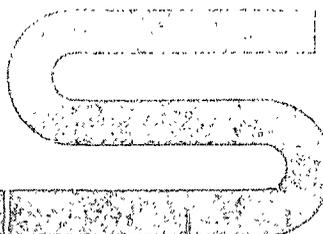


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REVIEW OF EXPERIENCE GAINED IN FABRICATING NUCLEAR GRADE
URANIUM AND THORIUM COMPOUNDS AND THEIR ANALYTICAL QUALI
TY CONTROL AT THE INSTITUTO DE ENERGIA ATÔMICA, SÃO PAU
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Introduction

As part of the national program for developing atomic energy for peaceful uses, directed by Brazilian Nuclear Energy Commission (CNEN), the Instituto de Energia Atômica (IEA), São Paulo, has giving great emphasis to a systematic development of research on establishing of the technology of ura nium and thorium, training of chemists and engineers, and production of some nuclear materials for further metallurgic work and fabrication of fuel ele ments for nuclear research reactors. This paper reviews the main activities on the recovery of uranium from ores, the purification of uranium and tho rium raw concentrates and their transformation in nuclear grade compounds. The design and assemblage of pilot facilities for ammonium diuranate (ADU), uranium tetrafluoride, uranium trioxide, uranium oxide microspheres, uranyl nitrate denitration, uranium hexafluoride and thorium compounds are discus sed. The establishment of analytical procedures as an important support to the technical work is emphasised.

Recovery of Uranium from Ores

For the establishment of the technology of U recovery from domestic resources, some work has been done on unexploited till now phosphate rock. The extraction and recovery of U from phosphates of Northeast Brazil were investigated by treating the ores with HCl. The process, developed on a large laboratory scale, was also applied to the phosphate rock leaching solutions obtained industrially /1/.

Several alternative schemes were evaluated for the extraction and separation of U, Mo and V values of sulfuric acid leach liquors, in connection with the development of an acid leaching process for the U-Mo ore from Poços de Caldas plateau, Minas Gerais State /2/. A separation method of U from Mo exploring the complex sulfates of U(VI) and Mo(VI), their affinity for anionic ion exchangers, and the adsorption of Mo on activated charcoal is published /3,4/.

Ion-exchange Ammonium Diuranate (ADU) pilot plant

Two pilot plant facilities were assembled for the production of nuclear grade natural ADU from Brazilian uranium concentrate as part of the program to develop a capacity for the production of fuel elements for research reactors. The establishment of the necessary technology and training of personnel were the main objectives of this research and development work. This program comprised several steps in transforming the yellow cake to nuclear purity uranium compounds.

The first facility set up for the purification of uranium is based on an ion-exchange process. A previous report /5/ described the concentrate, a sodium diuranate produced from the industrial processing of monazite /6/ and its dissolution for the preparation of uranyl nitrate solution. The main impurities considered in the concentrate are sodium, phosphate, silicium, iron and, of course, thorium and rare earth (RE). Great concern was given to the decontamination of Th and RE. The loaded strong cationic resin is washed with water and then with dilute HNO_3 and eluted with ammonium sulfate. ADU is precipitated by flowing the eluate into ammonium hydroxide and keeping the reaction medium at a pH not less than 6,5 to maintain the coprecipitated sulfate to a minimum level of 0.5%/U₃O₈ /7/. The precipitate is dewatered by filtration in a canvas filter and dried at 110°C. The equipment, operational flowsheet and results were described /5/. More information related to the technology of purification of U by cationic ion exchanger were pu

blished /8,9/. The behavior of Th present as main impurity was studied /8/. The decontamination of Th and the sorption of UO_2^{++} were studied /9/.

Solvent Extraction Pilot Plant

A second pilot plant facility set up for the purification of U is based on the conventional liquid-liquid extraction technique. The facility and its equipment, operational flow-sheet, performance and gained experience were published /10,11/. The facility comprises a section for the dissolution of the yellow cake and three pulsed columns with perforated plates for the extraction of uranyl nitrate (UN) with TBP-varsol, scrubbing and stripping of pure UN. Details are given /10/ for the pure ADU precipitation by continuous and discontinuous process and the instrumentation control panel. The pioneer work for the establishment of purification of U with pulsed columns was carried out some years before /12,13/.

