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LWR Fuel Reprocessing and Recycle Program Quarterly Report for Period January 1 to March 31, 1976

B. L. Vondra

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Printed in the United States of America. Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
Price: Printed Copy $4.50; Microfiche $2.25

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ORNL/TM-5447

Contract No. W-7405-eng-26

LWR FUEL REPROCESSING AND RECYCLE PROGRAM

QUARTERLY REPORT FOR PERIOD JANUARY 1 TO MARCH 31, 1976

B. L. Vondra, Program Manager

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MAY 1976

NOTICE This document contains information of a preliminary nature and was prepared primarily for internal use at the Oak Ridge National Laboratory. It is subject to revision or correction and therefore does not represent a final report.

OAK RIDGE NATIONAL LABORATORY
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FOREWORD

This quarterly report is the first in a series to be issued on "LWR Fuel Reprocessing and Recycle Program."

Eight tasks were assigned to UCC-ND with major efforts centered at the Oak Ridge National Laboratory. Because of the close relationship of the reprocessing activities with the ongoing LMFBR Fuel Reprocessing Program at ORNL, those tasks will be managed within a combined LWR/LMFBR Fuel Reprocessing Program under the direction of W. D. Burch, Program Director. B. L. Vondra, as LWR Program Manager, will coordinate all activities reported here. Responsibility for the coordination and collation of reports of all work at Oak Ridge was assigned to ORNL by SRO/SRL, who is designated as the lead organization for the LWR Fuel Recycle Program.

Projects for which budget and work plans have been established are as follows:

F-OR-02-001	SRO/LWR:	Assistance in Shear Development and Related Problems
F-OR-02-002	SRO/LWR:	Head-End Studies: Voloxidation and Dissolution
F-OR-02-003	SRO/LWR:	Off-Gases Fluorocarbon Absorption Studies
F-OR-02-004	SRO/LWR:	Purex Process Studies (Solvent Extraction)
F-OR-02-005	SRO/LWR:	Finishing Processes - MOX Fuel Fabrication
F-OR-02-006	SRO/LWR:	Uranium Hexafluoride Conversion
F-OR-02-007	SRO/LWR:	Economic Studies
F-OR-02-008	SRO/LWR:	Radiological Techniques for Environmental Assessments of the LWR Fuel Cycle

Progress is reported under the respective sections corresponding to the 189s. Each section heading is identified by the last digit in the 189 number. A scope is included for those projects which are just getting under way.

SUMMARY

An LWR Fuel Reprocessing and Recycle Support Program in support of the Energy Research and Development Administration (ERDA) program administered by Savannah River Operations - Savannah River Laboratory (SRO/SRL) has been established at Union Carbide Corporation's Nuclear Division (UCC-ND). A summary of the objectives and initial efforts for the eight assigned tasks is presented in this first quarterly report.

Two dissolutions were made using irradiated LWR fuel from the H. R. Robinson II reactor. Characterization of the solids and the resulting solutions is in progress. Preliminary data indicate that essentially all of the iodine was evolved during the dissolution.

General engineering design criteria have been completed for a model 1/2-ton-per-day voloxidizer.

1. ASSISTANCE IN SHEAR DEVELOPMENT AND RELATED PROBLEMS

C. D. Watson (Chemical Technology Division, ORNL)

The joint ORNL-SRL program, "Assistance in Shear Development and Related Problems," was initiated recently through two liasion meetings of SRL and ORNL staff members at ORNL. In the first meeting, a consensus of conduct between the two sites was agreed upon. In the second meeting, which was held on March 22, 1976, a preliminary program and a schedule for FY 1976, 1976A, and 1977 were outlined.

Basically, the initial effort will involve identification and determination of the unresolved LWR shearing and related head-end problems. A survey of industry and ERDA sites will be made for their respective inputs.

2. HEAD-END STUDIES: VOLOXIDATION AND DISSOLUTION

2.1 Voloxidation

D. C. Hampson (Chemical Technology Division, ORNL)

The voloxidation process is being developed as a head-end processing method for removing tritium from the fuel prior to aqueous processing. Based on experimental work, it appears that this objective can be met by heating the oxide fuel to about 450°C in flowing oxygen or air. Volatilization of tritium, and to a lesser extent some of the other fission products, is effected as the UO_2 becomes restructured during oxidation to U_3O_8. Early removal of tritium from the fuel into a relatively small volume is desirable to avoid extensive dilution of the tritium with water in the subsequent fuel dissolution step.

2.1.1 Experimental engineering studies

W. S. Groenier and C. H. Brown (Chemical Technology Division, ORNL)

The type of equipment being considered for voloxidation is a rotary kiln to which the sheared fuel is fed continuously and oxidized from solid UO_2 to a freely flowing U_3O_8 powder. During the oxidation and pulverization, a large fraction of the tritium should be released to a filtered off-gas system.

An existing small-scale kiln has been reactivated to obtain necessary engineering data on rotary kiln performance. The rotary kiln consists of a retort tube that is 7.5 ft (2.29 m) long and 6.5 in. (0.17 m) in diameter. Six mixing flights, which are 1 in. (0.025 m) deep, are equally spaced radially about the inner diameter of the retort tube and extend along the entire length of the tube.

A clamshell furnace which is about 2.5 ft (0.76 m) long and is rated at 16 kW at a maximum temperature of 2000°F (1100°C) surrounds the kiln tube. This furnace, which is positioned immediately downstream of the feed inlet, provides a heat source for the high-temperature oxidation reaction. Immediately downstream of the heated zone is an external water spray cooler which will provide cooling of the kiln tube and its charge. The available area for heat transfer in the cooler is about 2.2 ft^2 (0.21 m^2). Cold process water can be metered to the spray cooling jacket at flow rates up to 4.3 gpm (2.71×10^{-4} m^3/sec). Some heat transfer and residence time-distribution data, to be obtained over a period, will comprise the experimental program with the unit.

2.1.2 Component development

D. C. Hampson and E. L. Nicholson (Chemical Technology Division, ORNL)

A larger-scale experimental rotary kiln is being designed to provide engineering data on seals and heat transfer for the pilot-plant scale model (1/2 ton/day). The criteria for this unit have been completed. Efforts to process the unit through an industrial firm experienced with such equipment will begin immediately.

2.2 Dissolution Studies

D. O. Campbell and S. R. Buxton (Chemical Technology Division, ORNL)

An experimental effort, including hot-cell studies, was initiated in January 1976 in support of LWR fuel reprocessing. As a result of prior work, several potential problem areas had been recognized. These areas were primarily related to insoluble phases and nonionic species in process solutions. Some fully irradiated PWR fuel was dissolved

at ORNL about 1 year ago, and one conclusion from this work was that the presence of solids in real process solutions did cause difficulties.

The goals of this program include the study and definition of several suspected problem areas, as well as the identification of potential problems which have not been recognized in prior work. Recognized problems include (1) the presence of solids in dissolver solution because of the existence of material in irradiated UO_2 which does not dissolve in HNO_3; (2) the subsequent appearance of precipitates because of interactions of fission products or other instabilities in process solutions; (3) the generation of insoluble material due to process operations such as valence adjustment, denitration, or evaporation; (4) the existence of inextractable species, particularly plutonium, and (5) several long-standing questions such as the fate of iodine, tritium, and ^{14}C during processing. The reasons for concern about solids, which have previously been tolerated in chemical processing, relate both to the effect of solids on plant operability (plugging of equipment and heat generation) and to potential losses of material associated with the solids (especially actinides for which losses may have to be extremely small).

Accomplishment of the goals for this program requires highly accurate analytical data and physical measurements to characterize the materials. Because of the high radiation level of the irradiated fuel, severe handling problems may be encountered; therefore, an additional task is the development of methods to obtain representative

samples, which will be analyzed and characterized by the best means available at ORNL. Initially, several methods are being studied and cross-checked in an effort to obtain accurate and reliable results.

2.2.1 Hot-cell dissolution

During January 1976, glass equipment was assembled and tested for effecting the dissolution of irradiated UO_2 and scrubbing the off-gas from dissolution with NaOH. Two dissolutions of unirradiated, depleted UO_2 were made in the laboratory to test the equipment. The apparatus was then installed in a cell mock-up, and necessary modifications were made to facilitate manipulator operation. The equipment was subsequently assembled in Cell 4 of Building 2026 (High-Radiation-Level Analytical Laboratory), and a dissolution of unirradiated UO_2 was made in-cell.

