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QUARTERLY REPORT

TECHNOLOGY OF NON-PRODUCTION REACTOR FUELS REPROCESSING

BUDGET ACTIVITY 2790

Compiled by the Redox Technology Operation from contributions of the Chemical Processing Department and the Hanford Laboratories Operation of the Hanford Atomic Products Operation.

Submitted by V. R. Cooper

Research and Engineering Operation
CHEMICAL PROCESSING DEPARTMENT

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QUARTERLY REPORT

TECHNOLOGY OF NON-PRODUCTION REACTOR FUELS REPROCESSING
BUDGET ACTIVITY 2790

This report summarizes the research and development work carried out during March, April, and May, 1959, for Budget Activity 2790 - Separations Development for Non-Production Reactors. The effort on Activity 2790 will enable Hanford to begin reprocessing in January, 1962, the fuel elements from power reactors which employ depleted or slightly enriched uranium fuels.

COORDINATION

Shipping

Activity on the problems associated with the shipment of fuel elements included:

(1) Visit to Hanford by M. Gleason of the MARAD-AEC and H. T. Loeser of Electric Boat Division of General Dynamics Corporation to discuss shipping and reprocessing of irradiated fuels from the Nuclear Ship Savannah. They also observed demonstrations and exhibits of remote handling equipment and techniques.

(2) Trips to Washington D. C. by R. G. Geier on May 4-6 and by N. Ketzlach on May 25 to discuss shipping cask criteria.

(3) Submission on various aspects of the shipping problem were made as follows:

  (a) "Cask Acceptance Criteria, Appendix C-HAPO" was issued on May 8, 1959.

  (b) "Design Criteria 213-U Building Cask Transfer Facility," by M. E. Yates, was issued for comment on June 19, 1959.

PROJECT ACTION

AEC Directive, HW-483, in the amount of $700,000 authorizing complete design for Project CG-830, was issued March 30, 1959.

Architect-engineer service requirements were set forth in a letter from H. P. Shaw to D. J. O'Neil, U. S. Atomic Energy Commission, "Architect-Engineer Services for Reprocessing Power Fuels," dated May 12, 1959, with attachments including HW-60247 "Preliminary Description of the Plant for Design of a Non-Production Fuels Reprocessing Facility," by L. B. Bradley, dated May 8, 1959.

HW-60436, "Parameters of Dissolver Equipment Design," by W. A. Graf was issued on May 20, 1959.

HW-59345 Rev. 2, "Scope Design Basis Non-Production Fuels Reprocessing", by M. L. Oldfather was transmitted to J. T. Christy, U. S. Atomic Energy Commission, with a letter of same title by H. P. Shaw, on June 11, 1959.

HW-60553, "Project Proposal, Revision I, Plant Modifications for Reprocessing Non-Production Reactor Fuels", was issued on June 9, 1959.

The development work required for support of the scope design effort was studied, and a listing of pertinent information required was compiled and published by the Hanford Laboratories Operation. Assignment and scheduling of test work to obtain the required information is in progress.

CURRENT PROCESSING CONCEPTS

Current planning calls for location of the receiving and storage, mechanical treatment, dissolution, clarification and solution storage facilities at the Uranium Recovery Plant. Dissolver solution is to be piped to the Redox Plant for separation and decontamination of the uranyl and plutonium nitrates. The conversion of the uranyl nitrate to UO_3 will be carried out in a geometrically-safe agitated trough calciner to be installed at the present uranium calcination facility. No further treatment of the plutonium nitrate is planned in the present project.

Research and development work is being narrowed to apply the major effort toward the more promising and immediate goals with lesser consideration for the less attractive or very long range goals. The scheme outlined below appears to be the most suitable one for satisfying Hanford's responsibilities in the non-production fuel reprocessing program.

Receiving and storage facilities will provide a cask transfer station so that an incoming cask can be transferred to a captive railroad car for subsequent unloading in the Uranium Recovery Plant. Fuel elements will be stored in large tanks converted for this purpose. Minor decontamination of the empty cask, if required, will be carried out at the cask transfer station.

The primary objective of the mechanical treatment facility will be the removal of end fittings and minor disassembly to permit charging of the fuel element to a dissolver. Somewhat less emphasis will be placed on development of a shear, the purpose of which would be size reduction to (a) permit a larger charge to the dissolver and, hence, greater processing capability, and (b) permit core leaching should fuel elements be received which resist applicable chemical decladding processes.

Initially, one geometrically-safe batch dissolver employing solution recirculation will be installed. The geometry of the dissolver system will assure nuclear safety up to an equivalent enrichment of 5 per cent U^{235}. Chemical decladding of zirconium or stainless steel-clad whole fuel elements, employing either the Zirflex or Sulfex processes, respectively, will be followed by chemical dissolution of the core with nitric acid, or a nitric acid-ferric nitrate mixture in the case of U-Mo alloys. Core leaching of chopped fuel elements followed by chemical or mechanical removal of cladding chips will also be possible. The dissolver will be constructed of Hastelloy-F to assure compatibility for decladding of zirconium and stainless steel-clad fuel elements.

Liquid-solid separation equipment will be provided for recovery of solid fissile material from the cladding solutions. The geometry of the centrifuge must assure nuclear safety for equivalent enrichments up to 5 per cent U^{235}. Vendors of centrifugation equipment will be contacted for possible development work on this type of equipment.

MECHANICAL TREATMENT

Friction Sawing Studies - Sawing studies were made during March using a dry friction blade for cutting Zircaloy tubing. In addition to the basic sawing studies, an off-gas filtration system including a rotoclone and a filter assembly was installed to permit studying of the particulate material generated during the friction cutting operations. Although quantitative information is not currently available from these tests, the following pertinent observations were made:

1. Some burning of small chips and fines occurred in the saw hood during the friction cutting operation. Burning did not propagate along the Zircaloy tube proper.
2. All particulate material generated was not oxidized or burned. Up to 10 per cent of the material collected in the rotoclone hopper was metallic zirconium.
3. Approximately 45 square inches of Zircaloy tubing (approximately 3/16-inch thick cut) were cut during the test. This represents roughly the cutting required in trimming the "hardware" from one ton of power reactor fuel uranium. Although approximately 85 per cent of the Zircaloy powder was trapped in the rotoclone, sufficient 3 to 10 micron particles passed through the cyclone to plug the filter assembly.

