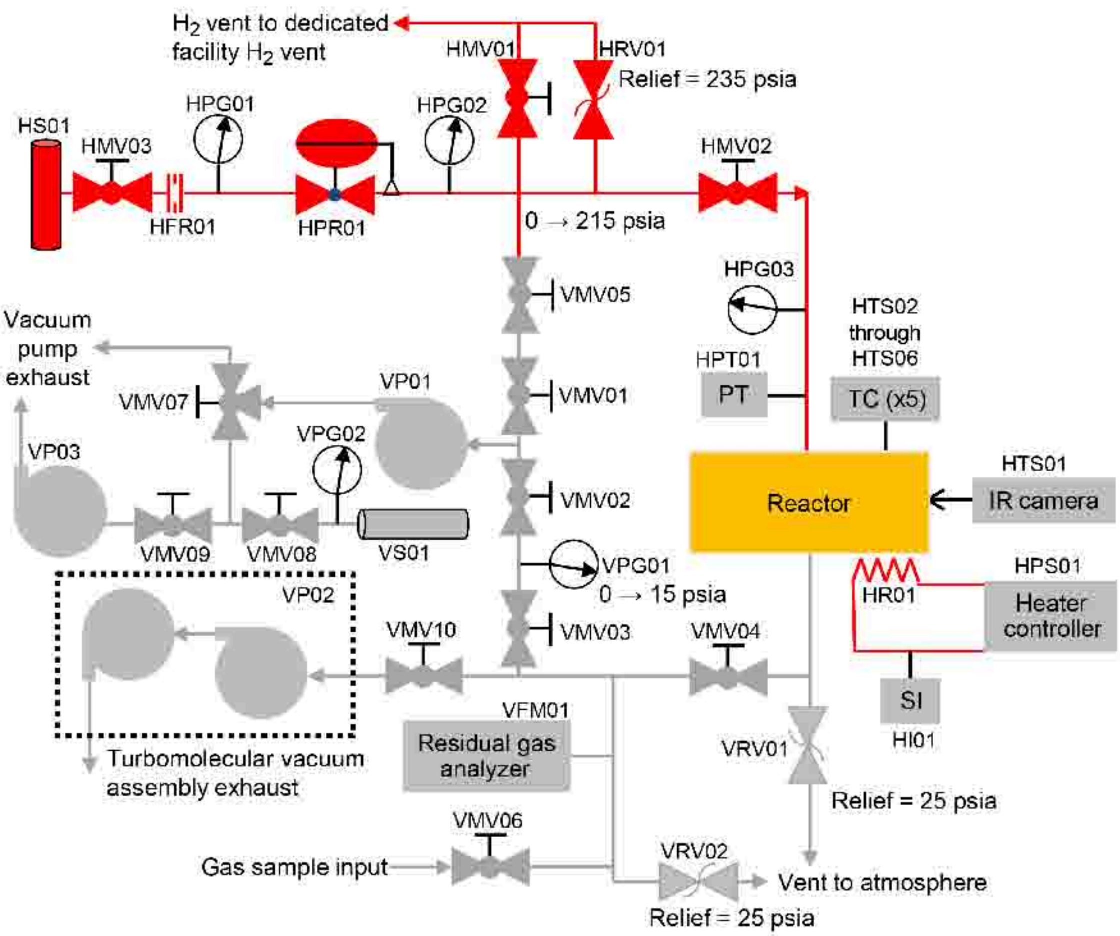

Figure 1.—Diaphragm reactor test system. IR is infrared. PT is pressure transducer. SI is current sensor. TC is thermocouple.

