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**Examination of Fast Reactor Fuels, FBR Analytical
Quality Assurance Standards and Methods, and
Analytical Methods Development—Irradiation Tests**

October 1—December 31, 1975

Compiled by

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ABSTRACT

This is the sixth quarterly report on the Examination of Fast Reactor Fuels and FBR Analytical Quality Assurance Standards and Methods at the Los Alamos Scientific Laboratory. Since the first quarterly report (LA-5798-PR), the Analytical Quality Assurance Standards and Methods project has been divided into the following two projects.

FBR Analytical Quality Assurance Standards and Methods

Analytical Methods Development-Irradiation Tests

Most of the investigations discussed here are of the continuing type. Results and conclusions described may therefore be changed or augmented as the work continues. Published reference to results cited in this report should not be made without obtaining explicit permission to do so from the person in charge of the work.

PROJECT AL-001

EXAMINATION OF FAST REACTOR FUELS

Person in Charge: R. D. Baker
Principal Investigators: J. W. Sculite
K. A. Johnson
G. R. Waterbury

I. INTRODUCTION

This project is directed toward the examination and comparison of the effects of neutron irradiation on LMFBR Program fuel materials. Unirradiated and irradiated materials will be examined as requested by the Reference Fuels System Branch of DRRD. Capabilities are established and are being expanded for providing conventional preirradiation and postirradiation examinations. Nondestructive tests will be conducted in a hot cell facility specifically modified for examining irradiated prototype fuel pins at a rate commensurate with schedules established by DRRD.

Characterization of unirradiated and irradiated fuels by analytical chemistry methods will continue, and additional methods will be modified and mechanized for hot cell application. Macro- and microexaminations will be made on fuel and cladding using the shielded electron microprobe, emission spectrograph, radiochemistry, gamma scanner, mass spectrometers, and other analytical facilities. New capabilities will be developed in gamma scanning, analyses to assess spatial distributions of fuel and fission products, mass spectrometric measurements of burnup and fission gas constituents and other chemical analyses.

Microstructural analyses of unirradiated and irradiated materials will continue using optical and electron microscopy and autoradiographic and x-ray techniques. Special emphasis will be placed on numerical representation of microstructures and its relationship to

fabrication and irradiation parameters. New etching and mounting techniques will be developed for high burnup materials.

II. EQUIPMENT DEVELOPMENT

A. In-Cell Equipment

(F. S. Abeyta, G. R. Brewer, A. L. Bridge, W. R. Carter, F. J. Fitzgibbon, M. E. Lazarus, J. M. Ledbetter, P. A. Mason, A. G. Nicol, J. A. Romero, T. J. Romero, D. S. Shaffer, L. N. Vikdal, J. J. Villareal)

1. The automatic centering system for the DP-West optical profilometer is now operational. Those elements with bows up to 25 mm (1 in.), which could not be "profiled" before, are now in the process of being measured.

2. The new Data Acquisition - Computer System for optical profilometer is being installed and should be ready for operation by the end of January. As reported previously, the new system will greatly simplify profilometry data acquisition.

3. The new Baratron head for the DP-West Fission Gas Sampling System has been calibrated and installed. During the initial installation, the head developed a large zero drift and since it was installed in a contaminated system, the repairs had to be made at LASL. The MKS Baratron Company provided repair parts and instructions. The repairs were made successfully and the DP-West Fission Gas Sampling System is now back in operation.

4. The new laser drilling/welding design for fission gas sampling has been completed. Construction has started and should be complete by the end of January.

Installation and testing should be completed by the end of February. The advantage of this system over the early design is the ability to easily reweld fission gas sampling holes for future element storage or reinsertion in a reactor.

5. The remote control cart and shielded operating table have been installed in Cell 5. The cart will be used to store up to 400 fuel elements. A 50-mm-(2 in.-) thick lead shield is being installed under the cell working table to minimize radiation damage to the cell window.