Dissolution of Yellow Cake and Extraction of UN

The dissolution of the yellow cake is accomplished into a reactor of 300 l capacity. The digestion of DUS is made with 2M HNO_3 at 90-100°C for the complete flocculation of silica. The filtered UN has a concentration of 475 g U/l and is adjusted to 300 g U/l, 1M in HNO_3 and $NaNO_3$ as salting out agent for the extraction. To overcome the troublesome presence of Th and RE, controlled addition of sodium sulfate is done /12,13/. The pulsed columns were dimensioned according the experience gained in theoretical and practical work /14,15,16/. The extraction is done in countercurrent with a org/aq ratio of 2.2. The loaded organic phase (OP), with 135 gU/l, is scrubbed with 0,2 M HNO_3 with org/aq ratio of 10. The washed OP (110-115 g U/l) is stripped with water using an aq/org ratio of 1.6, resulting a uranyl nitrate solution of 70-105 g U/l. Stripping the OP at 60°C raised the UN concentration to 100 g U/l /10/.

Precipitation of Ammonium Diuranate

The ADU is obtained by bubbling undiluted anhydrous NH_3 gas into a UN of 100 g U/l, at 60°C, final pH of 7.0-7.5. The pilot plant is equipped to perform the precipitation in a batchwise way and as continuous operation as well. The ADU is dewatered into a vacuum canvas filter and has a umidity ranging from 45 to 50% /10/. Experience gained with the continuous precipitation is described /17/. The reactor is fed at a rate of 2 l/min, at 60°C, and NH_3 bubbled at a rate of 56 l/min. The final pH is 4.5 or 7.0-7.5

During the preparation of UO_3 as feeding for the UF_4 plant some problems arose concerning the mechanical resistance of the agglomerate oxide.

By suggestion of the UF_4 pilot plant adviser (Mr. Paul Vertès, AIEA's expert), the precipitation of ADU was done at a final pH of 4.0-4.5. The ADU precipitated at higher pH is sent to the ceramic grade UO_2 fabrication. Moist cakes of both ADU type were batch-tray dried at 110°C.

UO_3 Facility

A facility /10/ for the conversion of ADU to UO_3 comprises a continuous, electrically heated, belt furnace. The ADU is calcined into stainless steel trays, each one with a capacity of 6 kg ADU. The furnace has a temperature gradient from 110 to 500°C. The trioxide is used as feeding for the UF_4 plant or sent to the Metallurgical Division for further transformation in ceramic UO_2 . The capacity of the furnace is of one tray each 12 minutes, with a residence time of about two hours.

Treatment of Aqueous Effluents

The uranium pilot plants give rise to some solutions containing U, Th and RE. All effluents are collected and treated with NaOH. The dried precipitate assessed about 25%U and 14%Th, Fe, Ti and RE being also present.

Selective Sorption of UO_2Cl_2 into Strong Anionic Resin

As mentioned before, the unique Brazilian U concentrate used until present time has as peculiar characteristics the presence of Th and RE. A technique for the transformation of crude SDU into nuclear grade ADU was developed and carried on a large lab scale /18/. HCl acid is used for the dissolution of SDU with the purpose of exploring the selective sorption of uranyl anionic chlorocomplexes on a strong anionic resin. Uranyl chloride is eluted with deionised water, giving a pure UO_2Cl_2 solution averaging 100-105 g U/l; therefore, reasonably higher when compared with TBP-extraction /10/ or cationic ion exchanger pilot plant /5/.

Extraction of Uranyl Chloride by Tri-n-Octylamine (TOA)

The extraction of UO_2Cl_2 by TOA and the decontamination of several impurities with the aid of thiourea (tu) is investigated at a lab scale. The effect of tu giving rise to unextracted cationic species of several metallic ions and its consequence in the extraction of UO_2Cl_2 with TOA is described /19/.

UF_4 Pilot Plant

Some previous works as a programmed study of preparation of UO_2 appropriate for conversion to UF_4 on a large laboratory scale (20 kg per batch), starting from ADU and using NH_3 as reducing agent were carried out /20,21/. The study for the preparation of UF_4 by hydrofluorination of UO_2

had as main purpose to acquire experience in this technology and found out the best operational conditions, envisaging the installation of a pilot unit /21/. After those preliminar studies, a pilot plant facility to acquire the necessary technology on the UF_4 production for further uses in the reduction to U metal and preparation of UF_6 was set up /22/. The establishment of this unity had the collaboration and technical assistance and some financial support from the International Atomic Energy Agency (IAEA). The project was designed by the IAEA's expert, Mr. Paul Vertès. The starting material is UO_3 , reduced to UO_2 by cracked NH_3 and anhydrous hydrogenfluoride for the conversion to UF_4 .