	ITEM	COMPLETE
P.	**PRELIMINARY SETUP**	
P.1.	Update AEC Sample Travel Log	______________
P.2.	Power on and verify flow through the test cell HVAC vent	______________
P.3.	Install gas bottle into bottle holder	______________
P.4.	Connect gas bottle to high-pressure line	______________
P.5.	Connect ground strap between gas bottle and the facility ground	______________
P.6.	Verify that the electrical resistance between the gas supply bottle and ground is $\leq$1.0 Ω	______________
P.7.	Plug in the test stand power strip	______________
P.8.	Turn on the test stand power strip	______________
P.9.	Perform a visual inspection of all connections (e.g., tubing, valves, etc.)	______________
P.10.	Verify the following manual valves are closed: HS01 _____ VMV01 _____ VMV05 _____ VMV09 _____ HMV01 _____ VMV02 _____ VMV06 _____ HMV03 _____ VMV03 _____ VMV08 _____	______________
P.11.	Verify the following valves are open: HMV02 _____ VMV04 _____	______________
P.12.	Verify that the 3-Way valve VMV07 is turned to the "Atmosphere" port	______________
P.13.	Turn on facility test indicator light by turning the key on the Facility Operator E-Stop Interface Panel to the "On" position	______________
P.14.	Turn on the roughing pump at the main power switch	______________
P.15.	Open valve VMV02 to evacuate the line to the chamber	______________
P.16.	Wait until vacuum gage VPG01 reaches <–30 inHg	______________
P.17.	Open valve VMV03 to connect roughing pump to main chamber	______________
NOTE:	Continue to the next step. From this step, run vacuum pump either 20 min or until RGA pressure instrumentation indicates that the chamber pressure is $<10^{-2}$ torr.	
P.18.	Verify that all reactor bolts are properly tightened	______________
P.19.	Install insulation around reactor heater	______________

P.20. Verify that the external hard drive is attached to Computer #1 ______________

P.21. Turn on laptop Computer #1 ______________

P.22. Turn on laptop Computer #2 ______________

P.23. Remove lens cap from IR camera ______________

P.24. Turn on IR camera ______________

P.25. From Computer #1, start FLIR® software

P.26. Within the FLIR® software, navigate to the *Instruments* display ______________

P.27. Within the FLIR® software, engage the LiveStream by clicking on the icon with the lightning bolt ______________

P.28. Within the FLIR® software, set the Emissivity to 0.33 ______________

P.29. Within the FLIR® software, set the Distance to 0.2 m ______________

P.30. Within the FLIR® software, set the Frame Rate to 7.5 frames per second ______________

P.31. Within the FLIR® software, set the Temperature Range to 0 – 650 °C ______________

P.32. Set image display range to Auto ______________

P.33. Within the FLIR® software, verify that the images are saved within the external hard drive folder ______________

P.34. On Computer #2, start the Extorr *VacuumPlus* software ______________

P.35. Within the *VacuumPlus* software, verify that an active Com Port is selected ______________

P.36. Within the *VacuumPlus* software, verify that baud rate is 115200 ______________

P.37. Turn on the data logger ______________

P.38. Verify that the thermocouples and pressure transducers are operational (all display ambient readings) ______________

P.39. Install flash drive into data logger ______________

P.40. Verify that the data logger is set to store data on the flash drive ______________

P.41. Verify that the data logger stores the data in the ".csv" data format ______________

P.42. Record the data logger channel names and calibration values ______________

P.43. Turn on the turbo pumping station VP02 main switch ______________

P.44. When green “Status” light on turbo pumping station begins to blink, press the Start/Stop button to activate the pumping station ____________

P.45. Wait for the vacuum system pressure on the RGA to drop below 5×10^{-4} torr ____________

P.46. Close vacuum isolation valve VMV03 ____________

P.47. Close vacuum isolation valve VMV02 ____________

P.48. Verify that supply bottle valve HMV03 is closed ____________

P.49. Open manual valve HMV01 ____________

P.50. Wait until HPT01 is ≤2 psig ____________

P.51. Close manual valve HMV01 ____________

P.52. Open manual valve VMV01 ____________

P.53. Open manual valve VMV05 ____________

P.54. Completely back out, then completely turn in pressure regulator HPR01 ____________

P.55. Wait for at least 60 s ____________

P.56. Close VMV05 ____________

P.57. Close VMV01 ____________

P.58. Completely back out pressure regulator HPR01 ____________

P.59. Turn off roughing pump VP01 ____________

P.60. Locate the x-ray head in the desired position adjacent the diaphragm ____________

