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DETERMINATION OF CARBON, HYDROGEN, AND TRITIUM IN IRRADIATED REACTOR FUELS AND CLADDING MATERIALS

by

C. B. MacDougall, T. K. Marshall,
G. M. Matlack, and G. R. Waterbury

ABSTRACT

A reliable gravimetric method for determining carbon and hydrogen was adapted for analyses of irradiated fuels and claddings in a hot cell. The sample was burned in oxygen at 1000°C in a furnace system within the hot cell to oxidize hydrogen to water and carbon to carbon dioxide. The evolved gases were swept through heated tubing into magnesium perchlorate to trap the water and into Ascarite to trap the carbon dioxide, and the traps were subsequently weighed. The bias in measuring either carbon or hydrogen was consistently -2.5%, and the relative standard deviations were 4.4% in measuring hydrogen and 3.0% in measuring carbon. The same furnace system was used to determine tritium in a separate portion of the sample. This portion was reacted with moist oxygen at 1000°C, and the tritiated water was collected in a spiral trap cooled to -80°C. This trap was then heated to 200°C, and the tritiated water was swept by oxygen into a volumetric flask containing distilled water. An aliquot of the water was pipetted into a scintillation vial filled with Aquasol and the mixture was counted. The relative standard deviation of this method was $\pm 10\%$ for measuring 0.1 μg of tritium, and the bias was -4%.

I. INTRODUCTION

The significant effect on the properties of reactor fuels and claddings caused by interstitial impurities, including carbon and hydrogen, necessitated measuring these elements as part of the chemical characterization of irradiated reactor material. Previous experience with a combustion-gravimetric method^{1,3} was very satisfactory and suggested modification for use in a remote facility. To use economically the equipment and space in a hot cell, the same combustion furnace and gas lines were used in determining tritium retained in fuels and other irradiated materials.

To measure carbon and hydrogen, the sample is burned in oxygen at 1000°C in a furnace system within the hot cell to oxidize hydrogen to water and carbon to carbon dioxide. The evolved gases are swept through heated tubing into magnesium perchlorate to trap the water and into Ascarite to trap the carbon dioxide. The traps are then weighed. The bias in measuring either carbon or hydrogen is consistently -2.5%, and the relative standard deviation is 4.4% in measuring hydrogen and 3.0% in measuring carbon.

The modifications required for adapting this method to hot cell use were all concerned with equipment design. These changes included a new-type

fused silica furnace tube, a different reaction crucible, and a furnace-tube support system so that samples can be handled with a manipulator.

Tritium is measured in a separate portion of the sample by reacting the sample with moist oxygen at 1000°C and by collecting the tritiated water in a cold trap. This trap is then heated, and the tritiated water is swept by argon into a volumetric flask containing distilled water. An aliquot of water is pipetted into a scintillation vial filled with Aquasol, and the mixture is counted. The relative standard deviation of this method is $\pm 10\%$ for measuring $0.1\ \mu\text{g}$ of tritium, and the bias is -4.0% .

These methods were used to analyze (uranium,plutonium) dioxides, plutonium dioxides, stainless steel cladding samples, and (uranium,plutonium) carbides. The equipment proved to be versatile. It was able to determine carbon and hydrogen or tritium by changing the trapping and measuring systems. The equipment was installed in a hot cell facility similar to that described by Lilienthal⁴ and by Burwell, Schulte, and Wilson.⁵

II. APPARATUS AND REAGENTS

A. Apparatus

The following list describes the apparatus used in our experiment.

Absorption Tubes. 100-ml Schwartz (four required). Fill two-thirds of each of two tubes with Ascarite, and the remaining third with anhydrous magnesium perchlorate. Fill the other two tubes with anhydrous magnesium perchlorate. Pack the openings to all tubes with glass wool to prevent physical loss of dust from the tubes.

Balance, analytical, with nonbrowning glass, Ainsworth Right-a-Weigh, or similar.

Balance, analytical, for weighing absorption tubes, Ainsworth BCT, or similar, with set of S-grade weights.

Base Plate, for sealing furnace tube (Fig. 1).

