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ORGDP FUEL REPROCESSING STUDIES
SUMMARY PROGRESS REPORT
JULY THROUGH DECEMBER, 1965

AUTHORS:

S. H. Smiley
D. C. Brater
J. H. Pashley

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Engineering Development Department
Technical Division

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UNION CARBIDE CORPORATION
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Oak Ridge Gaseous Diffusion Plant
Oak Ridge, Tennessee

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A B S T R A C T

The Oak Ridge Gaseous Diffusion Plant Technical Division is participating with Argonne and Oak Ridge National Laboratories in studies of a group of processes aimed at purifying and recovering valuable uranium and plutonium from spent nuclear reactor fuels by volatilization as the respective hexafluorides. The ORGDP portion of the program includes two main phases: (a) preparation of conceptual plant studies with concomitant definition of problem areas associated with the process technology; and (b) component development, including scale-up and testing of crucial process equipment and auxiliaries. The current report is the second in a series of progress reports to be issued semiannually. Topics covered include (a) status of the conceptual study of the multipurpose fluid-bed plant for high enrichment fuels; (b) status of low enrichment fuel flow sheet studies; (c) composition of irradiated fuels; (d) theoretical aspects of uranium hexafluoride-plutonium hexafluoride-fission product separation by distillation; (e) design of a reactor for producing bromine pentafluoride from bromine trifluoride; (f) sizing of condenser systems for collection of bromine pentafluoride-uranium hexafluoride mixtures; (g) sodium fluoride fluid-bed sorber criticality calculations; (h) corrosion testing and related studies; (i) process and reactor studies, including cold semiworks plant design; (j) outlet gas filter testing; (k) product purification sorption-desorption system using batch sodium fluoride pellet traps; (l) connector testing; (m) status of valve selection; (n) peripheral and diaphragm compressor evaluation; and (o) reactor fabrication procedures.

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TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION	15
SUMMARY	15
ENGINEERING STUDIES	15
HIGH ENRICHMENT FUELS	15
LOW ENRICHMENT FUELS	15
Systems Analysis	16
Composition of Irradiated Fuels	16
Uranium Hexafluoride-Plutonium Hexafluoride- Fission Product Fluoride Separation	16
Bromine Pentafluoride Converter	16
Bromine Pentafluoride-Uranium Hexafluoride Condenser System	16
Sodium Fluoride Fluid-Bed Sorber	17
PROTOTYPE TESTING AND EVALUATION	17
MATERIALS OF CONSTRUCTION - CORROSION TESTING AND RELATED STUDIES	17
PROCESS AND REACTOR STUDIES	17
OUTLET GAS FILTER STUDIES	18
PRODUCT PURIFICATION, SORPTION-DESORPTION SYSTEM	18
CONNECTORS	18
VALVES	19
COMPRESSORS	19
Peripheral Compressor	19
Diaphragm Compressor	19
REACTOR FABRICATION PROCEDURES - WELDING STUDIES	19

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	<u>Page</u>
DISCUSSION	20
ENGINEERING STUDIES	20
HIGH ENRICHMENT FUELS	20
LOW ENRICHMENT FUELS	20
Systems Analysis	21
Composition of Irradiated Fuels	22
Uranium Hexafluoride-Plutonium Hexafluoride- Fission Product Fluoride Separation	24
Bromine Pentafluoride Converter	26
Bromine Pentafluoride-Uranium Hexafluoride Condenser System	29
Sodium Fluoride Fluid-Bed Sorber	31
PROTOTYPE TESTING AND EVALUATION	35
MATERIALS OF CONSTRUCTION - CORROSION TESTING AND RELATED STUDIES	35
Filter Materials	35
Flange and Reactor Materials	35
PROCESS AND REACTOR STUDIES	39
Cold Semiworks Pilot Plant	39
OUTLET GAS FILTER STUDIES	46
PRODUCT PURIFICATION, SORPTION-DESORPTION SYSTEM	48
Cylindrical Pellet Trap	48
Annular Trap	49
CONNECTORS	49
VALVES	50
COMPRESSORS	54
Peripheral Compressor	54

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WAS INTENTIONALLY
LEFT BLANK

	<u>Page</u>
Diaphragm Compressor	55
REACTOR FABRICATION PROCEDURES - WELDING STUDIES	56
APPENDIX A	57

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WAS INTENTIONALLY
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LIST OF TABLES

<u>Table</u>		<u>Page</u>
I	Summary of Transuranic Decay Energies Based on Spent Yankee Reactor Fuel Data	25
II	Reactor Volumes	27
III	Equilibrium Conversion of Bromine Trifluoride to Bromine Pentafluoride and Bromine Fluoride at Various Temperatures	28
IV	Design Comparison for Flash Cooler and Reflux Condenser for Uranium Hexafluoride-Bromine Pentafluoride-Noncondensables Mixture	31
V	Radii of Infinitely Long Cylinders	33
VI	Critical Sphere Data	34
VII	Results of Corrosion Tests	36
VIII	Sieve Analyses of Alumina	47
IX	Inconel Filter Flow Measurements	47
X	Results of 4-Inch Sorption Trap Tests	48
XI	Effect of Discharge Pressure on Corblin Compressor Performance	56

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WAS INTENTIONALLY
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LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Corrosion Test 11	38
2	Corrosion Test 21	40
3	Corrosion Test 21	41
4	Process Flow Diagram, Fluoride Volatility Testing and Development	42
5	Two-Zone Primary Reactor Assembly and Details	43
6	Filter-Cooler Assembly and Details	44
7	ORGDP Double Milled Touchpoint Gasket	51
8	Gas-Filled Nickel O-Rings	52
9	Solid Nickel O-Rings in Grooved Flanges	53

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ORGDP FUEL REPROCESSING STUDIES
SUMMARY PROGRESS REPORT
JULY THROUGH DECEMBER, 1965

This report is the second in the series of semiannual summaries of Oak Ridge Gaseous Diffusion Plant work in the AEC-sponsored volatility process development effort. As stated previously, the work is performed in conjunction with National Laboratory programs, particularly those at Argonne National Laboratory and Oak Ridge National Laboratory. Much work still remains to be done by the National Laboratories in exploration of basic chemical and technical feasibility of new process opportunities and alternatives for low enrichment power reactor fuels. The ORGDP program supports and extends the National Laboratory efforts in two ways. First, conceptual plant studies leading to statement of technology problem areas, program analysis, and economic comparisons of alternative flow sheets are performed. Second, a component development program is being conducted to cover scale-up and testing of crucial process equipment and auxiliaries. Perhaps the most significant item is a semiworks fluid-bed reactor system to provide scale-up data for the main (head-end) fluid-bed reactor.

Emphasis has continued to be placed on study of low enrichment power reactor fuel reprocessing systems. Reporting will continue to be on the semiannual basis as in the present report, but as substantive completion is achieved in various areas, topical reports will be issued.

SUMMARY

ENGINEERING STUDIES

HIGH ENRICHMENT FUELS

Work has resumed on the report of the multipurpose fluid-bed plant for processing aluminum, zirconium, and carbide-base high enrichment fuels. The body of the report is complete. After the introductory and concluding chapters have been added, edited copies will be sent for comment to the National Laboratories.

LOW ENRICHMENT FUELS

Emphasis at present is centered on the study of processes for zircaloy-clad power reactor fuels. Because of the difficulties associated with the separation of the uranium hexafluoride-plutonium hexafluoride-fission product fluoride mixture after the reaction with fluorine, an alternative flow sheet was investigated in which bromine pentafluoride was used as the fluorination agent. Disadvantages with its use involve additional recovery equipment for the bromine pentafluoride and a separate collection system for plutonium hexafluoride.

Systems Analysis

Problem areas for the process, as defined by the reference (fluorine) flow sheet, are being summarized in a series of process questions with discussions. The work is intended to provide detailed background on the problems and to clarify what has been done so far to solve them, to suggest further work requirements, and to assist in the coordination of the necessary research and development efforts on an overall project basis.

Composition of Irradiated Fuels

The composition of spent irradiated power reactor fuels must be known for conceptual studies on fuel reprocessing plants to permit evaluation of the process problems associated with the fission products, transuranics, and daughter products that may be present. While burnup codes have been found which predict energy spectrums in irradiated fuel, none has been found which gives detailed fission product distributions. A computer code has been written at ORGDP to predict weights, activities, and heat releases for each of the many fission products of concern in volatility reprocessing studies on high enrichment uranium fuels. The code is being reworked for application to low enrichment fuels irradiated in power reactors.

Uranium Hexafluoride-Plutonium Hexafluoride-Fission Product Fluoride Separation

Distillation has been proposed as a means of separating plutonium and uranium from a mixture of uranium hexafluoride-plutonium hexafluoride-fission product fluorides. Computer programs are being developed to evaluate two process schemes incorporating the batch distillation. In the first scheme, a heel would be added to the reboiler; plutonium and uranium, essentially free of fission products, would be recovered overhead in separate cuts. In the second scheme, the plutonium with negligible impurities would be separated out in a thermal decomposer operating simultaneously with the still, while the uranium would be recovered from the condenser.

Bromine Pentafluoride Converter

Bromine trifluoride is formed in the bromine pentafluoride volatilization of uranium from the fluid-bed reactor. Converters have been sized to regenerate the pentafluoride from the trifluoride for recycle purposes. For a converter feed containing 10% bromine trifluoride, an equimolar amount of fluorine could be added in a flame-type reactor, 10 inches in diameter by about 20 feet long, to obtain over 95% bromine trifluoride conversion.

Bromine Pentafluoride-Uranium Hexafluoride Condenser System

Studies have been carried out on the condensation of bromine pentafluoride and uranium hexafluoride from gas mixtures containing large volumes of inert gases. A flash cooling or a reflux condensation operation using additional bromine pentafluoride will lower the liquidus temperature to

below minus 35°C., thus permitting condensation of more than 95% of the condensable material in the gases. For the reflux condenser, 85 sq.ft./kg.mole/hr. of feed condensables is required, while the flash cooler would require three contact stages. Different amounts of bromine pentafluoride are involved with the two processes, and the choice of operation will be based on economic factors associated with the final flow sheet.

Sodium Fluoride Fluid-Bed Sorber

Criticality computations have been made for the sodium fluoride fluid-bed sorber which would remove uranium hexafluoride from the off-gas stream of the two-zone fluid-bed reactor in the bromine pentafluoride flow sheet. It was assumed that $UF_6 \cdot NaF$ and $NaF \cdot HF$ complexes would be present in the equipment. Conservatively, the initial calculations were made for 6% uranium-235 assay, but additional calculations for 2 and 5% assays are now under way. The maximum allowable diameter for the 6% assay was found to be about 24 inches, considering finite length.

