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DEVELOPMENT AND STATUS OF LMFBR FUEL REPROCESSING IN THE FEDERAL REPUBLIC OF GERMANY [x)]

F. Baumgärtner and W. Ochsenfeld

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Abstract

The problems associated with reprocessing oxide LMFBR fuel elements result from the higher plutonium content, the higher fission product content and the higher level of thermal decay power; their consequences on the Purex process are well recognizable. Reprocessing LMFBR elements by the conventional Purex process technique is considered to be feasible.

If the cooling time of LMFBR fuel elements amount to one year or more, it is only the higher plutonium content which determines the process flowsheet. The necessary chemical steps for reprocessing co-precipitated oxides have been developed at Karlsruhe for this case. The mechanical head end still needs to be developed on a technical scale , as does the management of safety against criticality.

If reprocessing is to follow a cooling time of less than 200 days, as envisaged in the original planning for LMFBR fuel elements, the efficiency of offgas treatment and the reliable operation of fast contactors in the first extraction cycle still have to be tested.

Entwicklungsgang und Stand der Wiederaufarbeitung von LMFBR-Fuels in der Bundesrepublik Deutschland

Zusammenfassung

Die Probleme für die Wiederaufarbeitung von oxidischen LMFBR-Elementen ergeben sich aus dem höheren Plutoniumgehalt, dem höheren Spaltproduktgehalt und der höheren Nachwärmeleistung und sind für den Purexprozeß überschaubar. Die Aufarbeitung von LMFBR-Elementen nach dem herkömmlichen Purexverfahren wird für möglich gehalten.

Wenn die Abklingzeiten der LMFBR-Brennelemente ein Jahr oder länger beträgt, ist für die Prozeßdurchführung nur der erhöhte Plutoniumgehalt bestimmend. Für diesen Fall sind bei Mischoxiden die erforderlichen chemischen Maßnahmen zur Wiederaufarbeitung in Karlsruhe ausgearbeitet worden. Das mechanische Head-End ist im technischen Maßstab noch zu entwickeln, ebenso das Management zur Erhaltung der Kritikalitätssicherheit.

Falls die Wiederaufarbeitung nach Abklingzeiten von weniger als 200 Tagen erfolgen soll, wie es für LMFBR-Elemente ursprünglich geplant war, so sind die Effektivität der Abgasbehandlung und der zuverlässige Betrieb von Schnellextraktoren im ersten Extraktionszyklus noch zu erproben.

A) The Reprocessing Concept for the First Generation of Fast Breeder Reactors

In the Federal Republic of Germany studies on reprocessing fast breeder fuels were initiated in the sixties at the Institute für Heiße Chemie of the Karlsruhe Nuclear Research Center within the framework of the Fast Breeder Project founded at that time. In the early days there was no preference for a specific reprocessing technique. As in other countries, molten salt (1-4) and volatilization techniques (5) (chlorination (6-12) and fluorination (13,14)) were studied, but so was the aqueous extraction technique. These first fast breeder reprocessing studies were pursued with the objective of building a joint reprocessing and refabrication plant (15) (called Scharade) along the lines of the EBR II plant in Idaho, United States.

However, in 1964/65 the conviction grew at Karlsruhe that for the time being the only technique offering chances of implementation in reprocessing LMFBR fuel elements on a technical scale would be the aqueous extraction method (16-21). In our opinion, the non-aqueous techniques, have stood unsatisfactory chances of technical success. In non-aqueous techniques, e.g., criticality cannot be controlled through concentration, but only by limitation of the masses; product yields and decontamination factors are lower, the process flowsheets are operated in the batch mode, corrosion problems are more severe, etc.

On the other hand, the problems associated with reprocessing LMFBR elements in an aqueous extraction process can be predicted and give rise to the expectation of solutions being found within the framework of an advancement of the technically proven Purex method. For this reason, our studies of the non-aqueous processing techniques were terminated in 1965 and the development capacity of the Institut für Heisse Chemie has since been directed exclusively at the further advancement of the Purex process for application to LMFBR elements.

For this purpose, planning and construction of an experimental scale aqueous reprocessing plant (MILLI) was started which had its first high active operation in 1971 (22) (Fig. 1). This facility has a daily capacity of 1 kg of fuel (= 1 milliton) per extraction cycle, is safe against criticality by geometry, is α-tight sealed and can be remotely manipulated behind a shielding for 3×10^5 γ-Ci per cell. It consists of three extraction cycles equipped with mixer-settlers: one co-decontamination cycle, one separation cycle and one decontamination cycle in which plutonium and uranium are processed alternately (Fig. 2).

