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LMFBR FUEL CYCLE STUDIES PROGRESS REPORT FOR JULY 1972, NO. 41

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3. Title LMFBR Fuel Cycle Studies Progress Report for July 1972, No. 41.

Author(s): W. E. Unger, et al

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ORNL-TM-3952

Contract No. W-7405-eng-26

CHEMICAL TECHNOLOGY DIVISION

LMFBR FUEL CYCLE STUDIES PROGRESS REPORT FOR JULY 1972, NO. 41

AQUEOUS FUEL REPROCESSING

W. E. Unger, R. E. Blanco, D. J. Crouse, A. R. Irvine, C. D. Watson

SEPTEMBER 1972

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LMFBR FUEL CYCLE STUDIES PROGRESS REPORT FOR JULY 1972, NO. 41

ABSTRACT

This report continues a series outlining progress in the development of methods for the reprocessing of LMFBR fuels. Development work is reported on problems of irradiated fuel transport to the processing facility, the dissolution of the fuel and the chemical recovery of PuO_2-UO_2 values, the containment of volatile fission products, product purification, conversion of fuel processing plant product nitrate solutions to solids suitable for shipping and for subsequent fuel fabrication. Pertinent experimental results are presented for the information of those immediately concerned with the field. Detailed description of experimental work and data are included in the topical reports and in the Chemical Technology Division Annual Reports.

HIGHLIGHTS

Biphenyl (a proposed cask coolant) was found to react with sodium to produce voluminous black solids. (Sect. 1.9)

A new procedure was developed for volatilizing iodine from nitric acid solutions. Using this procedure, about 99.9% of the iodine was volatilized in a single step. (Sect. 6.2)

AQUEOUS FUEL REPROCESSING

(W. E. Unger, R. E. Blanco, D. J. Crouse, A. R. Irvine, and C. D. Watson)

1. SHIPPING (TASK 1)

(A. R. Irvine, C. D. Watson, L. B. Shappert, J. H. Evans, G. A. West, D. E. Willis, D. E. Horner, and W. H. Baldwin)

The objective of Task 1 is to ensure that an economic and safe method of shipment of LMFBR spent fuel will be available when needed for transport of fuel from the demonstration and early commercial LMFBR's. The work involves analytical studies of the various facets of the problem; design, construction, and test of components, and of assemblies; and preliminary design of prototype casks.

The principal thrust of this effort is directed toward establishing that low-vapor-pressure liquid coolants can be adequately contained under conditions at least as stringent as those stipulated in the applicable regulations. In addition, the heat transport phenomenon inside a liquid-filled system is being studied. Experiments and calculations have demonstrated the overwhelming superiority of liquid sodium to gas and solid coolants from the standpoint of heat transport.

Reportable accomplishments include the following:

1.1 and 1.2 Heat Dissipation Studies and Tests (Tasks 1.1 and 1.2)

Fabrication of a new tube bundle continued for the electrically heated mockup of a fuel subassembly. Completion is now expected by about September 1.

The ducting for routing cooling air over the surface of the 37 subassembly cask mockup has been designed. A similar air ducting system has been designed for the single subassembly cask test. This system will provide an accurate indication of heat discharge from each portion of the simulated cask surfaces.

1.3 and 1.4. Cask Integrity Studies and Tests (Tasks 1.3 and 1.4)

A 2-in. Conoseal gasketed closure was thermally cycled three times without causing any leakage in excess of ~ 10^{-10} cm^3 He/sec (the lower limit of detection). The rate of change in metal temperature was commensurate with the calculated rate of temperature change of cask body temperature when the cask is exposed to a regulatory fire. The maximum temperatures that were attained were 400, 600, and 900°F, respectively, for tests 1, 2, and 3. The lowest temperature, 400°F, is the calculated maximum temperature that would be attained in a 30-min-duration fire.

Gray Tool Company completed the initial equipment setup for determining stresses in a closure seal plate. A photo-elastic technique is being employed. Initial trials showed good visualization of the stress pattern and gave an indication that stresses in the area of concern will be sufficiently low to allow the desired reduction in metal thickness. A reduction in the interference between the sealing surfaces will be required in order to avoid overstressing the plastic model and to permit good visualization of the stress pattern when the seal plate is in the fully closed position.

The design of a prototype cask access port and its actuating and testing tools is continuing. Tests to determine holdup of helium in crevices, sealing grease, etc., were unsuccessful owing to the presence of helium sorbers (rubber gaskets) in the leak detection equipment. These rubber parts have now been replaced by copper gaskets.

Completion of fabrication of two model casks was delayed, but both have now been completed. These two models utilize a thin member, backed up by a mechanically retained heavy section plug, for retention of cask contents at the fuel access end of the cask. Testing will be accomplished during the next report period.

1.9 Miscellaneous (Task 1.9)

Studies of the Reaction of Biphenyl with Metallic Sodium
(D. E. Horner and W. H. Baldwin)

Biphenyl is being considered for use as a heat transfer medium in casks used to transport irradiated LMFBR fuels to the reprocessing plant. Residual amounts of sodium, which will be used as the reactor coolant, will adhere to the fuel element when it is withdrawn from the reactor and will be available for reaction with the cask coolant. For this reason, we are studying the reaction between biphenyl with metallic sodium at elevated temperatures. In a preliminary test, sodium metal and biphenyl (~ 0.25 mole per mole) were allowed to react in an argon atmosphere at ~ 250°C for 6 hr. An estimated 50% or more of the sodium slowly disappeared with formation of a heavy black precipitate. No gaseous products were evolved. Because of the possibility that this precipitate may have resulted from the reaction of sodium with impurities in the biphenyl rather than with the biphenyl itself, a vacuum-distilled centercut of the reaction mixture (as a purified sample of biphenyl) was allowed to react again with more sodium. Again a heavy black precipitate formed, leading to the conclusion that the biphenyl itself must be reacting. We now plan to separate and identify the reaction products by various means and to determine the extent of reaction as a function of time and temperature. Later, other potential organic coolants will be studied.