PROTOTYPE TESTING AND EVALUATION

Emphasis of the program during the report period has been on the three-step process, i.e., hydrogen chloride decladding, uranium dioxide pellet oxidation, and fluorination treatment, developed primarily by the Argonne National Laboratory for handling spent zircaloy-clad uranium dioxide fuels.

MATERIALS OF CONSTRUCTION - CORROSION TESTING AND RELATED STUDIES

Corrosion tests show that commercially available sintered Inconel filter material will withstand the volatility process conditions if the fluorination outlet gases are cooled to 100°C. before filtering. It is unlikely, however, that this material will be satisfactory if fluorination of the filter at 350°C. is required periodically to convert solid plutonium compounds to plutonium hexafluoride.

Exposure tests have shown that sintered nickel filter material is only slightly attacked by fluorine at 350°C. The only noticeable effect of a 20-hour treatment was a small gain in weight.

Studies with samples submerged in a fluidized bed of alumina indicate that both nickel and Duranickel will be satisfactory materials of construction for the volatility process. The corrosion rates should be 30 to 50 mils per year at the expected conditions.

PROCESS AND REACTOR STUDIES

Efforts in this area have been centered about the cold semiworks head-end system which is now under construction at ORGDP and is intended to furnish engineering-scale information for use in the design of the final fluidized bed volatility plant. Process vessels, including the two-zone fluid-bed reactor, the filter cooler, and a Monel gas absorber, are in fabrication,

and an operating area is being prepared for the installation.

The reactor is constructed of low carbon nickel with Duranickel flanges in accordance with Section VIII of the ASME pressure vessel code. It is equipped with air cooling coils, electric heating elements, and temperature controls for every 12 inches of height. The filter cooler is constructed of so-called commercially pure nickel ($< 0.15\%$ carbon). In the bottom, there is a fluid bed for cooling reactor off-gases, while there are nine porous metal filters in the upper section to catch fines. This vessel is also designed in accordance with Section VIII of the ASME. Cooling coils and electric tubular heaters are attached to the exterior walls for maintenance of temperature control.

OUTLET GAS FILTER STUDIES

Purolator seamless sintered nickel filter tubes held up well under process conditions; however, two of the five tubes purchased had loose end caps. Work with these tubes was discontinued when it was learned that the vendor had stopped manufacturing them.

Evaluation of Micrometallic sintered powder Inconel tubes and Huyck sintered fiber nickel tubes showed that both of these filters are suitable for 315°C . hydrochlorination, 100°C . oxidation, and 100°C . fluorination. Both types of tubes had been rolled and welded longitudinally. The sintered fiber tubes plugged less than any sintered powder tubes studied to date; i.e., about 20% as compared with 80% loss in permeability.

PRODUCT PURIFICATION, SORPTION-DESORPTION SYSTEM

Studies of the sorption of uranium hexafluoride by sodium fluoride pellets show a large effect of gas velocity. Increasing the gas velocity through a packed bed of pellets from 0.08 to 0.19 foot per second decreased the effective sorptive capacity* from about 0.31 to 0.15 pound of uranium hexafluoride per pound of sodium fluoride. Sorptive capacities were essentially the same for cylindrical and annular traps.

CONNECTORS

Satisfactory sealing has been obtained with flat-faced Duranickel flanges compressing Johns-Manville No. 945 nickel gaskets. This type connector stands up well in an inert atmosphere with temperatures cycled between 100 and 370°C .

Good sealing was also obtained with solid nickel O-ring gaskets compressed into grooves milled into the flanges. Alignment should be very critical with this unit. Gas-filled O-rings were unsatisfactory, even with a grooved flange, and removal of the gasket after use was difficult.

* Sorptive capacity is defined as the pounds of uranium hexafluoride sorbed per pound of sodium fluoride when the concentration of uranium hexafluoride in the trap outlet gas first exceeds 1 ppm.

VALVES

A search has been started for a valve suitable for use in the reactor solids discharge line. Ball, plug, and slide-type valves are being considered.

COMPRESSORS

Peripheral Compressor

The prototype compressor has been constructed and installed in a test loop. Preliminary results obtained with air indicate that the unit is meeting design requirements and that it is operating smoothly. Tests will be made with air, hydrogen chloride, fluorine, and uranium hexafluoride under a range of pressures, temperatures, flows, and speeds.

The test rig used previously to evaluate the compressor designs was installed in another facility for life testing at design conditions. The rig was operated at 11,000 rpm. and at the flow rate of the hydrogen chloride application. In air, the suction volume flow rate was about 105 std.cfm., and the two-stage unit delivered a pressure ratio of 2.3, while consuming 27 hp. Smooth operation was achieved for 2,180 hours before being stopped for a high bearing temperature. It is planned to modify the rig to accept a new bearing design which should give even longer service.

Diaphragm Compressor

The Corblin diaphragm compressor was operated for nearly 1,400 hours without a diaphragm failure. Atmospheric suction pressure and a flow of 12 std.cfm. were maintained with a compression ratio of 2. Increasing the compression ratio to 6 had essentially no effect on flow rate. No further studies with this compressor are planned. It appears that the unit will perform as claimed by the manufacturer.

REACTOR FABRICATION PROCEDURES - WELDING STUDIES

A formal procedure has been published for training and qualifying welders to make acceptable welds of nickel-201 to nickel-201 using nickel-201 rod. In addition, welding techniques have been developed for joining Duranickel to nickel-200 and nickel-201 using both INCO-61 and nickel-201 rods.

DISCUSSION

ENGINEERING STUDIES

J. H. Pashley, R. B. Schappel, D. L. Breton, D. I. Dunthorn
C. C. Littlefield, J. R. Merriman, T. Shapiro

HIGH ENRICHMENT FUELS

The study of a multipurpose fluid-bed plant for processing aluminum, zirconium, and carbide-base high enrichment fuels was resumed after suspension for a time to allow concentration on low enrichment fuel processing. Capital and operating cost estimates have been prepared and are being reviewed. The body of the report has been written, but the introduction, summary, conclusions, and recommendations are only in preparation. When the report is complete and has been edited at ORGDP, copies will be transmitted to the National Laboratories for comment. The final copy will subsequently be prepared and published.

LOW ENRICHMENT FUELS

Low enrichment flow sheet alternatives are being investigated. Efforts are being concentrated on processes for zircaloy-clad uranium dioxide power reactor fuels. As described previously, the standard reference flow sheet, which is based on the fluid-bed studies at the Argonne National Laboratory, involves use of hydrogen chloride for decladding at 400°C. with the volatilized zirconium tetrachloride and stannous chloride being pyrohydrolyzed to form solid waste suitable for storage.

The dense uranium dioxide pellets which are essentially unaffected by the hydrogen chloride treatment fall to the bottom of the reactor. In the next step, diluted oxygen gas is employed at about 400°C. to convert the uranium dioxide to powdered urano-uranic oxide which is carried by the fluidizing gas flow into the upper part of the fluid bed of diluent alumina. There, the uranium oxide is reacted with diluted fluorine to produce uranium hexafluoride. For thermodynamic reasons, conversion of the uranium present to volatile uranium hexafluoride occurs rather than fluorination of the plutonium present. More extended fluorination with high concentrations of fluorine at temperatures up to 550°C. is required to complete the plutonium removal. Enough plutonium is, however, removed with the uranium to require separation and recovery from the uranium hexafluoride stream.

The product purification procedure employed involves a combination of distillation for separation of fission products and thermal deposition of plutonium tetrafluoride from the uranium hexafluoride-plutonium hexafluoride stream. Because of the problems involved with these operations, an alternative flow sheet is being investigated which utilizes bromine

pentafluoride for volatilization of uranium hexafluoride free of plutonium. The plutonium hexafluoride cannot form with this reagent for thermodynamic reasons.

The high reaction rate of urano-uranic oxide with bromine pentafluoride also indicates another possible advantage in that the uranium fluorination may be conducted at a lower temperature than possible with the fluorine, and as a result of this milder treatment, the volatilization of plutonium hexafluoride on subsequent reaction with fluorine might be more complete.

Offsetting these advantages are the additional operations necessary with the bromine pentafluoride procedure. These include reconversion of the reaction products, bromine trifluoride and bromine, to bromine pentafluoride by reaction with elemental fluorine; separation of bromine pentafluoride and uranium hexafluoride; and recovery of the bromine pentafluoride, as well as the uranium hexafluoride. Also, a separate fluorination product collection system must be provided for handling the plutonium hexafluoride. Design procedures have been evolved for the converter for producing bromine pentafluoride, for the condenser system for collecting a liquid mixture of bromine pentafluoride and uranium hexafluoride from a dilute gas stream, and for distillation of multicomponent mixtures. Another possibility for separation of bromine pentafluoride-uranium hexafluoride mixtures is the use of a stirred fluid bed of sodium fluoride for removal of the uranium hexafluoride. Future work includes a comparison of the various alternatives for the bromine pentafluoride type flow sheet with the reference flow sheet.

Systems Analysis

Perhaps the most important phase of the engineering studies being conducted is the systematic analysis of proposed flow sheets. The basic objectives of the systems analysis work are (a) the definition and discussion of problem areas; (b) assessment of current state-of-the-art, i.e., status; (c) the determination of further work needed; and (d) the coordination of technical effort to meet process and schedule requirements.

The basic approach being taken is the formulation of questions covering facets of problems for each flow sheet. The effort is to break down each general problem into questions covering part of the overall problem so that each question may be readily handled and discussed with reasonable brevity. For example, a general question might be, "How difficult will the main fluid-bed reactor be to operate?" Questions falling under this general area would include "Will there be a caking problem in the fluid bed?" and "What is the maximum allowable gas velocity to avoid excessive elutriation?" Of course, there are others, and there are questions concerning the charging operation and so forth.

The questions are organized by vessel system and problem category. Vessel systems include the main fluid-bed reactor, reactor off-gas cooling and filtration, pyrohydrolysis fluid bed, hydrogen chloride recovery system, cold trapping system, plutonium tetrafluoride production apparatus, and

distillation system. The problem types considered are operation, process, design, safety, construction, and accountability. For the main reactor, listing is also given by step; i.e., charging, hydrochlorination, dechlorination, oxidation-fluorination, bed dumping, and sampling. Overall flow sheet questions are listed in a general section.

Work is now in progress on preparation of brief write-ups on each question and collation of the questions to obtain workable assignment recommendations. This work will be released in the form of charts on which work locations, target dates, and status are stated as part of the question listing.

The program analysis effort will be closely related to conceptual processing studies in which flow sheet alternatives will be analyzed for feasibility, versatility, and cost.