P.61. Open supply tank isolation valve HMV03 ____________

P.62. Turn on heater controller power strip ____________

P.63. Verify that heater control setpoint is 15 °C ____________

P.64. Verify that the heater control mode “S10t” is set to “OnOff” ____________

A. TESTING

A.1. Start recording on the data logger ____________

A.2. Start recording on the FLIR® thermal camera ____________

A.3. Take a baseline RGA data scan ____________

A.4. Use HPR01 to set the system pressure as indicated by HPT01 ______________

Target Pressure: ______ psig Final Pressure: ______ psig

A.5. Use the heater controller to set the reactor temperature as indicated by TC channels #1 on the data logger ______________

Target Temperature: ______ °C Final Temperature: ______ °C
NOTE: Set "SP1" to a temperature that is 30 to 50 °C below the target temperature as there is a significant overshoot.

A.6. Once desired temperature setpoint is reached, change heater control mode "S10t" from "OnOff" to "Puls" with a time constant value of less than "4" ______________

Temperature at Set Point Change: ______ °C

NOTE: This step is accomplished by pressing and holding for 5 seconds the "Enter" and "↑" buttons. Once the screen flashes, press "Index" until the "S10t" submenu appears. Use the "↑" key to select "Puls" and press "Enter". Push "Enter" a second time to confirm the time constant value. It is also beneficial to enable the control parameters continuous learning mode. To do so, simultaneously press "Enter" and "↑". Do not hold this time. Press "Index" to step through the submenu until "Strt". Use the "↑" key to select "YES". At any time, one can return to the main display by repeatedly pressing "Index".

A.7. All personnel exit the test cell ______________

A.8. Close the interlocked cell sliding door ______________

A.9. Commence x-ray operational protocol ______________

A.10. Follow test plan: Update pressure and temperature set points as required by approved test plan ______________

Test Start Time: ______________ am/pm Test Stop Time: ______________ am/pm

A.11. Use "Save As" to capture RGA scan at the beginning and end of each test condition ______________

A.12. Stop recording FLIR® video at the end of each test condition ______________

A.13. Start new FLIR® video at the beginning of each test condition ______________

B. SHUTDOWN

B.1. Complete x-ray shutdown procedure ______________

B.2. Change heater controller set point to 15 °C ______________

B.3. Change the "Strt" controller self-tuning mode to "no" ______________

NOTE: See step A.6 for controller operation details.

B.4. Change heater control mode “S10t” to “OnOff” ______________

B.5. Turn off the heater controller power strip ______________

B.6. Wait until diaphragm temperature is less than 200 °C ______________

B.7. Open test cell door ______________

B.8. Verify that the gas supply bottle isolation valve is closed ______________

B.9. Don protective gloves ______________

B.10. Remove thermal insulation surrounding reactor ______________

B.11. Wait for reactor temperature to drop below 100 °C as indicated by channels #1 through #3 on the data logger ______________

B.12. Press the Start/Stop button on the turbo pumping station ______________

B.13. Stop recording the FLIR® video ______________

B.14. Turn off the FLIR® camera ______________

B.15. Open HMV01 ______________

B.16. Wait for HPT01 to drop below 5 psig ______________

B.17. Close HMV01 ______________

B.18. Turn off facility test indicator light by turning the key on the Facility Operator E-Stop Interface Panel to the “Off” position ______________

B.19. Stop recording data on the data logger ______________

B.20. Turn off the turbo pumping station VP02 main switch ______________

B.21. Turn off test stand power trip ______________

B.22. Unplug test stand power strip ______________

C. DATA PROCESSING

C.1. Review video in FLIR® Tools+ software. Capture and annotate images of note ______________

C.2. Safely exit from FLIR® Tools+ software on Computer #1 ______________

C.3. Safely exit from the *VacuumPlus* software on Computer #2 ______________

C.4. Turn off data logger ______________

C.5. Remove USB flash drive from data logger and install in into Computer #2 ______________

C.6. Copy RGA files from “C:\Gas Sample Tests” from the day’s tests ______________

C.7. Safely remove the USB flash drive from Computer #2 ____________

C.8. Install the USB flash drive from Computer #2 into Computer #1 ____________

C.9. With the external hard drive installed on Computer #1, create a folder for new data on the external drive with the name format ____________

