

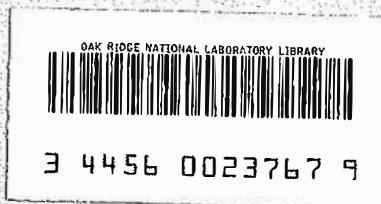
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RESULTS OF THE ICPP-ORNL DAREX COORDINATION MEETING
at Idaho Chemical Processing Plant, November 20-21, 1961

E. L. Nicholson and J. A. Buckham

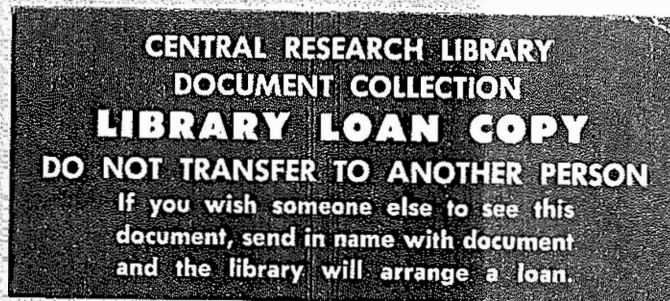
ABSTRACT

This report summarizes the agreements reached in a meeting at the Idaho Chemical Processing Plant on November 20-21, 1961, between ORNL and Phillips Petroleum Company technical personnel in regard to criteria for the proposed Darex Pilot Plant. The objectives of the pilot plant will be to develop processes suitable for production use for low- and highly-enriched U-235 fuels and for thorium fuel. A pilot plant capable of both batch and continuous processing with a highly-enriched fuel capacity of 6 to 10 kg/day U-235 and equivalent volumetric throughput for low-enriched fuels is planned. Flowsheets were reviewed and final experimental and engineering studies were assigned to permit preparation of a tentative schedule for Darex design, procurement, and installation.

This document has been reviewed and is determined to be
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Name/Title: Leesa Laymance/ORNL TIO

Date: 1/23/2021



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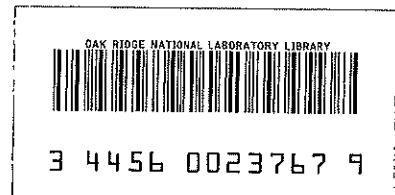
RESULTS OF THE ICPP-ORNL DAREX COORDINATION MEETING
at Idaho Chemical Processing Plant, November 20-21, 1961

Present: ORNL - F. L. Culler, F. G. Kitts, E. L. Nicholson
ICPP - J. A. Buckham, P. Burn, H. V. Chamberlain, R. G. Denney,
D. K. MacQueen, J. A. McBride, C. M. Slansky, J. I. Stevens,
F. M. Warzell, M. E. Weech

1.0 OBJECTIVES OF THE DAREX HOT PILOT PLANT INSTALLATION

The objectives of the Darex Hot Pilot Plant program were stated to include the following:

- (a) The principal objective is to develop processes suitable for production use and to obtain data necessary for design of a production facility. As such, the hot pilot plant Darex installation must be an experimental unit operable under a wide variety of conditions. Use of this unit itself as a production facility is a secondary objective.
- (b) Demonstration of the Darex flowsheet for fuels that would be of interest to a private fuel reprocessing company. This includes slightly enriched uranium and thorium fuels.
- (c) Demonstration of the Darex flowsheet on an existing backlog of highly enriched fuel both to demonstrate the process and, as a secondary objective, to eliminate the backlog.
- (d) Demonstration of continuous Darex processing with provisions also included for batch processing of fuels presenting unusual problems such as those fuels having a high silicon or niobium content or containing $\text{ThO}_2\text{-UO}_2$ ceramic.
- (e) Demonstration of satisfactory removal of chloride from solvent extraction plant feed solutions prepared by the Darex Pilot Plant.
- (f) Demonstration of the removal of chloride to innocuous levels from gaseous wastes that are routinely discharged from the Darex Pilot Plant. Attempts will be made to eliminate routine discharge of chloride-bearing radioactive waste so a new waste storage system may be avoided. Dumping of the plant chloride inventory prior to plant repair might be by removal in drums after distillation to remove activity.
- (g) Possibly, the demonstration of the use of soluble nuclear poisons as a primary criticality control.



2.0 DAREX PILOT PLANT THROUGHPUT

A capacity between 6 and 10 kg/day for continuous processing of highly enriched fuel or an equivalent capacity of low enriched fuel was stated to be a satisfactory target, pending more exact information on the solvent extraction system capability. The ICPP and ORNL continuous flowsheets satisfied this condition. Reduced throughput with UO_2 - ThO_2 fuels is unavoidable but not detrimental to process demonstration. The capacity attainable with batch operations for highly enriched fuel requires further study and is dependent on criticality control methods.

3.0 LOCATION OF THE DAREX PILOT PLANT AND FUEL CHARGING

The Darex Pilot Plant will be located in an ICPP Hot Pilot Plant cell, 11'-6" x 17'-6" x 30' high. The previously proposed method of fuel element charging through existing curved charge tubes through a partition wall was discarded because it would not permit charging long power reactor fuels. The fuel will be charged vertically downward from the roof of the pilot cells. Studies will be made by ICPP of the problems of handling casks for long fuel elements in this area. A closure will be required on the slug chute to confine fumes within the dissolver.