In the first dissolution of irradiated fuel which was made in February (Run I), 250 g of irradiated PWR fuel (UO_2 fragments) from Westinghouse was dissolved. This fuel which has a somewhat uncertain history, is probably not representative of PWR reference fuel because the initial enrichment was higher and the irradiation time may have been shorter. The fuel was discharged from the reactor approximately 5 years ago.

In the second dissolution (Run II), which was made in March, 240 g of fully irradiated fuel (UO_2 fragments, about 30,000 MW/MTU) from the H. B. Robinson reactor was dissolved. Since this fuel was discharged in April 1974, its cooling time is as short as that for any fuel which will need to be processed commercially for many years to come. We have obtained a moderate supply of this fuel, including fully irradiated

UO_2 fragments and several sections of cladding of lower irradiation from the ends of rods, for use in several subsequent experiments.

Dissolution conditions were selected to approximate reasonably the projected conditions for the Allied-General Nuclear Services (AGNS) plant at Barnwell, South Carolina, whose proposed operating conditions are well defined. Air was drawn into the dissolver solution through a sparge tube at a flow rate of about 0.4 dissolver solution volume per minute. Off-gas from the dissolver flowed successively through a condenser, a condensate receiver, a backflow trap, two NaOH scrubbers in series, a control valve, and a small vacuum pump. One-half of the UO_2 was dissolved in one-half of the acid while the system was heated to about 90°C. Subsequently, the remainder of the UO_2 was added and the last half of the acid was slowly added as dissolution proceeded. The dissolution progressed very smoothly in all cases; no particular problems were encountered.

2.2.2 Radioactivity in off-gas

There is a considerable interest in the distribution of certain radioactive products, particularly ^{129}I, ^{3}H, and ^{14}C, between the dissolver solution and off-gas. Attempts are being made to measure the concentrations of these isotopes, as well as all gamma-emitting fission products, in each solution in the dissolution and off-gas train. At this time, the data for ^{129}I are complete and some results for ^{3}H have been received; methods for determining ^{14}C are still under development.

The distribution data presented in Table 1 show that greater than 99% of the ^{129}I can be removed from the dissolver solution into the off-gas. The total amount of ^{129}I found is given for (1) the dissolver, (2) the condensate plus wash solutions from the condenser and backflow trap, (3) the first NaOH scrubber, and (4) the second NaOH scrubber. In Run II the air sparge rate was lower, especially during times of maximum dissolution rate, and the dissolution time was shorter. Since less total air flowed through the system during this run it was expected that more ^{129}I would be left in the dissolver solution. Run I more nearly matched projected AGNS conditions.

Table 1. ^{129}I distribution during LWR dissolution studies

	Amount of ^{129}I found			
	Run I		Run II	
	mg	% of total	mg	% of total
Dissolver	0.15	0.52	0.32	0.97
Condenser[a]	0.039	0.14	0.36	1.08
1st NaOH trap	27.8	99.26	32.4	97.4
2nd NaOH trap	0.023	0.08	0.19	0.57
Total	28.0	100	33.27	100
ORIGEN[b] calculation	~34-?		33.0	

[a]Condensate plus washings from all equipment between the dissolver and the first caustic scrubber.

[b]For a discussion of the ORIGEN code, see M. J. Bell, ORIGEN - The ORNL Isotope Depletion Code, ORNL-4628 (May 1973).

The analytical method depends on oxidation of the ^{129}I and a stable iodine carrier to iodate in basic solution, acidification, and reduction to elemental iodine, which is extracted into CCl_4. Both ^{129}I and ^{127}I are determined by activation of this separated product. Because there was some question that equilibrium with the iodine carrier might not be established in the few minutes normally allowed prior to analysis, the dissolver solution from Run II was reanalyzed after being allowed to stand for several days. The reanalysis gave a value 80% of that shown in Table 1, which is reasonable since some ^{129}I may have been lost from solution during the three weeks between analyses.

The total amount of ^{129}I found in the system is in remarkably good agreement with ORIGEN calculations, especially when compared with the agreement obtained for other fission products. Reproducibility is rather good; thus this method appears to be adequate.

The limited data obtained for tritium are in agreement with the transport of water from the dissolver. The fractions of tritium found in the dissolver, the condensate, and the two NaOH traps were 98.4, 1.2, 0.4, and 9×10^{-4}% respectively. The total tritium found from 250 g of UO_2 was 7.3×10^{10} dis/min, which is appreciably lower than the amount calculated by the ORIGEN code. There are two sources of tritium in the ORIGEN calculations: ^{3}H present as a fission product from heavy elements, and ^{3}H formed by neutron capture in ^{6}Li impurity. Each source is predicted to contribute about 2×10^{11} dis/min.

2.2.3 Insoluble dissolution residue

All the dissolver solutions are black because of the presence of a small amount of finely divided insoluble material. In these studies, the solutions were transferred from the dissolver and centrifuged. Some of the solids that remained in the dissolver vessel were washed out with 3 M HNO_3 and similarly recovered by centrifugation. Several larger pieces of solids, including a Zircaloy end piece and several brown-colored fragments up to 0.1 in. across were present in Run I; in Run II there were no larger particles.

The total weight of solids (excluding the Zircaloy) was approximately 0.3% of the weight of UO_2 in each case. This is in general agreement with other data for fuel of this burnup. The solids are extremely radioactive, primarily as the result of the presence of ruthenium. The in-cell monitor indicated approximately 4000 R/hr per gram of residue for the 5-year-cooled fuel, and much more for the 2-year-cooled fuel.

The solids were leached repeatedly by refluxing in 3, 6, or 8 M HNO_3 solutions, each of which dissolves a fraction (but not all) of the material. This treatment gave a highly radioactive, red-colored solution characteristic of ruthenium species. Subsequently, portions of the leached solids were fused in Na_2CO_3 and dissolved in HNO_3; again, a red solution was obtained. It is not certain that the solids were completely dissolved by this treatment; thus dissolution procedures are still under development.

Preliminary gamma spectroscopy results show that the radiation is predominantly due to the ^{106}Ru-^{106}Rh pair and, in some cases, a

surprisingly large amount of ^{125}Sb. Samples are being examined by scanning electron microscopy (SEM). Microprobe analyses of those samples which had suitable radioactivity levels indicate the presence of molybdenum, ruthenium, and technetium. Alpha analyses demonstrate that these solids contain very little plutonium, americium, or curium after leaching (i.e., generally 0.01% of the total present in the initial fuel).

Analytical data are not yet complete for any run, and many reruns and cross-checks are being made for verification. The characterization of the solids is receiving major attention because they could cause severe problems from heat generation if they should segregate or accumulate in process equipment.

2.2.4 Solution stability

A second problem involving solids arises from the precipitation that occurs in clarified solutions during processing. Numerous samples of dissolver solution were aged for several weeks at temperatures of 70 to 90°C after adjustment to HNO_3 concentrations of 3 to 5 M. Most of the uranium and plutonium was extracted with tributyl phosphate from part of the dissolver solution, and samples of the raffinate were similarly aged. In all cases, small amounts of solids appeared in the solutions within a few days.

It was observed that solutions with higher acid concentrations produced solids which adhered to the glass walls of the sample tubes. Solutions of the lower acid concentrations did not show this tendency, but solids formed more rapidly. Although a number of solids samples have been submitted for analysis and SEM examination, results are not

yet available in most cases. Some of these solids appear to have a composition quite similar to a 2:1 molybdenum:zirconium compound precipitated from synthetic waste under similar conditions.

Both the dissolver solution and the extraction raffinate spontaneously change color from very dark brown to a much lighter color over a period of several days. This color change presumably results from the interaction of ruthenium with a radiolysis product; similar results can be obtained by the addition of nitrite ion to synthetic solutions.

Radiolysis products may also effectively adjust the plutonium valence for extraction since excellent extraction behavior was observed in both runs. Solutions were extracted after standing for several days; more than 99.7% of the plutonium was readily extracted.*

All interfacial "crud" from the TBP extractions was collected. This mixture of black "crud," organic, and aqueous solutions was washed successively with 3 M HNO_3 and dodecane to remove the entrained liquid phases. A final wash with acetone yielded a single liquid phase from which solids could be recovered by centrifugation. Only a few milligrams of solids resulted from each of the two runs, and this material has been prepared for analysis. It is possible that some solids were dissolved during the rather extensive washing to remove the entrained liquid phases.

---

* Detailed extraction studies are being made with some of these solutions in the Waste Stream Processing Program, which is supported by DPR, ERDA.