Composition of Irradiated Fuels

Knowledge of the composition of spent irradiated fuel is necessary for the design of a fuel reprocessing plant. Volatility plant studies require a breakdown by individual nuclide for transuranics, fission, and daughter products to allow consideration of contamination levels and chemical effects in the fluid-bed reactor and in product handling and purification operations.

A high degree of accuracy is not required for conceptual plant studies, since reactor operating conditions, such as burnup, flux levels, and temperatures, are actually, within practical limits, arbitrary to the reactor operator. The basic fuel composition chosen as feed, however, must be representative of a spectrum of fuels generated, and the recovery plant must be suited to process fuels of at least similar composition and burnup.

Perhaps not surprisingly, the amount of information available in the literature as to either calculated or experimentally determined fuel compositions is limited. Some existing computer programs, such as burnup codes, can be employed to provide part of the desired information, but none of the codes surveyed seems to be focused directly on the problem of predicting the chemical composition of spent fuel elements both in detail and with input requirements compatible with parametric studies. For this reason, it was necessary to prepare a general purpose computer program which would provide estimates of the desired quantities.

Discussions of some points of interest related to irradiated fuel composition follow.

Fission Product Compositions. Predictions of fission product compositions in spent power reactor fuel are necessary to determine the eventual dispositions of the materials in the fluoride volatility scheme and to provide bases for choices of removal or separation methods.

Molybdenum can be cited as an example. The element tends to follow

uranium through sodium fluoride sorption-desorption and to form compounds with volatilities similar to that of uranium hexafluoride. Preliminary calculations for one case of interest, made using the ISOCRUNCH code, show that the molybdenum concentration in the fuel studied varies almost linearly with exposure from about 1,000 ppm. (initial uranium basis) at 10,000 Mwd./tonne to 3,000 ppm. at 30,000 Mwd./tonne.

A computer code has been written which predicts fission product and released energy distributions for spent high enrichment fuels. Buildup and decay equations for the various nuclides are solved by a FORTRAN IV version of the CALP subroutine used in the ISOCRUNCH code. Most of the values predicted by the code agree with values computed by laborious hand calculations within 5%. As a result, the code is in use in the preparation of fission product flow sheets for studies involving high enrichment fuels.

Further work is being aimed toward revising the code for use with power reactor fuels. The central problem involved is one of estimating the effects of resonance absorption and temperature on the effective cross-sections of the heavy nuclides. A cross-section calculation subroutine is being added to the code, and at present, it appears that the Westcott procedure for estimating effective cross-sections at various temperatures, modified to correct for resonance self-shielding, will be satisfactory. A preliminary limited check of the subroutine was encouraging, with values for total plutonium production agreeing within 5% of published values obtained using a more sophisticated approach*. Satisfactory agreement also was obtained for individual plutonium isotopes, where values were generally within 15% of the published numbers. Currently, the subroutine is being tested further by comparing more results with values obtained from other sources.

Transuranic Decay Energies. Computations have been made to determine typical decay energies of the several thousand grams of transuranium elements which are present in a tonne of spent power reactor fuel. The transuranic contents of irradiated fuel rods from the Yankee reactor, as reported by Roberts and Van Tuyl†, were used to provide a reasonable order of magnitude for the amounts used in these calculations.

All of the nuclides present are major alpha emitters except plutonium-241, which decays chiefly by β^- . The alpha particles generally have energies between about 4.5 and 6 mev.; also, most of the gamma photons given off in any significant quantities during decay have energies below 0.1 mev., as compared with an estimated average gamma energy near 0.6 mev. for the fission products.

* McConiga, A. F., Fuel Cycle Analysis Chained Computer Programs Guide. A FORTRAN II, Version 2 Monitor System, General Electric Company, December 1, 1963 (HW-79951).

† Roberts, F. P., and Van Tuyl, H. H., Promethium-146, Fission Product, and Transuranium Isotope Content of Power Reactor Fuels, Comprehensive Chemical Analyses, Battelle-Northwest, March, 1965 (BNWL-45).

Neutrons are released from most of the nuclides by spontaneous fissioning and by (α, n) reactions with such light elements as aluminum, oxygen, and fluorine. Of the three elements, neutron yields are highest with fluorine. To obtain the most conservative (α, n) rates, neutron releases were computed for the four irradiation cases assuming all the alpha particles interacted with the fluoride portion of the nuclide tetrafluorides.

A summary of the results of the computations for decay energies and neutron releases are shown in table I. It appears that the (α, n) route for neutron release is generally more significant than spontaneous fission and, for the cases studied, gives totals which are greater than the totals for self-fissioning by at least an order of magnitude. Moreover, the neutron release from transuranics which form nonvolatile fluorides, such as americium and curium, will probably be great enough to preclude the possibility of determining by a direct neutron detection the plutonium concentration remaining in the fluorinated alumina bed.

Uranium Hexafluoride-Plutonium Hexafluoride-Fission Product Fluoride Separation

The thermal decomposition of plutonium hexafluoride to solid plutonium tetrafluoride has been shown to provide a good separation of plutonium hexafluoride from uranium hexafluoride; but when spent fuel fission products are included in the mixture, contamination of the plutonium tetrafluoride deposit is likely as the result of codeposition of fission product fluorides. With an additional distillation step, the fission products can be separated out prior to decomposition. Accordingly, the partition of uranium hexafluoride-plutonium hexafluoride-fission product fluoride mixtures by distillation is under study. The system would be operated at about 2 atmospheres so that uranium hexafluoride could be condensed as a liquid. Initial efforts are centered about batch operations to carry out the separation.

Since the boiling point of uranium hexafluoride is below that of plutonium hexafluoride, the standard procedure for batch distillation would be to remove low boiling fission product fluorides from the top of the still first, then uranium hexafluoride, and finally plutonium hexafluoride, leaving the high boiling fission products as bottoms. This, however, would require that the still reboiler be capable of handling large quantities of material at first and minute quantities during later stages of operation. In addition, the temperature of the still pot would rise considerably in the latter stages of operation as a mixture of high boiling fission products and plutonium hexafluoride is left behind, increasing the rate of plutonium hexafluoride thermal decomposition in the still pot.

One method of countering this problem is to add a high boiling compound to the still charge to act as a reboiler heel in the latter stages of the process. The material must be stable in a strong fluorinating, nuclear radiation environment and have a boiling point somewhat higher than plutonium hexafluoride. The boiling point, however, must not be so high as to accelerate plutonium hexafluoride decomposition or allow passage of

TABLE I

SUMMARY OF TRANSURANIC DECAY ENERGIES
BASED ON SPENT YANKEE REACTOR FUEL DATA

	Irradiation, Mwd./tonne of Uranium			
	13,700	17,700	24,300	31,800
Total Activity, curies/tonne	54,900	70,100	133,600	158,000
<u>Energy Release Rates</u>				
Alpha Release, mev./sec.-tonne	2.91×10^{14}	3.24×10^{14}	1.44×10^{15}	2.13×10^{15}
Beta Release, mev./sec.-tonne	1.39×10^{13}	1.77×10^{13}	3.28×10^{13}	3.83×10^{13}
Gamma Release, mev./sec.-tonne	7.34×10^{11}	8.86×10^{11}	1.24×10^{12}	1.42×10^{12}
Total Release, mev./sec.-tonne	3.06×10^{14}	3.43×10^{14}	1.47×10^{15}	2.17×10^{15}
<u>Estimated Energy Release Rates</u>				
From (α, n) Reactions, neutrons/sec.-tonne	1.57×10^8	1.77×10^8	1.02×10^9	1.55×10^9
From Spontaneous Fission, neutrons/sec.-tonne	6.00×10^6	9.43×10^6	7.31×10^7	1.47×10^8

high boiling fission products out of the reboiler.

An alternative procedure is to take effective advantage of the fact that plutonium hexafluoride-uranium hexafluoride distillative separation is difficult. In essence, as uranium hexafluoride is boiled up into the column from the reboiler, it will carry plutonium hexafluoride with it, but most of the high boiling fission products, being much easier to separate from uranium hexafluoride, will be left in the bottom few stages of the column. An essentially pure mixture of uranium hexafluoride-plutonium hexafluoride will result in the upper part of the column where it may be withdrawn to a thermal decomposer as a side stream.

Calculation methods in the form of digital computer programs are being developed for both techniques. A program has been written which describes the operation of a batch column under steady state, total reflux conditions, the probable starting point for the separation. A first approximation to transient operation during which a side stream is recycled from the column through a thermal decomposer has been carried out as a series of steady state calculations in which total column holdups of plutonium hexafluoride are less in successive steps. The steady state computer program has been incorporated in a larger program designed to give a more accurate picture of the dynamic situation through the use of column differential mass and enthalpy balances. This program, already written, is in the debugging stage at present.

It appears that the system can be made to work either with or without an added reboiler heel. Without a heel, the column side stream to the decomposer may contain plutonium hexafluoride in starting concentrations of less than one-fourth that of the charged material, while the use of a heel may easily allow the same stream to have double the plutonium hexafluoride concentration of the charged material.

Bromine Pentafluoride Converter

The bromine pentafluoride used in the fluidized bed reactor as the fluorinating agent to form volatile uranium hexafluoride is reduced to bromine trifluoride as a result of the reaction. This bromine trifluoride is to be reacted with fluorine in a separate vessel, a converter, to regenerate the pentafluoride for recycle purposes.

The conversion reaction precedes the separation of the halides from uranium hexafluoride to avoid solidification of bromine trifluoride in condensers or cooling equipment. About 100 cfm. of reactor off-gases and fresh fluorine would be fed to the converter at a pressure of 1.4 atmospheres. The reactor off-gases would contain bromine fluorides, uranium hexafluoride, and a high concentration of inerts. Utilization of the bromine pentafluoride in the fluidized bed reactor will average about 80%. The feed gases to the converter will run around 10 mole percent fluorine and 10 mole percent bromine trifluoride. About 1.7 pound moles of bromine trifluoride is to be processed per hour. The converter sizing was considered both for the isothermal and flame-type reactors. The isothermal converter volume required at 400°C., assuming equimolar

fluorine and bromine trifluoride in the feed gases and isothermal conditions, would range between 2 and perhaps 20 cubic feet, depending upon the degree of conversion sought. The reactor volumes required for various conversion fractions at selected reactor temperatures are listed in table II, based on 1 cfm. of feed. The volume requirement decreases as higher reaction temperatures are employed. The equation used for the volume computations was based on a plug flow reactor and is as follows:

$$V_R = \frac{V_o}{kC_{BrF_3}^o} \left[\frac{(1 + \epsilon)^2}{(1 - X)} X + 2\epsilon(1 + \epsilon) \ln(1 - X) + \epsilon^2 X \right]$$

where

V_R = converter volume, cu.ft.

V_o = inlet volumetric gas flow at converter temperature and pressure, including fluorinating gas added (i.e., fluorine plus associated inerts), cu.ft./hr.