6. A welding fixture was designed and fabricated for the remote welding of modified alpha tubes to comply with the off-site regulations on shipment of fuels. A procedure was also developed and used twice successfully. However, a modified welding fixture and procedure are being developed to increase the reliability and consistency of the welds.

7. An alpha box previously used for the Spectrograph Arc-Spark Source is being modified to permit the installation of the General Electric O/M Potential Meter Apparatus. Liquid and gas supply lines, electrical power and instrumentation conductors, and inert gas purge and pressure control systems are being installed. Seals are being installed at all the box penetrations to permit the maintenance of an inert gas atmosphere.

8. Twenty-seven, new-type, Filter Rack Assemblies were designed and fabricated to simplify the replacement of HEPA filters in the Hot Cell Venting System.

9. An additional Vacuum Potting Fixture with heater assembly has been fabricated to improve the turn-around time of the Sodium Distillation Furnace. This unit will permit the removal of fuel samples from the furnace immediately after the distillation rather than utilizing the furnace to supply heat for curing of the sample potting resin.

A defective, time-delay relay was replaced in the control system of the Sodium Distillation Furnace Vacuum Pumping Station.

10. A replacement Macrophotography Sample Holder with black anodized surface was fabricated for the Metallography Section. The black surface is expected to improve the quality of the metallography sample macrographs by reducing reflected light.

11. A special welding fixture for the automatic welding of Residual Fuel Storage Capsule end caps in an inert atmosphere has been designed. The fixture is capable of being mated to any of the inert atmosphere cells via the "18-Inch Transfer Port." The fixture is capable of sealing stainless steel capsules of 12.7 mm (1/2 in.) or 19.0 mm (3/4 in.) o.d. by 0.89 mm (0.35 in.) wall thickness. Tubing lengths up to 1.55 m (61 in.) can be handled. The sealed capsules will provide inert storage for residual fuel sections and recapsulation of fuel components for off-site shipment. Fabrication is expected to begin next quarter.

B. Manipulator Maintenance

(A. L. Bridge, P. A. Mason, E. L. Mills)

1. A telescoping tube hoist cable required replacement on a General Mills Model 300 manipulator.

2. The hand adapter and the grip and wrist rotate drive motors of a GM Model 300 manipulator were accidentally hit and damaged by a hot cell shielding door while it was being raised. A spare manipulator was "cannibalized" to provide replacement parts.

3. A defective limit switch was replaced on the bridge drive of a GM Model 300 manipulator.

4. Six manipulators required major repair work during the last quarter.

C. Cell Window Maintenance

(F. J. Fitzgibbon, G. R. Herrera, J. M. Ledbetter, R. J. Reynolds, J. J. Villareal)

A procedure used by ORNL to remove deposits from the interior surfaces of lead glass windows was used successfully to clean two hot cell window assemblies at Wing 9. The window cleaning was performed by a filling and draining technique while the windows remained in place. The windows were restored nearly to the original state of clarity as judged by visual observation.

Following the draining of the mineral oil, a 1:1 mixture of toluene and isopropyl alcohol was used to rinse the oil from the glass surfaces. Glacial acetic acid then removed the deposit from the glass; rinses with toluene-isopropyl alcohol removed the acetic acid. This procedure required five days for each window assembly. This is a relatively inexpensive procedure as compared to the estimated \$5,000 cost per window for an outside contractor.

D. Shipping Casks

(F. J. Fitzgibbon, P. A. Mason, J. W. Schulte)

Construction of the new Radiography Cask continued during this quarter. Completion of the cask, which is being constructed under Quality Assurance Procedures, is expected next quarter. Approval will then be obtained for off-site shipments of radioactive materials.

Modifications to the last two (ten total) "Rover Casks" have been completed. The modifications will bring the casks into compliance with ERDA requirements. A LASL representative will inspect two casks at HEDL, prior to painting, to ascertain compliance of the casks as intended with the proposed alterations.