Just recently the process of obtaintion of UO_3 was changed. The ADU precipitated at high pH (7.5) for ceramic grade UO_2 is calcined to UO_3 in the continuous belt furnace and then pelletized in spheres of about 4-6 mm, dried again and used as feed material in the L reactor. This new type of UO_3 exhibited excellent mechanical properties.

Facility for Denitration of Uranyl Nitrate

Development studies on a fluidized bed process for conversion of uranyl nitrate solution to uranium trioxide and recovery of nitric acid has been done. It will comprise a 3" \emptyset reaction chamber containing a bed of UO_3 powder fluidized by upward flowing air and heated by external electric heaters. Construction of the equipment, installation and operational tests are scheduled for 1977-1978 period. The acquired technology and experience will be transferred to the denitration of thorium nitrate and denitration of disposable waste solutions.

Electrolytic Fluorine Generator and UF_6 Preparation

Solely as part of training program several experiments were carried out for the preparative obtaintion of UF_6 by oxidative decomposition of UF_4 with dry oxygen.

Considerable efforts were expended by the IEA to set up facilities for electrolytic generation of fluorine and UF_6 preparation. Part of the equipment is under construction. The first work tests will be done at an operational level of 100-120 amperes hour fluorine cell and 0.5 kg UF_6 per hour.

Production of Spherical Nuclear Fuel Particles

Presently the production of UO_2 microspheres is being carried out at a large laboratory scale, by the H-Process /23/. Particles in a wide diameter range (50-800 μm) are obtained.

Installation of a Training Lab for Fuel Reprocessing

A program is designed for a research and training project in the field of nuclear fuel reprocessing. In particular it is foreseen to study the reprocessing procedures for shortly irradiated uranium and thorium oxides in the IEA-R1. The facilities will comprise a cold lab, a semi-hot and a hot lab, and instrumental room for analytical control by alpha and gamma-ray spectrometry, X-ray fluorescence spectrometry, potentiometry and molecular spectrophotometry. The analytical service facilities are already installed. The semi-hot lab presently works for the treatment of irradiated ThO_2 (1 to 3 grams irradiated during one day) for the recovery of ^{233}Pa and further decay to ^{233}U (obtained as tracer solution).

Pilot Plant for Purification of Thorium Nitrate

The alkaline process for breaking up monazite is being practiced in Brazil since 1948 on an industrial scale. A thorium concentrate is obtained in a form very suitable for solvent extraction purification /6/. A highly efficient Th-U separation is obtained by using an extractive-precipitation of thorium sulfate /6/.

Thorium nitrate solution of high purity obtained at the pilot plant is designed to be converted into thorium oxide for further use as thorium oxide pellets. To accomplish this aim the precipitation of thorium oxalate was exhaustively studied /24/. A laboratory scale dry-way preparation of ThF_4 by the action of anhydrous HF on ThO_2 was investigated /25/.

Then, supported by several years work at large laboratory scale in the purification of thorium nitrate, a pilot plant is under construction based on solvent extraction of thorium nitrate with 50% TBP-varsol /26/.

Analytical Quality Control

A rapid routine determination of natural U content in uranyl solution is done by gamma-ray spectrometry /27/ (^{235}U 185 KeV). A non-destructive method for U determination in low grade ores by gamma-ray spectrometry is based on the measurement of the ^{234}Th (93 KeV) photopeak and applied especially to the Morro do Agostinho ore, Poços de Caldas, Minas Gerais State /28/. The denitration of Th in monazite samples was made directly by measurement of the height of the ^{212}Pb photopeak (240 KeV) /29/.

A procedure was outlined for the direct determination of the U content of uranyl nitrate-TBP-organic phase /30/. The thermogravimetric behavior of ADU samples, the pyrophoricity grade of UO_2 powders and their O/U ratio were determined by recorded thermoanalytical curves which gave valuable

information on the stability of the oxides /31/. A chemical procedure for the O/U ratio in UO_{2-x} and $UO_{2-x} \cdot ThO_2$ sintered pellets was developed /32/. The analytical control of UF_4 was made by sequential analysis of the most probable products existing along with the tetrafluoride /33/. A colorimetric method for the determination of calcium in nuclear grade uranyl salts and uranium oxides was established /34/. The determination of microquantities of B in highly pure U and thorium compounds is done through the extraction of the colored complex of BF_4^- - monomethylthionine /35,36/. A procedure for the separation and concentration of extremely low amounts of Th and RE from uranyl solutions was developed based upon the sorption of those elements from solutions containing 0.3M HF onto a small column of alumina /37/. Using this technique the individual RE have been analysed by emission spectrography /38/. Separation of RE from thorium nitrate on cellulose-ether- HNO_3 system and their determination by emission spectrography was established /54/.