Neutron Shield Development

The facilities for testing a simulated section of a cask neutron shield were almost completed. The neutron shield is a mixture of LiOH-NaOH and serves as a heat sink and as thermal insulation during the early portions of an accidental fire. The test is expected to be run next week. Calculations have been made which indicate that this arrangement should be effective in minimizing the effect of exposure of a cask to fire.

2. RECEIVING AND STORAGE (TASK 2)

(A. R. Irvine, B. B. Klima, and C. D. Watson)

This task is concerned with the means for rapid, effective, economical, and safe operation of receiving and storage facilities for LMFBR fuels.

No work was performed on this task.

3. HEAD-END PROCESSING OF LMFBR FUELS (TASK 3)

(C. D. Watson)

The objectives of this task are to develop economic head-end processing steps, in preparation for Purex recovery methods, for long- and short-decayed fuels.

Reportable accomplishments include:

3.1 Decay Heat Dissipation

(R. L. Cox)

The preparation of a report, "Calculated Temperatures Attained by LMFBR Fuels from Decay Heat Emission," by R. L. Cox was continued.

Thermal Conductivity of Sheared Fuels (S. D. Clinton)

The preparation of a report, "The Thermal Conductivity of Sheared Prototype LMFBR Fuels," by S. D. Clinton and R. L. Cox was continued.

3.2 Dismantling of Multitubular Assemblies

(G. A. West and J. C. Rose)

The experiments with a plasma torch for cutting the corners of hexagonal shrouds have been temporarily suspended during revision of the torch guide mechanism. The design and fabrication of a single probe guide to control both vertical and horizontal movements was continued by the Cecil Equipment Co., Inc.

Dismantling by abrasive belt grinding and milling type cutting methods continue to be evaluated for hot-cell shroud removal operations.

3.3 Shearing

(G. A. West)

The shearing of sodium logged prototype fuel rods with the multirod shear in an inert atmosphere was successfully demonstrated, and the product collected for internal sodium deactivation and voloxidation studies. A total of eight prototype fuel rods (0.305-in.-OD x 0.015-in. wall x 38.375 in. long stainless steel tubes filled with 0.270-in.-OD

UO_2 pellets with ∿2.836 grams of metallic sodium around and atop a 30.5 in.^{2} column of UO_2) were sheared singularly in the ORNL multirod shear. The shear was encased in a vinyl plastic tent to maintain an inert atmosphere of argon. Glove ports were provided in the plastic for placing individual rods into the feed envelope and for handling, collecting, and canning the product in inert atmosphere filled containers for transfer to other experimental equipment.

Minor modifications to the ORNL 250-ton shear were continued for studies of intact shearing of compacted hexagonal subassemblies.

3.5 Sodium Deactivation Studies

(W. S. Groenier and H. A. Connelly)

Sodium deactivation experiments were begun in Bldg. 3503 to obtain data on the evaporation of sodium from a molten pool and also from sheared LMFBR fuel rods that were internally logged with sodium. Theoretical mass-transfer correlations have been previously reported.[1] For forced convection, the evaporation rate of sodium can be expressed using the Sherwood, Schmidt, and Reynolds numbers.

$$Sh = 0.664\ (Sc)^{1/3}\ (Re_x)^{1/2}. \qquad (1)$$

The main experimental apparatus consisted of six feet of 1-in.-OD stainless steel tubing. The first one-foot section was packed with stainless steel York mesh and enclosed by a tubing furnace. The argon sweep gas was preheated in this section. The next one-foot section was also enclosed in a tubing furnace. The sodium to be evaporated was placed in this section. A 3/4-in.-OD nickel boat, capable of holding one gram of sodium, was centered in this section for runs designed to determine evaporation rates from a molten pool. For runs using internally logged fuel rods, the section was filled with a 40 in. fuel rod sheared into 1/2 in. pieces as described in subsection 3.3. The last section of tubing was packed with York mesh. There was no heat supplied to this section so the sodium would condense out of the argon sweep gas.

The experimental procedure for preparing sodium utilized a dry box where the charges were cut and weighed. Care was taken to minimize the oxidation of the sodium. The fuel rods were sheared in Bldg. 4505 and stored in the dry box until needed. With the system charged, the furnaces were turned on. When operating temperature was obtained, the argon flow was begun for a measured interval of time. The argon flow was then stopped and a small flow of air was begun. This step oxidized the sodium in place. At the conclusion of the run, the tubing was dismantled and water added to the sections containing sodium oxide. The resulting sodium hydroxide solutions were titrated to determine the amount of sodium present in each section. From these data, evaporation rates could be determined.

The results of Runs 1-5 are given in Table 3-1. An error in determining the temperature of the sodium was discovered after Run 3. As can be seen from the corrected values given, the operating temperatures for Runs 1-3 were near or above the boiling point of sodium (1616°F). Therefore, the usefulness of these data is limited. Runs 1-4 used a molten pool of sodium. Run 5 used sheared fuel rods.