$C_{BrF_3}^o$ = inlet concentration of bromine trifluoride, lb.mole/cu.ft.)

ϵ = the fractional change in the volume of the system proceeding from no conversion to complete conversion

X = the fraction of the initial bromine trifluoride converted

k = the reaction rate constant = $RT \exp(17.6707 - \frac{8,411}{T})$ in which T = temperature in degrees Kelvin

TABLE II

REACTOR VOLUMES

Temperature, °C.	Reactor Volumes* Required for Various Percentages of BrF ₃ Feed Converted, cu.ft./cfm. of feed†		
	90%	95%	99%
250	0.931	1.93	9.86
300	0.229	0.48	2.43
350	0.071	0.15	0.74
400	0.026	0.054	0.28

* Assuming a plug flow reactor with equimolar fluorine and BrF₃ in the feed gases. The concentration of the BrF₃ is about 8 mole percent.

† The volume of feed to the converter is computed here at converter temperature and pressure. It should be obvious that the units as reported also represent process holdup time in minutes.

Since about 165,000 Btu./hr. will be released in the bromine trifluoride conversion reaction and considering the rapid increase of rate with temperature, it would appear to be quite difficult to design or operate an isothermal reactor. Provision of sufficient heat transfer area to avoid a temperature rise in the unit would be both impractical and unnecessary. Allowing the reaction temperature to rise should result in a more compact and efficient unit. There are limits on allowable temperature because of the effects on the equilibrium conversion. The effect of equilibrium conversion is shown in table III. The data were computed using the van't Hoff relationship, assuming 10 volume percent bromine trifluoride and fluorine with no bromine pentafluoride initially present.

TABLE III

EQUILIBRIUM CONVERSION OF BROMINE TRIFLUORIDE TO
BROMINE PENTAFLUORIDE AND BROMINE FLUORIDE AT
VARIOUS TEMPERATURES

Temperature, °C.	Fraction Converted to Bromine Pentafluoride	Fraction Converted to Bromine Fluoride
300	1.000	0.0
400	1.000	0.0
500	1.000	0.0
600	0.998	0.0
700	0.990	0.0002
800	0.962	0.0018
900	0.893	0.0106
1000	0.751	0.0438
1100	0.529	0.136
1200	0.279	0.317

The converter has been sized as a single 10-inch diameter pipe reactor with reaction heat removed by cooling at the wall. Computations were made assuming a three-step process:

1. The conversion reaction occurs rapidly in the first step as the gases are introduced. In the reaction volume required, there is a limited amount of surface available for heat transfer; therefore, the temperature rises sharply and essentially adiabatically as the

heat of the reaction is released. A flame reaction takes place.

2. In the second step, the gases are cooled by radiation from the reaction zone, thus permitting the reaction to proceed further, since conversion is favored at the lower temperatures. Both sensible and reaction heats are removed.
3. Further cooling is carried out in the third step, mainly by convective heat transfer.

When average plant flow sheet values and the equilibrium data in table III were utilized to prepare an adiabatic heat balance, the temperature of the reaction zone in the first step was computed to be 700 to 750°C., provided gases enter the converter at 100°C., and there was no preheat required. The temperature in the second and third steps decreased from 700 to 400°C. The vessel would be about 20 feet long overall and would include a 5-foot long internally finned section at the outlet. Thus, for average plant conditions, very close to complete conversion can be obtained.

On the other hand, if the concentration of the bromine trifluoride in the feed to the converter reaches 30%, the temperature in the adiabatic first step is expected to rise above 1000°C. Without temperature control, conversion would drop to less than 75%, and bromine fluoride and fluorine would appear in the outlet gases because of decomposition and incomplete conversion of bromine trifluoride.

Bromine Pentafluoride-Uranium Hexafluoride Condenser System

The off-gases from the fluid-bed reactor in the interhalogen flow sheets contain condensable bromine pentafluoride, bromine trifluoride, and uranium hexafluoride, together with an oxygen-nitrogen mixture which is in excess by a volume ratio of about 4 to 1 over the fluorides. In one version of this process, the gases pass through a converter in which the bromine trifluoride reacts with fluorine to form the pentafluoride and are eventually fed into a condenser for removal of the condensables. Separation by cold trapping at lower temperatures would be extremely efficient but would be objectionable, since a unit to collect solid or solid-liquid product would be large and, in all probability, relatively difficult to operate.

Liquid condensation of these gases could be carried out just short of the liquidus temperature of 0°C., the point at which solid begins to freeze out. Unfortunately, at this temperature, only 15% of the total condensables could be collected as liquid before saturation of the inert gases.

With the introduction of additional bromine pentafluoride to the system, the condensation becomes more efficient. The product liquidus temperature can be lowered to below minus 35°C., and more than 95% of the condensable material can be separated out as liquid. Approaches to condenser operation employing the dilution principle appear technically

feasible. These are as follows:

1. Flash cooling, in which enough cold liquid bromine pentafluoride is added to handle the heat load completely.
2. Reflux condensation, which requires internal heat transfer area and a minimum reflux of bromine pentafluoride to eliminate solid formation.

The liquid condenser product would be distilled to remove the uranium hexafluoride and to supply the bromine pentafluoride needed for recycle to the fluidized bed reactor and to the condenser system. The gas effluent from the condenser may be used as inert recycle to the fluidized bed reactor to use the retained condensables. These effluent gases would contain some bromine pentafluoride but only a little uranium hexafluoride due to the dilution and scrubbing effect of the bromine pentafluoride added. Most of the volatile fission products should be collected in the condensate except for the noble gases and possibly tellurium fluorides.

The condenser design choice depends largely upon economic factors associated with the final process flow sheet. In the end, countering costs and savings must be weighed, for while the uranium hexafluoride loss to the exit gas stream and the required condenser heat transfer area decrease as the bromine pentafluoride recycle rate is increased, the effects on the remainder of the system in terms of distillation and other equipment sizes, flow rates, and heat loads are to increase costs.

Condenser design equations have been programmed for the IBM 7090, and evaluations have been made for a wide range of cases.

In summary, the addition of five times more bromine pentafluoride than inlet condensables in the condenser feed gases will dilute the condensed phase to the bromine pentafluoride-uranium hexafluoride system eutectic and will thus result in the lowest possible operating temperature for the liquid condenser. The addition of more bromine pentafluoride would then only reduce the amount of heat transfer area required. For complete flash cooling, at least eight times more cold liquid bromine pentafluoride is required than the inlet condensables. Representative designs for a flash cooler and a reflux condenser, operating at assumed flow sheet conditions, are shown in table IV.

TABLE IV

DESIGN COMPARISON FOR FLASH COOLER AND
REFLUX CONDENSER FOR URANIUM HEXAFLUORIDE-
BROMINE PENTAFLUORIDE-NONCONDENSABLES MIXTURE

Feed Temperature, °C.	200	
Condensable: Noncondensable Mole Ratio in Feed	0.25:1	
BrF ₅ : UF ₆ Mole Ratio in Feed	4:1	
Condensable: Noncondensable Mole Ratio in Exit Gas	0.0125:1	
	<u>Flash Cooler</u>	<u>Refluxed Condenser</u>
Operating Pressure, atm.	1.5	1.5
Exit Gas Temperature, °C.	- 38	- 38
Liquid BrF ₅ Added: Condensables Originally in Feed, Mole Ratio	10:1	5:1
Temperature of Liquid BrF ₅ Added, °C.	- 50	- 52
Liquid Product Temperature, °C.	7	9
Mole Fraction UF ₆ in Product	0.018	0.033
Contact Stages Required	3	-
Heat Transfer Area Required, sq.ft./kg.mole of feed condensables per hour	-	85

Sodium Fluoride Fluid-Bed Sorber

Solid sorbent applications in the fluoride volatility process include fluidized beds of sodium fluoride for the isolation and purification of uranium hexafluoride in the bromine pentafluoride reactor off-gas stream and fixed sodium fluoride beds as traps to collect fission products or traces of uranium hexafluoride in vent gases.

In the bromine pentafluoride flow sheet, the concentration of uranium hexafluoride in the feed to such a sorber would probably be less than 5 volume percent. Criticality calculations have been made for a system containing uranium hexafluoride, hydrogen fluoride vapor, and sodium fluoride with an assumed uranium enrichment of 6% uranium-235. Hydrogen fluoride was included because there is a good chance it will be present in the process in small amounts as an impurity. The enrichment chosen is also conservative, since the fuel assays are expected to be lower than 6%. Calculations were made assuming the formation of HF·NaF and

$\text{UF}_6 \cdot 2\text{NaF}$ complexes in the fluid bed. While other complexes of sodium fluoride and hydrogen fluoride, such as $\text{UF}_6 \cdot \text{NaF}$, $\text{NaF} \cdot 2\text{HF}$, are known chemically, their formation is dependent on operating conditions. As applied to this flow sheet, the operating conditions would be chosen which would not favor the formation of the undesired complexes.

Although the amount of hydrogen fluoride present would be small under normal circumstances, computations were made to include concentrations of $\text{HF} \cdot \text{NaF}$ ranging from zero to 100 volume percent of the fluidized bed contents to cover even highly improbable operational errors.

The densities of the fluidized bed at different concentrations were based on a uniform homogeneous mixture of $\text{UF}_6 \cdot 2\text{NaF}$ and $\text{HF} \cdot \text{NaF}$ in which no voids were present. Voids between the $\text{UF}_6 \cdot 2\text{NaF}$ crystals were assumed to be filled completely by $\text{HF} \cdot \text{NaF}$ crystals and vice versa.

The density of $\text{UF}_6 \cdot 2\text{NaF}$ is 4.13 g./cc., and that of $\text{HF} \cdot \text{NaF}$ is 2.16 g./cc. In the pure uranium hexafluoride complex, the uranium density is 2.25 g./cc. based on the chemical formula above. Likewise, the hydrogen density in its complex is 0.035 g./cc., which is about one-third of the hydrogen density in water. Therefore, the density of any element in a chosen mixture of the complexes was obtained by multiplying the density of the element in its complex by the volume fraction of the complex in the mixture.

Calculations were made using the DTF transport code for the full range of mixtures of $\text{UF}_6 \cdot 2\text{NaF}$ and $\text{HF} \cdot \text{NaF}$ complexes. The maximum multiplication factor for infinite media, k_∞ , was found to be about 1.25 and to occur at a moderating ratio of about 150 atoms of hydrogen to one atom of uranium-235.

The estimated radii of infinitely long cylinders corresponding to effective neutron multiplication factors, k_{eff} , of 1.00, 0.95, and 0.90 are listed in table V for the optimum atomic hydrogen-to-uranium ratio range. In these calculations, a 30 cm. water reflector was assumed. The minimum critical radius appears to be about 36.5 cm. which occurs at a moderating ratio of about 160. The minimum radius for k_{eff} equal to 0.9 is about 27.5 cm.