Boat, sample, glass. Cut off the top half of a 30-ml beaker, so that the remaining cup is 2.8 cm (1.125 in.) high.

Bottle, for drying gases, 250 ml with fritted disk and stopper. Fill one-half of the bottle with concentrated sulfuric acid.

Bubbler, for safely trapping sulfuric acid, 50-ml capacity.

Combustion System, as shown in Fig. 2.

Fig. 1.
Base plate.

Fig. 2.
Combustion system.

Counter, scintillation, Packard Model 2101, Semi-Automatic Tri-Carb Liquid Scintillation Spectrometer System, or similar.

Crucible, zirconium dioxide, LECO #528-35, or similar.

Crucible Holder, consisting of a 5- by 10- by 1.27-cm dural plate having a 0.635-cm-diam, 0.635-cm-deep hole located 2.5 cm from one end and a 1.59-cm-diam, 10.16-cm-long rod located 2.5 cm from the opposite end.

Degasification System, consisting of an argon supply, hardware for mounting the spiral trap, volumetric flask, connecting tubing, and an air-natural gas torch.

Dewar, two required. One Dewar, 7.47-cm-i.d. and 20.96-cm-high, is for cooling the spiral trap, and one Dewar, 14.29-cm-i.d. and 26.67-cm-high, is for minimizing the melting of the ice in the humidifier.

Flask, volumetric, 100-ml.

Flow Gauge, three required, Hoke Bantam, or similar.

Furnace Tube, fused-silica (Fig. 3).

Furnace Tube, nickel, for assuring complete combustion of sample (Fig. 4). Fill the furnace tube with a mixture of 50% lead chromate and 50% copper oxide. Pack the end with quartz wool to prevent removal of dust from the furnace.

Fig. 3.
Fused-silica furnace tube.

Fig. 4.
Supplementary nickel furnace tube.

Furnace Tube, nickel, 40.6-cm-long 2.86-cm-diam, to fit resistance furnace, and with a screw cap fitted with a lead gasket to make a gas-tight seal.

Furnace tube, 3.18-cm-bore, 30.5-cm-long, Hevi-Duti, or similar.

Guide Tube, solder a 12.7-cm length of 0.635-cm-i.d. brass tubing to a 1.59- to 0.635-cm reducing coupling.

Heating Tape, heavy insulated Briskeat, or equivalent, 1.83-m length, three required, for heating exit lines from furnace.

Jar, filled with crushed ice for maintaining oxygen gas at constant humidity (Fig. 5).

Pedestal, fused silica, consisting of a 30-mm-diam, 3-mm-thick disk connected to a rod 115 mm in length and 6.35 mm in diameter.

Pipette, Drummond Microcaps, Kensington Scientific Corporation, or similar.

Pyrometer, graduated from 0 to 1000°C. Sim-Ply-Trol Assembly Products Incorporated, or equivalent, with Chromel-Alumel thermocouple.

Fig. 5.
Humidifying jar.

Fig. 6.
Spiral trap.

Sleeve, fused silica. Fuse two hooks at a distance of 17 mm (180° apart) from the bottom of a 96-mm-long, 32-mm-i.d., 2-mm-thick tube.

Thermometer, alcohol, graduated from -100° to $+50^{\circ}$ C.

Tongs, curved crucible, suitably modified for handling sleeve and pedestal remotely.

Torch, welding, for heating spiral trap, Prest-o-lite, or similar.

Trap, spiral (Fig. 6).

Trap, 15-cm length of 12-mm-i.d. tubing filled with 75-g polyethylene beads, for removing fission products.

Tweezers, 30.48-cm-long, suitably modified for easy handling by manipulators.

U-Tube, copper, for replacing spiral trap in apparatus during degasification process.

Vial, glass, for scintillation counting, with calibrated volume mark.

Variable transformers, 7.5-A, for controlling temperatures of furnace tubes and heating tapes, six required.

Wet Test Meter, for initial measurement of gas flow, Precision Scientific Company, or similar instrument.

Wire, nichrome coil, wind a 30.48-cm length of wire around the exit arm of the fused-silica furnace tube, and connect the leads to a variable transformer.

B. Reagents

The reagents used in this experiment are listed here.

