Chemical Processing and Equipment

VOLUME SIX

Chemical Processing and Equipment

UNITED STATES OF AMERICA

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SELECTED REFERENCE MATERIAL UNITED STATES ATOMIC ENERGY PROGRAM

Chemical Processing and Equipment



UNITED STATES OF AMERICA

Foreword States Atomic Energy

Interchange of scientific and technical knowledge will greatly facilitate the work of the scientists and engineers whose skills will be devoted to the future development of the peaceful uses of atomic energy.

The United States has made available to the world's scientific community a large body of such data. In honor of this historic Conference and to stimulate further exploration and development of the beneficial applications of nuclear energy, the United States Atomic Energy Commission has prepared this special collection of technical data for the use of the delegates and the nations represented.

The purpose of this collection is to provide information concerning the ways that we have found in which fissionable materials can be put to work in nuclear reactors for research purposes and for the production of power and radioisotopes.

It is our sincere hope that this material will be of practical value to the men and women of science and engineering in whose hands the great power of the atom is becoming a benign force for world peace.

Reus L Straum

Chairman, U.S. Atomic Energy Commission

SELECTED REFERENCE MATERIAL ON ATOMIC ENERGY

VOLUME ONE Research Reactors

VOLUME TWO Reactor Handbook: Physics

VOLUME THREE Reactor Handbook: Engineering

VOLUME FOUR Reactor Handbook: Materials

VOLUME FIVE Neutron Cross Sections

Volume six Chemical Processing and Equipment

VOLUME SEVEN Eight-year Isotope Summary

Volume eight Information Sources

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National Reactor Test Station (Phillips Petroleum Company)

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Oak Ridge National Laboratory (Carbide and Carbon Chemicals

Company)

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Contents

CHEMICAL PROCESSING OF REACTO	OR FU	VEL ELEMENTS AT THE IDAHO	
CHEMICAL PROCESSING PLANT	Γ	giliiningingiliinggaalaagaa	1
PROCESS	3	Samplers Product handling equipment	30
Process Requirements	4	Product handling equipment	
Fuel Cooling	4	Filter handling equipment	31
Fuel Dissolution	4	Instrumentation	31
Feed Preparation	5	DECONTAMINATION OF EQUIPMENT	
Solvent Extraction	10	FOR MAINTENANCE	00
Product Handling	11	5 20 months of math	33
Waste Handling	12	Necessity	33
Solvent Recovery	13	Decontamination Methods	34
Recovery of Uranium Salvage		Efficiency	34
		Advantages over Remote Maintenance	36
PLANT FACILITIES	14		
Design Philosophy	14	ANALYTICAL SECTION OF THE	
Arrangement	15	IDAHO CHEMICAL PROC-	
Main Process Building	15	ESSING PLANT	37
Laboratory facilities and administrative	-	Facilities TAMES AND	37
area	19	Equipment	37
Source of fissionable-material storage	1833		
canal ps. as psp 3. ps. page	19	HEALTH PHYSICS AT CHEMICAL	
Waste Disposal Building and Waste Tank	20	PROCESSING PLANT	38
Farm	21	Definition of Responsibility	38
Service Building	22	Permissible Radiation Levels	39
Ventilation System	22	Radiation Present	39
Vessel Vent System	22	Radiation and Contamination Protection	00
vood vone dystein	22	and Control	40
PROCESS EQUIPMENT	23	Personnel monitoring	41
Process Vessels	23	Instruments	42
Piping and Valves	25	Wastes	43
Mechanical Equipment	27		TO
Special Equipment	28	COSTS	44
Fuel element chargers	28	Distribution of Capital Cost	44

Contents

LABORATORY EQUIPMENT			45
Hot Laboratory Design Features ENCLOSURES FOR RADIOACTIVE OPERATIONS	47 51	Mixers Sample Manipulative Devices Miscellaneous Equipment	207 215 217
Shielded Cells, Caves, Barriers, Dry Boxes, and Portable Shadow Shields	51	PHYSICAL MEASUREMENT EQUIP- MENT	219
Unshielded Dry Boxes, Gloved Boxes, and Hoods Accessories for Enclosures	73 87	Apparatus for Weight Measurement Apparatus for Measurement of Physical and Mechanical Properties	219
VIEWING AND OPTICAL EQUIPMENT	95	MACHINE TOOLS	235
Shielding Windows Periscopes, Telescopes, and Cameras	95 103	MATERIALS-HANDLING EQUIPMENT	249
MANIPULATORS	113	Containers Container Openers and Sealers	249 261
General-purpose Manipulators Restricted-motion Manipulators Accessories for Manipulators	113 133 147	Transfer Equipment Storage Facilities	269 279
Miscellaneous Manipulators	159	DECONTAMINATION AND MONITOR- ING INSTRUMENT ARRANGE-	
CHEMICAL EQUIPMENT	161	MENTS	281
Fluid-sampling Devices Fluid-flow Devices	161 175	IRRADIATION FACILITIES	287
Vessels Heaters and Coolers	195	Radiographic Equipment	287