III. ANALYTICAL CHEMISTRY

A. Gamma Scanning

(J. R. Phillips, A. S. Murray, J. N. Quintana, J. R. Netuschil)

The primary data processing code has been modified by the addition of an option to detect automatically all the statistically significant gamma-ray peaks and to calculate parameters associated with each peak. At times, up to 800 individual gamma-ray spectra are processed with this new option providing the following information:

- (1) The average channel peak center and energy for all statistically significant gamma-ray peaks.
- (2) Identification of the gamma-ray peak and the correct energy.
- (3) The minimum and maximum channel numbers acceptable for plotting the particular isotopic distributions.
- (4) The number of individual spectra in which specific gamma-ray peaks occurred.
- (5) The slope and intercept of the energy versus channel plot for each spectra as well as an average slope and intercept value.

The new option has eliminated several manual data processing steps, thereby improving the efficiency of data analysis.

B. Electron Microprobe

(W. F. Zelezny, J. D. Farr, J. M. Hansel)

The shielded electron microprobe is being used in a preliminary investigation of the possible axial migration of burnup monitor Nd in mixed uranium-plutonium carbide

fuels. If neodymium migrates axially, the generally accepted mass spectrometric burnup technique would be adversely affected. In only one mixed carbide-sesquicarbide fuel, having undergone 7.8 at. % burnup, has any evidence been found for axial migration of neodymium and associated fission products.

In this particular sample a deposit of fission products on the top metallic (tungsten) insulator plug was determined to contain plutonium and significant concentrations of neodymium, cerium, praseodymium, palladium, and rhodium, but very little ruthenium, and no uranium. Ruthenium occurred in appreciably higher concentrations in the nearby fuel material.

These anomalous distributions of rare earth fission products were the first evidence of the axial migration observed by microprobe examination of irradiated carbide fuels. There has been some past evidence from gamma scanning data of the axial migration of cerium in mixed oxide fuels. Although these results are not conclusive, the anomalous rare earths migration indicates that this investigation should be continued in greater detail.

C. Measurement of Retained Fission Gases by Isotope Dilution Mass Spectrometry

(R. M. Abernathy)

An isotopic spike-mass spectrometric determination of fission product gases retained in irradiated reactor fuels was developed and put into semiroutine use. The internal standard is a mixture in argon of krypton and xenon enriched in their light isotopes. The mixture is prepared manometrically and verified by mass spectrometry. A precisely known amount of the internal standard mixture is added to the fission product gases as they are released during dissolution. The final mixture of gases is collected by cold-trapping and analyzed by mass spectrometry. The addition of the internal standard eliminates the requirement for quantitative collection of the gases and permits correction for any uncollected fraction. Quality Assurance procedures ANC-DE-1-MS-6, R 2 and ANC-DE-1-HC-11, R 2 describing this method were written and approved.

IV. MICROSTRUCTURAL ANALYSIS

(K. A. Johnson, E. C. Walter)

The technician cross-training program has continued this quarter in all the microstructural areas.

Ion gun etching is now being applied to irradiated carbide fuel on a routine basis.

A new (Mark VI) ion gun etcher incorporating operating experience has been designed and is now being fabricated.

Cut or roll film with automatic processing has been applied to the photocopy area. This leaves the remote metallograph the only photography area not yet using cut or roll film.

V. REQUESTS FROM DRRD

A. Examination of Irradiated Materials

(R. M. Abernathy, K. A. Johnson, M. E. Lazarus, R. A. Morris, J. R. Phillips, J. W. Schulte, G. R. Waterbury, W. F. Zelezny)

General Electric Company: Thirteen irradiated fuel pins were received during the second quarter of FY 76. Examinations performed on 15 fuel capsules are listed in Table 401-I.