2.2.5 Characterization methods

Analytical results obtained thus far offer encouragement that we can, in fact, obtain meaningful data; however, several problem areas have been identified. Promising analytical methods include conventional alpha count analysis (including some chemical separations and alpha-pulse analysis), gamma spectroscopy for those isotopes which are appropriate gamma-emitters, spark-source mass spectroscopy (SSMS) for essentially all isotopes present in appreciable quantities, and SEM examination combined with X-ray fluorescence or microprobe analysis for samples which are not too highly radioactive to permit handling in unshielded equipment. Special methods must be used for certain elements such as ^{129}I, ^{3}H, ^{14}C, and ^{99}Tc. In general, methods that require chemical separations (including emission spectroscopy) are less desirable because of the possibility of fractionation during the separation.

Considerable effort is being devoted to the SSMS method because of its great potential. Initial results included a few serious discrepancies and somewhat marginal accuracy in most cases. However, we are investigating the use of selected separated isotopes as spikes to permit the determination of several important fission products by isotopic dilution and, at the same time, to provide better-quality standards for the remainder of the fission products. It is anticipated that this method will yield much more accurate results once improved procedures are developed.

The SEM and microprobe appear to offer an excellent approach to the study of solids; preliminary results are very encouraging. Since a shielded instrument is not available for handling high levels of

radiation, it is necessary to use very small samples. Similarly, a sample with a very high radioactivity level will swamp the X-rays from the microprobe, thereby preventing elemental determination. The use of small quantities of material for analysis introduces the problem of obtaining a representative sample. The best method appears to be to prepare a rapidly stirred, dilute slurry of the solids and to transfer a very small droplet to the sample mount.

We believe that, once procedures are better optimized, the methods presently being used will be adequate for detailed study of fully irradiated UO_2 fuel. Certain radiation effects must be compensated for, and some chemical procedures will be unsatisfactory unless this is taken into account. It is apparent that radiolysis products interact with ruthenium, as indicated by the color changes of solutions; they probably also affect the valences of elements such as plutonium.

The Robinson reactor fuel that was discharged from the reactor about 2 years ago is sufficiently radioactive that sample handling problems are difficult; however, it appears that current methods will be adequate for a detailed study of this fuel. It is interesting that, with present trends in nuclear power development, processing of shorter-cooled fuel will not be required until near the end of this century; accordingly, there appears to be no incentive for handling more-radioactive fuel at this time.

3. PUREX PROCESS STUDIES (SOLVENT EXTRACTION)

B. L. Vondra (Chemical Technology Division, ORNL)

This program involves the integration of laboratory development studies, flowsheet tests employing synthetic solutions, and confirmatory investigations in a hot-cell system using irradiated LWR fuels.

3.1 Laboratory Studies

3.1.1 Solvent cleanup

O. K. Tallent and K. D. Williams (Chemical Technology Division, ORNL)

The solvent from the Purex process is presently treated with sodium carbonate and sodium hydroxide to remove impurities before it is reused. This treatment generates liquid waste with a high sodium content and may result in an insoluble plutonium phase; both of these results are undesirable. Solvent has been successfully regenerated using macroreticular resins.[1] Regeneration of the resin requires $HF-HNO_3$ reagent.

We hope to develop solvent cleanup methods which do not produce a high-sodium waste and which serve to recover the plutonium values or result in their isolation from other waste materials. Initial emphasis will be placed on further study of macroreticular resins to confirm their utility and to investigate alternative regeneration methods. If regeneration is not practicable, the capacity of the resins will be determined and possible resin disposal methods will be assessed.

3.1.2 Plutonium valence adjustment

O. K. Tallent and K. D. Williams (Chemical Technology Division, ORNL)

Reduction of Pu^{6+} to Pu^{4+} before solvent extraction can be accomplished by treatment with nitrogen oxides. However, the kinetics of the reduction as a function of temperature, nitric acid concentration, plutonium concentration, and variations in the NO/NO_2 ratio need further study. To initiate this investigation, we plan to install a system for supplying known mixtures of NO and NO_2 and contacting them with plutonium solutions in a controlled manner. The kinetics of Pu^{6+} reduction by various $NO\text{-}NO_2$ mixtures will then be determined at several temperatures. The acceptable range of conditions for reduction of Pu^{6+} will be established from these studies.

3.1.3 Methods for removing iodine from fuel solutions

J. C. Mailen and D. E. Horner (Chemical Technology Division, ORNL)

Methods for removing iodine from fuel dissolver solutions are normally based on the destruction of nonvolatile organic iodides such as iodoacetic acid (by ozone treatment) followed by adjustment of the iodine valence to yield volatile elemental iodine (e.g., by addition of NO_x or hydrogen peroxide). The ozonation step is disadvantageous since ruthenium may be volatilized. Limited hot-cell tests indicate that iodine is more easily removed from radioactive solutions than from nonradioactive solutions.

We have found that peroxynitrous acid is capable of removing iodine from iodoacetic acid, apparently by oxidizing it to iodic acid (evidenced by a colorless solution immediately after reaction of I_2 with peroxynitrous acid).

It is known that nitrous acid reacts with hydrogen peroxide in acids to give peroxynitrous acid:[2]

$$HNO_2 + H_2O_2 \rightarrow HOONO + H_2O.$$

The peroxynitrous acid then decomposes to yield free radicals:

$$HOONO \rightarrow OH + NO_2.$$

These radicals have been shown to be capable of effecting the oxidation of bromide ion to bromine under conditions where neither peroxide nor nitrous acid alone has sufficient power.[2] Iodoacetic acid has been identified as a nonvolatile iodinated organic which is formed from iodine and acetic acid (a known contaminant in recycle acid[3]) in nitric acid under irradiation.[4] After the initial oxidation occurs, iodine color slowly appears in the solution, probably due to the reduction of iodate to elemental iodine by excess peroxide or nitrous acid. It was further determined that the addition of nitrous acid to a solution initially containing H_2O_2 such that the peroxide was in excess during much of the addition gave more complete iodine removals from the organic iodide (70% vs 40%) than when the reagents were added in the reverse order. Both nitrous acid and hydrogen peroxide are produced in irradiated nitric acid, with the nitrous acid being produced in large excess. Thus, although some peroxynitrous acid will be formed (and would aid in the destruction of organic iodides), it seems likely that the complete removal of organic iodides would benefit from artificially introduced, larger quantities of hydrogen peroxide.

We plan to determine the conditions under which peroxynitrous acid is most effective in decomposing organic iodides. Our objectives are to understand iodine behavior in fuel solutions and, possibly, to develop

processes that take advantage of peroxynitrous acid reactions. These investigations will include simulations of fuel solution conditions, in the absence of irradiation, to examine our ability to test iodine processing systems in a nonradioactive environment.

3.1.4 Chemistry of ruthenium

Many areas of ruthenium chemistry are of importance in fuel reprocessing; of particular interest are the formation of ruthenium solids and volatile RuO_4 during the fuel dissolution and the removal of RuO_4 from gas streams. The formation of various complexes with fuel solution components and impurities and their solvent extraction behavior are also of interest. Considerable information is available concerning the complexes of ruthenium with nitric acid and NO_x and the behavior of these complexes in the solvent extraction system; however, less is known about the formation of complexes with impurities in recycle acid and their solvent extraction behavior.

3.1.5 Program planning - hot cells

J. H. Goode, R. G. Stacy, V. C. A. Vaughen (Chemical Technology Division, ORNL)

We have begun the preliminary planning relative to desired improvements of the Purex process. Our studies in this area will lead to the construction of a small-scale Purex solvent extraction system in a hot cell. In addition, batch solvent extraction tests will be made to extend the basic equilibrium distribution data, with the aid of SEPHIS code predictions on radioactive feeds. Feed treatments will be developed to adjust the plutonium valence, and to treat certain fission products as needed.

3.2 Building 4507 Hot-Cell Operations

V. C. A. Vaughen (Chemical Technology Division, ORNL)

3.2.1 Head-end studies with LWR fuels

J. H. Goode, R. G. Stacy, E. C. Hendren, and O. L. Kirkland (Chemical Technology Division, ORNL)

Planning of program details has been initiated; the intent is to coordinate specific tasks to be performed at SRL and ORNL. This program will include experimental studies that encompass a number of fuels of varying irradiation histories from several fuel fabricators and reactors.

3.3 Cold Studies

W. D. Bond (Chemical Technology Division, ORNL)

Two mixer-settler units are being installed in Building 3508. Flowsheet conditions will be studied using synthetic solution prior to tests in the hot cells.