Sawing studies made during April include comparisons of dry and wet sawing of Zircaloy tubing. Dry sawing was accompanied by a shower of white sparks and ejection of glowing metal fragments. Wet sawing (slight tube submergence accompanied by a blade water spray) was considerably less spectacular. As was expected, the sawdust was principally zirconium oxide (about 90 per cent) and contained a significant quantity of particles in the one to two micron size range.

Sodium and Sodium-Potassium Alloy Reservoir Studies - Studies of mechanical techniques for safely processing the sodium or sodium-potassium alloy (NaK) reservoirs present in some non-production fuels have continued. Tests have included both high-speed friction sawing and hacksaw cutting of NaK-filled capsules in the presence of water. Friction-saw tests were made with the capsules at the surface of the water and under an inert-gas blanket. Up to four capsules (6 gm NaK/capsule) were exposed in a single blade pass without fires or explosions when the saw hood atmosphere contained less than five per cent oxygen. Loadings up to 48 grams of NaK (12 gm/capsule) were exposed in a single blade pass (24 gm. maximum instantaneous exposure) with two-inch maximum water submergence and a helium-purged hood atmosphere containing less than five per cent oxygen. The usual dense white cloud of oxide fumes formed in the saw hood, but no fires or explosions occurred. The NaK exposure rate encountered in the above test is a factor of two greater than the rate contemplated during production cutting of the PRDC fuel rods. Thus, the success of this technique in eliminating NaK (or Na) reservoirs from the active sections of power fuels has been conclusively demonstrated.

In the hacksaw tests, single capsules were opened six inches to two feet below an air-blanketed water surface. The quantity of burning NaK reaching the surface decreased with increasing submergence, but the severity of underwater explosions increased.

Hydrogen was not detectable with a chromatograph on the saw hood after a cut of over 20 grams of NaK was made. The detection limit of the chromatograph is 0.2 per cent of H_2 in He.

Cold Saw Studies - Although high-speed friction-blade sawing has been demonstrated as an acceptable method for the removal of end fittings from non-production fuels, friction sawing requires cutting at relatively high temperatures and produces fine particles (1 to 2 micron) which must be safely separated from gas streams and stored. Consequently, "cold" sawing (i.e., low-speed multi-tooth blades which provide milling-cutter type operation) is currently being evaluated as an alternate method for end fitting removal. Initial cold-saw feasibility tests were conducted during April at the Motch and Merryweather Company in Cleveland, Ohio. During these tests simulated end fitting geometries (stainless steel tubing and pipe assemblies) were successfully cut using a 14-inch diameter blade (112 teeth, 0.39-inch tooth pitch) turning at 15 revolutions per minute (54 surface feet per minute). The "sawdust" produced during the test was considerably larger than that produced during friction sawing. Semi-quantitative analysis indicates that more than 99 per cent of the particulate matter produced is greater than 300 microns in diameter. On the basis of these attractive test results a "cold" saw is to be obtained to permit additional Hanford evaluation.

SHEARING

Shear NaK Studies - Studies are currently being made to develop safe methods for handling the small sodium and NaK reservoirs present in some of the fuels to be handled in the non-production fuel reprocessing program. In recent studies tubes containing sodium have been sheared underwater with inert gas blanketing the gas space above the shear blade. Although capsules containing up to 100 grams have been sheared in a single blade stroke without incident, the results are not considered representative of plant conditions. In these experiments the thin wall tubing collapsed and decreased the area of sodium-water contact. Consequently the reaction was not so violent as it would be under actual plant shearing conditions when greater than 50 per cent of the tube area would remain open.

To determine the violence of the NaK-water reaction when NaK is free to react completely with water, small bottles containing up to 8 grams of NaK were fractured by the shear. Although the hydrogen generation rate was considerably greater than in the sodium experiments discussed above (sufficient to force water, gas and some NaK particles from the shear feed port), it was found that the inert-gas blanket was effective in preventing burning and explosions in the shear gas space.

From these experiments it is concluded that sodium and/or NaK can be safely reacted under water employing inert gas blanketing as long as it is reacted a little (e.g., 5 or 6 grams) at a time. Consequently in the future, studies are to be made to develop sawing and shearing techniques which will permit meeting NaK a little bit at a time. Such techniques are compatible with the NaK-containing fuel elements Hanford is to process.

Shear Blade Performance - The equivalent of one ton of fissile material in typical non-production fuels geometry has been chopped with an alloy steel shear blade, without significant attrition of the blade. The blade, currently being life-tested, has made several thousand cuts totaling approximately 5000 sq. in. (cross-section area) of material cut.

Shearing Unirradiated Uranium-Bearing Fuel Rods - Stainless steel-clad swaged UO_2 rods and stainless steel-clad uranium-10 weight per cent molybdenum alloy (U-Mo) rods were sheared under water to investigate the mechanical and particulate handling problems associated with these two major components of the power reactor fuels reprocessing program. Moderate shear forces (25,000 and 9,500 lb./sq. in. for U-Mo and UO_2, respectively) and minor end closures (less than 10 per cent) were observed in shearing. As was expected, the U-Mo cut cleanly, with minimum fines generation, while up to 30 per cent of the UO_2 disintegrated to form a sludge in the bottom of the catch bucket. A small fraction of the UO_2 as 10 to 20 micron-sized particles was forced into suspension to produce in the water a brown turbidity. On standing, the particles settled in less than 16 hours. The studies indicate that the shear auxiliaries (e.g., product receivers, bath filters) must be designed to handle the fines and slurries produced.

DEJACKETING

Zirflex

The Zirflex process which employs aqueous ammonium fluoride to dissolve zirconium is the process selected for decladding of zirconium-clad fuel elements. Development work on the process is essentially complete.