C.10. Copy the data logger data files from the USB flash drive to the folder created in Step C.9 ____________

C.11. Create the a subfolder "…\RGA" in the folder created in Step C.9 ____________

C.12. Copy the RGA data files from the USB flash drive to the folder created in Step C.11 ____________

C.13. Create the a subfolder "…\FLIR Images" in the folder created in Step C.9 ____________

C.14. Copy the FLIR® image files from the external drive folder "…\FLIR" to the folder created in Step C.13 ____________

C.15. Safely remove the USB flash drive from Computer #1 and install it in the data logger ____________

C.16. Turn off Computer #1 and Computer #2 ____________

C.17. Update AEC Sample Travel Log ____________

D. SPECIAL PROCEDURE #1: HIGH VACUUM PRESSURE DURING OPERATION

D.1. Turn on roughing pump VP01 ____________

D.2. Open manual valve VMV02 ____________

D.3. Wait for VPG01 to read <30 inHg ____________

D.4. Open manual valve VMV03 ____________

D.5. Wait until RGA responds favorably ____________

NOTE: If RGA does not respond within 120 s, reduce hydrogen system pressure until the RGA responds favorably.

D.6. Close manual valve VMV03 ____________

D.7. Close manual valve VMV02 ____________

D.8. Turn off roughing pump VP01 ____________

E. SPECIAL PROCEDURE #2: DIFFUSION GAS SAMPLE COLLECTION

E.1. Verify that the Sample Under Test is at the desired temperature as indicated on the average of the thermocouple probes

E.2. Verify that the 3-Way valve VMV07 is towards the “Atmosphere” port

E.3. Turn on Evacuation Pump VP03

E.4. Open Sample Line evacuation valve VMV09

E.5. Wait for between 15±5 s

E.6. Open Sample Bottle Valve isolation valve VMV08

E.7. Turn on the roughing pump VP01 at the main power switch

E.8. Open valve VMV02 to evacuate the line to the chamber

E.9. Wait until vacuum gage VPG01 reaches <–30 inHg

E.10. Take and verify two RGA samples

E.11. Open valve VMV03 to connect roughing pump to main chamber

E.12. Verify that Sample Bottle Pressure Gage indicates <30 inHg

E.13. Close Sample Bottle Valve isolation valve VMV08

E.14. Turn 3-Way valve VMV07 towards the Gas Sample Bottle line

E.15. Wait for between 10±5 s

E.16. Open Sample Bottle Valve isolation valve VMV08

E.17. Close Sample Line evacuation valve VMV09

E.18. Close Turbomolecular Vacuum Pump Assembly Isolation Valve VMV10

E.19. Wait until the Sample Bottle Pressure Gage indicates >2 psig

E.20. Close Sample Bottle Valve isolation valve VMV08

E.21. Turn the 3-Way valve VMV07 towards the “Atmosphere” port

E.22. Wait for the vacuum system pressure to drop below $5\text{x}10^{-4}$ torr

E.23. Open Turbomolecular Vacuum Pump Assembly Isolation Valve VMV10

E.24. Close vacuum isolation valve VMV03

E.25. Close vacuum isolation valve VMV02

E.26. Turn off roughing pump VP01 ______________

E.27. Continue with the test as defined in the Test Plan ______________

F. SPECIAL PROCEDURE #3: INSTALLING SAMPLE INTO REACTOR

F.1. Collect required PPE which include the following: ______________

Protective gloves (Latex) _____Laboratory glasses _____

F.2. Collect required tools and materials which include the following: ______________