3.3.1 Development of in-cell equipment

In-cell reprocessing equipment previously designed and fabricated for on-going (intermediate 100-g scale) studies with LMFBR fuels will also be utilized for similar experimental operations involving fully irradiated LWR fuel. This equipment includes: (1) a new fuel shear with the capability of cutting 1/4- or 1/2-in.-diam rods into pieces up to 3 in. long, (2) a stainless steel voloxidation burner complete with furnace and rotary drive mechanism, (3) a stainless steel dissolver with its own paddle-stirring system and provisions for gas purging and solution refluxing, and (4) a continuous tritium monitor. This

equipment is depicted in the photographs shown in Figs. 1 through 5. A cold testing and modification program for the equipment is currently under way in the laboratory as the cell cleanout operation progresses at Building 4507.

A recent series of trial cuts was made using the fuel shear on 1/2-in.-diam porcelain-filled stainless rods simulating LWR-sized fuel. With each cut, only minimal deformation (pinching) of the ends was observed. We expect irradiated Zircaloy cladding to exhibit even more embrittlement (and hence less pinching) upon shearing.

Shakedown runs using chipped simulated fuel in the rotary voloxidizer have been made to test its mechanical operability and to obtain thermal profiles along its open end, where plateout of fission products from the burner off-gas is most likely to occur. Removable inserts for the collection of fission products will be used as part of deposition studies. Temperature measurements were made along this region for purge air flows from 50 to 500 cc/min and reaction chamber temperatures ranging from 550 to 700°C. Additional measurements at reaction chamber temperatures between 400 and 550°C are planned. The data that have been obtained indicate that the wall temperature profile can be estimated relatively closely.

Mechanical shakedown runs with the stirred dissolver have been made to eliminate problems caused by jamming of the sheared fuel. A series of nitric acid dissolutions of chopped (and voloxidized) pieces of cold UO_2 fuel rod is planned. These runs will serve to test the

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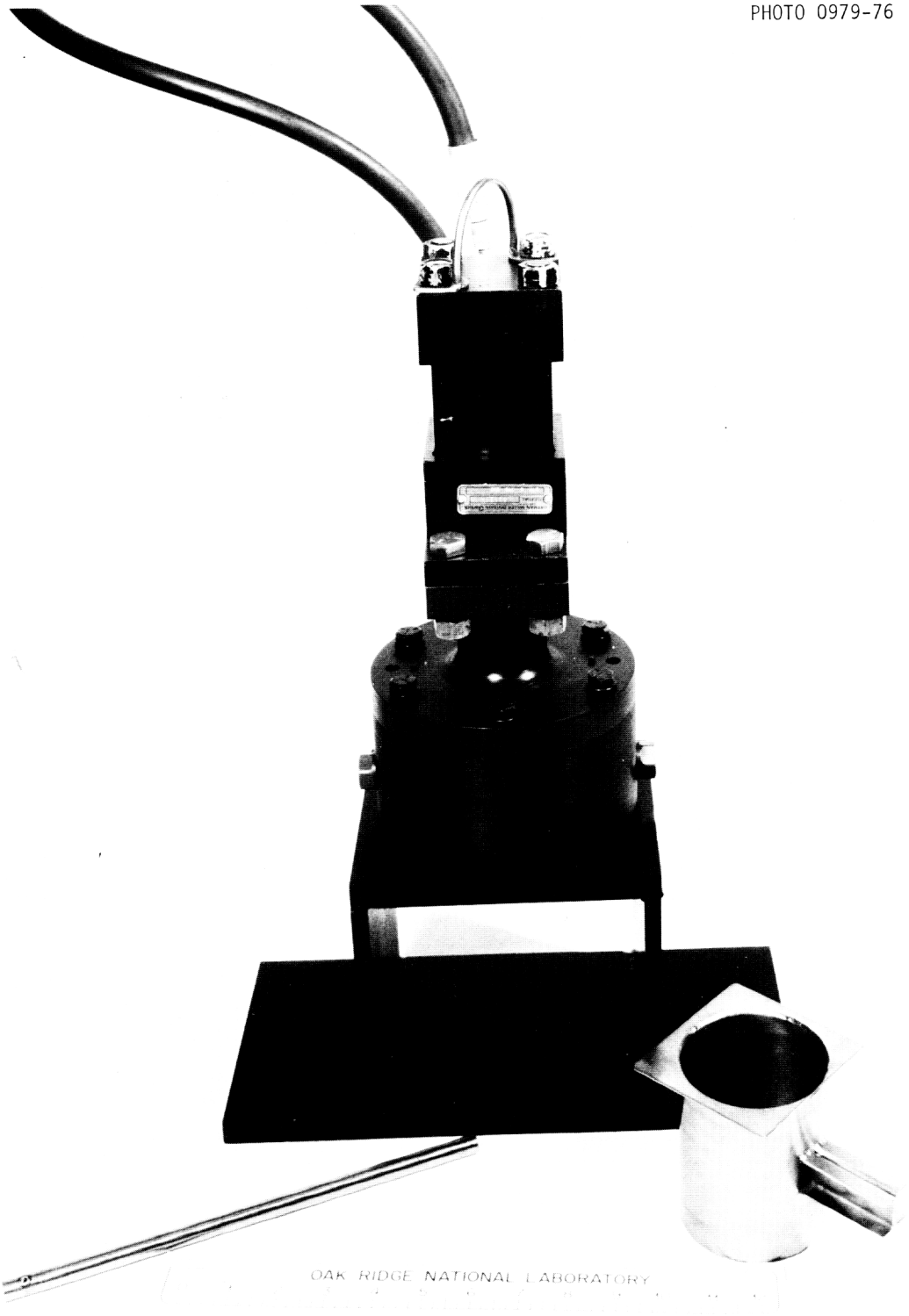

Fig. 1. Hydraulic shear for clad fuel.

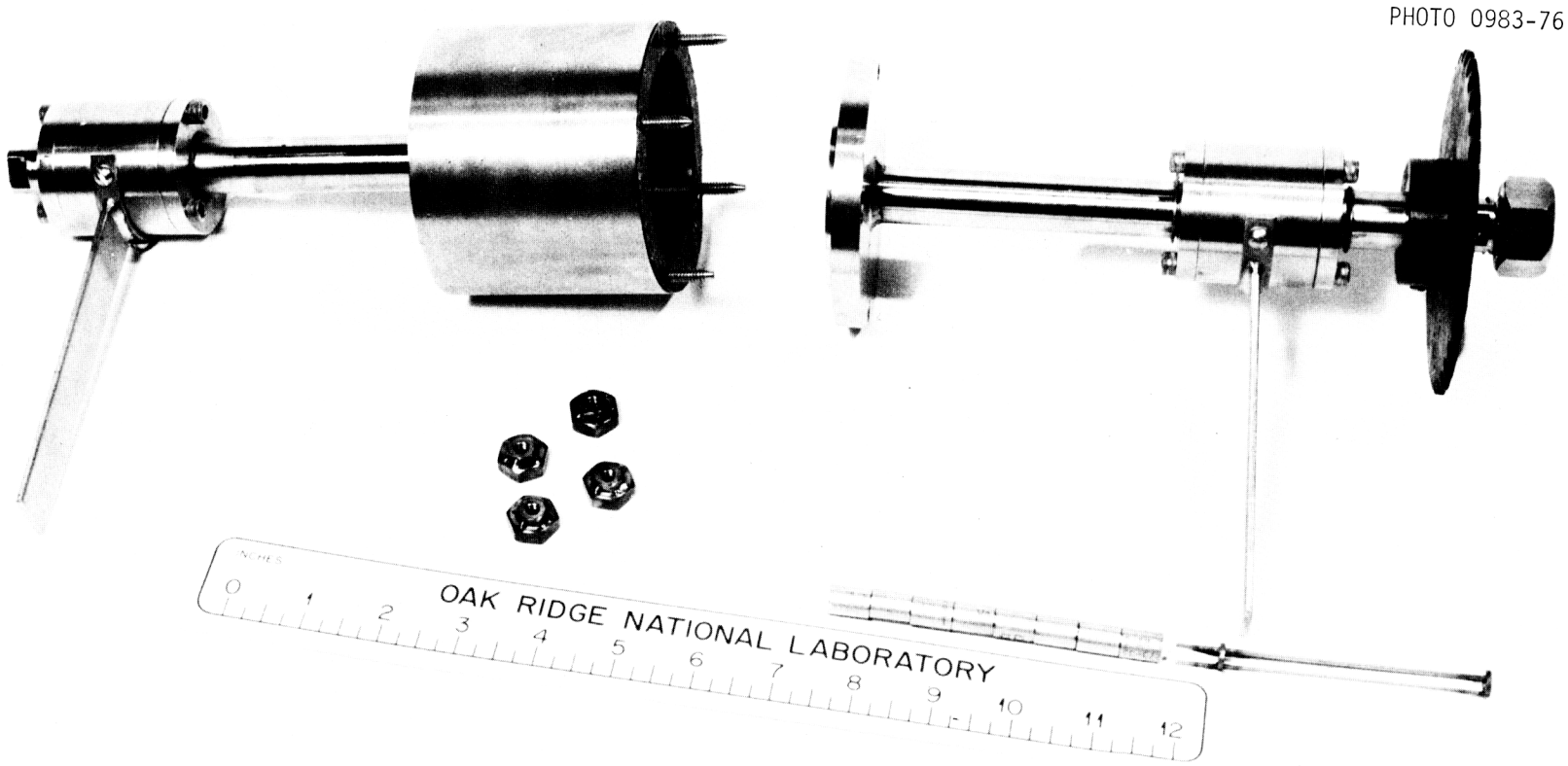

Fig. 2. Voloxidation burner (unassembled).