Behavior of Explosive Uranium-Zirconium Residues in Zirflex - Experience of other workers has demonstrated that annealing heat treatments of uranium-zirconium alloys which produce the delta phase (formerly designated the epsilon phase) lead to explosion hazards in nitric acid dissolution of such alloys. Attempted dissolution of alloys containing the delta phase results in isolation of the delta phase as a powder residue. This residue has been demonstrated to be pyrophoric and capable of explosively violent oxidation reactions when subjected to a sharp impact or an electrical spark.

Since a uranium-zirconium alloy will exist in the diffusion layer of uranium metal fuel elements metallurgically bonded to Zircaloy jackets, some concern was felt for hazards which might arise from formation of the delta phase in this diffusion layer.

Since the Zirflex process will be used to declad these fuels, experimental studies were initiated to determine if the Zirflex decladding process would dissolve or desensitize delta phase uranium-zirconium alloys. Results to date, while preliminary, are encouraging.

The technique which has been used for preparation of explosive residues is that described by Schulz, Scott, and Voiland, viz., heat treatment of uranium-zirconium alloys to form the delta phase followed by partial anodic dissolution of the sample in cold dilute nitric acid. The anodic dissolution results in fairly rapid dissolution of the bulk alloy. Powder residues are formed, however, which on drying and sparking with a Tesla coil oxidize in a thoroughly authoritative manner (i.e., "explode"). The results of the tests conducted thus far are not entirely clear-cut in that residues formed in this way do not dissolve entirely in the Zirflex decladding media (aqueous ammonium fluoride or ammonium fluoride - ammonium nitrate mixtures). However, the more reactive constituents do dissolve rapidly in Zirflex decladding media. This has been verified both by sparking tests and by comparison of X-ray diffraction patterns before and after treatment with the Zirflex reagents.

In all cases the residues remaining after Zirflex treatment are quite inert in the sparking test. Similarly, comparison of X-ray patterns shows in all cases rapid disappearance of the most prominent lines apparently associated with the reactive portion of the residues.

Quite recent results suggest that the Zirflex insoluble residues observed in these tests may be formed in the anodic dissolution procedure used to prepare the reactive powders (e.g., zirconium oxide). A bulk uranium-zirconium alloy subjected to a heat treatment known to produce delta phase did not produce powder residues when exposed directly to the Zirflex decladding medium, although corrosion of the alloy did occur. Thus there is considerable basis for optimism that the conventional Zirflex decladding operation will result in complete dissolution of the zirconium present in the diffusion layer on metallurgically bonded uranium fuel, thereby eliminating the possibility of isolation of powder residues in subsequent nitric acid core dissolution.

It should be recognized, however, that the Zirflex process will not be capable of dissolving the zirconium out of bulk high-uranium, low-zirconium alloys. The only known safe dissolution approach for such alloys entails inclusion of sufficient fluoride (in excess of 4 moles fluoride per mole zirconium) in the nitric acid dissolving medium to assure that the zirconium in the alloy is dissolved as rapidly as the uranium.

Zirflex Waste Mobility - Flow properties of neutralized Zirflex waste were determined in semi-works scale equipment (5-gallon head tank and 1/2-inch flow tube). The solids settled rapidly to 20 per cent of the total volume. Medium agitation was required to keep the solids suspended. The slurry was transported through the flow tube without difficulty when the flow was turbulent (Reynolds number greater than 2500).

Materials of Construction - Hastelloy-F weldments welded with Ni-o-nel show average corrosion rates of 0.22 mil/mo. during 50 hours' exposure to simulated Zirflex dissolver conditions.

Sulfex

The Sulfex process, which employs 3 to 4 molar sulfuric acid, has been selected for the decladding of stainless steel-clad fuel elements. Laboratory studies, which have previously demonstrated satisfactory penetration rates, terminal stainless steel concentrations and waste losses, are now seeking to define factors contributing to the waste losses which result from attack on the fuel core by the decladding solution.

Laboratory Studies - In dissolution of stainless steel cladding from UO_2 it was found that a more rapid initial UO_2 dissolution by sulfuric acid occurred if the UO_2 was exposed to the atmosphere. This more rapid UO_2 dissolution after exposure to the atmosphere is presumed to be due to surface oxidation of some UO_2.

Data obtained with no significant exposure of the UO_2 to the atmosphere between experiments indicate that the rate of UO_2 dissolution in the presence of stainless steel is dependent on the rate of stainless steel dissolution. It was shown with unirradiated UO_2 that, whereas the simultaneous dissolution of stainless steel or carbon steel decreased the rate of UO_2 dissolution, the simultaneous dissolution of 99.99 per cent iron had no effect on the UO_2 dissolution rate. From this it is concluded that hydrogen evolution is not responsible for the lower UO_2 dissolution rate.

It was found that a black solid of unknown composition is present during and for a while after the dissolution of carbon steel or stainless steel in sulfuric acid. The inhibition of UO_2 dissolution by the presence of the black solid isolated from these solutions indicates that it is the black solid rather than the steel itself which results in the lower UO_2 dissolution rate in the presence of dissolving steel. The weight of the black solid isolated by the dissolution of 304-L in boiling 4 M H_2SO_4 was ca. 0.3 per cent of the weight of the steel dissolved. This black solid is about 90 per cent dissolved in one hour in boiling 4 M H_2SO_4. The data obtained to date indicate that uranium waste losses of 0.1 per cent or less can be maintained in sulfuric acid dissolution of stainless steel cladding from UO_2.

Studies of the effect of radiolysis on the rate of attack of fuel core materials by the Sulfex medium (dilute sulfuric acid) were extended to more highly irradiated uranium dioxide. It was found that uranium dioxide irradiated to a (calculated) exposure of 3000 MWD/T and cooled five months was attacked by boiling 4 M H_2SO_4 at a rate of only 0.02 per cent per hour. This corrosion rate is essentially identical to that found in earlier work with 500 MWD/T uranium dioxide cooled nine months, despite the fact that the measured radiation does rate with the present material is greater by a factor of 7.5, and within a factor of probably two to three of that expected in the NPF program.