A semiquantitative routine spectrometric method was outlined for the direct determination of 18 elements in uranium compounds, using gallium oxide and sodium fluoride as carriers /39/. Traces of RE were spectrographically determined after separation of uranyl chloride with TBP /40/. A comparative study of the efficiency of some spectrochemical carriers was established for the quantitative spectrographic analysis as trace impurities in uranium /41/.

Vanishing small amounts of RE in U are determined by fluorescence spectrometry after separation onto an alumina column /42/. Zirconium was analysed by direct spectrofluorimetric determination in uranyl chloride using morin /43/ and spectrophotometrically with chloroanilic acid /44/. A procedure for direct fluorimetric determination of RE in thorium compounds was established /45/.

The separation of contaminants in U is achieved by extraction chromatography and the impurities determined by atomic absorption spectrophotometry (AAS) /46/. The extraction of Zn and Pd /47/ and of Bi, Cd, Pb, Hg, Au and Ag /48/ in nuclear grade uranium and their determination by AAS was investigated, burning directly the organic phase. The separation of several metal traces by solvent extraction from pure thorium and their determination by AAS was also carried out /49/. Preconcentration and determination of Cd, Pb, Tl, Bi and Cu in high purity U salts was accomplished by anodic stripping polarography /50/.

The determination of uranyl nitrate diffusion coefficients for

the calculation of the height of transfer unity was studied /51/. A routine method for mean crystallite size determination of U_3O_8 microspheres were established by X-ray diffraction line broadening technique /52/.

Conclusion

In the past several years we could say that the IEA, with the support of the CNEN, have acquired, with enthusiasm and success, a great deal of the technology of materials for nuclear industry. The main priority was emphasised on U and Th, but other strategic materials have been studied as well, among them Zr, RE and graphite. A set of works for U and Th purification carried out in those past years on a large laboratory scale and on pilot facilities was established according to the scheme: preparation of UN solutions from the concentrate, production of ADU by ion exchange and solvent extraction techniques, production of UO_3 , preparation of UF_4 and production of thorium nitrate, oxalate and oxide.

The necessary analytical procedures as support for the nuclear materials technology was also acquired. Efforts were concentrated on working out of the methods and schemes for health physics system for all works involving U and Th /53/.

Finally, but not less important, many professionals, including chemists and engineers, were trained and acquired reasonable good background in the nuclear material science.

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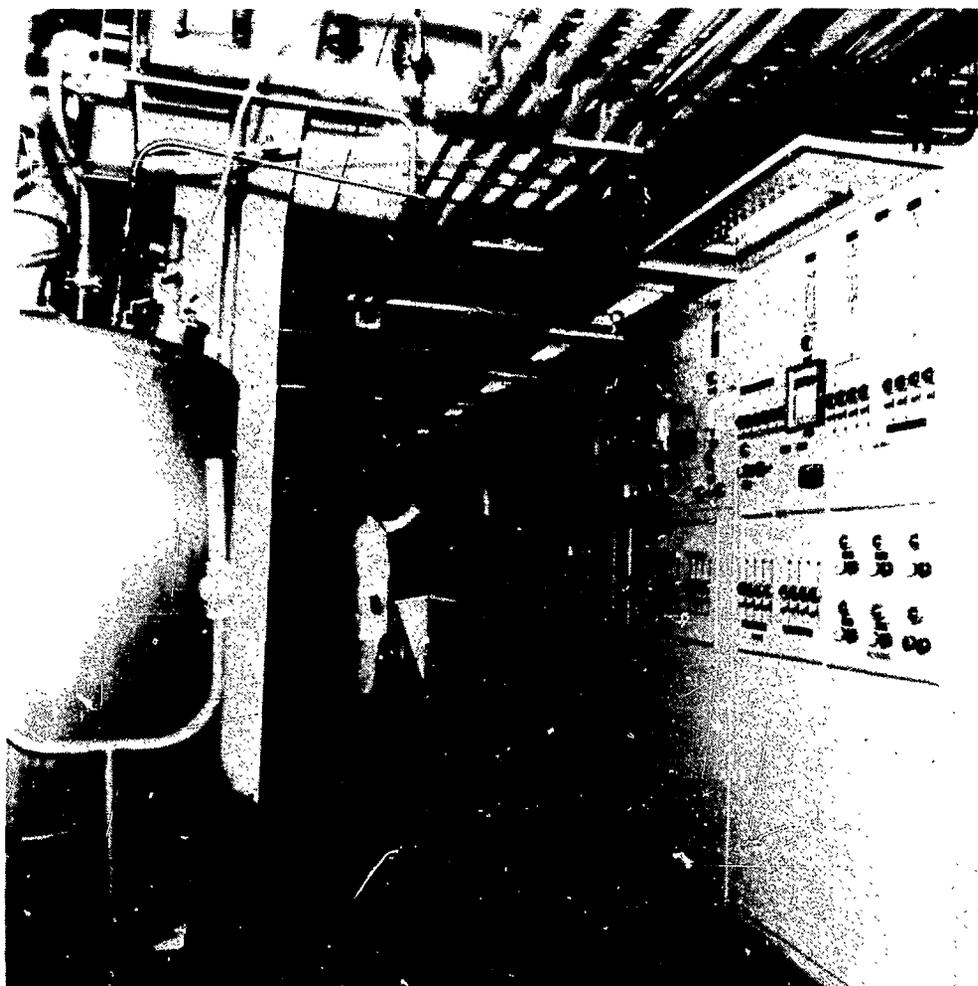