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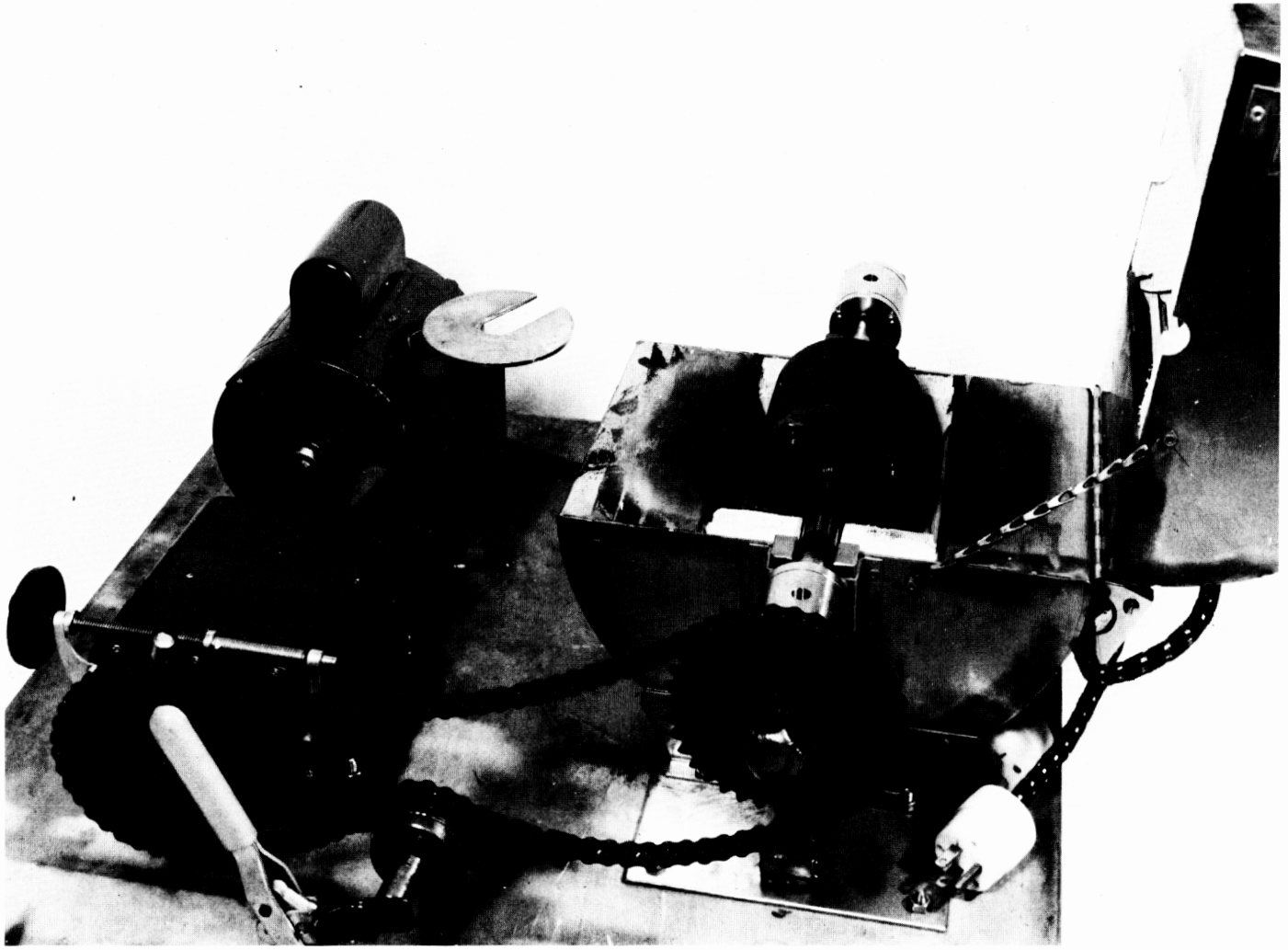

Fig. 3. Voloxidation burner with heater and rotary drive assembly.

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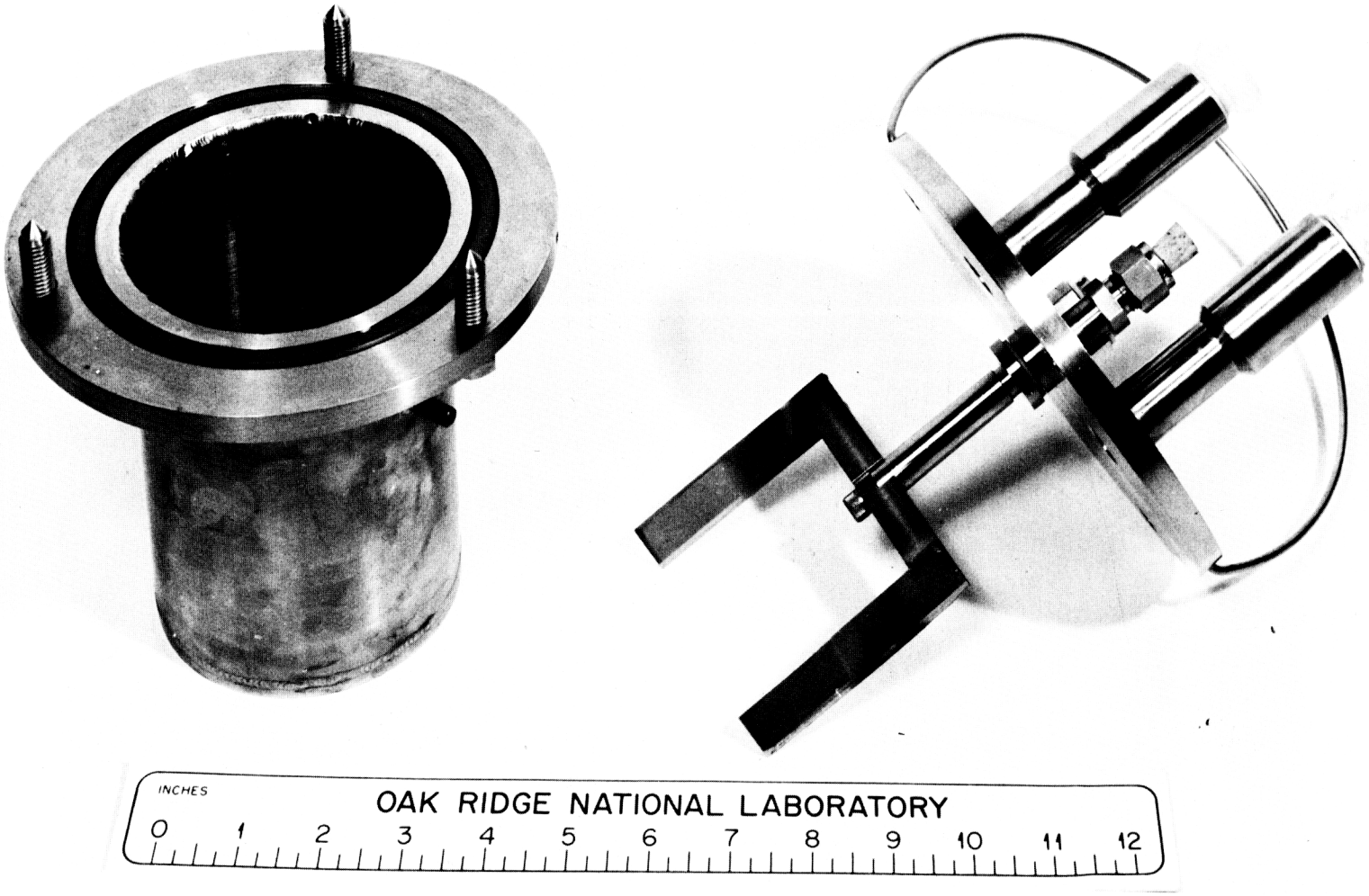

Fig. 4. Dissolver pot and paddle stirrer assembly.