Pilot Plant Studies - The modifications of Hastelloy-F dissolver to permit the dissolution of stainless steel using the Sulfex process have been completed. An acid addition tank to permit the controlled and measured addition of acid to the dissolver, and a recorder to permit the continuous monitoring of the hydrogen concentration in the off-gas stream were installed.

A proposal for operating the pilot plant Sulfex dissolver involves addition of 98 weight per cent sulfuric acid to dissolver previously charged with stainless steel and water. Annealed 304-L plate, annealed 304 tubing, and sensitized (one hour at 1250 C) 304-L plate were dissolved by this procedure. The 304-L samples started to dissolve at a sulfuric acid concentration of about one molar and the 304 tubing at about two molar.

Several other bench-scale runs were made of the sulfuric acid dissolution of stainless steel with similar results. No serious passivation was experienced when the stainless steel in mild steel wool was placed in boiling water and sulfuric acid was added. The stainless steel began reacting when the solution became 0.8 M H_2SO_4. With no mild steel present, the reaction with stainless steel began when the solution became about 5 M H_2SO_4. Cooling the solution caused no noticeable passivation of the stainless steel.

Materials of Construction - Two more heats of modified Hastelloy-F have been obtained for testing as possible welding material for use with Hastelloy-F base metal. One of these contains nine per cent molybdenum and one per cent niobium. The other contains 12 per cent molybdenum and no niobium. Nominal composition of Hastelloy-F includes six per cent molybdenum and two per cent niobium. Both heats showed poor corrosion resistance to nitric acid - hydrofluoric acid and to sulfuric acid - stainless steel solutions. Both suffered severe attack by nitric acid - hydrofluoric acid when used as weld filler with Hastelloy-F base metal.

Welded and annealed Haynes 25 tubing corroded at rates about the same as those previously observed for Hastelloy-F in H_2SO_4 and H_2SO_4-stainless steel solutions. Preferential weld metal attack occurred in the H_2SO_4-stainless steel solutions.

CORE DISSOLUTION

Uranium and Uranium Dioxide - Further studies on the dissolution of ingot uranium and sintered uranium dioxide in nitric acid and nitric acid - ferric nitrate solutions confirm a previously reported acceleration in dissolution rate by ferric nitrate. The instantaneous dissolution rates for a sample of fused uranium dioxide in 9 M HNO_3 and in 9 M HNO_3 - 0.1 M $Fe(NO_3)_3$ were about one-half those for sintered uranium dioxide in the same reagents.

Uranium-Molybdenum Alloy - Early in the month it was observed that the presence of ferric nitrate in the dissolvent during dissolution of uranium-molybdenum alloys permits dissolution, without solids formation, to higher terminal uranium concentration and lower terminal acidity than has been possible with either nitric acid alone or with nitric acid - aluminum nitrate - hydrogen fluoride combinations. This observation has been exploited as a means of preparing solvent extraction feeds from uranium-molybdenum alloys without the use of fluoride containing solutions. With one molar ferric nitrate present, uranium - 3 weight per cent molybdenum alloy could be dissolved to terminal uranium and free nitric acid contents of 1 M and 0.2 M, respectively, without appreciable solids formation. Similar results were obtained with uranium - 9 weight per cent molybdenum alloy. The free acid of such solutions, when cooled, can be reduced to less than zero with dibasic aluminum nitrate or caustic. Precipitation of ferric hydroxide appears to limit the extent of neutralization possible. The pH of the neutralized solutions is still less than zero. Plutonium is readily oxidized to Pu(VI) in these solutions by dichromate. A series of solutions prepared by dissolution of uranium-molybdenum alloys and containing varying uranyl nitrate, ferric nitrate, and free nitric acid has been set aside for observation of stability during long-term storage at 25 and 50 C.

It was further observed that the presence of ferric nitrate increases the dissolution rate of uranium-molybdenum alloys, uranium metal, and uranium dioxide by nitric acid. The most pronounced increase in dissolution rate occurs at low acid where the dissolution in nitric acid alone becomes very slow. This property may be useful to shorten time cycles in batch dissolution to no heel.

Further studies on the preparation of Redox-type feed solutions from uranium-molybdenum alloys by dissolution in nitric acid-ferric nitrate solutions may be summarized as follows:

1. Off-gases from the dissolution of uranium - 3 weight per cent molybdenum alloy in ferric nitrate - nitric acid solutions are primarily oxides of nitrogen. Only traces (less than 0.2 volume per cent) of hydrogen were found.

2. Sulfate ion at concentrations which might be present during core dissolution due to incomplete washing following Sulfex decladding had no significant effect on uranium-molybdenum alloy dissolution in ferric nitrate-nitric acid solutions.

3. A laboratory-scale recirculating dissolver has been used for dissolution of uranium-molybdenum alloys. This equipment was operated with storage vessel-to-dissolver volume ratios from 10 to 20. Solution was maintained at 85 C in the storage vessel and preheated before it entered the dissolver. Normal dissolution rates and solution stabilities (as compared to runs in pot-type dissolvers) were observed. Extensive (ca. 10 hours) recirculation of dissolver solution following dissolution to less than 0.2 M free acid caused precipitation of a brown solid, presumably ferric hydroxide.

4. Stability (toward precipitation) of the dissolver solutions, during long term storage at 25 and 50 C, as a function of acidity, ferric nitrate, and uranyl nitrate concentration is under study. The minimum acidity for stable solutions is governed by the ferric nitrate concentration. Lower temperature favors stability of the solutions.

5. Oxidation of plutonium by dichromate or other oxidizing agents will be necessary for Redox processing. Minimum acidity to prevent solids formation during the oxidation step appears to be about -0.4 M for solutions about one molar in uranium and in ferric nitrate. Such solutions have a pH less than zero.