Chemical Processing of Reactor Fuel Elements at the Idaho Chemical Processing Plant

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Chemical Processing of Reactor Fuel Elements at the Idaho Chemical Processing Plant

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Chemical Processing of Reactor Fuel Elements at the Idaho Chemical Processing Plant

It is not possible completely to burn up the fissionable material from the fuel elements in a nuclear reactor because the build-up of fissionproduct poisons, the depletion of the fissionable material, and the radiation damage to the elements eventually require their replacement. Economical operation of the reactor requires that the unconsumed fissionable material be recovered from the spent fuel elements. This section describes a successfully operating plant for the decontamination and recovery of fissionable material from fuel elements. Its purpose is to familiarize the reader with the problems connected with recovery operations and to give details of the process operations and plant facilities. As reprocessing operations must always be considered an integral part of any nuclear reactor research or power program, it is hoped that this information will be of value to those planning nuclear projects in the future.

The Idaho Chemical Processing Plant (ICPP) is designed to recover fissionable material from spent reactor fuel elements from various experimental and power-producing reactors in the United States. The plant is located at the National Reactor Testing Station near Idaho Falls, Idaho. The initial process development for the plant processes was carried out by the Oak Ridge National Laboratory (ORNL). The present operating contractor is the Phillips Petroleum Company of Bartlesville, Oklahoma.

This account is confined to describing the facilities, equipment, and process initially provided for uranium recovery from enriched uraniumaluminum alloy fuels such as those used in the materials-testing reactor (MTR) and the bulkshielding reactor. Much of this equipment is, of course, adaptable to the processing of other types of fuels.

The high value of the uranium in the fuel elements demands that the process be capable of essentially complete uranium recovery. In addition, the process is required to effect nearly complete separation of the fission products from the uranium.

This plant is the first Atomic Energy Commission processing facility that has been designed for direct, rather than remote, maintenance. No provisions have been made for remotely controlled removal or repair of the process equipment. Thus, in the event of equipment failure or the necessity of modification, chemical solutions must be used to decontaminate the equipment before personnel can enter the process areas to carry out maintenance or construction work. As the radioactive solutions processed contain as much as 150 curies of radioactivity per liter, effective procedures for decontamination are an absolute necessity for the successful operation of this type of plant.

PROCESS

The process consists of three main steps: (1) dissolution of the uranium-aluminum elements in nitric acid, (2) adjustment of the dissolver solution to a composition suitable for solvent extraction, and (3) separation of the uranium from the aluminum, fission products, and transuranic elements with which it is associated in the fuel elements. The separation is accomplished by continuous liquid-liquid extraction employing methyl isobutyl ketone (hexone) as the solvent.

A schematic flow sheet of the process is shown as Fig. 1.

by neutron capture in U²³⁶. It constitutes only a small fraction of the total activity at the time

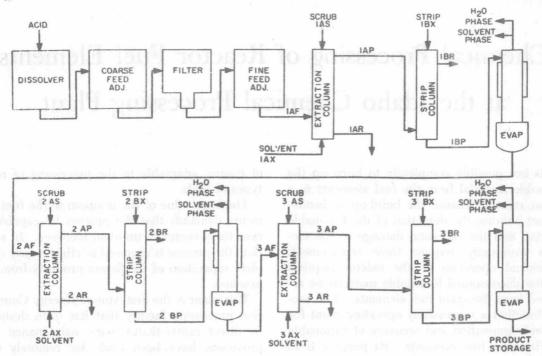


Fig. 1 Simplified feed preparation and extraction flow sheet.

Process Requirements. The process is required to recover greater than 95 per cent of the unconsumed uranium, produce a uranium product essentially free of plutonium, and reduce the fission-product activity to a level that will allow further processing of the uranium without shielding. In the case of MTR fuel, the fission-product concentration must be reduced by a factor of approximately 10⁶. These requirements have been successfully met in all processing runs.