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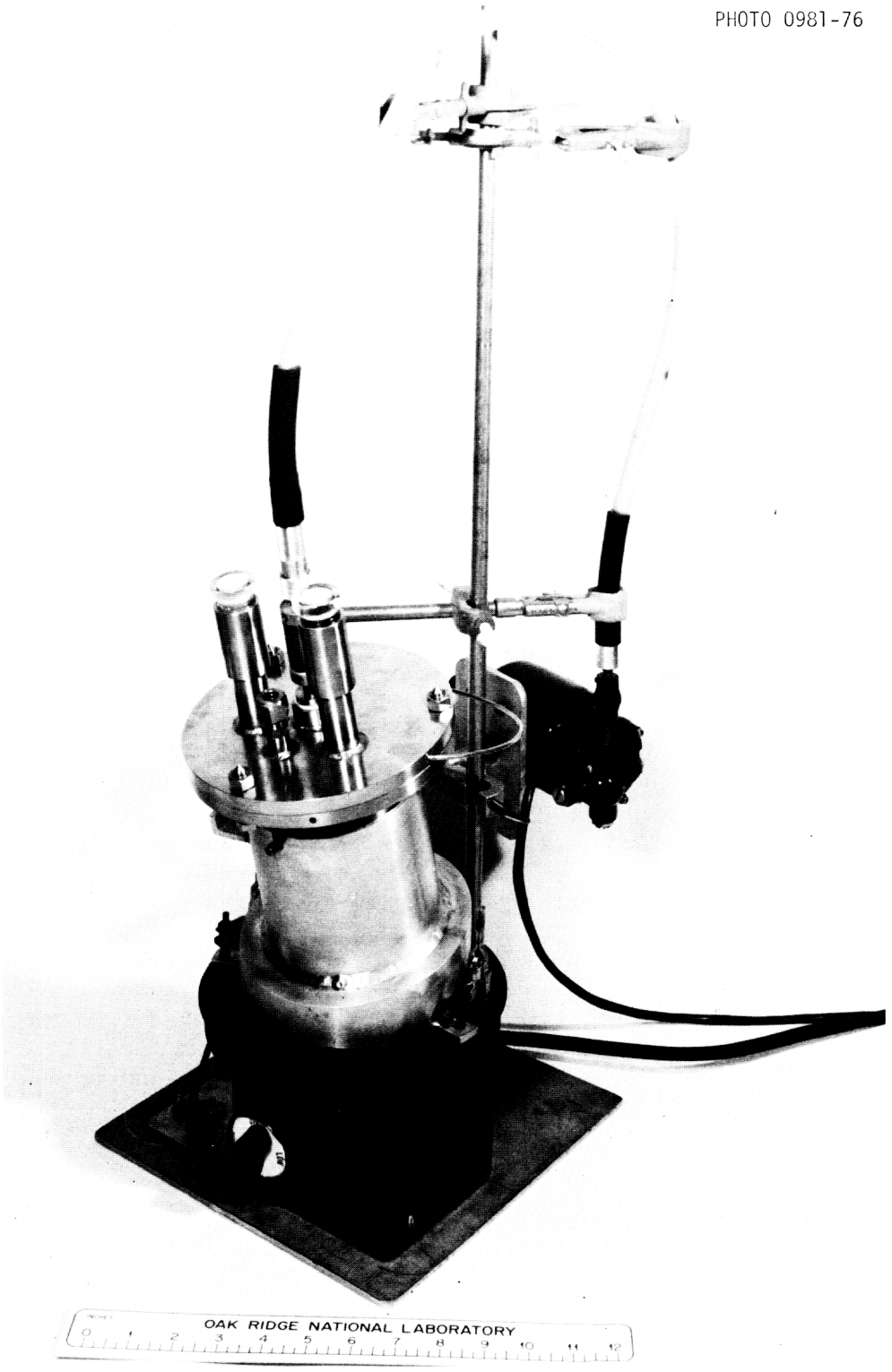

Fig. 5. Dissolver system (assembled) with heating system and direct-drive paddle stirrer.

integrity and durability of the burner and dissolver seals while providing data about the overall performance characteristics of the equipment.

3.3.2 Continuous tritium monitoring system

Developmental improvements and modifications are continuing to be made on the continuous tritium monitoring system (built by the Instrumentation and Controls Division) for the analysis of tritiated water (^3H_2O) in gas streams. Shakedown runs have shown that the monitor is capable of detecting the ^3H_2O content of simulated voloxidizer off-gases. The system provides for continuous measuring and recording of counting rates, and for integrating the total counts over a specified time interval. System response times to increasing concentrations of ^3H_2O in air are about 1 min. Additional calibration runs have been temporarily suspended until semipermeable dimethyl silicone membranes can be tested for their effectiveness in separating the tritium sample from ^{85}Kr (another component of voloxidizer off-gas). Our tests have shown that the counting signal from ^{85}Kr strongly overrides that of tritium even after the tritiated water has been scrubbed from the gas stream. A two-stage membrane system has been designed and is now under construction.

3.3.3 Equipment installation

The burner feed gas and off-gas systems have been designed. The layout of the voloxidation gas flow system to be used in subsequent hot-cell experiments is shown in Fig. 6. It provides for (1) a variety of feed or purge gases to be manifolded into the system, (2) measurement

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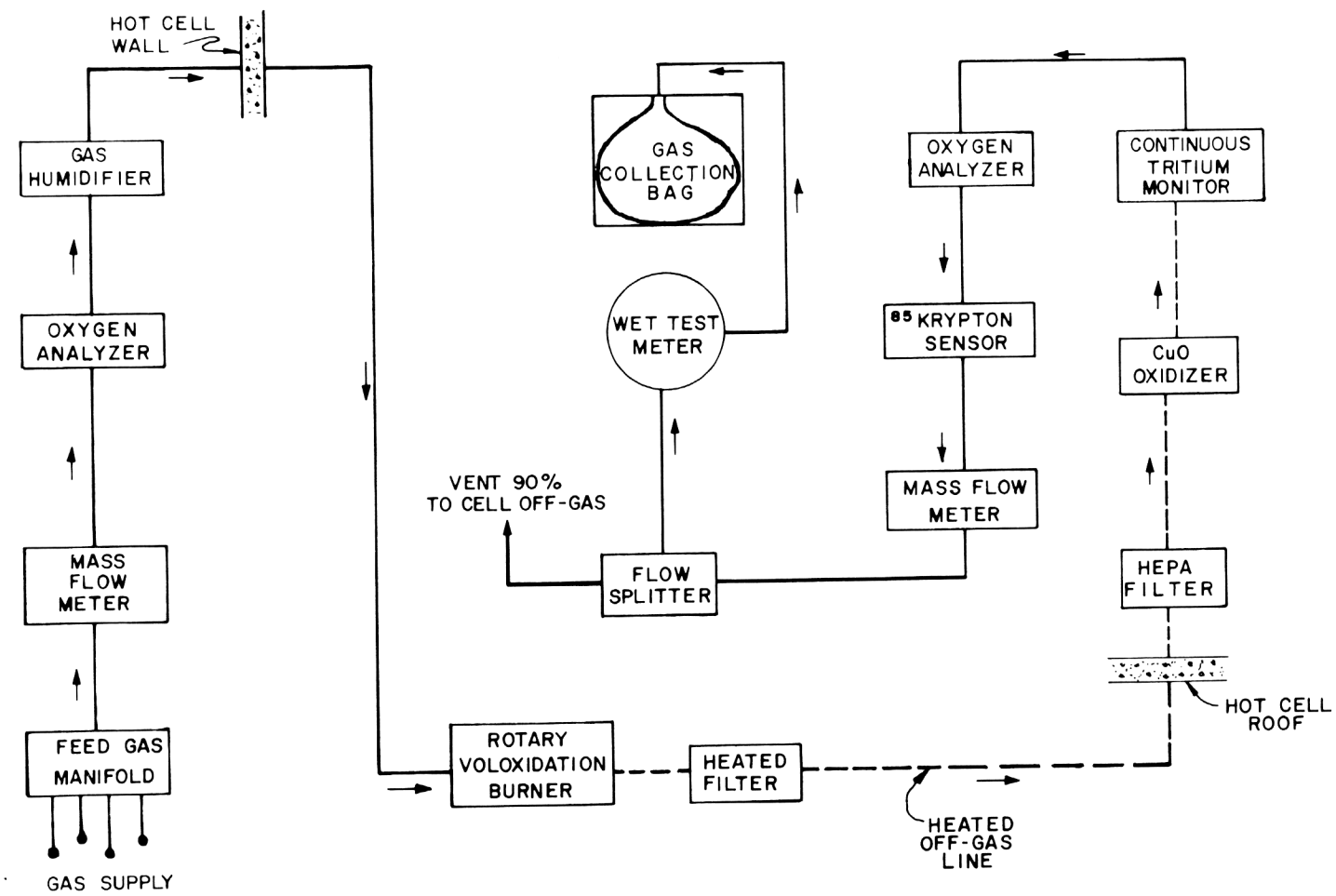