6. Aqueous ferric nitrate for use in this process can be prepared readily by dissolving mild steel in nitric acid. In dissolution rate studies covering ferric nitrate concentrations from zero to one molar and nitric acid concentrations from two to 10 molar, dissolution rates for 1020 carbon steel ranged from two to 12 g/sq. cm-hr. Ferric nitrate prepared in this manner has been used to dissolve uranium-molybdenum alloys and the dissolver solutions have been used for plutonium oxidation and batch contact solvent extraction studies. No observable differences between these dissolver solutions and those prepared using reagent grade crystalline ferric nitrate were found.

7. Exploratory studies attempting to define the mechanism by which ferric salts prevent precipitation of uranyl molybdate are in progress. Preliminary evidence points toward formation of a soluble iron-molybdenum heteropoly acid.

Further studies on the dissolution of uranium - 9 weight per cent Mo alloy in HNO_3-$Fe(NO_3)_3$ solutions were made. The maximum uranium concentration which can be achieved during dissolution without solids formation is dependent on the terminal HNO_3 concentration and increases from about 0.5 M at 0.5 M free acid to about 0.7 M at 2.0 M free acid. The acidity of the solutions at room temperature can be reduced to about -0.4 M free acid with Diban without solids formation.

Preliminary attempts to identify the iron-molybdenum complex formed when molybdenum or U-Mo alloy is dissolved in HNO_3-$Fe(NO_3)_3$ involved adsorption of the complex on anion exchange resin. In the absence of uranium, iron and molybdenum were adsorbed on the resin in a mole ratio of one to six (Fe/Mo). Salts containing the group $(Fe(MoO_4)6)^{-9}$ are reported in the literature. Data so far obtained with uranium present are confused due to analytical difficulties.

The dissolution rate of uranium - 3 weight per cent Mo alloy in aluminum jacket removal solutions (10 per cent NaOH - 20 per cent $NaNO_3$) was found to be very low. During 30-minute exposures to the boiling solution, the average dissolution rate was about 0.00015 gm/(sq.cm.)(hr.). Thus, NaOH-$NaNO_3$ decladding followed by core dissolution in HNO_3-$Fe(NO_3)_3$ appears to be a satisfactory procedure for dissolving the aluminum clad U-Mo alloy fuel scheduled for reprocessing at Hanford.

Materials of Construction - The corrosion of 304-L stainless steel and Hastelloy-F in nitric acid - ferric nitrate systems was studied. Corrosion rates in boiling solutions increased as the concentration of either ferric nitrate or nitric acid was increased. At nitric acid concentrations of five molar or less and ferric nitrate concentrations up to 0.6 M, corrosion rates were less than two and 0.3 mil per month for 304-L and Hastelloy-F, respectively.

Both Ni-o-nel and Hastelloy-F were corroded intergranularly in 5 M HNO_3 - 1 M $Fe(NO_3)_3$ solution. Base metal corrosion rates were relatively high (10-12 mils/mo.) for both alloys in this solution at boiling. The rates were reduced by decreasing either the ferric nitrate or the nitric acid concentration. These observations point to the need for controlled addition of one or both of these reagents during the dissolution of U-Mo alloys. Ni-o-nel weld metal showed no preferential attack in this system.

Dissolution of Uranium Zirconium Alloys - Exploratory experiments were done attempting to define the effect of aluminum nitrate and hydrogen fluoride on the formation of explosion sensitive surfaces during the dissolution of uranium-low zirconium alloys. The studies were done using a uranium - 5 weight per cent zirconium alloy. In the absence of aluminum, a fluoride-to-dissolved zirconium mole ratio of about four prevented sensitive surfaces. With aluminum nitrate present, the F/Zr mole ratio required to prevent sensitive surfaces is greater than four. With 0.5 and one molar aluminum nitrate present, sensitive surfaces were obtained at F/Zr mole ratios of eight and ten, respectively.

Sensitive surfaces formed by anodic dissolution of uranium - 5 weight per cent zirconium in one molar nitric acid were completely de-sensitized in less than 30 seconds' exposure to boiling 3 M NH_4F-0.5 M NH_4NO_3, Zirflex reagent.

Sodium Dissolution - Measurements of the instantaneous pressures developed in the reaction of submerged metallic sodium with cold 13 M HNO_3 were made using an unbonded strain gage and recording oscilloscope. Reactions were carried out in an 8-inch pipe (open at the top) employing submergences of 3-8 feet.

The reactions were characterized by an audible report and a decided movement of the 8-inch pipe, but little spray or violent gas evolution was noted at the top of the container. Interpretation of the oscillograms is now under way.

COMBINED CLADDING AND CORE DISSOLUTION

Although the initial reprocessing of non-production fuels will employ separate decladding and core dissolution steps, alloyed fuels may, in the future, require combined dissolution of the cladding and core.

Darex (Dilute Aqua Regia Process)

One method of preparing a solvent extraction feed from stainless steel-clad fuel elements consists of complete dissolution of the elements in a dilute nitric and hydrochloric acid mixture followed by concentration in the presence of strong nitric acid to effect chloride removal.

Chloride Removal - Analyses of the information obtained during chloride removal from Darex dissolver effluents have indicated the following trends:

1. During typical chloride removal conditions, the concentration of total chloride in the vapor phase exceeds that in the liquid if the acid concentration in the liquid is greater than the chloride concentration in the liquid.

2. The ratio of the vapor-to-liquid chloride concentrations is apparently inversely proportional to the chloride concentration in the liquid.

3. The presence of stainless steel hinders chloride removal.

4. Under "total" reflux conditions, the chloride removal rate varied markedly with boil-up rate, suggesting the possibility of a rate limiting reaction in the vapor phase.

5. The presence or absence of uranium has little effect on the terminal chloride concentration attainable.

Pilot Plant Studies - Additional studies of metallic uranium dissolution in dilute aqua regia were made. The dissolution rate is primarily a function of the nitrate-to-chloride ratio, decreasing gradually as the ratio increases to 4.5, and decreasing markedly at higher ratios. The rate appears to be a direct function of the acidity and stainless steel concentration and an inverse function of the uranium concentration, although all three of these variables have second-order effects.

