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ZIRCONIUM PROCESSING CAPABILITY
OF THE IDAHO CHEMICAL PROCESSING PLANT

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ABSTRACT

A versatile hydrofluoric acid dissolution process enables the Idaho Chemical Processing Plant to process zirconium- or Zircaloy-clad zirconium-uranium fuels. Use of dissolved boron for nuclear criticality control permits high capacity semi-continuous operation. The Monel dissolver and austenitic stainless steel waste storage tanks satisfactorily resist corrosion by the process solutions. The resulting high-level waste solutions can be converted to granular solids by fluidized bed calcination.

ZIRCONIUM PROCESSING CAPABILITY OF THE IDAHO CHEMICAL PROCESSING PLANT

SUMMARY AND INTRODUCTION

The Idaho Chemical Processing Plant (ICPP) is equipped to process zirconium- or Zircaloy-clad zirconium-uranium alloy fuels at a substantial rate in a completely integrated set of facilities. Storage basin capacity is available for many hundreds of kilograms of uranium as zirconium-uranium fuel; the dissolution and extraction system will handle up to 835 kg of zirconium and 17 kg of uranium (on a before burnup basis) per day; ample tanks for storage of process waste solutions are available with more being provided on a regular basis as tanks previously filled with aluminum nitrate wastes are emptied by operation of the ICPP Waste Calcining Facility. The ICPP hydrofluoric acid process is also adaptable to oxide-containing fuel cores (eg, advanced PWR fuel), and the aqueous zirconium wastes can be converted to solids in the existing calcining facility when this step becomes desirable.

This unique capability for processing zirconium based fuels resulted from study and modification [1] of the original ICPP aqueous hydrofluoric process [2] when it became evident that programs at other AEC sites [3] were not sufficiently advanced [4] to allow design of a full scale plant based on any other approach in the near future. Using the experience gained in the successful pioneering of continuous dissolution of aluminum fuel at the ICPP, a semi-continuous dissolution process was developed for zirconium based fuel, requiring relatively inexpensive changes to existing equipment. The modified ICPP facility takes full advantage of the proven technology and experience in successful processing of early zirconium-fuel cores [5] and adds a new technique in the use of dissolved boron as a neutron absorber to assure criticality control at high uranium processing rates. Unit processing costs have been reduced substantially by attainment of higher throughput rates and elimination of the need for new waste storage tanks.

SEMI-CONTINUOUS OPERATION GIVES ICPP A LARGE ZIRCONIUM PROCESSING CAPACITY

Semi-continuous dissolution permits high processing rates in the ICPP zirconium fuel processing facility as compared with the original batch process [2]. The semi-continuous mode of operation involves periodic dissolution of large batches of fuel in a continuously flowing aqueous hydrofluoric acid stream, followed by continuous processing of the resultant dissolver solution through one cycle of TBP extraction and two cycles of Hexone extraction. A simplified flow-sheet of the overall process is given in Figure 1.

FIG. 1 SIMPLIFIED FLOWSHEET OF THE ICPP ZIRCONIUM PROCESS.

The ICPP process will handle up to 835 kg of zirconium per day, limited by the capacity of the extraction system. This is equivalent to approximately 10 kg of uranium per day as PWR seed fuel elements. Conservative calculations indicate that as much as 17 kg of U-235 can be safely processed each day in fuel elements containing higher percentages of uranium [6] [7].

Dissolution is conducted on the upflow principle with acid passing through a bed of fuel held in a tubular basket centered in the batch dissolver. The principal innovation permitting the increased ICPP capacity was the use of dissolved boron as a neutron poison for primary criticality control. This, together with a dissolver basket to assure fuel geometry control, permits the addition of a full day's fuel load in one dissolver charge. Dissolution rate is principally dependent on the availability of acid and independent of surface area down to a very small metal heel, thus permitting continuous withdrawal of dissolver product of constant composition as acid is metered into the dissolver.

Adjustment of dissolver solution to extraction column specifications (for control of corrosion, solution stability, salting strength, and uranium oxidation state) [8] is carried out continuously in equipment previously used for batch processing. Dissolver solution and adjustment solution flow in proportionally controlled streams into the adjustment (or complexer) vessel, are mixed by air agitation, and overflow continuously into collection vessels which serve as sampling and measuring points and as feed vessels for the continuous extraction system.

First cycle solvent extraction employs the TBP process in the aluminum-fuel headend system which has considerably larger columns than the original zirconium-process equipment. Second and third cycle extraction by the Hexone process follow in the aluminum-process equipment.