Fig. 6. Hot-cell voloxidation gas flow system.

of total mass flows and oxygen contents of both inlet and outlet gas streams, (3) continuous monitoring of the off-gas stream for $^{3}H_2O$ and ^{85}Kr, (4) collection of semivolatiles using removable deposition inserts, (5) trapping of particulates on disposable in-cell filters, and (6) direct sampling of the off-gas. Provisions have been made to add ^{14}C measurements, molecular sieves and/or silica gel traps, hygrometers, etc., as they are needed. Procurement of voloxidation control and monitoring equipment has been completed, and the feed gas manifold has been constructed. Cell refurbishment and installation of off-gas lines will continue as the cell cleanout permits.

4. OFF-GASES - FLUOROCARBON ABSORPTION STUDIES*

W. E. Unger (Chemical Technology Division, ORNL)

This program is concerned with the development of the Krypton Absorption process on an accelerated basis. Funding is provided for expansion of the pilot-plant development, initiation of plant engineering design criteria, performance of a proposed system reliability analysis, and a study of the chemical effects of impurities in the fluorocarbon solvent on the process and equipment.

4.1 Fluorocarbon Absorption Process Development for Off-gas Decontamination*

M. J. Stephenson (ORGDP)

The ORGDP effort involves the operation and analysis of a development pilot plant and engineering studies related to application of the process.

4.1.1 Pilot-plant operation

Pilot-plant work is being directed toward defining the general process behavior of feed gas components, such as carbon dioxide, nitrogen dioxide, iodine, methyl iodide, and water, and the effects of these components on the general operability and overall performance of the process. Tests conducted last year under the LMFBR program showed that carbon dioxide is significantly more soluble than krypton in Refrigerant-12 (R-12) and carbon dioxide removals in excess of 99.9% were measured under the same conditions where krypton removals

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* Jointly funded by LWR Recycle and LMFBR Reprocessing programs.

of about 99% had been achieved. The concentrations of CO_2 and Kr were 500 to 1000 ppm and 0.1 ppm respectively. More definitive tests are now in progress to establish: (1) solubility values for carbon dioxide in R-12 as a function of temperature, pressure, and other components in the system; (2) process removal performance as a function of various operating variables, (3) fractionation requirements, (4) product collection efficiency, and (5) product fixation schemes. Recent pilot-plant tests continue to show that carbon dioxide is significantly easier to remove from the bulk carrier gas than krypton. Final product stripping of carbon dioxide from the solvent was found to be slightly more difficult but well within the capability of the pilot plant stripper. The solubility of carbon dioxide in R-12 is close to that observed for xenon. Recent pilot-plant tests demonstrated that ^{14}C can be separated from the krypton product by a second intermediate fractionation step. However, available data indicate that a batch cold trap cooled to ~ -250°F with liquid nitrogen would be a more efficient method for separating the two components. In addition, the use of the cold trap would also allow direct separation and isolation of by-product xenon. A product cold trap is currently being constructed for pilot-plant evaluation and is scheduled to be in operation by late summer.

Pilot-plant tests continue to show that NO_2 is compatible with the process solvent. In these recent studies, which lasted for more than a month, nitrogen containing 50 to 500 ppm of NO_2 was continually fed into the pilot plant 5 days per week. Performance calculations indicate that greater than 99.9% of the incoming NO_2 was being removed from the feed gas, although experimental verification of this extent of removal

could not be made without special analytical equipment which is capable of detecting low-ppm quantities of NO_2. Such equipment is now being installed. However, available equipment did show greater than 90% removal. The results of these tests indicate that overall pilot-plant operability is not affected by moderate levels of NO_2 in the feed gas and also that the solvent is still an effective means for removing the higher-boiling components from the recirculating solvent.

The pilot plant was recently approved for ^{131}I studies. This required plant modifications and the formulation of special operating precautions to minimize the chance of an inadvertent release of iodine. Preliminary determination of the process behavior of iodine and methyl iodide will be completed in the fourth quarter of this fiscal year. Process calculations based on available data indicate that very high iodine recovery efficiencies are possible. Preliminary work with tritiated water is planned for early summer.

4.1.2 Process application

Process modeling and optimization studies have been initiated to identify the relative effects and importance of individual process elements and operating conditions on the overall system function. This work clearly shows that the overall effectiveness and efficiency of the process can be varied, depending on the judicious selection of the process operating parameters. For example, the effectiveness of any selective absorption or stripping process is a function of certain solubility differences between various constituents of a gas mixture

and the process solvent. When attempting to isolate one component from another, the closer the two solubilities are, the more difficult the separation becomes.

Another parameter to consider is the amount of krypton retained relative to the amount of nitrogen. Figure 7 shows the effects of flash temperature and pressure on the relative krypton enrichment of the solvent passing through the flash unit. These calculations show that, on this basis, lower feed temperatures and lower flash pressures are favored. In the final analysis, obviously, a balance has to be achieved between the degree of solvent enrichment and the amount of krypton desorbed.

Flash unit calculations are based on the assumption that thermodynamic equilibrium is maintained, and rate equations are not necessary in the quantitative description of the flash phenomena. Comparison of the calculated fraction of krypton flashed with the available pilot-plant values is given in Table 2. This comparison suggests that either equilibrium conditions were not actually achieved or krypton and/or nitrogen is slightly more soluble than the available data imply. All of the earlier tests were conducted with a constant liquid feed rate.

Modeling studies are still in progress and, other process components are being analyzed. Other pilot-plant data will be integrated into the basic model as it becomes available and, ultimately, a detailed design code will be evolved for overall process optimization and application.

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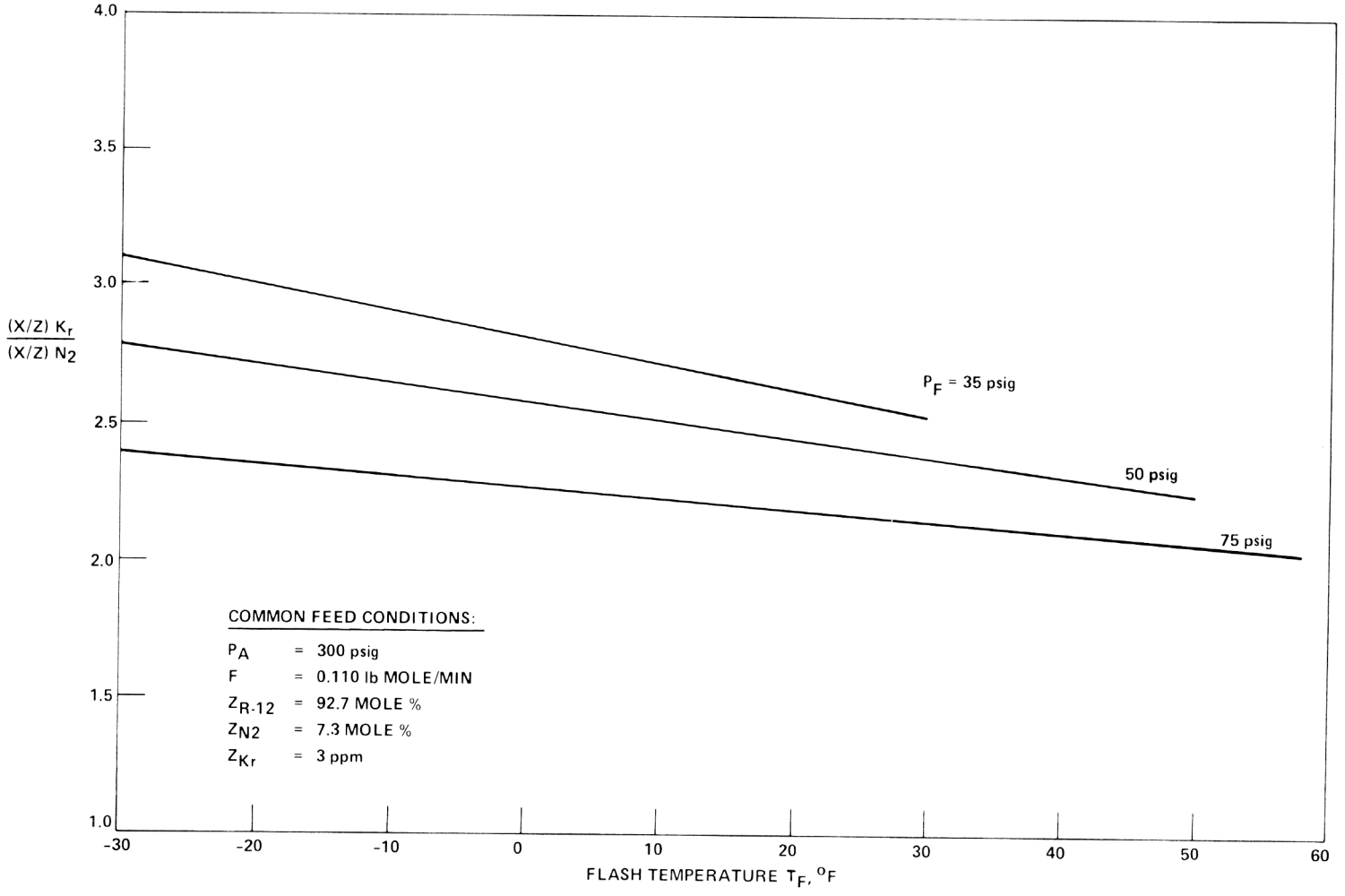