Analyses of off-gas from the downdraft condenser show H_2, N_2O, NO, and NO_2 present. The maximum hydrogen gas concentration was 2.5 per cent, with typical values less than 2 per cent for an air sparge rate of 1.5 scfm per square foot of initial uranium surface area. A typical, representative equation for the dissolution is:

$$U + 2.8\ HNO_3 + 1.1\ O_2 \longrightarrow UO_2(NO_3)_2 + 0.1\ H_2 + 0.5\ NO_2 + 0.15\ N_2O + 1.3\ H_2O$$

Chloride was volatilized by sparging gaseous N_2O_4 - NO_2 mixed with an equal volume of air into a boiling 2.2 M U, 4.0 M H^+, 2.8 M Cl^- solution. The off-gases passed through a downdraft condenser and a caustic scrubber for chloride removal. The condensate was refluxed. An average sparge rate of 0.6 gram mol NO_2 per liter of solution per hour produced 0.4 mol of acid and removed 0.25 mol chloride per mol of NO_2 added. No appreciable change in chloride removal rate was noted down to the terminal 0.2 M Cl^- concentration attained.

In another chloride removal study, a solution 2.2 M U, 4.0 M H^+ and 2.8 M Cl^- was concentrated by a factor of three, diluted to the original volume with 60 per cent HNO_3 and boiled under reflux for 2.5 hours. Excess HNO_3 was removed by concentration. A chloride decontamination factor of 10^4 was obtained.

Ten stainless steel clad (10-mil wall thickness) metallic uranium core (3/4-inch OD) fuel elements were dissolved smoothly and completely in 1 M HCl, 6 M HNO_3 in about seven hours.

Chloride was removed from an 0.85 M stainless steel and 0.8 M U solution by sparging with NO_2-N_2O_4 and air. Chloride volatilized at the rate of 0.1 mole chloride per mole NO_2 added. Acid was produced at the rate of 0.7 mole H^+ per mole NO_2 added until the solution reached 6.1 M H^+, after which no additional increase was obtained. Terminal chloride concentration was 1 M compared with 0.2 M reported above for solutions containing uranium but no stainless steel.

Materials of Construction - A Hastelloy-F probe with a heat transfer surface has been installed in the Darex pilot plant dissolver for seven weeks. After 623 hours at low temperature and 244 hours at or near boiling the probe showed a sharp corrosion band 1/16-inch deep over an area 1/16-inch wide corresponding to the vapor-liquid interface. Generalized pitting to depths of 1/16 of an inch was noted in an area surrounded by stagnant vapor. Areas exposed to liquid and vapor showed mild corrosion similar to that previously reported for the Hastelloy-F coupons exposed in Darex system.

A similar probe made of Haynes-25 showed comparatively little corrosion after six months in the Darex dissolver. Corrosion of Haynes-25 was most noticeable in areas sensitized by welding. No preferential attack was noted at the vapor-liquid interface.

A coupon of Hastelloy-F welded with Ni-o-nel showed general attack at a rate of about two mils per month during exposure in the 321 Building Darex dissolver.

Hastelloy-F samples were exposed to 2 M HCl-5 M HNO_3 at boiling and at 25 C with and without an air sparge. Severe interface attack occurred in the absence of boiling or air sparge. No observable interface attack occurred when the solution was boiling and air sparged.

Niflex

Pilot Plant Studies - Niflex (HNO_3 - HF) dissolution studies of 304 stainless steel have continued. Experiments designed to discover why the bottom portion of the stainless steel charges did not dissolve were unsuccessful. Charging ammonium bifluoride (F/SS ratio of 5) at the top or bottom of the boiling solution, changing the contact area between the stainless steel and Hastelloy-F, or charging all the stainless steel near the top or bottom of the dissolver, did not significantly change the dissolution pattern. Incomplete dissolution still occurred along the Hastelloy-F contact area and near the bottom end of the stainless steel charge.

Increasing the F/SS mole ratio from 5 to 5.5 or 6 with 2 M HNO_3 and 2 M fluoride increased dissolution of the stainless steel charge to approximately 99.9 per cent in a four-hour period. Dissolution of the areas in contact with Hastelloy-F was generally obtained.

Stainless steel-clad sintered uranium dioxide pellets (1/2-inch in diameter) were declad in 2 M nitric acid and 1 M ammonium bifluoride solution with a nominal F/SS mole charge ratio of 7. After a four-hour decladding period, aluminum nitrate and nitric acid were added to make the solution 2.3 M HNO_3, 0.66 M ANN, 1.33 M fluoride and 0.2 M stainless steel. Dissolution of the UO_2 was essentially complete in eight hours after the additions. At the end of the run one per cent of the stainless steel was undissolved. The undissolved uranium still encased in stainless steel was 0.4 per cent of the total uranium charged.

Dissolution of 1/2-inch-diameter stainless steel-clad uranium dioxide pellets and 3/4-inch-diameter stainless steel-clad uranium rods was completed on 100-pound batches. Neither a fluoride-to-stainless steel mol charge ratio of 7 nor a ratio of 8 gave complete dissolution of the stainless steel cladding. The stainless steel cladding was 96 to 99 per cent dissolved after four hours of operation. As expected, the end plugs were left intact, except for a 10 mil surface removal. (End plugs were approximately 23 per cent of the total stainless steel charged.) Approximately two weight per cent of the uranium core and three weight per cent of the uranium dioxide core dissolved in the Niflex solution (2 M HNO_3, 1 M NH_4HF_2, and 0.4 M stainless steel) during the four-hour decladding period. Uranium concentrations at this point in the dissolution steps were 5 to 13 grams per liter.