TABLE V
RADIi OF INFINITELY LONG CYLINDERS*

Volume Fraction $\text{UF}_6 \cdot 2\text{NaF}$	Moderating Ratio, H/U-235	Radius of Cylinders for Various k_{eff} , cm.		
		$k_{\text{eff}} = 0.90$	$k_{\text{eff}} = 0.95$	$k_{\text{eff}} = 1.00$
0.50	61.3	33.4	39.7	48.4
0.40	91.9	29.4	34.8	41.7
0.30	143	27.9	33.2	38.8
0.275	162	27.6	31.6	37.2
0.250	184	27.5	30.6	36.4
0.225	211	28.4	32.5	38.1
0.200	245	30.7	35.9	41.2
0.150	347	34.3	42.1	53.6

* 30 cm. water reflector.

The critical radii of spheres reflected with 30 cm. of water are given in table VI. Also, the critical mass of uranium for each of the spheres is included in the table. The minimum critical mass occurs at a moderating ratio of about 240 and is about 300 kg. of uranium.

TABLE VI
CRITICAL SPHERE DATA*

<u>Volume Fraction UF₆·2NaF</u>	<u>Uranium Density, g./cc.</u>	<u>Moderating Ratio, H/U-235</u>	<u>Critical Radius, cm.</u>	<u>Critical Mass, kg. of U</u>
0.400	0.901	91.9	56.1	668
0.300	0.676	143	53.8	440
0.250	0.563	184	51.4	320
0.200	0.451	245	55.4	320
0.175	0.394	289	58.4	328
0.150	0.338	347	70.2	490
0.1375	0.310	384	70.8	460

* 30 cm. water reflector.

The results indicate that, for the 6% uranium-235 assay, the maximum allowable diameter of a cylindrical absorber would probably be about 2 feet, considering vessel length. Where lower uranium assays may be assumed, allowable sizes would be greater. This work is being continued with calculations under way for assays of 2 and 5%.

PROTOTYPE TESTING AND EVALUATION

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MATERIALS OF CONSTRUCTION - CORROSION TESTING AND RELATED STUDIES

Evaluations of various materials of construction for use in a volatility reprocessing facility have continued using both the bench-loop and the fluid-bed systems. Studies have been made to determine the corrosion rates of potential reactor, flange, and filter materials under the conditions required for processing zircaloy-clad ceramic fuels. The results of the tests are summarized in table VII and are discussed below.

Filter Materials

The decision to limit the filter temperature conditions during the fluorination phase of the process to 100°C. so that plutonium hexafluoride decomposition is minimized allowed Inconel to be considered as a filter material. In test 10, samples of porous Inconel were exposed to hydrogen chloride and hydrogen at 400°C. and then to dilute fluorine at 100°C. for twenty cycles in the bench-loop unit. After 76 hours of hydrochlorination and 199 hours of fluorination, a slight discoloration of the material was noted, but no indication of deterioration was observed under magnification. These results indicate that Inconel may be acceptable as a gas filter material in a fuel reprocessing facility if the filter temperature during fluorination is maintained at 100°C.

Periodically, it may be necessary to subject the process system to high temperature fluorine so that any plutonium reduced to plutonium tetrafluoride may be reconverted to plutonium hexafluoride. To determine the effects of such conditions on filters, samples of porous nickel were subjected to a fluorine atmosphere at 350°C. Examination after 10 and 20 hours of treatment found the samples to be in good condition. Some slight corrosion was evidenced by a small weight gain.

Flange and Reactor Materials

In corrosion test 11, samples of nickel-201, nickel-201 welded with INCO-61 welding rod, and nickel-201 welded with nickel-201 welding rod were submerged in the alumina bed of the filter loop fluid-bed reactor and were exposed to hydrogen chloride and hydrogen at 400°C., to 10% oxygen in nitrogen at 540°C., and to 20% fluorine in nitrogen at 540°C. Seven cycles involving 35 hours of hydrochlorination, 68 hours of oxidation, and 58 hours of fluorination were completed. Based on weight loss, the corrosion penetrations were alarmingly high at about 600 mils per year. All samples appeared to be attacked uniformly, except that the INCO-61 weld material was markedly pitted. Microscopic examination of the nickel-201 indicated intergranular corrosion penetration as great as 3 mils, figure 1.

TABLE VII
RESULTS OF CORROSION TESTS

Test Number	Materials	Total Number of Cycles	Phase I			Phase II			Phase III			Corrosion Penetration, mils/yr.
			Gas Components	Exposure, Temperature, °C.	Total Exposure, hr.	Gas Components	Exposure, Temperature, °C.	Total Exposure, hr.	Gas Components	Exposure, Temperature, °C.	Total Exposure, hr.	
10	Porous Inconel	20	HCl, H ₂	400	76	F ₂ , N ₂	100	199				
11	Nickel-201 Nickel-201 With INCO-61 Weld Nickel-201 With Nickel-201 Weld	7	HCl, H ₂	400	35	O ₂ , N ₂	540	68	F ₂ , N ₂	540	58	615 650 603
12	Porous Nickel	1	F ₂	350	20							
13	Beryloco-440 Beryloco-440 1/2 HT Beryloco-440 1/2 H Nickel-201 Nickel-200	8	HCl, H ₂	400	30	F ₂ , N ₂	540	80				2.4 3.2 3.2 5.5 4.8
14	Nickel-200 Nickel-201 Nickel-201 With INCO-61 Weld Nickel-201 With Nickel-201 Weld Duranickel	5	HCl, H ₂	400	25	O ₂ , N ₂	500	46	F ₂ , N ₂	540	40	12 12 11 16 7
15	Nickel-200 Nickel-201 Nickel-200 With Nickel-200 Weld Nickel-201 With Nickel-201 Weld Nickel-200 With INCO-61 Weld Nickel-201 With INCO-61 Weld	6	HCl, H ₂	400	30	O ₂ , N ₂	500	60	F ₂ , N ₂	540	60	71 53 53 47 88 100
16	Nickel-200 Nickel-201 Nickel-200 With Nickel-200 Weld Nickel-201 With Nickel-201 Weld Nickel-200 With INCO-61 Weld Nickel-201 With INCO-61 Weld	5	HCl, H ₂	400	25	O ₂ , N ₂	540	50	F ₂	540	50	34 33 31 27 46 51
17	Nickel-200 Nickel-201 Duranickel	20	HCl, H ₂	400	77	O ₂ , N ₂	500	151	F ₂ , N ₂	540	151	5.6 2.4 3.7

TABLE VII (Contd.)

RESULTS OF CORROSION TESTS

Test Number	Materials	Total Number of Cycles	Gas Components	Phase I		Gas Components	Phase II		Gas Components	Phase III		Corrosion Penetration, mils/yr.
				Exposure Temperature, °C.	Total Exposure, hr.		Exposure Temperature, °C.	Total Exposure, hr.		Exposure Temperature, °C.	Total Exposure, hr.	
18	Nickel-200 Nickel-201 Duranickel	5	HCl, H ₂	400	25	O ₂ , N ₂	500	50	F ₂ , N ₂	540	50	40 65 17
19	Nickel-200 Nickel-201 Duranickel	5	HCl, H ₂	400	25	O ₂ , N ₂	500	50	F ₂ , N ₂	540	50	41 46 18
20	Inconel-X	1	HCl, H ₂	400	5	O ₂ , N ₂	500	10	F ₂ , N ₂	540	10	
21	Nickel-200 Nickel-201 Duranickel	27	HCl, H ₂	400	135	O ₂ , N ₂	500	270	F ₂ , N ₂	540	270	35 50 30

Nickel-201
100X Magnification

CORROSION TEST 11
Figure 1

Subsequently, a series of short test runs was made under similar conditions. The results of these experiments, tests 13 through 19, are shown in table VII. Corrosion test runs 14 and 17 were conducted in the bench-loop unit, while the remaining tests were made in the filter loop fluid-bed reactor. Temperatures were varied, and several types of alumina were utilized. Although the corrosion rates experienced in the fluid bed were consistently higher than those in the bench loop, the values were one-tenth of those observed in test 11.

The results of a longer exposure study, test 21, confirm the corrosion rates indicated in the short-term runs. The average expected corrosion penetration should be 30 to 50 mils per year for nickel-200, nickel-201, and Duranickel. Photomicrographs, figures 2 and 3, show that intergranular corrosion is minimal. All results indicate that both nickel and Duranickel will be satisfactory materials of construction for equipment in the proposed hydrogen chloride decladding, pellet oxidation, and oxide fluorination processes.

PROCESS AND REACTOR STUDIES

A smoothly operating fluid-bed halogenator is essential to a successful volatility process for power reactor fuels. The unit must process highly radioactive materials of varying physical and chemical makeup in corrosive atmospheres under poor fluidization conditions. Over a long operating period, only minor maintenance can be tolerated.

To eliminate major uncertainties that would result in the design of a large-scale reactor by basing such design on small-scale laboratory results, process evaluation will be undertaken in a semiworks head-end reactor pilot plant currently under construction at ORGDP. The system accommodates a two-zone halogenator which has a 5-inch diameter section for oxidation and a 10-inch diameter section for fluorination and is thus sized to be intermediate between the small-scale reactors and the anticipated full-scale unit.

Cold Semiworks Pilot Plant

The proposed pilot-plant site has been cleared, utilities have been routed to the area, instrument procurement is under way, and flow sheets and the necessary design work are relatively complete. The process flow diagram is shown in figure 4.