The material balances were expected to be less than 100% due to losses of sodium in the system. A possible reason for the loss of sodium is the reaction of sodium with the stainless steel tubing. For the fuel rod run, there exists the possibility of a sodium-uranium oxide being formed. The argon sweep gas passed through a water bubbler before exiting through the off-gas system. Examination of samples from this bubbler showed only trace amounts of sodium after the five runs, thus proving the sodium was not lost in the argon sweep gas.

From the knowledge gained in these initial runs about the operation of the system and the techniques involved in handling the sodium and the fuel rods, more accurate data are hoped for in subsequent runs. Future runs are planned for both molten pools of sodium and sheared fuel rods.

References for Sect. 3.

1. LMFBR Fuel Cycle Studies Prog. Rept. for October 1971, No. 32, ORNL-TM-3624, pp. 8-16 (December 1971).

Table 3-1. Results of Sodium Deactivation Experiments

Run No.	Superficial Argon Velocity (ft/sec)	Sodium				Run Time (hr)	Temperature (°F)	Evaporation Rate	
		Initially (g)	Vaporized (g)	Remaining Unvaporized (g)	Total Accounted for (g)			Theoretical (lb/ft^2-hr)	Experiment (lb/ft^2-hr)
1	10.1	1.0	0.705	0.151	0.856	2.5	1697	---	0.246
2	10.1	1.0	0.625	0.080	0.706	1.5	1886	---	0.364
3	10.1	1.0	0.644	0.243	0.887	1.0	1335	---	0.562
4	5.0	1.0	0.229	0.980	1.209	3.5	1026	0.18	0.0571
5	5.0	2.94	0.965	0.345	1.310	5.0	1046	---	---

4. VOLATILE FISSION PRODUCT REMOVAL (TASK 4)

(D. J. Crouse and C. D. Watson)

The objective of Task 4 is to develop a head-end processing method for removing tritium from the fuel prior to aqueous processing. Based on information in the literature and our preliminary tests, conceivably this objective can be met by heating the oxide fuel to some reasonable temperature (450 to 750°C) in flowing oxygen or air (this treatment is termed voloxidation). Early removal of tritium from the fuel into a relatively small volume of gas is desirable to avoid large dilution of the tritium with water in the fuel dissolution step. In addition, the oxidation treatment has the advantage of converting any residual sodium in the fuel to the oxide prior to dissolution.

There was no reportable progress on this task this month.

5. DISSOLUTION (TASK 5)

(D. J. Crouse and C. D. Watson)

The objective of Task 5 is to ensure that LMFBR fuels can be dissolved in nitric acid with high metal recoveries. Since the dissolution characteristics of the fuels can vary widely depending on their plutonium content, method of preparation, and irradiation histories, extensive leaching data are being obtained to define the effects of the many variables. A thorough understanding of iodine chemistry in the dissolver system is needed as a guide for providing effective iodine control. The dissolver equipment must be designed and operated within rather narrow limitations imposed by criticality control and off-gas considerations. It appears that satisfactory solution of these problems can best be accomplished using a continuous dissolver and this approach is being emphasized. Evolution of a successful dissolver will require development of equipment for dependably moving the sheared stainless steel hulls and other solids through the system, and development of seals for isolating the system to prevent excessive inleakage of diluent gases.

There was no reportable progress on this task this month.

6. FEED PREPARATION (TASK 6)

(D. J. Crouse)

The aqueous feed discharged from the dissolver will contain solids (undissolved fission products, corrosion products, etc.) and will probably require clarification prior to solvent extraction. Preparation of the feed for solvent extraction also will include adjustment of the plutonium valence and the nitric acid concentration, and a treatment to remove iodine.

This month a new and more effective procedure was devised for volatilizing iodine from the fuel solution.

6.2 Iodine Control

(G. I. Cathers and C. J. Shipman)

We are studying methods for removing essentially all of the iodine from dissolver solutions prior to solvent extraction since this would facilitate iodine control in the processing plant. In our most recent tests, highly effective removal of iodine was obtained by sparging nitric acid solutions with a relatively large volume of N_2O_3 at a temperature below the solution boiling point. In earlier tests,[1] use of a high N_2O_3 concentration in a pressurized system was not particularly effective, possibly because the temperature in those tests was at the solution boiling point or higher. The present tests were conducted at 95 to 105°C (boiling temperature is 108°C) in order to enhance the solubility of N_2O_3 in the liquid phase.

Data for five runs are tabulated in Table 6-1. The residual iodine concentration in the solution after the first 10% distillation cut was 0.52, 0.12, and 0.072%, respectively, for test temperatures of 105, 100, and 95°C.

The procedure (discussed in previous reports) of readding iodine carrier, treating the solution with ozone, and redistilling was checked using N_2O_3 sparging in the second distillation. In the first three runs (222, 223, and 224), carrier readdition by itself (no ozone sparging) only lowered the ^{131}I concentration in the solution slightly in subsequent distillations due to lack of isotopic exchange. In the last two runs (225 and 226) carrier readdition followed by ozone sparging and redistillation resulted in the lowest residual iodine levels (0.002 to 0.007%) that we have observed in any of our tests. As described previously, the ozone treatment apparently serves to destroy small amounts of organic impurities in the solution that react and tie up the ^{131}I in a form (organic iodides) that is not exchangeable with the added carrier.