TABLE 401-I

POSTIRRADIATION EXAMINATIONS OF CAPSULES AND PINS FROM GE

Examination	No. of Capsules or Pins Examined
1. Profilometry, Optical	6
2. Gas Sampling and Analysis	3
3. Sectioning	2
4. Burnup Analysis	1
5. Scribe Reference Marks	3
6. Diameter Measurements, Micrometer	1
7. Optical Microscopy ^a	3 (12) ^b

^aThe optical microscopy includes macrophotography, alpha autoradiography, beta-gamma autoradiography, and as-polished and etched photomicroscopy (including mosaics) in inert (N₂) atmosphere. Specimens from other experimenter's fuel pins were examined in like manner.

^bNumbers in parentheses in this and following tables refer to number of samples.

Hanford Engineering Development Laboratory:

Examinations performed on 21 irradiated fuel pins are listed in Table 401-II.

TABLE 401-II

POSTIRRADIATION EXAMINATIONS OF CAPSULES AND PINS FROM HEDL

Examination	No. of Capsules or Pins Examined
1. Preliminary Measurements	8
2. Profilometry, Optical	7
3. Radiography	1
4. Gas Sampling and Analysis	5
5. Sectioning	6
6. Density Measurements	1 (8)
7. Microprobe Analysis	2
8. Burnup Analysis	7 (21)
9. Scribe Reference Marks	1
10. U-Pu Analysis	4 (5)
11. ¹³⁷ Cs Analysis	5 (6)
12. Optical Microscopy	3 (36)
13. EMX Preparation and Photomicroscopy	5 (6)

Los Alamos Scientific Laboratory: This section contains carbide and nitride pins, the technical evaluation of which is being carried out by LASL personnel under the Advanced Fuel Program. Sixteen irradiated fuel pins were received during the second quarter of FY 76. Examinations performed on 31 fuel pins are listed in Table 401-III.

B. Other Requests

(K. A. Johnson, K. E. Dowler, F. J. Fitzgibbon)

Eight SEM samples of irradiated cladding were prepared and examined for ANL-W.

All required pre- and postirradiation data concerning 93 irradiated fuel pins slated for storage and eventual recovery at SRL have been received and assembled. Additional information related to the packaging safety and criticality analyses is still pending. However, per a request by HEDL, eight of these pins have since been returned for further examinations.

TABLE 401-III

POSTIRRADIATION EXAMINATIONS OF CAPSULES
AND PINS FROM LASL

Examination	No. of Capsules or Pins Examined
1. Visual Examination	18
2. Preliminary Examination	18
3. Profilometry, Optical	50
4. Radiography	2
5. Gamma Scan	
a. Gross	10
b. Multispectral	3
6. Gas Sampling and Analysis	3
7. Photography, Full Length	19
8. Photography, Incremental	19
9. Eddy Current	1
10. Sectioning	6
11. Density Measurements	9 (36)
12. Microprobe Analysis	2 (3)
13. Burnup Analysis	9
14. Pressure Leak Tests	2
15. Split Pin Cladding	2
16. NaK Removal	2
17. Optical Microscopy	5 (18)
18. EMX Preparation and Photo- microscopy	2 (4)
19. SEM of Irradiated Clad	3 (3)

VI. SUPPORTING WORK

Accountability of Fissile Material
(K. E. Dowler, F. J. Fitzgibbon)

The itemization of all fuel pins, fuel pin sections, and other accountable fissile material has been completed. Some portions of the existing computer program which were not applicable to the CMB-14 Group were used to tabulate and show location of nonaccountable items, such as metallography specimens and fuel pin hardware. This additional utilization of the computer program will be advantageous towards a complete itemized inventory of all fuel pin components and sections.

VII. QUALITY ASSURANCE

(L. E. Lanham)

General

1. A computer program has been prepared to list quality assurance procedures. The program has been prepared to give a better, more efficient method for quality assurance document control. The remaining task of entering procedures in the program will be completed as soon as manpower is available.