The head-end two-zone fluid-bed reactor and the filter cooler were designed according to Section VIII of the ASME pressure vessel code. Construction drawings of the two units are shown as figures 5 and 6, respectively. The fluid-bed reactor, figure 5, is basically of low carbon nickel construction with Duranickel flanges. The maximum operating temperature will be about 650°C. Special welding processes have been developed for welding low carbon nickel to itself and Duranickel to low carbon nickel, each employing low carbon nickel wire. The vessel stands about 17 feet high. The process operation description with engineering details for decladding and oxidation-fluorination has been described in

Nickel-200
250X Magnification

Nickel-201
250X Magnification

CORROSION TEST 21

Figure 2

Duranickel
250X Magnification

CORROSION TEST 21

Figure 3

PROCESS FLOW DIAGRAM
FLUORIDE VOLATILITY TESTING AND DEVELOPMENT

Figure 4

DESIGN CONDITIONS		VESSEL	COILS
1.	DESIGN PRESSURE	FULL VACUUM TO 30 PSIG	
2.	DESIGN TEMPERATURE	15 PSIG	100 PSIG
3.	OPERATING PRESSURE	15 PSIG	100 PSIG
4.	OPERATING TEMPERATURE	425 PSIG	600 PSIG
5.	HYDROSTATIC TEST	10 PSIG	100 PSIG
6.	SOAK TEMP TEST	NOT REQ'D	NOT REQ'D
7.	STRESS RELIEF	NOT REQ'D	NOT REQ'D
8.	WELD	SEE NOTE #3-D	NOT REQ'D
9.	ASME CODE STAMP	7/16" D	7/16" D
10.	CONNECTION BALANCE		NONE
11.	DESIGN DETAIL	THE VESSEL SHALL BE IN STRICT ACCORDANCE WITH SECTION VIII OF THE ASME CODE	
12.	DESIGN DETAIL	THE COILS SHALL BE IN STRICT ACCORDANCE WITH SECTION VIII OF THE ASME CODE	
13.	PLACES SHALL BE SQUARE AND PERPENDICULAR TO THE CENTERLINE OF THE VESSEL OR NOZZLES.		
14.	WELDING INSPECTION SHALL BE IN ACCORDANCE WITH SECTION VIII OF THE ASME CODE UNLESS OTHERWISE SPECIFIED. WELD EXAMINATION SHALL BE SPECIFIED		
15.	WELDING INSPECTION AND WELD EXAMINATION SHALL BE SPECIFIED		
16.	WELDING OF LOW CARBON WICKER SHALL BE AS SPECIFIED IN AWS 3-A-3.4-A		
17.	WELDING OF STAINLESS STEEL TO LOW CARBON WICKER SHALL BE AS SPECIFIED IN PG 29-2		
18.	WELDING OF LOW CARBON WICKER TO WICKER SHALL BE AS SPECIFIED IN PG 29-2		
19.	WELDING OF COILS TO VESSEL SHALL BE AS SPECIFIED IN PG 29-2		
20.	1. THE VESSEL AND COILING COILS SHALL BE THOROUGHLY CLEANED OF ALL OIL, GREASE, OXIDES, AND CLOSURES		
21.	2. ALL WELDING MATERIALS, AFTER CLEANING, ALL OPENINGS SHALL BE CAPPED WITH TEMPORARY		
22.	CLOSURES		
23.	3. AFTER WELDING DRAINAGE, THE PARTS SHALL BE CLEANERED NOTE D-1 AND HEAT TREATED IN A SULFUR		
24.	4. AFTER HEAT TREATING, STARTING FROM TOP OF VESSEL, HOLES SHALL BE CLEANED TO 1.000" HOLE		
25.	5. WELDING OF TOP TEMPERATURE FOR 6 HOURS. COOL AS SPECIFIED IN INC0 BULLETIN T-3-12 TO OBTAIN 25		
26.	6. WELDING OF COILS TO SHELL MAY BE DONE USING E80-61 RODS.		

Figure 5
TWO-ZONE PRIMARY REACTOR
ASSEMBLY & DETAILS

Figure 6
FILTER - COOLER
ASSEMBLY & DETAILS

a previous report*.

Temperatures for decladding with hydrogen chloride will be maintained at about 450°C., while decladding with hydrogen fluoride-oxygen will be done at about 600°C. The oxidation-fluorination steps will be conducted in the range of 400 to 550°C. Each temperature control zone is about 12 inches high and contains a thermocouple for measurement and control of the wall temperature, an electric resistance heater section fitted around the shell, and tubing welded to the shell for cooling with forced air.

The reactor will be charged directly from the top through a standard 10-inch pipe flange. Process off-gases pass out via a side line to the filter cooler. The off-gas line contains 300 psi. flanges for testing of nickel and other special gaskets.

The filter cooler, figure 6, is also a two-diameter vessel constructed of so-called commercial, pure nickel with INCO-61 welds. It is 10-1/2-feet high with a 12-inch diameter, 2-3/4-foot high bottom section and a 16-inch diameter top. Flanges on the hot side will be Duranickel-lined. The bottom will accommodate a fluidized bed of alumina for cooling incoming gases and is provided with an overflow for use in maintaining the height of the bed at an assigned level in the event that sizable amounts of fines pile up because of carryover from the reactor. There are nine porous metal filters in the upper section directly above the fluid bed. These are equipped with frequent short-term blowback using pressurized nitrogen introduced into the downstream side to jar the filter catch off and drop it back into the fluid bed. The filters are supported from the top of the vessel and may be changed out by disassembling a 16-inch pipe flanged joint on that upper section. The vessel is equipped with wall thermocouples and is wrapped with resistance heaters and cooling coils so that heat control can be maintained separately in the cooling and filter sections.

About 130 kg. of alumina will be charged to the fluid-bed section of the filter cooler through a 2-inch nozzle located at the side of the vessel. The gas flow from the fluid-bed reactor is used to maintain the bed in the filter cooler in the fluidized state. During oxidation and fluorination steps, the reactor off-gases will be cooled from 550 to 100°C. During decladding, the process off-gases will pass through the vessel at a temperature maintained at about 315 to 350°C. This higher temperature will prevent desublimation of zirconium tetrachloride and stannous chloride vapors which are present in dilute concentrations in the hydrogen chloride declad procedure. About the same temperature would be used to prevent condensation of aqueous hydrogen fluoride should the hydrogen fluoride-oxygen step be in use.

* Smiley, S. H., Brater, D. C., and Pashley, J. H., ORGDP Fuel Reprocessing Studies, Summary Progress Report, Fiscal Year 1964 Through Fiscal Year 1965, Union Carbide Corporation, Nuclear Division, Oak Ridge Gaseous Diffusion Plant, October 29, 1965 (K-1649).

In the semiworks facility, the reactor and the filter cooler will be placed at roughly the same elevation. The line connecting them will be in the form of an inverted "V", with the legs sloped at 20 degrees off the vertical to prevent solids holdup in the line. In all probability, the fluid beds from both vessels will be dumped after each test run for weighing and sampling. This placement was chosen to reduce headroom requirements, to expedite solids removal through shortening of drain lines, and to permit manual operation of the drain valves from a single alumina collection station below the equipment.

OUTLET GAS FILTER STUDIES

Tests of one make of molded (seamless) nickel filter tubes were discontinued when it was learned that the vendor had stopped manufacturing these units. The tubes had performed satisfactorily and exhibited good corrosion resistance to standard process conditions of hydrogen chloride and hydrogen at 315°C., oxygen at 100°C., and fluorine at 100°C.; however, inspection showed that 2 of the 5 tubes purchased had loose end caps.

Since bench-scale corrosion tests of Inconel indicated that this material might be acceptable as filter media, two commercially made 3-inch diameter by 3-foot long sintered Inconel powder tubes were installed in the filter test loop. The tubes were formed from porous sheet. Seam closure and attachment of solid end hardware was accomplished by welding with INCO-82T rod. The filters were exposed to the exit gases from a bed of alumina fluidized at a gas velocity of about 1 foot per second. The exposure cycle consisted of hydrogen chloride and hydrogen at 315°C. for 5 hours and then fluorine at 100°C. for 10 hours. Nineteen complete cycles were completed involving 95 hours of hydrochlorination, 190 hours of oxidation, and 190 hours of fluorination. An automatic blowback system provided a reverse flow of 0.12 cfm. of nitrogen per square foot of filter area every 12 minutes. Three types of alumina were used in the fluid bed: Alcoa Tabular (minus 60 mesh); Alcoa Tabular (48 to 100 mesh); and Norton RR Alundum (60 to 100 mesh). Sieve analyses of these materials are summarized in table VIII.

Filter flow data, which were taken throughout the test for each of the two tubes in the filter are presented in table IX. The results show an immediate loss in permeability of about 80%, followed by a gradual increase in porosity throughout the test. Visual inspection of the tubes found them to be in good condition, with no indication of chemical attack. An inspection of the interior of the tubes using a Boroscope showed no evidence of powder.

Two 3-foot long, 3-inch diameter filter tubes manufactured by Huyck Metals Company were tested under conditions similar to those described in the Inconel tube test. The Huyck tubes were constructed of sintered nickel fibers on nickel wire backing and were welded with an INCO-82 rod. Inspection after ten cycles of exposure showed that the tubes were in good condition with little evidence of corrosion attack. Flow measurements made before and after exposure indicated a 17% loss in permeability which is much lower than the losses (up to 80%) experienced with

TABLE VIII
SIEVE ANALYSES OF ALUMINA

Mesh Fraction	Minus 60-Mesh Tabular Alumina, %	48- to 100-Mesh Tabular Alumina, %	60- to 100-Mesh Norton Alundum, %
+ 35	0	0	0
- 35 + 60	0	7	90.7
- 60 + 100	7	72	9.3
- 100 + 140	13	17	0
- 140 + 200	14	0.5	0
- 200	66	3.5	0

TABLE IX
INCONEL FILTER FLOW MEASUREMENTS

	Air Flow at Indicated Pressure Drop, std.cfm.			
	1 Inch of Water	2 Inches of Water	3 Inches of Water	4 Inches of Water
<u>Tube No. 48</u>				
Prior to 1st Run	9.9	20.0	29.9	-
After 11th Run	2.7	5.5	8.2	11.0
After 16th Run	3.6	7.3	11.0	14.6
After 19th Run	4.2	8.5	12.7	17.0
<u>Tube No. 52</u>				
Prior to 1st Run	10.7	21.5	32.5	-
After 6th Run	1.4	2.9	4.3	-
After 11th Run	2.6	5.2	7.8	10.5
After 16th Run	3.6	7.2	10.8	14.4
After 19th Run	4.4	8.8	13.9	17.8

the samples of two brands of sintered nickel powder tubes. Further testing of these Huyck tubes is planned.

PRODUCT PURIFICATION, SORPTION-DESORPTION SYSTEM

Studies of product purification involving sorption-desorption methods were continued in the cylindrical pellet trap and the annular pellet trap. All the tests were concerned with uranium hexafluoride collection and recovery; studies of selective sorption of impurities from uranium hexafluoride are not planned at this time.

Cylindrical Pellet Trap

Several sorption runs were made in the 4-inch diameter by 7-foot long pellet trap at flow rates of 0.3 std.cfm. and 0.7 std.cfm. which correspond to linear gas velocities of 0.08 and 0.19 foot per second, respectively, assuming 100% voids. The sorbent used was made by heating 1/8-inch sodium bifluoride pellets to 540°C. for 5 hours. Sorptions were conducted at 120°C. with gas concentrations of 10% uranium hexafluoride, 15% fluorine, and 75% nitrogen. The desorption of the uranium hexafluoride from the sodium fluoride-uranium hexafluoride complex was accomplished by heating the trap to 400°C. for 14 hours. A small flow of fluorine was used to prevent formation of uranium pentafluoride. Results of the tests are shown in table X. The sorption ratio (pound of uranium hexafluoride per pound of sodium fluoride at breakthrough) was greatly reduced on increasing the gas velocity to the 0.19 foot per second value. Further studies are planned at higher velocities and lower uranium hexafluoride concentrations.