Fuel Cooling. At the time of discharge from a reactor, the fuel elements are intensely radio-active, owing primarily to approximately 100 short-lived fission products. It has been estimated that an MTR element will have about 2×10^8 curies of activity associated with it. This activity may be greatly reduced by storing (cooling) the irradiated elements and allowing these fission products to decay. Four days of cooling reduces the activity by a factor of about 10^3 , and 135 days by a factor of 10^4 . However, the activity that determines the minimum permissible cooling time is U^{237} , which is formed

the irradiated fuel is discharged, but, since it is not possible to separate the isotopes of uranium during chemical processing, the fuel assemblies must be cooled until such time as the U²³⁷ activity in the recovered uranium is reduced to a low enough concentration to permit product handling and further processing without shielding.

Fuel Dissolution. The first chemical operation after cooling is the dissolution of the assemblies in nitric acid. Nitric acid dissolves finely divided aluminum quite readily, but, owing to the formation of passive surfaces of aluminum oxide, thick pieces of the metal are incompletely dissolved. The presence of mercury catalyzes the reaction of nitric acid with aluminum and allows complete dissolution, presumably owing to amalgaination and galvanic effects. Dissolving procedures for all fuel elements that contain aluminum include the use of mercuric nitrate as a catalyst. Dissolution of uranium metal in dilute nitric acid involves the primary reaction

 $U + 4HNO_3 \rightarrow UO_2(NO_3)_2 + 2NO + 2H_2O$

The dissolution of aluminum in nitric acid involves a number of reactions which may be approximately summarized in the over-all reaction

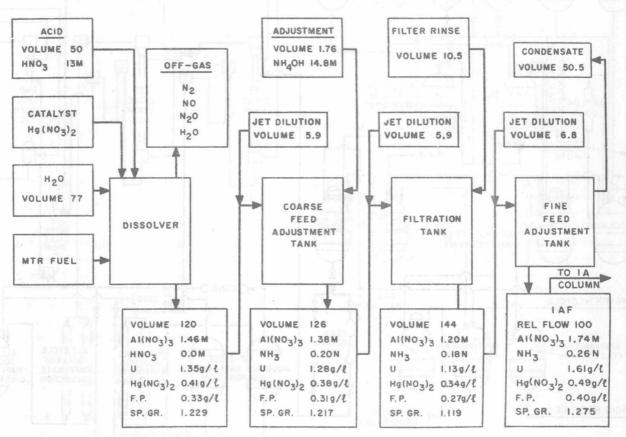
$$Al + 3.7HNO_3 \xrightarrow{Hg} Al(NO_3)_3 + 0.15NO + 0.10N_2O + 0.175N_2 + 1.85H_2O$$

All the above reactions and others occur in the course of a normal dissolving, and it is difficult to predict accurately the composition of the gaseous reaction products. However, the acid consumption is always about 3.7 moles per mole of aluminum dissolved.

In addition to the gaseous reaction products shown above, volatile fission products are liberated during dissolution.

and are dissolved in nitric acid (3.7 moles per mole of metal) containing a weight of mercuric nitrate equivalent to 1 per cent of the element weight. The dissolution is carried out at boiling temperature. MTR elements dissolve rapidly; and to control the reaction rate, only one-half of the acid is added initially and the balance is metered in over a 2-hr period. The dissolver batch is digested for a total period of 7 hr at boiling, at which time the dissolution is 100 per cent complete.

Feed Preparation. The solution from the dissolvers is adjusted to a composition suitable for extraction-column feed in the feed-preparation equipment. These operations are carried out in



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Fig. 2 MTR dissolution and feed preparation flow sheet.

MTR elements are dissolved in a batch operation as shown on the chemical flow sheet (Fig. 2) The elements are charged to the dissolvers batches following the chemical flow sheet for MTR elements shown in Fig. 2. The equipment for these operations is shown in Fig. 3.