Process wastes are stored in tanks originally installed for aluminum nitrate wastes. These tanks have been shown to be resistant to corrosion by the aluminum-complexed zirconium wastes [6]. Storage space is adequate for present needs and more is being made available steadily as the Waste Calcining Facility (WCF) processes existing wastes. The WCF, operating on a regularly scheduled basis, emptied a 300,000-gallon tank of waste between December 1963 and April 1964 and, through continued operation, is expected to eliminate the need for any new waste tank construction in the foreseeable future.

Calcination of the complexed zirconium wastes appears to be feasible in the ICPP Waste Calcining Facility [9], and eventual conversion of the wastes to solids by this process is planned. Addition of calcium nitrate to the zirconium wastes suppresses fluoride volatilization during the calcining operation which achieves a sevenfold reduction in waste volume and results in a safer, less mobile material for storage.

DISSOLUTION PROCESS IS UNIQUE

The unusual features of the ICPP zirconium process are found in the headend steps [8] [9]. A schematic diagram of the principal components of the dissolution and first cycle extraction system and a chemical flowsheet of process conditions keyed to the equipment, presented in Figure 2, reveal these features [9].

Fuel elements are charged, batchwise, to the Monel dissolver after it is filled with water (containing 7.0 g/l minimum of natural boron as a nuclear poison) to cushion the force of falling elements. Up to 835 kilograms of zirconium are charged per batch. After fuel charging is complete, the cushioning solution is jetted to a storage vessel and retained for reuse in subsequent chargings. Dissolving acid is then added and a sixteen-hour dissolution cycle commences; after dissolution is initiated, dissolvent (6.5M HF, 0.03M HNO₃, and 0.35M H₃BO₃) is fed continuously, while the temperature is controlled at approximately 60°C. Acid flowing to the dissolver is monitored by a neutron attenuation instrument which checks the boron content of the solution. This instrument consists of a neutron source and two neutron detectors, all enclosed in Monel sleeves suspended in the acid feed tank. The instrument output controls a solenoid valve in the acid feed line to the dissolver and will not permit acid flow unless boron is present at the minimum desired concentration of 3.8 g/l.

The dissolver operates full of solution; product is withdrawn through a seal loop to a nitrogen-gas lift which transports the solution to the coarse feed tank [Carpenter-20 (Cb) stainless steel] for continuous complexing. Complexing, or adjustment, establishes proper conditions for uranium extraction and waste storage [8] [11]. Aluminum nitrate is added to complex free fluoride ions, thereby preventing corrosion during subsequent processing in the extraction system which is constructed of austenitic stainless steel; aluminum nitrate also acts as the salting agent for extraction of uranium. HF is added as necessary to establish a stable aluminum-zirconium-fluoride ratio and prevent precipitation from the solution. Chromic acid, added with the complexer solution, oxidizes uranium (IV), produced in the dissolver, to the extractable uranium (VI) state. The complexer vessel is operated full of solution (approximately 2500 liters) and is air agitated. Solution overflows continuously from this vessel to tanks in the aluminum-process headend which provide surge capacity for continuous extraction and opportunity for sampling and input measurement. Subsequent extraction and waste handling steps follow conventional flowsheet and processing practices in the continuous aluminum-fuel processing equipment.

STREAM	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17 & 18	19	20	
DESCRIPTION	FUEL CHARGE	DISSOLVENT	CHARGE WATER	DISSOLVER PRODUCT	COMPLEXING SOLUTION	COMPLEXED DISSOLVER PRODUCT	SCRUB RECYCLE I/B	I A F	I A X	I A R	I A P	I B S	I B P	I C X	I C R	I C P	I D X	I D R	I D P	FIRST CYCLE PRODUCT
FLOW, l/hr	517(a)		452(a)	127(a)	472(b)	60	530	500	530	500	60	500	200	500	1200	10	200	2.1		
TBP, %								10.0		10.0		10.0		10.0						
DILUENT								Amsco		Amsco		Amsco		Amsco		Amsco				
Zr ⁺⁺⁺⁺ ,M			1.07		0.80		0.71		0.71											
F ⁻ ,M	6.54		6.35		4.76		4.22		4.22											
Al ⁺⁺⁺ ,M				1.95	0.37	1.5	0.50		0.50		1.50									
H ⁺ ,M	5.52		0.98	1.46	1.02		0.90		0.78	0.13	-1.13(d)		0.005		0.005		0.005	0.5		
NO ₃ ,M	0.03		0.015	7.30	1.41	4.5	1.75		1.63	0.14	3.36	0.007	0.005		0.022		0.022	2.0		
Sn, g/l			1.53		1.14		1.01		1.01											
Cr, g/l				5.20	0.99		0.88		0.88											
B, g/l	3.80	7.0	3.84		2.90		2.58		2.58											
U, g/l			1.17(c)		0.88	0.001	0.78		<7.8x10 ⁻⁴	0.84		0.84			2.10		2.10	200		
DENSITY AT 25°C	1.035		1.186	1.360	1.215	1.230	1.220	0.794	1.200	0.802	1.200	0.795	0.998	0.794	1.002	0.757	1.002	1.275		
U, kg/batch	10(c)																			
Zr, kg/batch	835																			
Sn,kg/batch	13																			