Dispenser bottle with Isopropyl Alcohol _____KimWipes, one box _____

¼" VCR Gasket, (x1), P/N SS-4-VCR-2 _____3/4" Open Ended Wrench_____

½" VCR Gasket, (x1), P/N SS-8-VCR-2 _____13/16" Open Ended Wrench_____

0.834" Copper Gasket, (x1), P/N GA-0133_____1-1/16" Open Ended Wrench_____

8 mm hex nut driver (x1)_____Can of Compressed Air (x1)_____

¼" Allen Wrench (x1) _____3/16" Allen Wrench (x1) _____

#8-32UNF by 3/4"Socket Head bolts (x6) _____ConFlat Threaded Plates_____

Vacuum Support Disk, (x1), P/N AEC-1078_____ Test Sample (x1)_____

5" diameter x ½" wide Ring Clamp (x2)_____Band Heater (x1)_____

F.3. Verify that the Diaphragm Rig is unpowered and unpressurized ______________

F.4. Verify that valves HMV02 and VMV04 are closed ______________

F.5. With the can of compressed air, blow down the Diaphragm Reactor to remove contaminants ______________

F.6. Clean the test sample and 0.834" Copper Gasket using the isopropyl alcohol and KimWipes ______________

F.7. Hold the Vacuum Body Weldment (AEC–1073) such that the sample opening is oriented upwards ______________

F.8. Place the Band Heater over the Vacuum Body Weldment such that the electrical wires protrude downwards (towards the site glass) ______________

F.9. Place the Vacuum Support Disk (AEC–1078) into the sample opening recess with the disk support ridges upwards ______________

F.10. Place the 0.834" Copper Gasket (GA–0133) into the space surrounding the support disk frame. This gasket should have space for slight lateral (~0.003") movement but no vertical movement ______________

F.11. Place the Test Sample onto the platform created by both the Copper Gasket and the Vacuum Support Disk. Be sure to orient the sample according to the test plan ______________

F.12. Hold the Pressure Body Weldment (AEC-1071) oriented with the sample opening downwards over the Vacuum Body Weldment ______________

NOTE: Care should be taken not to move or damage the inserted thermocouples through the Pressure Body Weldment thermocouple feed-through.

NOTE: It facilitates assembly to leave the small thermocouple ring clamp on to the Pressure Weldment during the assembly process. Do not tighten until it is time to firmly affix the thermocouples.

F.13. Rotate the Pressure Body Weldment around the vertical axis until the two-(2) VCR ports are in the same vertical plane ______________

F.14. Lower the Pressure Body Weldment onto the Vacuum Body Weldment maintaining the common vertical plane of the two-(2) VCR ports and the alignment of the test sample with Copper Gasket ______________

F.15. Verify the alignment of the test sample between the Weldments ______________

F.16. Bolt the Weldments together with the #8-32UNF x ¾" bolts and the ConFlat threaded plates using the ¼" Allen Wrench ______________

NOTE: It facilitates assembly to have the bottom two-(2) bolts inserted from the Vacuum Weldment side and the upper four bolts inserted from the Pressure Weldment side.

NOTE: This bolted assembly is known as the "Diaphragm Reactor".

F.17. Place a 5" diameter ring clamp over each VCR fitting on the Diaphragm Rig with the hex adjustment bolt facing the front of the test cart ______________

F.18. Place the ¼" VCR gasket (SS-4-VCR-2) on the pressure side male VCR fitting from the Diaphragm Rig ______________

F.19. Place the 1/2" VCR gasket (SS-8-VCR-2) on the pressure side male VCR fitting from the Diaphragm Rig ______________

F.20. Lower the Diaphragm Reactor on to the Diaphragm Rig such that the ¼" female port of the Pressure Body Weldment aligns with the ¼" male port of the Diaphragm Rig and the ½" female port of the Vacuum Body Weldment aligns with the male port of the Diaphragm Rig ____________

F.21. Verify alignment of sealing gaskets ____________

F.22. Tighten ¼" VCR fittings on the Pressure Body Weldment using the ¾" open end wrenches ____________

NOTE: Do NOT impose a torque on the Diaphragm Rig Plumbing. Move only the ¾" wrench on the VCR female nut in relation to the ¾" wrench on the VCR bulkhead while holding the wrench on the bulkhead in constant orientation relative the Diaphragm Rig plumbing.

F.23. Tighten ½" VCR fittings on the Vacuum Body Weldment using the 13/16" and 1-1/16" open end wrenches ____________

NOTE: Do NOT impose a torque on the Diaphragm Rig Plumbing. Move only the 1-1/16" wrench in relation to the 13/16" wrench while holding the 13/16" wrench in constant orientation relative the Diaphragm Rig plumbing.