Effect of Fluoride and Aluminum. — Addition of fluoride to the dissolver solution may be required to obtain complete dissolution of plutonium. Several tests were run to determine if the fluoride might interfere with iodine volatilization. Since aluminum would probably be added subsequent to dissolution (to complex the fluoride and minimize corrosion), aluminum was also added to the system. The tests showed that iodine volatilization was decreased somewhat but that this apparently was due to the aluminum rather than to the fluoride.

The results of the tests are presented in Table 6-2. In run 229 with solution that was 0.05 *M* in F^- and 0.1 *M* in Al^{3+}, the amounts of residual ^{131}I in the solution after the first and second distillation cuts were 0.34% and 0.33%, respectively; these amounts were higher by a factor of 3 to 4 than the values obtained under the same conditions but without fluoride and aluminum present (runs 223 and 224 of Table 6-1). Results

Table 6-1. Effect of High Rate of N_2O_3 Sparging on Volatilization of Iodine from 4 *M* HNO_3

Procedure: 250 ml of 4 *M* HNO_3 with 2.5×10^{-4} *M* I_2 (added as KI and traced with ^{131}I) was distilled at 95 to 105°C while sparging with 300 ml/min of N_2O_3.

Run No.	Test Description	Temperature of N_2O_3 Sparging (°C)	Solution Distillation Rate (ml/min)	H_2O/N_2O_3 Mole Ratio in Vapor Phase	Residual ^{131}I in Solution (% of initial)
222	After first 10% cut	105	2.0	8.3	0.52
	After second 10% cut				0.33
	Readded carrier before third 10% cut				0.21
223	After first 10% cut	100	0.8	3.3	0.12
	Readded carrier before second 10% cut				0.082
224	After first 10% cut	100	0.8	3.3	0.12
	Readded carrier before second 10% cut				0.073
225	After first 10% cut	100	0.8	3.3	0.10
	Readded carrier and used ozone treatment[a] before second 10% cut				0.0069
	Readded carrier and used ozone treatment[a] before third 10% cut				0.0021
226	After first 10% cut	95	0.28	1.2	0.072
	Readded carrier and used ozone treatment[a] before second 10% cut				0.0072

[a]Ozone treatment consisted of 20-min sparge with ozonized oxygen at 75°C.

Table 6-2. Effect of Fluoride and Aluminum on Volatilization of Iodine from Nitric Acid

Procedure: 250 ml of 4 *M* HNO_3 with 2.5×10^{-4} *M* I_2 (added as KI and traced with ^{131}I) was distilled at 100°C while sparging with 300 ml/min of N_2O_3.

Run No.	Concentration (*M*) F^-	Concentration (*M*) Al^{3+}	Description	Residual ^{131}I in Solution (% of initial)
229	0.05	0.1	First 10% cut	0.34
			Readded carrier before second 10% cut	0.33
230	0	0.1	First 10% cut	0.38
			Readded carrier before second 10% cut	0.33
231	0	0.2	First 10% cut	0.51
			Readded carrier before second 10% cut	0.41

with only aluminum present in the solution (runs 230 and 231) were about the same as when the solution contained both fluoride and aluminum, suggesting that the aluminum rather than the fluoride was causing iodine retention.

References for Section 6

1. W. E. Unger et al., LMFBR Fuel Cycle Studies Progress Report for February 1972, No. 36, Sect. 6.2, ORNL-TM-3759.

7. SOLVENT EXTRACTION (TASK 7)

(D. J. Crouse and C. D. Watson)

The objective of Task 7 is to establish that LMFBR fuels can be processed successfully by solvent extraction methods.

Work on Task 7 has been suspended for FY 1973.

8. PLUTONIUM PURIFICATION (TASK 8)

(D. J. Crouse)

This task covers the process steps in purifying plutonium, starting with the plutonium product solution from the first Purex cycle and continuing through preparation of a product solution of adequate purity and concentration for delivery to the fuel refabrication operation.

Work on Task 8 has been suspended for FY 1973.

9. WASTE TREATMENT AND STORAGE (TASK 9)

Progress on this task is reported separately.

10. OFF-GAS TREATMENT (TASK 10)

(D. J. Crouse, O. O. Yarbro, and C. D. Watson)

Retention of iodine will be the major problem in treating the off-gas from processing of short-cooled LMFBR fuels since plant retention factors of 10^7 to 10^8 will be required. Retention of most of the xenon, krypton, and tritium also may be required for future processing plants. All promising off-gas treatment methods will be evaluated and additional chemical and engineering data developed where necessary. The most promising iodine trapping methods will be evaluated in test racks and, to some extent, at full-scale in the ORNL Transuranium Facility which processes short-cooled

irradiated targets. Studies are active in the areas of (1) scrubbing iodine species from gas streams with aqueous solutions, (2) decomposition of organic solvent vapors and organic iodides by catalytic oxidation, (3) testing of solid iodine sorbents, (4) separation of iodine and tritiated water, (5) study of the chemistry of uncommon volatile iodine species, and (6) removal of krypton and xenon from process off-gases by a selective absorption process.

This month additional data are presented for the scrubbing of methyl iodide from air with concentrated nitric acid solutions (Iodex process) and mercuric nitrate--nitric acid solutions. The rate of oxidation of I_2 to iodate in concentrated nitric acid was determined as a function of the acid concentration.