The concept of shop assembly and piping of equipment in groups on frames and lowering the completed equipment group into the cell appeared desirable to minimize field welding and expenses. Studies will be made by ICPP to determine the maximum frame size that can be moved into the pilot plant.

4.0 PROCESS FLOWSHEETS

Extensive discussion was held on the proposed flowsheets: CPP-E-2962 (Phillips); E-45975, E-45976, E-45978, E-45977 (ORNL). The Phillips flow-sheet is for continuous processing of highly enriched stainless steel fuel using 60% nitric acid makeup for stripping the chloride from dissolver product. The ORNL flowsheets covered both batch and continuous operation for highly enriched stainless steel fuel using 90% nitric acid as the stripping acid for the batch systems and for spiking recovered acid to make up ~70% HNO_3 stripping acid for the continuous systems. The ORNL flowsheets also included a continuous Yankee Atomic Processing scheme and a batch scheme for Consolidated Edison fuel.

The Phillips system envisioned feeding acidified dissolver product directly to the chloride stripping column in which stripping vapor was provided by a reboiler operating on the column bottoms. Rather high HNO_3 content reboiler effluent (chloride free) was diluted to give solvent extraction feed solution. Column overheads were then fractionated to give aqua regia overhead for recycle to dissolution and 60% nitric acid bottoms for recycle to feed acidification. The Phillips chloride removal system was based on extrapolation of laboratory liquid-vapor equilibrium data taken in the presence of iron salts.

The ORNL continuous system envisioned surging dissolver product in the batch system feed adjustment tank and feeding from this reservoir at a constant rate to the chloride stripping column in which stripping vapor was provided by a boiler supplying 15.8 M nitric acid to the bottom of the column. The highly acidic column bottoms (chloride free) were evaporated in an external evaporator to yield strong nitric acid overhead (for butting with 90% HNO_3 and recycle to the 15.8 M HNO_3 boiler) and a low acid content salt solution which was diluted to give solvent extraction feed. Chloride stripper tower overheads were combined with dissolver off gases and condensed to produce dilute aqua regia for adjustment and recycle to the dissolver. The ORNL chloride removal system was based on limited unit operations scale continuous experiments with an integrated dissolver, chloride stripper, and feed evaporator system processing stainless steel fuel.

Basic differences in approach to such problems as criticality control, off-gas treatment, liquid wastes, requirements for batch processing, etc. were resolved or are to be the object of further study.

Main points of agreement are:

1. The system will be capable of both batch and continuous operation. Fuels will include selected high and low enrichment uranium and thorium fuels.
2. The feed adjustment tank will be incorporated in the system, thereby permitting batch operation also. The criticality problem of this vessel for the continuous flowsheets must be worked out. Soluble poisons should be considered.

3. The use of 90% HNO_3 simplifies operations and offers a saving in waste disposal for batch operation; it also makes possible the use of higher strength stripping acid and, hence, more complete chloride removal for the continuous system. It will be used. A dilute acid throwaway will be avoided if possible.
4. The general scheme embodied in the ORNL flowsheets will be adopted as a basis for process design by ICPP.
5. The ORNL flowsheets included a caustic scrubber to remove traces of chloride from the off gases. The Phillips group thought that the amount of chloride in off gases might be low enough that it could be handled by dilution in the main stack. It was agreed to provide space for the caustic scrubber but leave it out pending results of cold startup testing of the pilot plant.
6. Solids removal from the system by mechanical methods (filtration, centrifugation, etc.) should be avoided. Fundamental studies on Si and Nb and a limited amount of laboratory scale stripping experiments with solids present will be done by ORNL. ICPP will examine the possibility of testing the effects of the FAT tank high temperature hold period on solids during their stripper experiments, if these experiments are performed with dissolver effluents rather than synthetic solutions. Solids are the outstanding single problem interfering with a successful continuous Darex process.
7. ORNL will consolidate and analyze existing data applicable to the use of soluble poisons.
8. A study of the dynamics of the ORNL stripping system will be made at ORNL to resolve questions involving response time and system stability.
9. Previous ORNL Darex equipment design work should be incorporated wherever possible to speed the job. Complete sets of drawings should be prepared.
10. The dissolver should be made as large as possible (i.e., as large as criticality control will permit) to minimize splitting of large fuel elements. It can be made with a square cross-section if necessary. ICPP will investigate this.
11. Stripper sizing may have to be deferred until completion of cold pilot plant tests. Stripper reboiler will be a separate vessel to permit versatile operation. ICPP will perform the necessary additional pilot plant tests on the chloride stripping process. Such tests will include evaluation of the relative advantages of bottom and top addition of nitric acid to the stripper. Data regarding behavior of chlorine-containing compounds in the stripper should also be obtained from these studies.

12. ORNL will revise their chemical and material balance flowsheets in accordance with the results of this meeting.

5.0 SCHEDULE

Scheduling studies indicate that some instruments, most piping materials, and some vessels might be procured by the end of this fiscal year.

After reviewing the flowsheets and digesting the results of this meeting, Idaho will prepare a tentative schedule for the Darex design, procurement, and installation.

E. L. Nicholson, ORNL and J. A. Buckahm, ICPP

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