F.24. Position Heater thermocouples under the ring clamp along the axis of the Pressure Body Weldment such that the tip of the thermocouples securely impinge on the Pressure Body Weldment flange ____________

F.25. Tighten the Ring Clamp with the 8 mm nut driver to hold the thermocouples in position ____________

F.26. Verify all bolts and fittings are properly tightened ____________

F.27. Center the Band Heater over the joint between the Weldment flanges and tighten Band Heater Clamp using 3/16" Allen Wrench ____________

F.28. Install the fiberglass insulation around Diaphragm Reactor. Wrap the insulation to a diameter of 4.88±0.13" around the Band Heater between the vertical plumbing lines ____________

F.29. Run the surface mount thermocouple through the 5" Ring clamp on the Pressure Weldment side and place on the top of the insulation ____________

F.30. Move the 5" diameter Ring clamps over the insulation and tighten the adjustment bolt using the 8 mm nut driver ____________

F.31. Plug in the test stand power strip ____________

F.32. Turn on the test stand power strip ____________

F.33. Perform a visual inspection of all fluidic connections ____________

F.34. Verify the following manual valves are closed: ______________

HS01 _____ VMV01 _____ VMV04 _____ VMV07 _____
HMV01 _____ VMV02 _____ VMV05 _____ VMV08 _____
HMV03 _____ VMV03 _____ VMV06 _____ VMV09 _____

F.35. Verify that the 3-Way valve VMV07 is turned to the "Atmosphere" port ______________

F.36. Turn on the roughing pump at the main power switch ______________

F.37. Open manual valves VMV01 and VMV02 ______________

F.38. Open manual valves VMV03 and VMV05 ______________

F.39. Open manual valves HMV02 and VMV04 ______________

F.40. Wait for at least 60 s ______________

F.41. Record the pressure on VPG01 ______________

VPG01: __________ inHg

F.42. Close manual valves VMV01 and VMV02 ______________

F.43. Wait for at least 300 s ______________

F.44. Record the pressure on VPG01 ______________

VPG01: __________ inHg

F.45. If the pressure rises by more than 5 inHg between Steps F.41 and F.44, track down leak, resolve, and repeat Steps F.36 thru F.44. If a pressure rise of less than 5 inHg is observed, continue to Step F.46.

F.46. Close manual valves VMV03 and VMV05 ______________

F.47. Turn off the Roughing Pump ______________

F.48. Turn off the test stand power strip ______________

F.49. Unplug the test stand power strip ______________

G. SPECIAL PROCEDURE #4: REMOVING SAMPLE FROM REACTOR

G.1. Verify sample analysis personnel are available for immediate testing after removing the sample from the Diaphragm Reactor ______________

G.2. Collect required PPE which include the following: ______________

Protective gloves (Latex) _____ Laboratory glasses _____

G.3. Collect required tools and materials, which include the following: ______________

8 mm hex nut driver (x1)_____ 3/4" Open-ended wrench (x2) _____

Can of compressed air (x1)_____ 13/16" Open-ended wrench (x1) _____

Transportation Container for Test Sample_____

1-1/16" Open-ended wrench (x1)_____¼" Allen wrench (x1) _____

3/16" Allen wrench (x1) _____

Container for #8 bolts and Threaded Plates _____ Digital Camera _____

G.4. Verify that the Diaphragm Rig is unpowered and unpressurized _____________

G.5. Verify that the Diaphragm Reactor is below touch temperature _____________

G.6. Verify that the test stand power strip is unplugged _____________

G.7. Roll the Diaphragm Rig test cart out of the fume hood, taking care not to disturb the vent lines or electrical wires _____________

G.8. Don PPE _____________

G.9. Loosen insulation Ring clamp using 8-mm nut driver _____________

G.10. Slide loosened insulation Ring clamps outboard and remove insulation _____________

G.11. Loosen, but do not remove, thermocouple Ring clamp using 8-mm nut driver _____________

G.12. Move the Heater thermocouples out of the field of work taking care to not stress the thermocouple electrical wiring _____________

G.13. With the can of compressed air, blow down the Diaphragm Reactor to remove contaminants _____________

G.14. Take a picture of Diaphragm Reactor _____________

G.15. Loosen, but do not remove, ¼" VCR fittings on the Pressure Body Weldment using the ¾" open end wrenches _____________

NOTE: Do NOT impose a torque on the Diaphragm Rig Plumbing. Move only the ¾" wrench on the VCR female nut in relation to the ¾" wrench on the VCR bulkhead while holding the wrench on the bulkhead in constant orientation relative the Diaphragm Rig plumbing.