Engineering Studies of the Iodex Process
(W. S. Groenier and D. E. Spangler)

In an attempt to correlate decontamination factors (DF) observed in our experiments with methyl iodide and nitric acid, the logarithms of the DF values were plotted against the liquid to gas volumetric flow ratio $\left(\frac{L}{G}\right)$ that existed within the packed column (Fig. 10-1). This is the same approach that was used for elemental iodine and nitric acid (reported last month). Straight-line fits of the elemental iodine data resulted for each temperature and nitric acid concentration. For the methyl iodide data, the plot indicates that the relationship may be nonlinear.

In the case of elemental iodine, the theory describing mass transfer by gas absorption accompanied by rapid chemical reaction in the liquid phase predicts that

$$\ln(DF) = \frac{L}{G} D_a N \qquad (1)$$

where D_a is the mass distribution coefficient for absorption and N is the number of transfer units. Analysis of the methyl iodide process must include the absorption equilibrium of methyl iodide, the chemical reaction to form elemental iodine, the absorption equilibrium of this species, and the chemical reaction to form iodic acid. If we make use of simplifying assumptions of fast, first-order chemical reactions, the resulting equation is

$$DF = \left[\frac{D_{a_1}\frac{N_1}{N_2} + D_{a_2}}{D_{a_1}\left(1 + \frac{N_1}{N_2}\right) + D_{a_2}}\right]\left[\frac{1}{e^{-\frac{L}{G}D_{a_1}N_1} - e^{\frac{L}{G}D_{a_2}N_2}}\right] \qquad (2)$$

where subscripts 1 and 2 refer to CH_3I and I_2, respectively. Note that a ln(DF) vs $\left(\frac{L}{G}\right)$ correlation would not necessarily be linear.

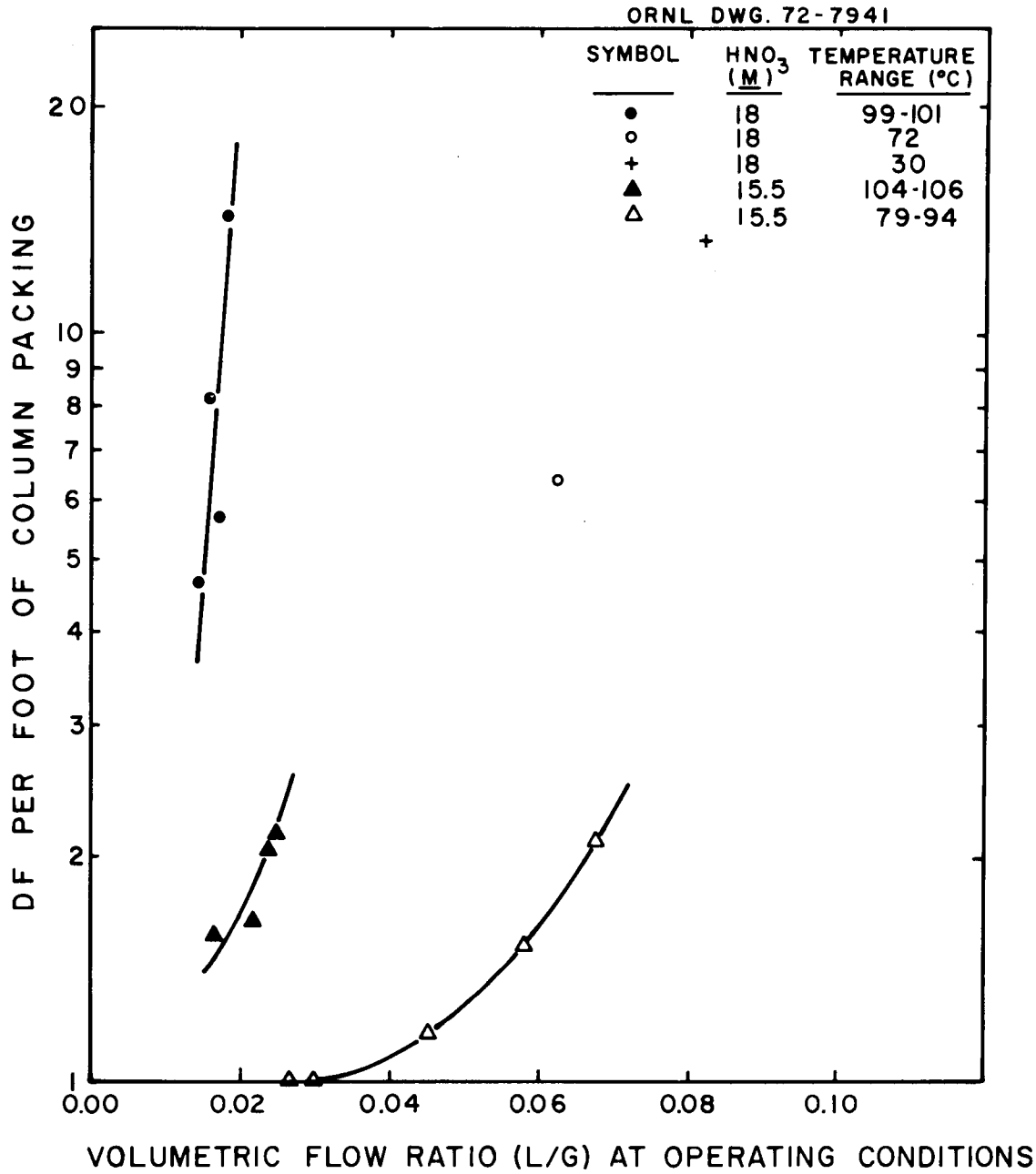

Fig. 10-1. Correlation of Methyl Iodide Decontamination Factors in the Iodex Process with Flow Ratio. Nitric acid flow was approximately 300 ml/min in all runs.