Aquasol, universal LSC cocktail, reagent grade.

Alcohol, ethyl, denatured.

Argon, tank, purify this gas by passage through the gas supply system shown in Fig. 2.

Ascarite, 8- to 20-mesh.

Carbon Dioxide, dry ice.

Cupric Oxide, wire form, analytical reagent grade.

Lead Chromate, analytical reagent grade.

Magnesium Perchlorate, anhydrous, analytical reagent grade.

Oxygen, tank.
Polyethylene Beads, 8-mesh.
Sulfuric Acid, concentrated, analytical reagent grade.
Vacuum Grease, Fluorlube GR-362.
Water, triply distilled.

III. RECOMMENDED PROCEDURE

Health and safety rules for handling irradiated materials must be followed rigidly, and adequate protection of operators must be ensured by using a suitably shielded facility. In addition, all exit lines for analyzing tritium must be helium leak-checked to prevent the release of tritium to the atmosphere. Degasification of the spiral trap must be performed in a hood.

IV. SAMPLE PREPARATION

A. Fuels

Visually inspect the irradiated oxide or carbide fuel in the shielded facility for contamination. Clean or discard samples containing foreign material.

B. Cladding

Visually inspect the sample for surface corrosion or contamination. In particular, be certain that no fuel adheres to the cladding. Remove adhering fuel by abrasion.

V. SAMPLE ANALYSIS

Tritium

1. Remove the drying bottle and bubbler from the line, and insert the gas-humidifying equipment where indicated. Place the copper U-tube in the equipment (Fig. 2).

2. Fill the humidifying jar and the Dewar, which surrounds the jar, with ice.

3. Turn on power to the tapes that heat the exit lines, to the nichrome coil that heats the exit arms of the reaction furnace, and to the resistance furnace that heats the nickel furnace tube.

4. Turn on the flow of oxygen, and adjust the flow to 1.67 ml/s by adjusting valve V_1 .

5. Close valves V_4 and V_{12} , and open valves V_3 , V_5 , V_6 , and V_{11} .

6. Turn on the power to the fused-silica furnace tube, and allow the furnace to heat to 1000°C.

7. Flush the lines for 30 min with oxygen.

8. Close V_1 , shut off power to the fused-silica furnace tube, remove the cap from the furnace, and allow the furnace to cool for 15 min.

9. Accurately weigh (within ± 0.1 mg) approximately 100 mg of sample into a weighed sample boat.

10. Close valves V_5 and V_6 , remove the copper U-tube from the line, and replace it with the spiral trap cooled by the ethyl alcohol-dry ice bath at $\sim -80^\circ\text{C}$.

11. Slide the guide tube into the fused-silica furnace tube, and drop the sample through the guide into the crucible.

12. Remove the guide tube and replace the cap on the furnace; twist the cap slightly to ensure that it is seated firmly.

13. Turn the flow of oxygen to 1.67 ml/s by adjusting valve V_1 .

14. Open valves V_5 and V_6 , and turn on the power to the fused-silica furnace tube.

15. Allow the sample to react for 50 min.

16. Close valves V_5 , V_6 , and V_1 , and remove the spiral trap from the line.

17. Replace the copper U-tube in the equipment.

18. Turn the oxygen flow back to 1.67 ml/s by opening V_1 , and allow the furnace to flush for 10 min.

19. Transfer the spiral trap to a hood, and insert the trap into the degasification system.

20. Add 75 ml of distilled water to a 100-ml volumetric flask, place it over the exit line of the degasification system, open valves V_5 and V_6 , turn on the argon so that bubbles are visible in the flask.

21. Heat the trap to 200°C with the torch to transfer the tritiated water to the volumetric flask.

22. Allow the trap to cool, close valves V_5 and V_6 , turn off the argon flow by closing the supply valve, and remove the volumetric flask from the equipment.

23. Fill the volumetric flask to the mark, stopper, and mix thoroughly.

24. Fill a glass scintillation vial to the mark with Aquasol, and pipette a 10- μl sample aliquot into the Aquasol.

25. Stopper and shake the vial to mix the sample thoroughly.

26. Scintillation count the weak $\beta(0.0186\text{ MeV})$ from the ^3H , and calculate the dis/s for the 10- μl sample aliquot.