TABLE X

RESULTS OF 4-INCH SORPTION TRAP TESTS

Run Number	Gas Velocity, ft./sec.	Sorption Time, hr.	Desorption Time, hr.	Sorption Ratio*, lb. UF ₆ /lb. NaF
F-1	0.08	7.0	14	0.35
F-2	0.08	6.1	14	0.33
F-3	0.08	4.8	14	0.26
F-4	0.08	4.3	14	0.31
G-1	0.19	1.8	14	0.24
G-2	0.19	1.0	14	0.15
G-3	0.19	1.3	14	0.15
G-4	0.19	1.0	14	0.14

* Pound of uranium hexafluoride per pound of sodium fluoride at breakthrough.

Annular Trap

A prototype sorbent trap of annular design consisting of concentric 5- and 12-inch diameter Monel pipes and having a 60-inch bed height has been under test. In several of the early test runs on this large trap, operating data indicated possible gas channeling through the pellet bed. In order to reduce this possibility, intermittent vibration and rapping was applied to the bottom and sides of the trap while the sodium fluoride pellets were being charged to the unit. As a result, about 10% more sorbent material was charged than in previous attempts.

Sorption ratios, however, were no better than those obtained previously. The average sorption ratio for six runs was 0.16 pound of uranium hexafluoride per pound of sodium fluoride using a gas velocity of 0.19 foot per second and concentrations of 10% uranium hexafluoride, 15% fluorine, and 75% nitrogen. Since this value is comparable to the results obtained with the same gas velocity in the 4-inch trap, there appears to be no deleterious effect of the annular design.

CONNECTORS

Service conditions for joints or connectors in the fluoride volatility process will be quite rigorous due to the high temperatures and the corrosive gas atmospheres involved in the operation. Design work on this problem during this period has centered on the adaptation of age-hardened Duranickel flat-faced flanges compressing various configurations of dead soft nickel gaskets. Provisions have been made to use double gasketing so that the pressure between the seals could be monitored to detect leaks.

In all tests, the flange loading required for satisfactory sealing of the joint was determined by bolt elongations using the following equation:

$$\text{Loading, lb.} = \frac{\Delta L \ E A \ n}{L}$$

where

A = cross-sectional area of the bolts, sq.in.

E = modulus of elasticity of bolt material, psi.

L = length of bolt elongated, in.

ΔL = average bolt elongation, in.

n = number of bolts used

After a connector unit was assembled, the joint was placed in a furnace and heat cycled between 100 and 370°C. maintaining the temperature for 2 hours at each temperature. The sealing performance was monitored by means of a pressured gas buffer on the gasket.

A Johns-Manville nickel gasket No. 945 withstood the heat cycling satisfactorily for twenty cycles. This gasket is of the multiple milled touchpoint design of four concentric rings and required a flange loading of 200,000 pounds on the 4-inch, 150-pound flanges to effect a tight seal. Since this loading seemed objectionably high, a two touchpoint gasket, figure 7, similar to the Johns-Manville gasket No. 945, was fabricated in the ORGDP shops. This connector required a flange loading of 60,000 pounds, and it withstood the heat cycling treatment satisfactorily for twenty cycles.

Attempts to accomplish a satisfactory seal using gas-filled nickel O-rings as shown in figure 8 were unsuccessful. This type joint has two major disadvantages: (a) a grooved flange of critical dimensions is required for the O-rings, causing the gaskets to stick in the grooves and presenting problems in removal by means of remotely controlled manipulators; and (b) the flange surfaces must be very clean for a satisfactory seal.

Two-joint assemblies were also made using double O-rings of solid 1/8-inch nickel wire which had been annealed dead soft. One unit was assembled at about 100,000 pounds loading force, and the other was assembled at 237,000 pounds force on the flange. After ten heat cycles, each assembly had exhibited a leak rate of approximately 0.02 cc. of air per minute. Disassembly of the joints failed to reveal the leak paths.

A joint consisting of two 4-inch Duranickel, 150-pound flanges compressing solid nickel O-rings into grooves milled in the flanges, figure 9, remained leak-tight for seventeen cycles of heat treatment. A total of 130,000 pounds of flange loading force was required for this seal. Alignment of grooves and O-rings would be critical in this assembly.

A flanged valve unit, utilizing grooved flanges and heavy copper O-ring type gaskets, proposed for use in the ORNL hot pilot plant, was heat-cycled in the flange testing unit to determine the sealing capabilities for its application in the fluorination system. The unit remained leak-tight after twenty-two cycles. It was somewhat difficult to remove the oxidized copper rings from the unit on completion of the test.

Selection of the proper bolt material is apparently important in proper joint assembly for application in areas of considerable temperature change. While heat cycling of the various types of connectors in the temperature range of 100 to 370°C. caused no problems, leaks developed when the maximum temperature was increased to 540°C. Differences in elongation of the materials over the wide temperature range were probably responsible for the difficulty. The search for an ideal bolt material for this application will be pursued.

VALVES

Periodically, it will be necessary to empty the contents of the fluoride volatility fluid-bed reactor to reduce the radiation level and possibly to withdraw unreacted materials involved in the process. A preliminary

PHOTO NO. PH-66-102

ORGDP DOUBLE MILLED TOUCHPOINT GASKET

Figure 7

GAS FILLED NICKEL O-RINGS

Figure 8

SOLID NICKEL O-RINGS IN GROOVED FLANGES

Figure 9

survey of possible vendors for an applicable valve was made using the following specifications as a basis for the discussions:

Type: Straight-through configuration, radiation resistant, hermetically sealed, remote operator required.

Size: 2 to 8 inches

Materials of Construction: Nickel, Duranickel, halogen-resistant ceramics

Operating Pressure: 20 psig.

Operating Temperature: 540°C. maximum

About thirty manufacturers were verbally contacted, with most of them admitting that the service was much too extreme for their units; however, several vendors requested an opportunity to bid on such a valve. Therefore, specifications are being formulated for ball, plug, and slide-type valves, which may be tested in the component test loops. These specifications will be submitted to prospective vendors for preliminary bids.

COMPRESSORS

In the volatility process, recycle of process gases will be required in the fluorination steps and probably during the hydrogen chloride dechlorination. Two types of recirculating pumps were chosen for testing: a Corblin diaphragm compressor and a peripheral unit developed at ORGDP.

Peripheral Compressor

Additional optimization testing of impeller designs for the application was completed. Tests on several impellers with blade angles of 90, 75, 60, and 45 degrees (measured from the plane of the impeller) showed that the 60-degree bladed impeller was best. This design was incorporated into the prototype compressor design. Fabrication, assembly, and installation of the prototype compressor was completed, and evaluation of its performance is in progress. The compressor was equipped with high precision (ABEC-9) Fafnir angular-contact ball bearings packed with Andok 260 grease and preloaded with 100 pounds force. The rotor, including the motor rotor and Duranickel impeller, was precision balanced before assembly in the unit to ensure smooth operation at 11,000 rpm. The compressor was installed in a loop in which its characteristics could be determined under various conditions simulating expected requirements in the fuel reprocessing facilities. It will be tested in air, hydrogen chloride, fluorine, and uranium hexafluoride under a range of pressures, temperatures, flows, and speeds. Preliminary results obtained in air indicate that the compressor is meeting design requirements and that it is operating smoothly.

The test rig used to evaluate the compressor designs was installed in another facility for duration testing at design conditions. The main

purpose of the test was to evaluate further the performance of the bearings and grease. The use of grease-packed bearings in these applications is near the upper limit in speed and size usually employed (DN = 550,000 in the load bearing); however, the advantages of using grease rather than oil lubrication make the attempt worthwhile. The rig was operated at 11,000 rpm. and the flow rate of the hydrogen chloride application. In air, the suction volume flow rate was about 105 cfm., and the two-stage unit delivered a pressure ratio of 2.3 while consuming 27 hp. Bearing, gas, cooling water, and casing temperatures were continuously monitored; gas pressures, power, speed, and water flows were recorded once per shift; and shaft and impeller clearances were observed each day. The rig operated in this manner for approximately 2,180 hours before a high bearing temperature shut it down. The temperatures of the bearings in this rig, also packed with Andok 260 grease, generally ran about 50 and 70°C. for the rear and load bearings, respectively, with about $\pm 2^\circ\text{C}$. variation, depending upon ambient conditions. It is planned to modify the test rig to accept a newer bearing design which should give even longer life.

Diaphragm Compressor

The Corblin diaphragm compressor tested in this program was designed to operate either as a single or dual head unit. The unit was assembled with both sets of 26-inch diaphragms, and the fluid volume between the diaphragms in the primary and remote heads was filled with fluorocarbon oil. The compressor was then operated without a diaphragm failure for nearly 1,400 hours compressing fluorine about 60% of the time. Atmospheric suction pressure and a flow of 12 std.cfm. were maintained with a compression ratio of 2. Delicate balancing of the fluids in the pump unit essentially eliminated the vibration problems incurred in the early operation of the pump. Adjustments to the primary pump fluid relief valve and to the fluorocarbon oil volume were also necessary to obtain the maximum flow rate.

Additional testing with air showed that the mass flow rates of this compressor are essentially unaffected by discharge pressure over the range investigated. The results of these studies are shown in table XI. The pump suction pressure was atmospheric in all cases, and the discharge pressure was limited to 75 psig. to protect the instruments used in the test loop.

TABLE XI

EFFECT OF DISCHARGE PRESSURE ON
CORBLIN COMPRESSOR PERFORMANCE

<u>Discharge Pressure,</u> <u>psig.</u>	<u>Air Flow,</u> <u>std.cfm.</u>
10	10.6
15	10.5
20	10.3
25	10.1
30	10.1
40	10.5
50	10.4
60	10.6
70	11.1
75	11.0

No further studies with this compressor are planned. It appears that the unit will perform as claimed by the manufacturer. A separate report on the tests is being prepared.

REACTOR FABRICATION PROCEDURES - WELDING STUDIES

A formal plant welding procedure has been published for training and qualifying welders to make acceptable welds of nickel-201 to nickel-201 with nickel-201 welding rod*. In addition, welding techniques have been developed for joining Duranickel to nickel-200 and nickel-201 using both INCO-61 and nickel-201 welding rods. Face bend and root bend tests showed no cracking, radiographs indicated no porosity, and tensile strength tests gave acceptable values. Cleanliness was of great importance in the attainment of an acceptable weld in all cases.