All data were obtained using the 4-cm-diam by 56-cm-long column packed with 1/4-in. beryl saddles. The DF values per foot of column packing were calculated from the DF across the entire 56-cm column and may not be uniform from each 1-ft-long section to the next. Future operation of a longer column is expected to clarify this point.

During the next month, runs will be made to ascertain if the DF decreases much with increased iodate concentrations in the acid. A recycled acid stream will be used successively in many runs, each of which will feature a separate measure of the DF using elemental iodine.

Spectrophotometric Study of the Rate of Oxidation of I_2 to HIO_3 in Concentrated Nitric Acid
(G. I. Cathers and A. H. Kibbey)

Study of the rate of conversion of free iodine (I_2) to iodic acid (HIO_3) in high concentrations of nitric acid is difficult because of the production of nitrous acid (or equivalent amounts of mixed nitrogen oxides) and the probability that the equilibrium is being approached. It can probably be assumed that, initially at least, the oxidation reaction is first-order. Determination of the first-order rate constants for different conditions is needed in interpreting engineering-scale data that are being obtained for the Iodex process. It appeared possible that a Cary spectrophotometer could be used to determine reaction rates since free iodine has an absorption band in the visible range. However, nitrite also has a visible absorbance, and it was not obvious that the two bands could be resolved sufficiently for quantitative work. Preliminary work with water and nitric acid solutions resulted in determination of the absorbance peak as a function of acid strength (Table 10-1). Since the absorbance of nitrite is in the range of 300 to 350 nanometers, this seemed to confirm that the spectrophotometric method might be used effectively. By using the same nitrite concentration in the acid that was placed in the reference cell of the double-beam spectrophotometer, the interference of nitrite was further reduced.

Table 10-1. Absorbance Wavelength of I_2 at Different Concentrations of Nitric Acid

HNO_3 Concentration (M)	Absorption Peak (nanometers)
0	460[a]
4	467.5
8	475
12	482.5
14	486
16	490

[a]Determined by W. Davis, Jr., of ORNL.

Attempts to determine the relationship between I_2 concentration and absorbance, i.e., to determine the molar absorptivity, ϵ, in 16 *M* acid were not successful since the I_2 converted quickly to iodic acid. Values of ϵ ($\epsilon = \frac{A}{lC}$ where $A = \log_{10} \frac{1}{T}$, l is path length in cm and C is the molar concentration) varied from 450 to 580 even in the presence of 0.1 *M* $NaNO_2$ which was added to prevent I_2 oxidation. An absorbance-time test was then made with 16 *M* nitric acid; the results are shown in Fig. 10-2 in the form of a semilogarithmic plot. The linearity of log absorbance (equivalent to log concentration) during the first part of the run indicates a first-order reaction rate. Similar tests were made with 14, 12, and 8 *M* acid. Essentially no reaction occurred at 8 *M*. The reaction half-times for 16, 14, and 12 *M* acid were, respectively, about 25, 160, and 450 min. The zero-time absorbances for 16, 14, and 12 *M* acid were identical at 0.7. For the concentration and cell lengths used, this gives a molecular absorptivity of 700 in all three cases, which compares with the value of 620 for the iodine-water system.

Effect of Exposure to Simulated Off-Gas on Service Life of Sorbents
(R. D. Ackley and W. H. Hinds)

Operation of the laboratory facility for investigating the effects of long-term exposure to simulated fuel reprocessing off-gas on the radioiodine trapping performance of various sorbents was continued. (Results obtained thus far, together with the pertinent experimental details, were presented in preceding issues of this report series.) Plans for this period had included determination of a sixth set of results corresponding to periodic injection of ^{130}I-labeled methyl iodide. However, some delay was encountered in obtaining the radioactive source, and, consequently, no additional results are available at this time.

Scrubbing of Iodine from Air Streams with Mercury Solutions
(J. M. Schmitt, D. J. Crouse, and W. B. Howerton)

Studies were continued of factors that affect the release of iodine from mercury scrub solutions. Previously[1] it was shown that the presence of hexane in air that was sparged through mercury solutions increased the amount of iodine released; the amount of iodine volatilized increased as the acid concentration was increased from 10 to 14 *M* and was larger when the iodine was in the iodate form than in the iodide form.

In the present test series, the mercuric nitrate--nitric acid solutions were sparged with ozone, prior to addition of the iodine, to remove organic contaminants. Also, the air stream was passed through CuO at 750°C prior to use to remove organic materials. In some cases, the sparge air (after CuO treatment) was contaminated with octane, hexone, or NO. With 0.4 *M* Hg^{2+}--10 *M* HNO_3 solution containing iodine as iodide, the measured value of D_G^L when sparging with pure air was slightly higher than 1×10^8 and this value did not change when the air was contaminated with octane or hexone (Table 10-2). With the iodine as iodate, the values with pure air were slightly lower ($4\text{-}7 \times 10^7$). In this case, the addition of octane or hexone