G.16. Completely loosen ¼" VCR fittings using fingers _____________

G.17. Remove all six of the #8 flange bolts using the ¼" Allen wrench placing all bolts and threaded plates in the designated container _____________

NOTE: It is helpful to have a hand below the flanges as the test sample can fall out of the assembly.

G.18. Loosen, but do not remove, ½" VCR fittings on the Vacuum Body Weldment using the 13/16" and 1-1/16" open end wrenches ____________

NOTE: Do NOT impose a torque on the Diaphragm Rig Plumbing. Move only the 1-1/16" wrench in relation to the 13/16" wrench while holding the 13/16" wrench in constant orientation relative to the Diaphragm Rig plumbing.

G.19. Slowly and carefully lift the ¼" VCR female nut on the Pressure Body Weldment and slide the Pressure Body Weldment away from the Vacuum Body Weldment ____________

NOTE: It is helpful to have a hand below the flanges as the Pressure Body Weldment is moved because the test sample can fall out of the assembly.

G.20. Take pictures of the Pressure Side of the Test Sample in the Vacuum Body Weldment to fully document the condition of the Test Sample ____________

G.21. Remove Test Sample from the Vacuum Body Weldment ____________

G.22. Take pictures of the Vacuum Side of the Test Sample to fully document the condition of the Test Sample ____________

G.23. Place the test sample in the sample travel container ____________

G.24. Verify that the ¼" VCR gasket is on the Diaphragm Rig ¼" VCR bulkhead ____________

G.25. Place the Pressure Body Weldment on the Diaphragm Rig ¼" VCR bulkhead and tighten the ¼" VCR female nut to finger tight ____________

G.26. Roll the test cart back into the fume hood ____________

G.27. Remove PPE and cleanup work area ____________

G.28. Update the travel log book ____________

G.29. Place the travel log book and the sample travel container with the test sample in a zip-lock bag ____________

G.30. Take travel log book and test sample to be analyzed within 15 min of being removed from the Diaphragm Reactor ____________

H. SPECIAL PROCEDURE #5: SYSTEM SHUTDOWN FOLLOWING POWER FAILURE

NOTE: If power failure is only a momentary fault, proceed with normal shutdown and test evaluation as described in Section B. Else, continue with step H.1.

H.1. Power off reactor heater using the remote switch ____________

H.2. Ensure the x-ray system is disabled ____________

H.3. Perform all achievable data processing steps in Section C ____________

H.4. Open test cell door ______________

H.5. Close the gas supply bottle isolation valve ______________

H.6. Turn off test stand power strip ______________

H.7. Unplug test stand power strip ______________

H.8. Exit the test cell and wait until diaphragm temperature is less than 200 °C ______________

H.9. Turn off the FLIR® camera ______________

H.10. Don protective gloves ______________

H.11. Remove thermal insulation surrounding reactor ______________

H.12. Wait for reactor temperature to drop below 100 °C as indicated by channels #1 through #3 on the data logger ______________

H.13. Open HMV01 ______________

H.14. Wait for HPT01 to drop below 5 psig ______________

H.15. Close HMV01 ______________

H.16. Turn off facility test indicator light by turning the key on the Facility Operator E-Stop Interface Panel to the "Off" position ______________

H.17. Stop recording data on the data logger ______________

H.18. Power off data logger ______________

E.2 X-Ray System Operation Procedures

Unit Specifications and Operation

Manufacturer/Model	Maximum Voltage Ratings	Normal Operation Voltage Ratings	Target Materials
Muller / MG150	150 kV	150 kV or less	Ceramics, composites, low Z metals, AEC Target Materials
Norelco / MG300	300 kV	290 kV or less	Steel and most other metal alloys, AEC Target Materials

Operating Procedure:

1. Complete the "Pre-operational Checks" and note on the daily utilization log. Any unsatisfactory responses need to be corrected prior to operation of the x-ray units.