27. Calculate the amount of tritium in the sample from the following equation.

$$^3\text{H} = \frac{C}{3.57 \times 10^4}$$

where

H = weight of tritium in μg .

C = count rate of 10- μl aliquot in dis/s.

28. Analyze the next sample by repeating steps 8 through 27.

Carbon and Hydrogen

1. Insert the sulfuric acid trap into the combustion system where indicated.

2. Turn on the power to the furnace that heats the cupric oxide, to the furnace that heats the nickel tube containing the lead chromate and the cupric oxide, and to the heating tapes.

3. Close valves V_3 , V_5 , V_6 , and V_{11} , and open valves V_4 , V_7 , V_8 , V_{10} , and V_{12} .

4. Lower the base plate, and place a zirconium oxide crucible into the fused-silica furnace tube.

5. Raise the base plate into position and adjust valve V_1 to pass 1.67 ml/s of oxygen.

6. Turn on the power to the coil heating the fused-silica furnace tube, and allow the furnace to heat to 1000°C.

7. Allow the crucible and furnace to bake for 30 min. If two crucibles are to be used, heat the second crucible for 30 min also.

8. Analyze a sample as follows.

a. Turn off the power to the reaction furnace, and allow the crucible to cool for 5 min. Close valve V_1 .

b. Lower the base plate, remove the pedestal and sleeve from the furnace, and place them in their respective positions on the crucible holder.

c. Weigh (within ± 0.1 mg) a sample no larger than 5 g and transfer it into a pretreated crucible (see step 7).

d. Place the crucible into the quartz sleeve, and reassemble the sleeve and pedestal in the furnace tube.

e. Raise the base plate into position, and seal the reaction furnace.

f. Adjust the flow of argon to 1.67 ml/s using valve V_2 , and allow the system to flush for 5 min at room temperature.

g. Close valve V_2 and adjust the flow of oxygen to 1.67 ml/s using valve V_1 .

h. Close valves V_7 , V_8 , V_9 , and V_{10} , and remove these collection tubes from the apparatus.

i. Place preconditioned and preweighed collection tubes in the line, and open valves V_1 , V_8 , V_9 , and V_{10} . Adjust the flow of oxygen to 1.67 ml/s using valve V_1 .

j. Turn on the power to the reaction furnace and allow it to heat to 1000°C.

k. Wait 50 min, turn off the power to the coil heating the fused-silica tube, and close valves V_1 , V_8 , V_9 , and V_{10} .

l. Remove the collection tubes from the analysis line, wipe them with a damp chamois, wait 20 min, and then weigh the tubes within ± 0.1 mg.

9. Determine a blank by following steps 8.a through 8.l but omit addition of sample in 8.c.

10. Calculate the amounts of carbon and hydrogen in the sample as follows.

$$\text{Carbon (ppm)} = \frac{(12.01)}{(44.01)} \frac{(W_1 - W_2)10^3}{W}$$

where

W_1 = weight increase in CO_2 collection tube, in milligrams, during sample analysis,

W_2 = sample blank, in milligrams, and

W = weight of sample, in grams.

$$\text{Hydrogen (ppm)} = \frac{(2.02)}{(18.02)} \frac{(W_3 - W_4)10^3}{W}$$

where

W_3 = weight increase in H_2O collection tube, in milligrams, during sample analysis, and

W_4 = sample blank, in milligrams.

VI. EXPERIMENTAL

The chemical aspects for determining carbon and hydrogen are well known.¹⁻³ Our major problem was to design new hardware to operate and maintain the equipment for hot cell use. The fused-silica reaction furnace was designed with a wide, flanged, bottom opening for easy removal of the crucible (Fig. 3). This design permitted us to use large (up to 5 g) samples that were transferred directly into the crucible. The entire assembly was then replaced in the furnace for the analysis. Small (100 mg) solid samples were added through the opening at the top of the furnace. The base plate supported the quartz sleeve and pedestal, and made a seal between the base and the furnace tube. The reaction furnace was heated by a nichrome coil controlled by a variable transformer. Reaction furnace temperatures were

measured with a thermocouple outside the cell before in-cell use. A second furnace containing a one-to-one mixture of copper oxide and lead chromate maintained at a temperature of 600°C assured complete oxidation of gases from large samples. The stainless steel exit lines from the furnace were heated at 120°C to facilitate total recovery of the water produced from the combustion of hydrogen. All seals were glass-to-glass and were lubricated with Fluorlube GR-362, which remained pliable under irradiation and also maintained leak-tight seals at the operating temperature of the apparatus.