2. Because of the demands placed on the quality assurance in production areas, some reductions have been made in the manpower effort for the examination and analysis areas. This has been accomplished without reducing the quality assurance surveillance and document review efforts, but has delayed some quality assurance activities such as scheduled audits.

Analytical Chemistry

1. An audit was conducted by LASL Quality Assurance for chemical analysis operations being performed for the Examination of Fast Reactor Fuels and the More Advanced Fuels Program. A report has been written and approved by Program Management. Corrective actions have been taken on all findings of this audit.

2. The following documents have been reviewed and approved by Quality Assurance.

ANC-DE-1-MS-2, R2, Addendum 1 Determination of Atom Percent Fission in Uranium and Plutonium Fuel

ANC-DE-1-PA-4, R2, Addendum 2 Gravimetric Determination of Hydrogen and Carbon in Oxide or Carbide Fuels and Cladding Materials

ANC-DE-1-MS-3, R2, Addendum 1 Determination of the Isotopic Compositions of Uranium and Plutonium

ANC-DE-1-UA-34, R2 Determination of Carbon in Graphite

ANC-DE-1-RA-35, R2 Determination of Carbon in Sodium Metal

Hot Cell Operations

1. An audit was conducted by LASL Quality Assurance for hot cell operations being conducted for the Examination of Fast Reactor Fuels. An audit report was

written and approved by Program Management. Corrective actions have been taken on all findings of this audit.

2. The following document has been reviewed and approved by Quality Assurance.

401-DE-14-05, R3 The Use of the Optical
Profilometer on Fuel Elements
Microstructural Analysis

1. A quality assurance review of the completed microstructural data package has been initiated and is being documented on quality assurance checklists. This is in addition to the surveillance of operations and review of the data generated as each operation is being performed.

2. The following documents have been reviewed and approved by Quality Assurance.

MAS-DE-11-2, R3 Preirradiated Microstructural
Examination Quality Assurance Program
MAS-PP-11-3, R1 Photocopy Process
Nondestructive Testing

1. Revised checklists for nondestructive testing activities for both the Examination of Fast Reactor Fuels and the More Advanced Fuels Program have been prepared and are being used by Quality Assurance.

2. The following document has been reviewed and approved by Quality Assurance.

M-1-RDT-AR-1-R-1 Autoradiographic Procedure
to Confirm Pellet Fuel in U-Pu-C or U-Pu-N
Reactor Fuel Pin Assemblies

PROJECT AL 003

FBR ANALYTICAL QUALITY ASSURANCE STANDARDS AND METHODS

RESEARCH AND DEVELOPMENT

Person in Charge: R. D. Baker
Principal Investigator: G. R. Waterbury

I. INTRODUCTION

Reliable chemical characterizations are essential to the development of high-quality fuels, control rods, and other reactor components in the FBR Program. The objective tasks are designed to assure the quality of the chemical characterizations necessary to evaluate reactor components relative to specifications. Tasks include: (1) the continual preparation and distribution of carefully characterized calibration materials and quality control samples for use in quality assurance surveillance programs of vendor and purchaser analytical chemistry laboratory operations, (2) the development of and the guidance in the use of quality assurance programs for sampling and analysis, (3) the development of improved methods of analysis, and (4) the preparation of continuously updated analytical method manuals. At this time, the emphasis is on the FFTF. The program will be extended, as appropriate, to the LMFB demonstration and large production facilities in the future.

II. ANALYTICAL CHEMISTRY PROGRAM FOR LOW-FRICTION HARD SURFACES (J. E. Rein, R. K. Zeigler, G. R. Waterbury)

The objective of this HEDL-LASL cooperative effort is to provide an analytical chemistry program for the chemical characterization of chromium carbide hard surfaces to be applied as a molten blend to various structural components of the FFTF reactor. The analytical methods, a manual containing these methods, a QA plan covering the production operation, and most of the needed reference materials were developed or obtained

previously. High-purity chromium carbide powder has not yet been obtained. Two batches of powder from each of two vendors have failed to meet specifications. The second vendor has notified that no attempt now will be made to prepare the material and that the paid funds will be returned.