FIGURE 2. PILOT PLANT FOR PURIFICATION OF URANIUM: INSTRUMENTALON PANEL.

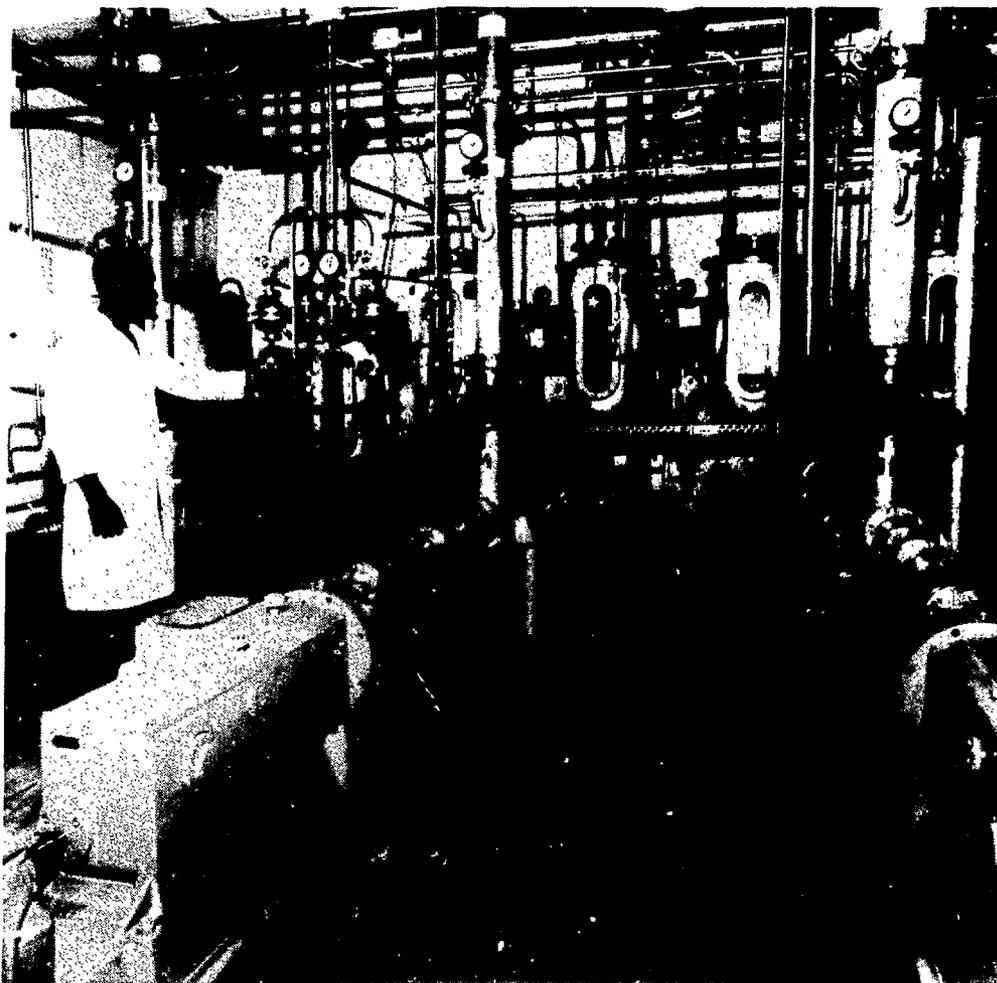


FIGURE 1. A GENERAL VIEW