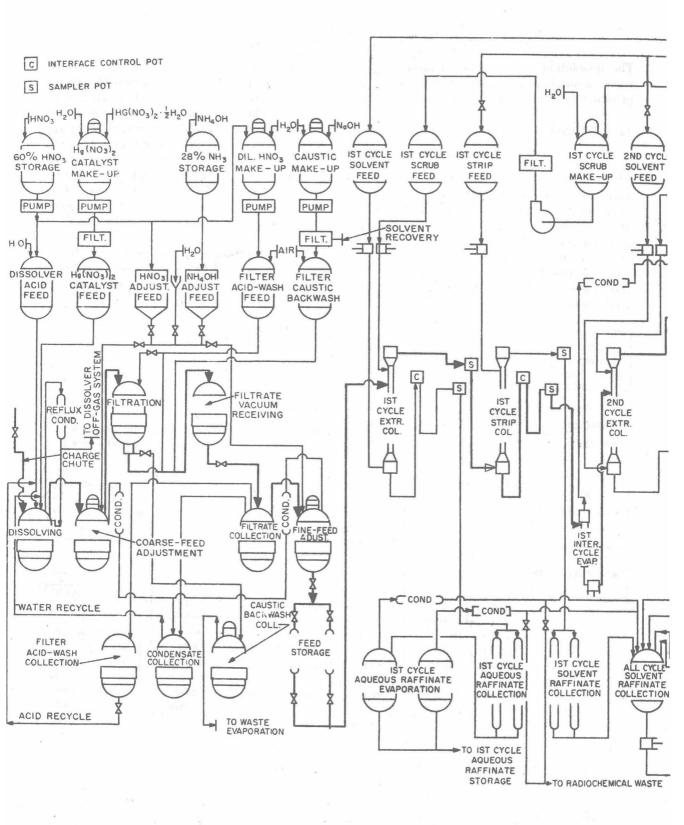


Fig. 3 Over-all process equipment flow

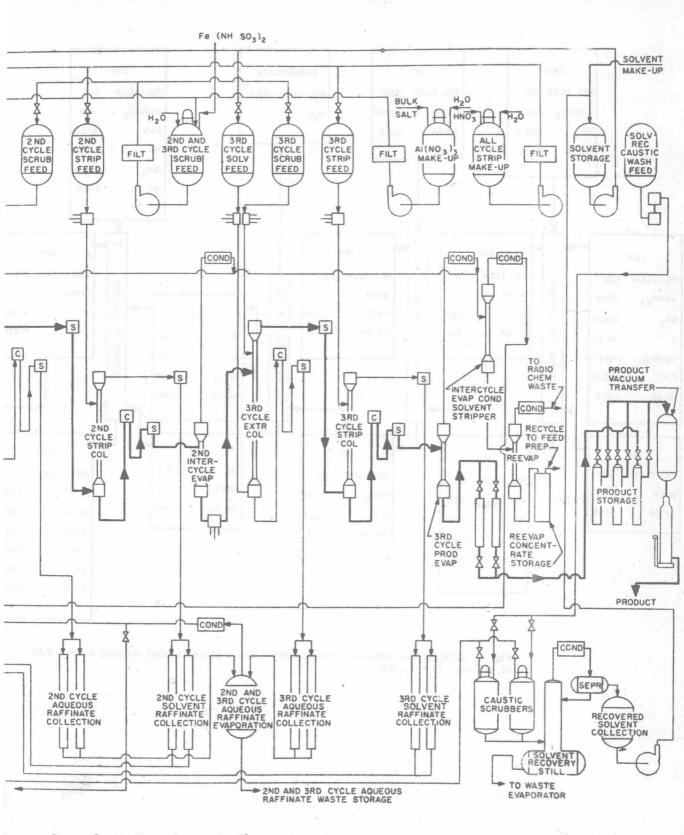


diagram for the Chemical Processing Plant

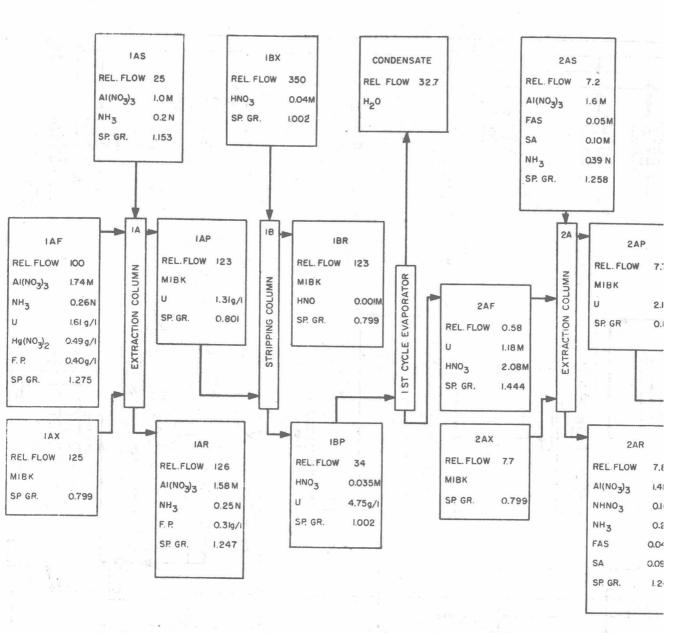


Fig. 4 MTR solvent extraction chemical flow sheet. MIBK, methyl isobutyl ketone; FAS, M, molarity; N, normality.