For economical use of equipment and space in the hot cell, and because of the fundamental similarity of the chemical reaction, the same combustion furnace and gas lines were used for determining tritium. Modifications to the equipment for measuring carbon and hydrogen were necessary to measure tritium. A humidifying unit was added to moisten the oxygen before introducing oxygen into the reaction tube and a cold trap was substituted for the magnesium perchlorate trap to collect the tritiated water. The humidifying unit was necessary because of the extremely small quantity of tritium (about 0.1 μ g) to be measured. This additional moisture, in effect, washed the tritiated water along the heated exit lines and into the spiral trap. Heating the exit lines was not sufficient when handling this small quantity of tritiated water. A stainless steel trap packed with glass beads (for greater surface area) and cooled to -80°C was used to trap the tritiated water. With a sweep flow of 1.67 ml/s, a collection time of 50 min, and a humidifying temperature of 0°C (ice), the total quantity of water to be trapped was about 24 mg. The spiral trap was designed to remove effectively twice this amount.

The radioactive nature of the tritiated water made measurements of microquantities possible. Health-safety considerations precluded a gravimetric finish for analyses, unless all operations were done in-cell. Scintillation counting of tritiated water was the established technique chosen for this measurement.^{6,7}

Numerous small tools were designed for adapting these processes to hot cell use. These tools included tongs for sample handling and guide tubes for transfers, which are described more fully in Sec. II.

VII. RELIABILITY

The method and apparatus were evaluated for determining carbon and hydrogen in fuels and cladding using sucrose as the standard (Table I).

The average recovery of carbon, based on the stoichiometric amount of carbon in the samples, was 98% with a relative standard deviation of 3%. The average recovery of hydrogen was 98% with a relative standard deviation of 4%. In-cell analyses compare reasonably well with those made out-of-cell as shown in Table I.

In addition to the analyses of the "standard" sucrose, tantalum and stainless steel samples that had been analyzed previously out-of-cell by independent methods were analyzed in the hot cell (Table II). The independent method for determining hydrogen consisted of heating the sample to 1200°C in a vacuum, oxidizing the hydrogen to water, and measuring the water with a McLeod Gage after removing extraneous gases. The independent method for determining carbon consisted of burning the sample in oxygen at 2000°C and measuring the carbon dioxide evolved by thermal conductivity.

The results obtained for carbon and hydrogen by the different methods agree well. Unfortunately, irradiated fuel and cladding standards of known carbon and hydrogen contents were not available, but analyses of the sucrose and metals indicated that the bias of the method was no greater than 2%.

TABLE I
MEASUREMENT OF CARBON AND
HYDROGEN IN SUCROSE

	C Recovery (%)	H Recovery (%)
Out-of-cell	100 $\pm$ 2	99 $\pm$ 2
In-cell	98 $\pm$ 3	98 $\pm$ 4

TABLE II
DETERMINATION OF HYDROGEN AND
CARBON IN TANTALUM AND
STAINLESS STEEL

Sample	In-Cell Measurements		Independent Measurements	
	H,ppm	C,ppm	H,ppm	C,ppm
Ta	5	30	2	35
Stainless Steel	10	230	6	235

Four irradiated oxide fuels were then analyzed for carbon and hydrogen (Table III). The precision of the measurements was not good, but this depends, of course, on the sampling and homogeneity of the sample. Out-of-cell analyses of similar materials can yield a spread in values of this magnitude. The PuO_2 sample showed gross contamination with free carbon, and was extremely heterogeneous.