(a) AVERAGE RATE FOR A 16-HOUR DISSOLUTION CYCLE.

(b) AVERAGE RATE FOR CONTINUOUS EXTRACTION (24-HOUR DAY).

(c) ASSUMES A 40% BURNUP OF URANIUM. (CRITICAL SAFETY ASSURED IN ABSENCE OF BURNUP, i.e., FOR UP TO 16.7 Kg FULLY ENRICHED URANIUM.)

(d) THE USE OF DIBAN IN THE SCRUB MAKES IT 1.13M ACID DEFICIENT.

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FIG. 2 SCHEMATIC AND CHEMICAL FLOWSHEETS OF ZIRCONIUM FUEL DISSOLUTION AND FIRST CYCLE EXTRACTION.

After the sixteen-hour dissolution cycle is complete, the residual solution in the dissolver is jetted to the complexing vessel. The dissolver is then refilled with the cushioning solution for another fuel charging and dissolution cycle.

REDUCED COSTS AND MINIMUM DIFFICULTIES ARE ASSOCIATED WITH THE NEW ICPP PROCESS

Decreased unit production costs result from the manyfold increase in processing rate brought about by the new ICPP zirconium processing technique, since operating and capital costs are not significantly increased at the higher production rate. Operating problems have not been increased over the original ICPP zirconium process which had a successful history of operation [5].

The column feed (adjusted dissolver product) is a stable solution over only a limited range of fluoride, aluminum, and zirconium concentrations in both the old and new flowsheets, but the addition of boron in the current flowsheet exerts no appreciable effect on the boundaries of the stable region [8] [10]. Boron shows no effect on the adjustment or extraction steps [8] [11]; all boron added in the dissolver feed is always found in the dissolver product; adjustment is complete in two minutes or less while stirring at room temperature; and boron does not extract with the uranium but remains in the raffinate. The use of nitric acid to dissolve tin and the use of chromic acid to oxidize uranium (IV) to the extractable uranium (VI) valence state have been studied extensively [11]; careful control of the complexing operation is necessary to avoid undue corrosion, but there are no additional difficulties with boron present in the flowsheet.

DISSOLVED BORON ASSURES NUCLEAR SAFETY

The present processing capacity of the ICPP for zirconium fuels is dependent on the use of natural boron as a neutron absorber (poison) in certain of the process streams. Since this is the first reported application of the use of a soluble poison for primary criticality control in a fuel recovery process, novel aspects of the process are discussed in some detail.

Nuclear criticality within the dissolver and succeeding process vessels, in the uranium concentrations called for in this flowsheet, can be prevented by the presence of approximately 3.8 g/l of natural boron in the process solutions [6] [7]. This amount of boron is added to the dissolver acid feed as boric acid. The presence of the nuclear poison is assured by use of monitoring instruments as well as administrative control procedures. Each batch of hydrofluoric acid dissolvent is spiked with boric acid in the makeup tank, and the presence of boron in the requisite amount is confirmed by laboratory analysis before use. In addition, the dissolvent feed tank is equipped with dual neutron attenuation instruments that measure boron concentration in solution. If the output of these instruments does not correspond to the desired 3.8 to 4.0 g boron per liter, a solenoid valve in the tank outlet will not open, and dissolvent cannot flow to the dissolver. As an additional precaution, all heat exchanger jackets contain boric acid solution to prevent possible dilution of the neutron poison in case of leakage.

Fuel Geometry is Controlled in the Dissolver

The dissolver being used for this process (Figure 3) is a Monel vessel having a lower cylindrical section, 24-inch diameter x 40-inch high, surmounted by an enlarged cylindrical section, 42-inch diameter x 50-inch high [7] [12]. A concentric crib made from 16 bars equally spaced on a 14-inch diameter circle is centered in the dissolver and connects with a 14-inch diameter charging chute coming down through the cell ceiling. Since the gaps between the bars comprising the center crib are 1-3/8 inches wide x 10 inches long and could permit partially disintegrated fuel pieces to fall out of the crib into the annulus, a situation undesirable both from the criticality standpoint and for most effective dissolution, a full length Monel liner, or basket is placed inside the crib and charging chute. The liner, 13-1/2-inch O. D. x 12-3/4-inch I. D., is provided with 1/4- x 6-inch slots (Figure 3) to allow acid circulation while retaining all but the finest fragments of fuel elements undergoing dissolution.