The proposed round robin evaluation of analytical capabilities still awaits transfer from HEDL to LASL of batches of Cr_3C_2 powder, nichrome powder, and hard surface product.

III. ANALYTICAL CHEMISTRY PROGRAM FOR METALLIC CORE COMPONENTS

(W. H. Ashley, E. A. Hakkila, M. E. Smith, J. E. Rein, G. R. Waterbury)

The draft of the HEDL-LASL Prepared PDT Standard F11-3, "Analytical Chemistry Methods for Metallic Core Components," was approved for issue by RRD with no significant changes.

IV. ANALYTICAL CHEMISTRY PROGRAM FOR BORON CARBIDE

(W. H. Ashley, R. D. Gardner, A. L. Henicksmann, F. R. Roensch, J. E. Rein, G. R. Waterbury)

The QA program associated with the production of boron carbide pellets as the FFTF control rod absorber material continues satisfactorily. The use of a selected pellet batch, pulverized and characterized at LASL, in the vendor and HEDL laboratories has essentially resolved the discrepancy in the soluble carbon determination. Based on evaluations using this material, HEDL adjusted conditions in the empirical method of analysis to obtain

results that are in agreement with the results obtained by the vendor and by LASL. These same conditions produce results for production materials that also agree with the vendor results. However, an important and difficult-to-control variable is the degree of pulverization of pellet samples. The finer the particles produced, the higher the results since the rate of the chemical reaction involving an oxidation of carbonaceous material with KMnO_4 increases. For example, our results for the special pulverized batch of pellets that had been provided to the vendor and HEDL laboratories were twice higher for < 200 mesh particles than for particles in the 100-200 mesh range. HEDL proposes to set aside production pellets which have been analyzed with agreement by the vendor and HEDL. Any new analyst will be trained with this material. Although very subjective, any alternate scheme, such as analyzing a measured particle distribution blend, becomes economically unfeasible.

V. ANALYTICAL CHEMISTRY PROGRAM FOR MIXED OXIDE FUEL

A. Calibration and Quality Control Materials

(J. V. Pena, F. R. Roensch, J. E. Rein)

The distribution of materials was continued to ARHCO as the supplier of the plutonium source materials,

to the FFTF fuel vendors, and to HEDL. Overcheck characterization of a new series of calibration blends of nonmetal impurities in $(\text{U}, \text{Pu})\text{O}_2$ matrix verified makeup values. For a new series of calibration blends of non-metal impurities in PuO_2 matrix, the overcheck characterization showed high and erratic fluoride levels. The fluoride level of all other PuO_2 matrix material on hand is too high to be useful. HEDL is attempting to obtain a batch of PuO_2 with suitable low levels.

A second batch of about 1000 $(\text{U}, \text{Pu})\text{O}_2$ pellets, received from HEDL, was packaged for use as quality control samples for surveillance of the O/M determination. The packaging involved hot-wire sealing of individual pellets in glass ampoules in a glovebox maintained in a dry, argon atmosphere. A third batch of pellets, to be sent by HEDL, will complete the packaging of this new series of quality control materials.

B. Reference Analysis

(J. W. Dahlby, C. H. Ward)

A second series of $(\text{U}, \text{Pu})\text{O}_2$ pellets randomly sampled from a production lot was analyzed for O/M ratio. The results for this series and for an earlier series were provided to HEDL for the purpose of establishing the cause for a small but consistent difference in the O/M measurements between a vendor and HEDL.

PROJECT AL 014

ANALYTICAL METHODS DEVELOPMENT - IRRADIATION TESTS

Person in Charge: R. D. Baker
Principal Investigator: G. R. Waterbury

I. INTRODUCTION

Necessary to the development of high-quality fuels and other reactor components required by the FBR program are highly reliable analytical methods for the measurement of burnup, oxygen-to-metal (O/M) ratio, and various gases in irradiated fuels.