Three stainless steel cladding samples from pins having 2.7 to 10% burnup were analyzed (Table IV). No difficulties that could be attributed to the radiation levels were observed, and the calculated precision compared well with similar out-of-cell analyses.

To verify the accuracy of the tritium determination, we used a zirconium tritide target as a standard. This target was made to specifications by Oak Ridge National Laboratory (Table V). We overchecked these specifications by dissolving weighed portions of the target by the sealed-tube method⁸ and scintillation counting the resulting solutions. An average measured value of 1.34 Ci/target was found.

The distribution of tritium over the target area was within $\pm 10\%$ of this average value. Repeated analyses of this standard by combustion of the sample, followed by scintillation counting of the tritiated water produced (Table VI), yielded an average tritium recovery of $96 \pm 9\%$.

The precision of the method is of the same order as the homogeneity of the standard.

Samples of irradiated carbide and oxide fuels were then analyzed (Table VII). Again, no suitable standards were available for comparison, but difficulties were not observed during these analyses. The precision of analysis depends upon methods of sampling, sample homogeneity, and in these examples, on the extremely small quantities of tritium being measured. A relative standard deviation of $\pm 30\%$, or 3σ , at the lower limits of determination of the method is expected. In the range between 0.1 and 10 ppm, the relative standard deviation should be the same ($\pm 10\%$) as that obtained for the standard foil.

TABLE III

CARBON AND HYDROGEN MEASURED IN IRRADIATED FUELS

Sample	Burnup (%)	Carbon Found (ppm)	Hydrogen Found (ppm)
1. (U,Pu) O_2	8.5	370, 160	150, 70
2. (U,Pu) O_2	8.5	60, 200	20, 80
3. (U,Pu) O_2	2.7	60, 20, 60	20, 20, 10
4. PuO_2	---	8.10%, 11.6%	<5, <5

VIII. SUMMARY

A method for measuring carbon and hydrogen has been successfully adapted to use in a hot cell to measure the carbon and hydrogen in irradiated fuels and claddings. This same equipment, with a few modifications, is used to determine tritium in these materials. By judicious sampling, hydrogen in the 0.02- to 25-mg range and carbon in the 0.05- to 50-mg range are measured. Quantities of tritium ranging from 0.001 to 50 μg are determined. Approximately six analyses (C and H or ^3H) can be performed in 8 h.

TABLE IV

CARBON AND HYDROGEN MEASURED IN IRRADIATED CLADDING

Sample	Burnup (%) ^a	Carbon Found (ppm)	Hydrogen Found (ppm)
SS cladding	2.7	390, 390, 400	120, 60, 120
SS cladding	8.5	1490, 1660, 1640	220, 120, 230
SS cladding	10.0	590, 840, 750	70, 30, 40

^aTaken from pins having indicated fuel burnup.

TABLE V

SPECIFICATIONS OF ZIRCONIUM
TRITIDE TARGET

Backing	Copper, 10 mil
Diameter	3.20 cm
Zr/ ³ H atomic ratio	1:1
Activity/Target	1.36 Ci

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TABLE VI

RECOVERY OF TRITIUM FROM ZIRCONIUM-TRITIDE TARGET

Sample Wt (mg)	Theoretical Tritium (μg^a)	Tritium Found (μg)	Recovery
2.7	0.19	0.20	105.3
3.8	0.27	0.24	88.9
3.0	0.22	0.21	95.4
2.7	0.19	0.17	89.5
1.8	0.13	0.11	84.6
3.0	0.22	0.18	81.8
4.4	0.32	0.33	103.1
3.1	0.22	0.23	104.5
2.2	0.16	0.14	87.5
3.8	0.27	0.28	103.7
2.0	0.14	0.14	100.0
2.7	0.19	0.20	105.3

Av recovery = $96 \pm 9\%$.

^aCalculated from sample weight.

TABLE VII

TRITIUM MEASUREMENT IN IRRADIATED FUELS

Sample	No. Analyses	Tritium (ppm)	Burnup (%)	Rel Std Dev (%)
(U,Pu)C ₃	6	0.005	5.0	60
(U,Pu)C ₃	3	0.008	5.0	20
(U,Pu)O ₂	5	0.0005	2.7	20

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