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FIG. 3 ICPP ZIRCONIUM FUEL DISSOLVER.

precipitate does not carry uranium with it in any significant amount [11]. Finally, while the central basket is designed with small perforations, small quantities of undissolved metal particles can still pass through these perforations and collect in the annulus. All of these possibilities were considered in the criticality evaluation [6] [7] [12].

The curve in Figure 4 indicates the safety of the process. A maximum k_{eff} of approximately 0.8 would occur under the most reactive metal distribution possible (a metal volume fraction of 0.6) with dissolved uranium at a concentration

Interacting Chemical and Physical Effects are Considered

Evaluation of the nuclear safety of the dissolver [6] [7] [12] required consideration of a number of factors. While the exterior geometry is fixed by the dissolver walls, the interior composition continually changes in an unpredictable manner. The fuel undergoes size degradation during dissolution; smaller particles fall down between larger particles. Furthermore, the chemical system is not stable over all concentration ranges; a large excess of free fluoride results in the precipitation of $UF_4 \cdot 3/4 H_2O$, while low concentrations of free fluoride result in a precipitate of $Zr(OH)F_3$. Fortunately, the zirconium

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FIG. 4 K_{eff} VALUES FOR POSSIBLE DISSOLVER OPERATING CONDITIONS.

ASSUMED CONDITIONS: CENTRAL CYLINDER (13-INCH DIAMETER) CONTAINS 3 PERCENT U^{235} -ZIRCONIUM ALLOY IN VARIOUS DENSITY DISTRIBUTIONS (MVF), SURROUNDING ANNULUS (42-INCH DIAMETER) AND LIQUID VOLUME FRACTION CONTAINS 4 g/l DISSOLVED URANIUM AND 3.8 g/l DISSOLVED BORON; TOTAL URANIUM IN DISSOLVER 17 kg.

of 4.0 g/l, well beyond the maximum of 3.0 g/l attained in the laboratory and the 1.2 g/l expected under flowsheet conditions.

Boron is Soluble Under all Process Conditions

The solubility of boron in the dissolver solutions imposes no limitation on the process. The solubility limit for boric acid or fluoboric acid in the process solution was not accurately determined but, after the desired level of approximately 4.0 g/l of boron had been established by calculations, the laboratory and pilot operations were conducted with solutions containing up to 6.0 g/l of boron; no loss or separation of boron from the process solutions occurred under any test conditions [11]. Additionally, dissolver solutions were evaporated to various extents, even to dryness, but on reconstitution the boron was found to be in solution and essentially in the original concentration. Precipitates of either uranium or zirconium fluorides, brought about by deliberate departure from flowsheet requirements, did not carry boron out of solution [11]. Indeed, no precipitation or anomalous behavior of boron was detected in any of the laboratory and pilot plant studies in the development of this process [11].

Effective Boron Concentration Cannot be Lost

To prevent any dilution of the boron content of process solutions and consequent reduction in the safety of the process, all decontamination and chemical addition lines were removed or blanked. A closed loop supplies boron-containing water to the dissolver jacket and off-gas condenser to eliminate hazard in the event of a jacket leak. An intermediate heat exchanger is installed between this loop and the plant water or steam systems; thus, any leak in a heat transfer surface will not result in dilution of the boron content of the process solution. The boron loop solution is not replaced during a processing campaign and its boron content is checked periodically by chemical analysis. Any change in volume of the coolant may be detected by a liquid-level alarm on a surge tank, and any leakage of process solution into the coolant will be detected by a radiation alarm.

The cushioning water, used to partially absorb the shock of fuel elements being charged to the dissolver, also contains boron. This water is cycled between the dissolver and an in-cell storage tank and does not have to be made up fresh for each charge. Its preparation is subject to the same supervisory control and laboratory checks that are required for dissolver solution.

CORROSION EXPERIENCE IS VERY FAVORABLE

Extensive tests indicate that corrosion is not a particular problem in this process. The Monel dissolver has been much more resistant to corrosion than originally anticipated in the design of the initial ICPP process for these fuels and will be satisfactory for extended operation. Corrosion in waste storage tanks has also been much less severe than originally anticipated; the regular Type 304L or Type 348 stainless steel tanks, provided for waste from aluminum fuels, have been found satisfactory for zirconium fuel raffinate storage.