All the above procedures are being used in the assembly of the semiworks reactor and filter-cooler now being constructed in the ORGDP shops. The techniques will be used also in the fabrication of units for the hot pilot plant being installed at ORNL.

* See Appendix A.

APPENDIX A

Engineering Specification No. ESP 4.3-44A

June 29, 1965

PROCEDURE SPECIFICATION FOR THE TIG WELDING OF
LOW CARBON NICKEL WITH LOW CARBON NICKEL FILLER METAL

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UNION CARBIDE CORPORATION
NUCLEAR DIVISION
ORGDP, Oak Ridge, Tennessee

Number ESP 4.3-44A

Date June 29, 1965

PROCEDURE SPECIFICATION FOR THE TIG
WELDING OF LOW CARBON NICKEL WITH LOW
CARBON NICKEL FILLER METAL

Revised

Page 1 of 6

SCOPE

This specification covers the welding procedure and the welder qualification tests for the TIG welding of low carbon nickel plate or pipe for application in extremely corrosive environments. This procedure is applicable to metal thicknesses 0.125 inch through 0.436 inch.

PART A - WELDING PROCEDURE

WELDING QUALIFICATION

All welding performed in accordance with this procedure shall be done by welders qualified in accordance with Part B of this standard. Individual welder qualifications shall expire 30 days after the last use of this procedure for Code quality work.

BASE METAL

The base metal shall be low carbon nickel pipe, plate or shapes, INCO Grade 201.

FILLER METAL

The filler metal shall be bare low carbon nickel wire, INCO Grade 201.

INERT GAS

Helium with a minimum purity of 99.995% shall be used for both the torch shielding gas and the backup gas.

ELECTRICAL CHARACTERISTICS

Welding shall be done using direct current, straight polarity; the base metal shall be connected to the positive terminal.

POSITION

Welding may be done in all positions.

PREPARATION FOR WELDING

The ends of the plate or pipe to be joined shall be beveled by machining or grinding in accordance with the groove details on page 6.

Number	ESP 4.3-44A	60
Date	June 29, 1965	
Revised		PROCEDURE SPECIFICATION FOR THE TIG WELDING OF LOW CARBON NICKEL WITH LOW CARBON NICKEL FILLER METAL
Page	2 of 6	

Cleanliness at the weld site and of the metal is essential to final weld quality. The welding site shall be clean and free of oil and grease. The metal to be welded must be degreased in a fast evaporating organic solvent; paint, marking inks or crayons or other potential contaminants must be removed from the metal surface at the immediate weld zone.

Prior to welding, both the filler wire and the base metal at the joint shall be resurfaced to remove any oxide film. This can be accomplished by machining, grinding, grit blasting, hand rubbing with an emery cloth, or chemical cleaning. When possible, flash pickling is recommended. This should be done in a solution composed of 20% H₂O, 30% H₂SO₄, 50% HNO₃ (volume ratios) plus 60 grams of NaCl per liter, successively dipping and water rinsing the metal until the surface attains a bright lustre.

White gloves shall be worn on handling cleaned metal and filler wire to avoid contamination of the weld joint surface or filler wire with skin oils. Clean, grease-free gloves shall be used during welding.

The gap opening of the assembled joint shall be uniform. Components shall be secured by either tacks or with low carbon nickel spacer lugs. The materials to be joined shall be supported to prevent stresses on the joint during the welding operation.

WELDING TECHNIQUE

Welding shall be done in accordance with the parameters shown on page 6. Inasmuch as the filler wire is virtually unalloyed, melting and solidification occur abruptly without incidence of a sluggish puddle. For this reason, the best welding control combination is a high heat input with a fast travel rate. The wire shall be fed into the puddle in fast, short jabs, and then pulled quickly and cleanly away. Approximately 1/8 inch of wire shall be fused off with each jab. The tip of the wire shall not be pulled outside of the inert gas shield of the torch.

Since the filler metal does not contain deoxidizers, special precautions must be taken to assure good tie-ins. Oxide films on the base metal or welding wire will disrupt the metal flow and affect bonding. The tip of the filler wire shall be clipped to remove any part exposed to the preceding bead heat. All new arcs shall be struck on freshly ground surfaces or surfaces precleaned with a motorized rotary stainless steel brush. Before each start-up on the critical first pass, the lead-off edges of the bead and the tack edges shall be feathered approximately 3/8 inch long by

PROCEDURE SPECIFICATION FOR THE TIG
WELDING OF LOW CARBON NICKEL WITH LOW
CARBON NICKEL FILLER METAL

grinding to remove any oxide film developed with the preceding deposition. The flow of inert gas from the torch shall be retained at each stopping point until the metal returns to its natural color. The back purge flow shall be retained until the metal has cooled to prevent internal oxidation.

The tungsten electrode shall be ground to a sharp tip and shall protrude approximately 1/4 inch outside the cup. The tip shall be kept clean and resharpened when it becomes blunt.

All welding shall be performed in a draft-free zone, protected by shields if necessary.

DEFECTS AND CLEANING

Areas containing cracks, blowholes, or tungsten contamination shall be cut out with a disc grinder before depositing an overpass. The metal surface shall be brushed with a clean stainless steel brush to remove oxides and grinding dust as frequently as is needed during welding.

PRE- AND POST- HEAT TREATMENT

None required.

X-RAY

Radiographs, where specified, shall be made in accordance with Paragraph UW-51, Section VIII of the ASME Code.

GENERAL

It shall be the responsibility of the inspector and welding supervisor to determine that all welds meet the requirements of this specification. Either shall have the right to require the welder to requalify when his weld quality is substandard to this specification.

PART B - WELDER PERFORMANCE TEST PROCEDURE

QUALIFICATION REQUIREMENTS

Each welder shall pass the tests described in this section to qualify for welding in accordance with this procedure.

Number	ESP 4:3-44A	62
Date	June 29, 1965	
Revised		PROCEDURE SPECIFICATION FOR THE TIG WELDING OF LOW CARBON NICKEL WITH LOW CARBON NICKEL FILLER METAL
Page	4 of 6	

WELDING REQUIREMENTS

The welder shall follow the procedure defined in Part A above. All test welding shall be witnessed by an inspector who may refuse acceptance whenever details of this standard are violated.

TEST MATERIALS

Four 4-inch long by 3-inch diameter schedule 40 Nickel 201 pipe spools are required; one end of each spool shall be machined beveled $37\frac{1}{2}^{\circ}$ as shown on page 6. All spools and the $3/32$ -inch bare filler wire shall be flash pickled as described in Part A.

TEST WELDS

Grooves for these tests shall be fitted in accordance with the joint details on page 6.

Test No. 1. 2G Position - Make a vee-butt weld with the pipe axis vertical and the weld groove horizontal. Stencil the numbers 1, 2, 3, and 4 on one butt end at 90° intervals, respectively.

Test No. 2. 6G Position - Make a vee-butt weld with the pipe axis horizontal and the weld groove vertical. The pipe shall be fixed, and not rotated or tilted during welding. After fixing in the test position, stencil one end with the numbers 1, 2, 3, and 4 at the 1:30, 4:30, 7:30 and 10:30 o'clock positions, respectively.

NON-DESTRUCTIVE TEST AND INSPECTION

The finished test welds shall have a workmanlike appearance, without voids, cold laps, cracks, or undercut. The reinforcement crown shall be generally uniform in width and height, and shall be $1/16$ -inch $\pm$ $1/32$ -inch high.

Weld penetration at the root shall be complete and without undercut; the root crown height shall not exceed $1/16$ inch.

Each test weld shall be radiographed in accordance with paragraph UW-51, Section VIII of the ASME Boiler Code. The size and distribution of porosity shall not exceed the ASME Porosity Limits for $1/4$ -inch weld thickness.

DESTRUCTION TESTS

Welds meeting the visual and radiographic requirements shall be subjected to bend tests. Saw cut four 1-inch wide by 8-inch

PROCEDURE SPECIFICATION FOR THE TIG
WELDING OF LOW CARBON NICKEL WITH LOW
CARBON NICKEL FILLER METAL

longitudinal coupons from each spool, each coupon to be centered at an original stencil number. Both root and reinforcement crowns shall be ground or machined flush with the associated base metal surface and the coupon edges rounded to an approximate 1/16-inch radius.

Coupons 1 and 3 of each spool shall be face bent and coupons 2 and 4 root bent in a guided bending jig proportioned for 0.216-inch thick metal.

TEST RESULTS REQUIRED

The bent coupons shall have no cracks or other open defects on the convex surface exceeding 1/16 inches measured in any direction; cracks on the corners shall not be considered unless they originate from defects in the weld.

RETESTS

In case a welder fails this test, a retest may be taken under the following conditions: An immediate retest consisting of two complete welds of either or both positions failed; or a complete test of the entire procedure following one additional week of supervised training.

ASSIGNMENT OF CODE

A welder qualified under this performance test shall be qualified to weld in accordance with ESP 4.3-44A.

PERIOD OF EFFECTIVENESS

The qualification of a welder passing this test shall be considered in effect indefinitely, but shall expire (1) 30 days after the last use of the procedure as defined in Part A, or (2) if the capability of the welder to make welds comparable to this standard is questionable. In either case, qualification can be regained by passing 2G position test.

RECORDS

Records certifying test results shall be initiated by the inspector and maintained on file by the Welding Engineering Department.

Number	ESP 4.3-44A	64
Date	June 29, 1965	
Revised		
Page	6 of 6	
PROCEDURE SPECIFICATION FOR THE TIG WELDING OF LOW CARBON NICKEL WITH LOW CARBON NICKEL FILLER WIRE		

DETAILS FOR PIPE TEST WELDMENTS
(3" Diameter)

2G POSITION
(Test No. 1)

5G POSITION
(Test No. 2)

SCHEDULE 40		PASS NO.	FILLER WIRE SIZE	AMPS	HE FLOW CFH	
SIZE	INCH				TORCH	PURGE
1-1/2	.148	1 2-3	3/32 3/32	90 95	25 25	40* 20
3	.218	1-3 4-6	3/32 3/32	100 120	25 25	40* 20
OVER 3"	TO .436	1-3 4-UP	3/32 3/32	105 125	25 25	40* 20

PASS NO.	FILLER WIRE SIZE	AMPS	HE FLOW CFH	
			TORCH	PURGE
1 2-3	3/32 3/32	90 95	25 25	40* 20
1-3 4	3/32 3/32	100 120	25 25	40* 20
1-3 4-UP	3/32 3/32	105 125	25 25	40* 20

Cup Size - No. 7

Tungsten Electrode - 3/32" dia., thoriated

Amperage may be varied $\pm 10\%$.

*Flow rate of 40 cfh on 1st pass only; use 20 cfh on all subsequent passes.