Fig. 7. Calculated amount of krypton enrichment during flash operation as a function of flash temperature and pressure.

Table 2. Comparison of flash calculations with measured values

Run series	Flash temperature (°F)	Flash pressure (psia)	Calculated fraction flashed		Measured fraction of krypton flashed	Percent of calculated
			Nitrogen	Krypton		
1-8-75	-15	64.3	0.850	0.606	0.516	85.1
1-21-75	-22	64.3	0.861	0.611	0.507	83.0
1-23-75	-24	64.3	0.863	0.610	0.490	80.3
2-25-75	-19	64.3	0.833	0.566	0.518	91.5
2-26-75	-19	64.3	0.825	0.551	0.515	93.4
2-27-75	-8	64.3	0.839	0.598	0.550	92.0
3-11-75	-9	64.3	0.865	0.645	0.544	84.4
3-12-75	-8	64.3	0.866	0.650	0.549	84.5
3-14-75	-9	64.3	0.881	0.677	0.556	82.2
3-19-75	-5	64.3	0.857	0.636	0.569	89.4
4-2-75	-4	64.3	0.873	0.670	0.559	83.4
4-3-75	-8	49.3	0.911	0.744	0.671	90.1
4-4-75	-11	49.3	0.898	0.711	0.654	91.9
4-8-75	-9	49.3	0.915	0.752	0.667	88.7
4-16-75	-3	49.3	0.908	0.747	0.686	91.9
4-17-75	-4	49.3	0.906	0.741	0.685	92.5
4-18-75	-3	49.3	0.908	0.747	0.676	90.5
4-22-75	-8	49.3	0.899	0.719	0.673	93.6
4-23-75	-2	49.3	0.928	0.800	0.720	90.0
4-24-75	0	49.3	0.906	0.748	0.705	94.3
4-25-75	-1	49.3	0.928	0.795	0.704	88.5
					Average	88.6

4.2 Chemical Studies of Contaminants in LWR Off-Gas Processing

L. M. Toth and J. T. Bell (Chemistry Division, ORNL)

We have begun an investigation which will utilize spectrophotometric techniques to study the chemistry of LWR fission products and processing products in the off-gases of LWR fuel reprocessing plants. The chemistry of I_2, H_2O, NO_2, CH_3I, and other off-gas contaminants in the chlorofluorocarbon solvents used in krypton-xenon isolation system is the primary interest. Reactions that cause corrosion or form solids will be particularly emphasized, and gas-liquid distribution coefficients for the various components will be determined. These studies are closely related to on-going design and testing of the krypton-xenon isolation system and will be used in future development of that system.

4.3 Reliability Studies

W. E. Unger (Chemical Technology Division, ORNL)

A reliability analysis of the krypton selective absorption pilot plant has been initiated. A subcontract has been awarded to Kaman Science Company, Colorado Springs, Colorado, for the study.

4.4 Engineering Design Criteria Studies

R. W. Glass (Chemical Technology Division, ORNL)

Work has begun on the development of engineering criteria which will lead to the design of a conceptual plant system.

5. FINISHING PROCESSES - MOX FUEL FABRICATION

T. N. Washburn (Metals and Ceramics Division, ORNL)

This program was initiated during this quarter. A meeting was held with representatives of SRL to define our role in the total MOX fuel refabrication effort. Visits were made to HEDL, General Electric, Exxon, NUMEC, and Westinghouse to discuss the current status of their efforts in MOX fabrication. A form 189 was prepared and submitted to ORNL and program management.

The major objective of this program is to assist SRL by providing technical consultation for the development of an overall program plan for LWR MOX fuel refabrication, for the establishment of design philosophy, and for the development of design criteria for MOX refabrication facility. An additional objective is to develop remote manipulation of viewing components for use in a LWR recycle facility.

6. URANIUM HEXAFLUORIDE CONVERSION

J. H. Pashley, J. E. Vath, R. P. Milford, J. Dykstra (ORGDP)

Uranium recovered in a nuclear fuel reprocessing plant must be converted to UF_6 if it is to be fed to uranium-isotope separation plants for reenrichment. Although UF_6 has been made from ore concentrate material for many years in satisfactorily operating plants, the processing of recovered uranium from power reactor fuels is currently not on a full-plant scale in the United States. Major differences arise with regard to virgin uranium because of the fission product and transuranic contents of purified uranyl nitrate hexahydrate (UNH) recovered from irradiated LWR fuel, even though the activity level may be relatively small. The objectives of this work are to evaluate existing technology for conversion of UNH to UF_6 in the light of these somewhat different problems posed by the use of recovered UNH as the input, to identify needs for development in the main steps and ancillary processes, including health and safety requirements, to pursue small-scale development on a limited basis, and, ultimately, to prepare cost-effective exemplary flowsheets and criteria for use in the design of UF_6 conversion plants.

The immediate goal is to accumulate available information on the processes involved in the conversion of UNH to UF_6 so that preliminary flowsheets may be formulated and equipment choices compared. Identification of needed development areas will then be possible based on this systems study which has just been initiated.

Preliminary discussions were held with staff members involved with uranium isotope separation at British Nuclear Fuels, Ltd. (BNFL) regarding

their experience with UF_6 recovered from irradiated fuels. Control of the transuranic problem, particularly for neptunium, and of fission products such as ruthenium were mentioned as matters of concern. Neptunium forms a volatile hexafluoride which is intermediate between UF_6 and the relatively easily decomposed PuF_6 in stability, while ruthenium not only forms a quite volatile fluoride but has a complex chemistry. Sampling and analysis for very low concentrations of these contaminants are problems of considerable technical difficulty. The possible use of cobaltous fluoride (for neptunium), magnesium fluoride (for technetium), and cryolite (for ruthenium) trapping was discussed.

7. ECONOMIC STUDIES

J. M. Morrison (ORGDP)

This task which was authorized on March 25, 1976, will support the fuel cycle development and demonstration programs being conducted by ERDA contractor organizations. Primary emphasis will be placed on the LWR fuel cycle, its relationship to the other nuclear fission cycles, and the interfaces among the various ERDA contractors as well as private industrial organizations involved.

8. RADIOLOGICAL TECHNIQUES FOR ENVIRONMENTAL ASSESSMENTS OF THE LWR FUEL CYCLE

S. V. Kaye, R. E. Moore and G. S. Hill (Environmental Sciences Division, ORNL)

Structures such as homes, factory buildings, offices, and schools, in which man spends most of his time, provide radiological protection from airborne radioactivity. Concentrations of radionuclides are less indoors than outdoors, resulting in a reduction of radiation dose through inhalation, air submersion, and exposure to contaminated surfaces. This project will evaluate the effect of man's largely indoor environment on estimates of population doses compared with the usual assumption of continuous out-of-doors residence. Equations were derived relating ratios of indoor-to-outdoor concentrations of radionuclides in air and surface concentrations. A computer code is being written to carry out a sensitivity analysis of the many parameters involved.

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