The tasks in this program include: (1) the development and proof testing of burnup methods based on chemical separation-thermal ionization mass spectrometry, on analyses using inexpensive apparatus, and on spark source mass spectrometry for rapid, precise measurements, (2) the development of methods for the measurement of oxygen-to-heavy metal atom ratio (O/M) of unirradiated and irradiated fuels, and the establishment of conditions for the preparation of reference materials and (3) the development of methods for the measurement of gases and their release rates as functions of temperature and time.

II. DEVELOPMENT OF BURNUP METHODS

Burnup measurement methods are being developed and evaluated to complement isotope dilution mass spectrometry (ISDM)^{1,2} for applications that do not warrant the high precision ($\leq 0.5\%$ relative standard deviation) or in laboratories where a mass spectrometer is not available. The development of an initial chemical procedure in a planned series was completed and described as part of a review paper³ on burnup measurement techniques.

A. Chemical Techniques Using Inexpensive Apparatus (S. F. Marsh, M. R. Ortiz, J. E. Rein)

In the microtitrimetric technique currently being investigated, measurement of the total rare earth group (as the fission monitor) by reverse titrations of excess chelon with standard cadmium solution to a cadmium selective electrode end point provided insufficient sensitivity for submilligram levels of rare earths. A direct titration of a rare earth mixture (in proportions simulating FBR fission products) has been evaluated using ethylenediaminetetraacetic acid (EDTA) as the titrant, equimolar Cu-EDTA as an indicator, and a copper selective electrode as the end point sensor. At the end point, excess EDTA affects the copper-EDTA equilibrium which is sensed by the electrode. The system has measured 56 nanomoles of fission - simulated rare earths with a relative standard deviation of 1%.

A similar technique will be investigated for measuring plutonium, preferably as Pu^{3+} ion. This requires use of a reducing agent to produce and maintain Pu^{3+} that will not itself affect the copper-EDTA equilibrium and the end point detection. Uranium, reduced to U^{4+} , also would be determinable. A simpler system would be the determination of uranium in the stable (VI) condition state. It appears that the EDTA complex of uranium (VI) is not sufficiently strong, even at high pH, to be useful. Other complexants are being investigated.

B. Spark Source Mass Spectrometry (R. M. Abernathy, W. D. Spall, J. E. Rein)

A spark source mass spectrometer, equipped with both photoplate and electrical detector systems, was received at the end of this reporting period. Specification testing is under way.

III. DEVELOPMENT OF GAS MEASUREMENT TECHNIQUES

(W. D. Spall, R. M. Abernathey)

Operation of the dynamic gas release apparatus using NBS SRMs of metals with certified gas component contents has disclosed that gases released by furnace components produce an unsatisfactory background level. This condition was aggravated each time the furnace section was opened for introducing a sample. The design was such that the sample was placed directly into the crucible, thereby preventing an outgassing to attain a low blank level. The furnace section has been modified so that the sample can be introduced into a position above the crucible and then dumped into the crucible after a satisfactory blank level is attained.

The temperature programming portion of the induction furnace became completely operable. Various time-temperature profiles have been devised and their reproducibility tested. System temperature stability and reproducibility is about 2%.

Long-term pressure stability is about 3% caused mainly by fluctuating temperature in the sampling area. This stability may have to be improved for gas release experiments conducted over long time periods approaching 8 h.

Compositional measurements of evolved gases by gas chromatography have shown this to be a reliable technique provided that the gas composition is fairly well known before analysis to permit proper selection of the analysis conditions. The quantity of sample currently required for an analysis is 50 μ l. The measurement precision is about 5% standard deviation at this level using syringe injection. An improved method of sample injection is needed. It may also be necessary to use an internal standard technique to attain a desired precision level approaching 1-2%.

IV. REFERENCES

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