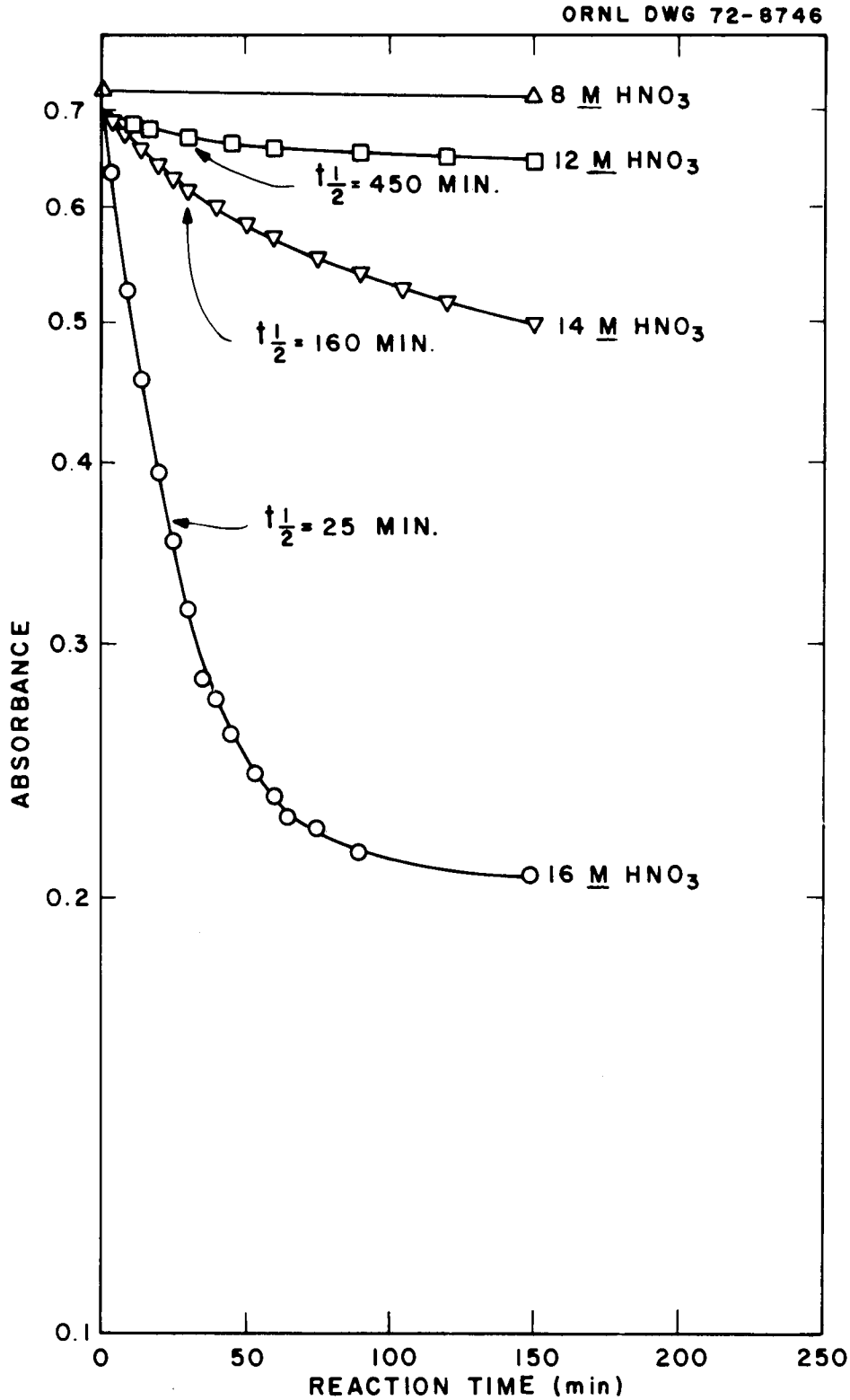

Fig. 10-2. Effect of Nitric Acid Concentration on the I_2 to IO_3^- Oxidation Rate. Initial I_2 concentration was 5×10^{-4} M; initial NO_2^- = 0. Cell length = 2 cm.

Table 10-2. Sparging of Iodine from Mercuric Nitrate--Nitric Acid Solutions with Air

Procedure: $Hg(NO_3)_2$--HNO_3 solutions containing 0.1 g of iodine (as I^- or IO_3^-) per liter were sparged with air that had been passed through CuO at 750°C to destroy organic materials and cooled; in some tests the CuO-treated air was contaminated with octane, hexone, or NO. Temperature was ~25°C in all tests.

Solution	Form of Iodine in Solution	Sparging Period[a]	Contaminant in the Sparge Air	Iodine Distribution Coefficient, D_G^L, for Incremental Sparging Period
0.4 *M* Hg^{2+}--10 *M* HNO_3	I^-	First	None	1.1×10^8
		Second	None	2.4×10^8
		Third	None	1.6×10^8
		Fourth	Octane	7.9×10^7
		Fifth	Octane	1.1×10^8
		Sixth	Hexone	2.1×10^8
		Seventh	Hexone	2.1×10^8
0.4 *M* Hg^{2+}--10 *M* HNO_3	IO_3^-	First	None	3.8×10^7
		Second	None	6.8×10^7
		Third	Octane	3.4×10^6
		Fourth	Octane	7.0×10^6
		Fifth	Hexone	3.6×10^6
		Sixth	Hexone	2.3×10^6
0.4 *M* Hg^{2+}--8 *M* HNO_3	I^-	First	None	1.0×10^8
		Second	Octane	1.4×10^8
0.4 *M* Hg^{2+}--8 *M* HNO_3	IO_3^-	First	None	6.9×10^8
		Second	None	4.8×10^8
0.4 *M* Hg^{2+}--8 *M* HNO_3	(I^-)[b]	First	None	1.9×10^8
0.4 *M* Hg^{2+}--8 *M* HNO_3	(IO_3^-)[b]	First	None	1.5×10^8
		Second	Octane	1.9×10^8
0.4 *M* Hg^{2+}--8 *M* HNO_3	(I^-)[b]	First	(NO)[c]	1.8×10^8
0.4 *M* Hg^{2+}--8 *M* HNO_3	(IO_3^-)[b]	First	(NO)[c]	1.7×10^8

[a]All sparging periods were 90 min.