The uranium dioxide dissolved at average rates of 30 to 60 mils per hour when aluminum nitrate was added to complex the fluoride and the solution was adjusted to 1.5 - 2 M HNO_3. Dissolution of the uranium dioxide cores was 75 to 99 per cent complete in eight hours, with a 1.5 and a 2 M HNO_3 concentration, respectively. The final solution of uranium dioxide contained approximately 0.4 to 0.5 M U, 4.8 M NO_3^-, 1 M Al^{+3}, 1 M F^-, and 0.2 M stainless steel. In the metallic uranium dissolution, average rates of 15 to 30 mils per hour were obtained; although the acidity became quite low and an unstable solution formed at concentrations of about 0.94 M U, 0.2 M H^+, 4.7 M NO_3^-, 0.16 M stainless steel, and 1.3 M Al. This "cut" dissolved 53 per cent of the total uranium present.

Pilot plant studies to date on the Niflex process have developed the following information:

1. The reaction with 304 stainless steel involves 0.6 mole of HNO_3 and three moles of HF per mole of stainless steel in a downdraft dissolver system.
2. Charge ratios, i.e., moles of fluoride per mole of stainless steel, above five are required for complete dissolution.
3. Dissolution of 304 stainless steel averages 10 mils per hour during the first hour of dissolution.
4. Approximately 0.1 mole of hydrogen was liberated per mole of stainless steel dissolved. Peak hydrogen evolution rate was 5×10^{-4} lb. mole/(hr)(sq. ft.) of stainless steel.
5. Corrosion of Hastelloy-F containment vessel was severe.

Materials of Construction - A series of Hastelloy-F weldments (welded with Hastelloy-F filler wire) were annealed for one hour at 2100 F and water quenched. The weldments were then given an additional one-hour anneal at temperatures ranging from 1400 to 1900F. In every case, the additional heat treatment reduced corrosion resistance of the weld metal to boiling 1 M HNO_3 - 1 M HF. Samples annealed at 1600 and 1700F showed, also, severe intergranular attack on the base metal.

Hastelloy-F weldments prepared by electron beam welding and by metal arc welding in a gloved box purged with helium showed the same degree of preferential weld metal attack in HNO_3-HF solutions as those welded by the usual tungsten-inert gas techniques. Attempts to determine composition of the segregated phase in Hastelloy-F weld metal by means of the microarc emission spectrograph were not successful.

SOLVENT EXTRACTION

Hexone Processing

SOLVENT EXTRACTION

Hexone Processing

Laboratory Studies - Feed solutions prepared by dissolution of uranium-molybdenum alloys with nitric acid-ferric nitrate as solvent resemble Darex feed solutions in their solvent extraction behavior. Batch contact studies showed little if any extraction of molybdenum or iron into hexone. Limited fission product distribution data show similarity to those obtained with Darex feeds.

Further mini-mixer-settler runs with feeds prepared by dissolution of uranium three weight per cent Mo alloy were made to study the effect of feed acidity and salting strength on decontamination attainable by solvent extraction. As with Niflex and Darex feeds, increasing acidity resulted in reduced gross beta and gamma decontamination in the first cycle. With feeds at -0.3 M free acid (0.80 M U, 0.87 M Fe, 0.3 M Al), the gross beta and gamma decontaminations were lower by factors of about 15 and 30, respectively, than obtained in control runs using acid-deficient feeds. Ruthenium contributed most of the radioactivity in the product streams. Changes in salting strength had much less effect on over-all decontamination than variations in acidity. Uranium and plutonium recovery was generally good (<0.2 per cent loss) indicating successful oxidation of plutonium. Greater than 90 per cent of the neptunium present appeared in the product streams. Molybdenum concentration in the product streams was less than five ppm parts uranium. Extraction of dichromate ion was not observed in these runs in contrast to observations during runs with high acid Darex and Niflex feeds. Aqueous raffinates were stable toward precipitation if feed acidity was -0.3 M or higher.

Semiworks Studies - Two Redox-type HA-HC Column solvent extraction runs processing simulated Darex feeds were performed at Oak Ridge National Laboratory. The first run tested the effect on HA Column flooding rate of 100 ppm "Mistron" de-emulsification agent in high-silicon-content feed (0.015 M Si). The use of "Mistron" (insoluble magnesium silicate in fine powder form) increased the flooding rate by a factor of 2.7 compared to the same test performed without the additive. The increased flooding rate is about 40 per cent greater than that of the acid-deficient flowsheet tested earlier. Visual observation indicated that the majority of the "Mistron" was carried up the column by the organic stream to the upper organic-aqueous interface. The organic product stream remained clear and the column packing remained essentially clean. Long-term effects will not be determined during the remainder of the tests.

The second run tested the effect of an intermediate concentration of silicon (0.004 M Si) in a Darex feed solution on HA Column flooding rate. This run flooded at a rate approximately two-fold higher than a similar run with 0.015 M Si and at approximately the same rate as a standard acid-deficient run.

Alternate Solvents

It has been previously reported that diethers in which the two ether oxygens are separated by 4 to 6-CH_2-groups offer certain advantages over hexone as a solvent. Specifically, they have significantly higher flash points than hexone and exhibit considerably better resistance toward nitric acid.

Recent work indicates these compounds may offer some advantage over hexone from the standpoint of fission product decontamination, as well. Batch contacts were made comparing uranium, zirconium-niobium, and ruthenium distribution from identical aqueous solutions into hexone and nine diethers. With a dilute uranium solution containing 1.5 M aluminum nitrate and adjusted to 0.2 M acid-deficiency, uranium distribution ratios ranged from about unity with methoxybutoxy pentane to 27 with dimethoxy pentane, compared with about 4 for hexone. Ruthenium distribution ratios ranged from 0.0037 with methoxybutoxy pentane to 0.019 with dimethoxy pentane, compared with 0.011 with hexone. Zirconium-niobium distribution ratios varied in a random fashion but were roughly bracketed by the values of 4.1×10^{-5} and 5.8×10^{-4} obtained for hexone with two different feed batches. With an acidified dilute uranium feed containing 1.5 M aluminum nitrate, and 0.2 M nitric acid, uranium distributions ranged from 2 for methoxybutoxy pentane to 48 for dimethoxy pentane, compared with 15 to 19 for hexone. Ruthenium distributions ranged from 0.12 for methoxybutoxy pentane to 2.4 for dimethoxy hexane, compared with 0.6 to 0.7 for hexone. Zirconium-niobium distributions were again scattered, ranging from 0.016 for methoxybutoxy pentane to 0.5 for dimethoxy pentane, compared with 0.05 to 0.07 for hexone.