Monel Dissolver has Ample Corrosion Allowance for Long Life

Monel equipment has been tested for 4000 hours in typical circulating dissolver solutions ^[13]. Cumulative corrosion penetration data indicate general rates ranging from 0.6 to 5 mils/month for different parts of the test loop; the penetration rate associated with the deepest pit found on microscopic examination was 7 mils/month.

The existing dissolver is designed with a 3/16-inch corrosion allowance ^[2]. Examination of specimens from this dissolver after 244 hours of operation ^[2] indicates a maximum corrosion rate of 7 mils/month, equivalent to an operating life of 18,500 hours.

Complexer and Extraction Columns Perform Standard Service

No corrosion problems are anticipated with the complexer or with the extraction columns; the complexer [Carpenter-20 (Cb)] is performing the same service for which it was designed, and the aluminum-process extraction columns used with the current zirconium flowsheet are of the same material (Type 304L stainless steel) as the original zirconium-process columns.

Existing Waste Storage Tanks are Corrosion Resistant

Excellent resistance of Type 304L stainless steel to zirconium-fuel process wastes has made possible the use of existing tanks, originally provided for aluminum nitrate wastes, for storage of aluminum-complexed zirconium-process

raffinates. Extensive corrosion studies [6], both laboratory tests and examination of specimens immersed in ICPP raffinate waste tanks containing the original zirconium-process wastes, indicate that the rate of attack (0.04 mils/month) is much less than originally anticipated. This low rate of attack, together with the fact that existing tanks are being emptied of waste by the fluidized bed calcining operation, eliminates the need for construction of additional tanks at the ICPP for zirconium fuel processing in the foreseeable future.

THE ICPP ZIRCONIUM PROCESS IS HIGHLY VERSATILE

While the ICPP now has a completely integrated facility of suitable capacity for handling currently anticipated zirconium based fuels, it also has the potential for even greater effectiveness, following a modest additional development effort. The processing of advanced zirconium oxide-uranium oxide fuels by an aqueous process in the existing ICPP equipment appears feasible, as does the conversion of aqueous zirconium wastes to granular solids in the ICPP Waste Calcining Facility when increased processing requirements make these steps desirable.

Oxide-Containing Fuels may be Processed

The processing of oxide-containing cores (eg, advanced PWR fuel composed of zirconium oxide-uranium oxide wafers clad in Zircaloy) appears feasible in an aqueous process adaptable to the ICPP Monel dissolver. Recently reported ICPP laboratory results ^[14] indicate success of a three-step batch dissolution process for the Zircaloy-clad, mixed-oxide core. The fuel is declad rapidly in 5M hydrofluoric acid-0.12M potassium fluoride solution at temperatures below 70°C. After decanting the solution, the zirconium is leached from the fuel core by boiling with 20M hydrofluoric acid-0.1M potassium fluoride for 2.5 hours. The solution is again decanted, and the uranium-containing residue is finally dissolved in boiling 0.1M chromic acid. The combined uranium losses in laboratory tests have been less than 0.1 percent, and all process solutions appear to be compatible with the existing Monel dissolver; thus, adaptation of the ICPP process to the advanced oxide-containing fuels appears promising.

Zirconium Wastes may be Calcined in the WCF

Calcination of the hydrofluoric acid process zirconium wastes appears to be possible in the ICPP Waste Calcining Facility (WCF) when sufficient wastes are generated to justify operation of the unit for this purpose. Liquid wastes may be stored for a considerable period in existing stainless steel storage tanks which are being made available by calcination of the present ICPP aluminum-process wastes; however, when large volumes of zirconium-process liquid

wastes begin to accumulate, it will be desirable to take advantage of the seven-fold reduction in volume and added safety in storage made possible by the conversion of the waste to solids. Both laboratory and pilot plant results indicate that the WCF should handle the zirconium fluoride wastes without difficulty ^[9]. Fluoride is converted to a nonvolatile form by addition of calcium nitrate; the slurry formed is readily pumped and passes through the calciner feed nozzles without difficulty. Fractional percentages of fluoride which are released from the calciner vessel are complexed and retained by excess aluminum and zirconium in the off-gas scrubbing system. Corrosion studies on actual pilot plant equipment, as well as on test specimens, indicate that the austenitic stainless steel from which the WCF is constructed is almost completely resistant to the environment in the calciner and scrubbing system while calcining these wastes. The calcined granular product is free-flowing and easily transported to solids storage bins. Initial tests indicate that this product is stable at storage temperatures much higher than the calcination temperature.

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