[b]A new batch of iodine tracer was used to prepare the solution for these tests.

[c]The sparge gas contained about 7% by volume of NO.

to the system decreased the value of D_G^L, i.e., increased the iodine volatilization, by a factor of about 10. In tests with 0.4 *M* Hg^{2+}--8 *M* HNO_3 solution, the values of D_G^L were slightly above 1 x 10^8 and no effect from octane was observed when the iodine was in either the iodide or iodate form. Also, adding nitric oxide (~7% by volume) to the air did not change the rate of iodine volatilization.

Tests in Packed Scrub Columns. — Some additional data were obtained for scrubbing methyl iodide from air with 0.4 *M* $Hg(NO_3)_2$--10 *M* HNO_3 solution in a 1.85-in.-diam column packed with 30 in. of 1/4-in.-ceramic beryl saddles or 1/4-in.-ceramic Intalox saddles (Table 10-3). These packings gave about the same performance. The gas throughput capacity of the column for a given decontamination performance using these packings is only about 40 to 50% of that obtained earlier[2] with stainless steel packing. Surprisingly, increasing the solution flow did not increase the decontamination efficiency. The performance was essentially the same with cocurrent flow (run 50) as with countercurrent flow (run 51).

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1. W. E. Unger *et al.*, LMFBR Fuel Cycle Studies Progress Report for April 1972, No. 38, Sect. 10.1, ORNL-TM-3823.

2. W. E. Unger *et al.*, LMFBR Fuel Cycle Studies Progress Report for June 1972, No. 40, Sect. 10.1, ORNL-TM-3924.

Table 10-3. Scrubbing Methyl Iodide from Air with 0.4 *M* $Hg(NO_3)_2$--10 *M* HNO_3 Solution in a Packed Column

Procedure: Air containing about 0.05 mg of CH_3I per liter was passed countercurrent to scrub solution which was pumped from bottom to top of the packed column (1.85-in.-ID column with 30 in. of packing); solution inventory was about 600 ml; all runs were for 1 hr at 25°C; about half of the solution replaced with fresh solution every third run to prevent excessive buildup of iodine.

Run No.		Flow (liters/min)		DF	DF per Foot of Packing
		Air	Solution		
43[a]	1/4-in. beryl saddles	10	0.10	86	5.94
44[a]		10	0.35	86	5.94
45[a]		7.5	0.10	263	9.29
46		7.5	0.35	267	9.35
47	1/4-in.-Intalox saddles	10	0.10	110	6.55
48		10	0.35	79	5.74
49		10	0.06	64	5.27
50[b]		10	0.10	60	5.14
51		10	0.10	65	5.31

[a]Data reported earlier[2] included for comparison.

[b]Cocurrent flow of solution and gas.

11. ENGINEERING STUDIES (TASK 1)

(A. R. Irvine)

The purpose of this task is to examine the overall problem of LMFBR spent fuel recovery plant design and to relate the individual problems involved in fuel recovery to the overall problem and to the other individual problems. The plant design surveys which are to be made include adaptation of existing LWR reprocessing facilities for recovery of fuel from FFTF and initial demonstration reactors, possible adaptation of later LWR reprocessing facilities to accept LMFBR spent fuel from commercial reactors during the first decade (or more) of their existence, and finally, preparation of a conceptual design for a facility that will be needed when a large number of breeder reactors are in use. Individual problems to be evaluated range from special problems in safety and containment (which are peculiar to reprocessing of LMFBR spent fuel) to equipment design and operational procedures.

11.2 "Zero Release" Plant Study (Task 11.2)

A final topical report is being written summarizing the results of "Zero Release" study of fuel reprocessing.[1] The study identifies significant sources of activity to gaseous and liquid process waste streams and attempts to predict the behavior of important fission products as related to effluent treatment. The overall results of the study indicate that reduction of effluent activity orders of magnitude below present practices should be possible, assuming that the new fission product trapping systems now under development prove successful and that liquid and gaseous effluent volumes can be practically reduced.

Some of the more difficult development problems to be met in attaining these results include ways of getting the bulk of material into and out of the cell bank during routine operation while maintaining low air inleakage and good containment. Carrying out maintenance operations while preventing the loss of significant activity is a real challenge. If the cell bank is to be opened up and ventilated for maintenance, extensive decontamination will be required. Remote or semiremote maintenance while maintaining the "sealed cell" containment may be a better approach. Another area that needs more attention is the decommissioning phase of the plant's life cycle, at which time the inventory of tritium and other activities must be disposed of in such a way which will not release levels of activity significant in comparison to the total released during the effective lifetime of the plant.

11.3 Integrated Engineering Facility Study

(E. L. Nicholson, A. R. Irvine et al)

Work has continued on process definition, process criteria, and design selection of equipment related to the above subject. The various steps are being studied to determine those features which require plant-type demonstration as opposed to those which have been adequately demonstrated previously and can be accomplished in the most convenient method applicable to the operation. Equipment flowsheets are in preparation and preferred options for the various steps are being selected.

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