Distribution ratios for plutonium(IV) and plutonium(VI) have not yet been measured. If these prove operable, it is planned to evaluate via miniature mixer-settler studies the decontamination potential for such solvents in an acid first cycle followed by one or more acid-deficient cycles.

INSTRUMENTATION

Boron^{10} Monitor - Development tests to determine optimum geometry, cell thickness, materials of construction, moderator size, and source intensity, are about 80 per cent complete. Cylindrical lucite cells ranging from 3-inch to 6-inch diameters were tested to determine optimum cell size. The maximum sensitivity to boron concentration in the 1 gm/liter range occurs with a 4-inch diameter cell. Thin-wall stainless steel tubing and reactor grade zirconium pipe are being evaluated for use in the plant prototype.

The thermal neutron density as measured in these tests is about 15 per cent greater with paraffin moderator than with water. The maximum counting sensitivity is observed with the neutron source located in the moderator as near to the measuring cell as possible. The data obtained thus far indicate that a source intensity of about 10^5 neutrons/second will be adequate, and will produce a dose rate of approximately 4 mrem per hour outside the moderator.

Chromatograph Installation on NPF Cut-Off Saw Hood - A process chromatograph was calibrated for gas samples containing 0 to 100 per cent air and 0 to 10 per cent hydrogen. The unit was installed to sample the atmosphere in the hood surrounding the NPF cut-off saw in order to determine the gas composition before and after a cut was made on NaK bonded fuel elements. The chromatographic data indicated rather quickly that the sputtering and flaming which had occurred while sawing bonded NaK elements was due to insufficient purging of the hood with inert gas. No evidence of reaction was noted with gas mixtures of 20 per cent air in helium.

Strain Gauge-Dissolver Weigher - A strain gauge weighing system for a continuous dissolver is now being readied for use on a prototype dissolver. The system has an accuracy of ±0.25 per cent for loads from 40 to 200 pounds at temperatures between 15 F and 115 F. Tare weights (basket) up to 100 pounds may be suppressed.

CRITICALITY STUDIES

Experiments proceeded during the past quarter for determining the criticality parameters of 3 per cent enriched uranium applicable to the nuclear safety in the reprocessing of power reactor fuels. Experiments were conducted with heterogeneous and homogeneous systems. The experiments with the heterogeneous systems, which involved enriched uranium rods in light water, were designed to evaluate the extrapolation lengths of the water reflected systems and to facilitate a comparison between the critical approach experiment and the exponential pile measurement. The details of these measurements are given in the Physics and Instrument Research and Development Monthly Reports(1,2,3) and the Nuclear Physics Research Quarterly Reports.(4,5)

Further k_∞ measurements were made in the P.C.T.R. of UO_3 + H_2O mixtures for determining the maximum value of k_∞ of this material. The maximum value of k_∞ has been evaluated but the nuclearly safe concentration has yet to be determined.

In FY 1959 critical mass data were obtained with the three rod sizes initially chosen; these were 0.925 inch, 0.600 inch, and 0.300 inch. In order to determine the minimal critical mass and maximum buckling (largest always safe container) it is necessary to extend the measurements to include a fourth and smaller rod size; the rod diameter will be 0.174 inch; the critical approach experiments with this rod size will be undertaken during the first quarter of FY 1960.

The k_∞ measurements with the UO_3 - H_2O mixtures were largely completed in FY 1959. During FY 1960 the experiments will be extended to include measurements with $UO_2(NO_3)_2$ - H_2O mixtures. The infinite multiplication constant (k_∞) will be measured for the uranyl-nitrate mixtures as a function of the H/U ratio; the maximum value of k_∞ will be determined, and the nuclearly safe concentration limit will be established for the uranyl-nitrate mixtures. Further measurements will be done with the UO_3 - H_2O mixtures during August and September to evaluate the nuclearly safe concentration limit for this material. The first k_∞ measurements with the 3 per cent $UO_2(NO_3)_2$ - H_2O mixtures will be undertaken during November.

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1. HW-59717 B (Secret), March, 1959.
2. HW-60233 B (Secret), April, 1959.
3. HW-60505 B (Secret), May, 1959.
4. HW-60220 (Unclassified), January, February, March, 1959.
5. Report for April, May and June, to be issued.

NUCLEAR MATERIALS MANAGEMENT

Reactor Calculations

Studies of the correlation between Chalk River predicted values for the SSNM content of NRX fuel rods and Hanford measured values were completed. The basis and reliability of Chalk River calculated values, and possible causes of the observed between-sites discrepancies are summarized in document HW-60517 (Richey, C. R., "Report on an Investigation of the Product Differences Found in Processing Chalk River NRX Fuel Rods," May 26, 1959).

Volume Measurement Studies

Development studies were initiated to investigate the precision and accuracy attainable in measuring large volumes of process solutions. A 3000 gallon process tank equipped with a high precision dip tube-manometer system is currently being used for this work. Operations Research and Synthesis Operation has reviewed the planned level measurement studies and will analyze the data from the tests. A four-line dip tube system will be calibrated with water and a heavy metal solution. A calibration with water and a conventional dip tube system will also be carried out. A sensitive electronic relay connected to a gaging rod will be used as an accuracy standard to determine the height of liquid above the bottom of the tank. A hand operated lead-screw drive moves the gaging rod up and down. Two 150-inch precision manometers are used to read the tank level, and one standard 50-inch manometer is used to read the specific gravity. A stainless steel 43-gallon heavy water drum with dome ends is used for the solution weigh-in measurements. Shake-down tests of the level measurement equipment are now under way.

Hanford SSNM Measurement Summary

A document discussing the SSNM measurement procedures which will most likely be used for the Hanford processing of non-production fuels has been issued (Schneider, R. A., "Measurement of the Nuclear Materials Content of Non-Production Fuels," HW-60533, May 25, 1959).