OF THE PULSED COLUMNS FOR
URANIUM PURIFICATION

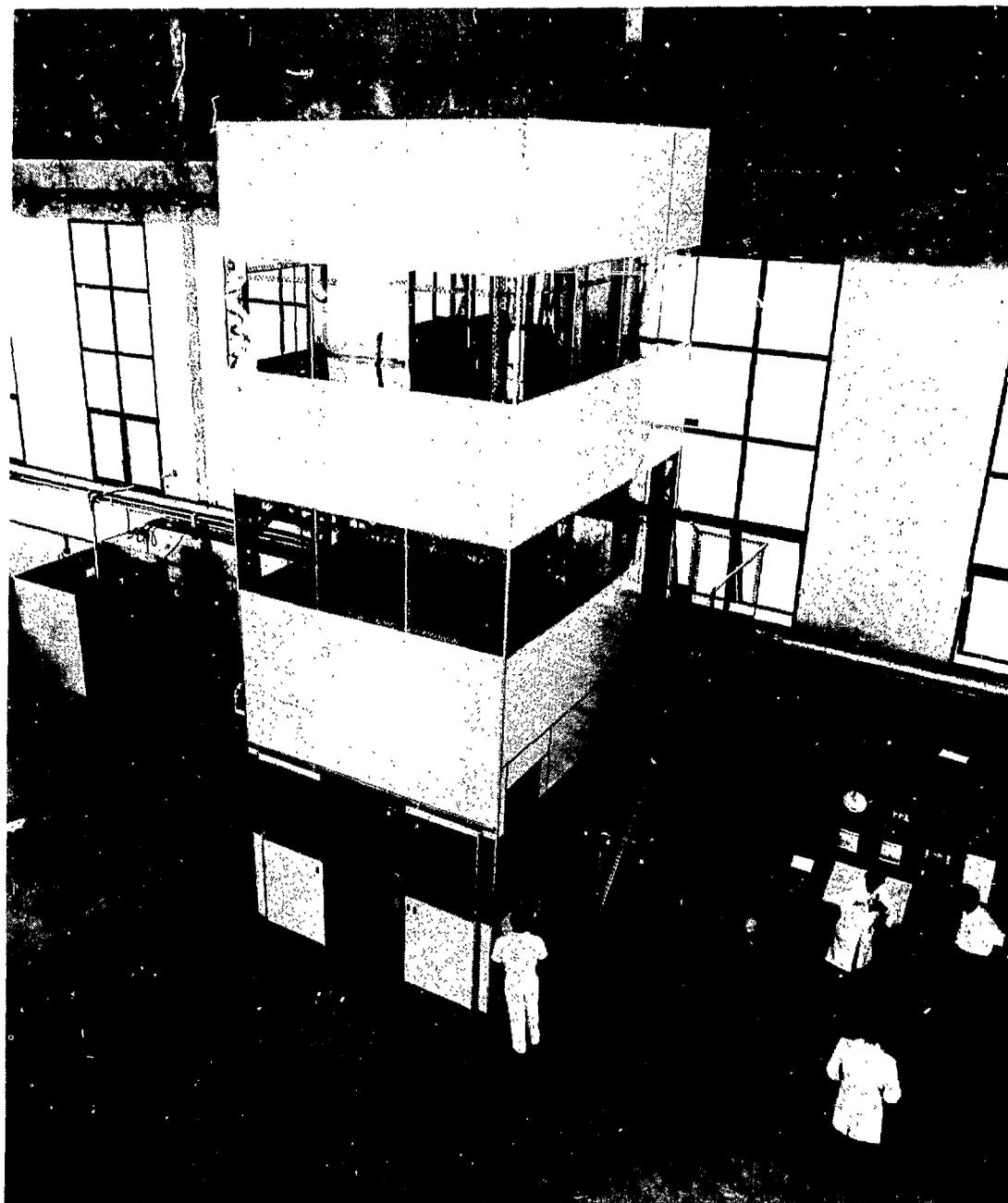


FIGURE 3. A GENERAL VIEW OF THE UF₄ PILOT PLANT