2. Establish cooling medium supply to the x-ray unit and verify integrity of this system.

3. Enter cell and position specimen and imaging media as required.

4. Exit cell and close cell door. Verify cell is not occupied.

5. Install key in control unit.

6. Turn on unit and adjust voltage and current to desired settings.

7. Set timer and energize x-ray to perform shot.

8. Daily, when in use, perform "Operational Checks" listed below and on daily utilization log.

9. Following the x-ray irradiation test, verify radiation field has been secured by checking that the Radiation Alarm audible signal and the x-ray controller "X-Ray On" lights are off.

10. Enter cell to retrieve sample, film, etc., and set up for next shot/test.

11. If additional shots/tests will be conducted, repeat steps 3 through 10 until complete. (Step 8 is required only once per day.)

12. When testing is complete, remove and secure key for x-ray unit.

Date: ______________________________

Test Cell Number:____________________

Operator: __________________________

Start Time: _________________________

Pre-operational Checks: (Performed once prior to testing)

- ☐ Radiation Survey of Room within past 12 months.
- ☐ Mezzanine Area Cleared, Secured and Posted
- ☐ Whole Body Dosimeter (worn)
- ☐ Radiation Monitor/Alarm Check (installed, powered, and inspected)
- ☐ Door Interlock Operational Check
- ☐ X-ray Unit Integrity Check (oil leaks, water leaks, wiring, etc.)
- ☐ X-ray warmup procedure followed. Verify both x-ray lights on controller are operational during warmup.

Pretest check (performed prior to each test—*indicate time that test commenced*)

- ☐ Cell Occupancy Check ___________

Warmup Procedure (daily use)

1.) Adjust voltage to 200 kV 5 mA
2.) Energize x-ray source
3.) Increase kV level in 5 kV increments every 30 s until full power is reached
4.) If kV level drops suddenly during increase to next level, reduce kV to previous value and hold for 5 min before proceeding

Warmup Procedure (system unused for 1 week or more)

1.) Adjust voltage to 200 kV 5 mA
2.) Energize x-ray source
3.) Increase kV level in 5 kV increments every 5 min until full power is reached
4.) If kV level drops suddenly during increase to next level, reduce kV to previous value and hold for 10 min before proceeding

References

1. Fralick, Gustave C.; Decker, Arthur J.; and Blue, James W.: Results of an Attempt To Measure Increased Rates of the Reaction $^{2}D + {}^{2}D \rightarrow {}^{3}He + n$ in a Nonelectrochemical Cold Fusion Experiment. NASA TM–102430, 1989. http://ntrs.nasa.gov/
2. Iwamura, Yasuhiro, et al.: Detection of Anomalous Elements, X-Ray, and Excess Heat in a D_2-Pd System and Its Interpretation by the Electron-Induced Nuclear Reaction Model. Fusion Technology, vol. 33, 1998, pp. 476–492.
3. Iwamura, Yasuhiro; Sakano, Mitsuru; and Itoh, Takehiko: Elemental Analysis of Pd Complexes: Effects of D_2 Gas Permeation. Jpn. J. Appl. Phys., vol. 41, 2002, pp. 4642–4650.
4. Liu, B. et al.: "Excess Heat" Induced by Deuterium Flux in Palladium Film. Condens. Matter Nucl. Sci., 2006, pp. 75–79.
5. Biberian, J.P.; and Armanet, N.: Excess Heat During Diffusion of Deuterium Through Palladium. Presented at the 13th International Conference on Condensed Matter Nuclear Science, Sochi, Russia, 2007.
6. Li, X., et al.: Progress in Gas-Loading D/Pd System—The Feasibility of a Self-Sustaining Heat Generator. Presented at the 10th International Conference on Cold Fusion, Cambridge, MA, 2003.