Since 1971 it has supplied chemical experience of reprocessing advanced fuel elements by the Purex technique on the basis of test irradiation samples from Dounreay and LWR fuel specimens with high burnup (maximum 40 MWd/kg) (23-28).

Between 1967 and 1973, the Gesellschaft für Kernforschung (GfK) and the Gesellschaft für Wiederaufarbeitung von Kernbrennstoffen (GWK) pursued a joint development program focusing on fuel reprocessing (Entwicklungsprogramm Brennstoff-Aufarbeitung = EBA). This cooperation served the purpose of establishing the preconditions, by joint research and development work, to process advanced fuel elements, especically oxidic LMFBR elements (of the SNR-300, 9 t/a of UO_2 + PuO_2), at the Karlsruhe Reprocessing Plant (WAK) with an advanced Purex flowsheet (29-31). The most important objectives of this scheme, as far as breeder reactors are concerned, were BRAUSE (32-39) (a breeder head-end stage for WAK) and PUDER (40,41) (for increase of plutonium throughput).

Since 1969 also CEN/SCK Mol has been participating in this development work under the cooperation between Germany, Belgium, the Netherlands and Luxemburg in the field of fast breeder development. Mol proposed and performed further studies in the head-end sector (voloxidation (42,43), dissolution of oxide fuel in molten salts (44,45), and decanning in molten metals (46) and with HF/O_2 (13).

Under the impact however of the development work still to be carried out on a 1500 t/a LWR reprocessing facility in the Federal Republic of Germany, the EBA project was integrated into the Reprocessing and Waste Management Project (PWA) at Karlsruhe in 1974. At the present time, this project does not involve problems specific to breeder reactors. At that time however, the chemical development of the Purex process had reached already the stage of test campaigns.

Therefore, in the first half of 1974, LMFBR fuel specimens from Dounreay with originally 15 % PuO_2 and a burnup of 60 MWd/kg were processed in the MILLI facility by the Purex process (23-28). Newly dissolution tests and additional reprocessing test campaigns of high burnup fuel specimens from the in-pile program of the Fast Breeder Project are envisaged for MILLI for the period 1976/77.

B) Differences Relevant to Reprocessing of LMFBR-Oxide and LWR Fuel Elements

From chemical view, the differences existing between oxide fuel elements of advanced light water reactors and the core of a LMFBR are not of a fundamental, but only of a gradual nature; the plutonium content differs by a factor of 20, the burnup by a factor of 2 to 3, the fuel power density by a factor of 3 to 5 (47-49).

In order to close the gap between the reprocessing problems of LWR and LMFBR fuel elements as much as possible, the concept of joint reprocessing of core and blanket elements has been pursued in the Fast Breeder Program.

Thus, a core-blanket mixture corresponding to the discharge rhythm of an LMFBR, namely, two core plus axial blanket and one radial blanket fuel inventory, after 365 days of cooling time does not differ in terms of its fission product activity from that of an advanced LWR fuel which has cooled for 200 days.

Table 1: Pu- and Fission Product Content and Radioactive Decay Heat of Fuels Following Burnup

	LMFBR		LWR
	Core	2 core and axial blanket + 1 radial blanket fuel inventory	
% Pu	18	10	1
% fission products	8	3.5	3-4
Radioactive decay-heat /W/kg/ after cooling time			
30 d	180	75	48
150 d	80	34	20
365 d	38	16	11

However, there is one difference which cannot be offset, i.e. the plutonium content, which is about 10 times that of an LWR fuel (Table 1).

After mixing of core and blanket solutions in the feed tank, the total quantities of fission products of LMFBR and advanced LWR fuel elements are about the same order of magnitude, approximately 3.5 %. But even in reprocessing LWR fuel elements the handling of this quantity of fission products has not yet become a routine matter; hence, the problems involved with this amount of fission products will be listed below within the fast breeder problems, too.

For evidence the development problems to be solved in reprocessing LMFBR elements by the Purex process shall be subdivided according to their origin from characteristic differences namely plutonium content, burnup, specific power.

I. Problems Arising from the High PuO_2 Content

(1) Complete dissolution of the PuO_2 fraction.

(2) Complete co-extraction of the plutonium together with uranium.

(3) Plutonium/uranium separation without major expansion of the process volume by excess of reducing agent.

(4) Extractive plutonium decontamination with a minimum of process volume (for criticality reasons).

(5) Effective techniques of plutonium concentration (extraction or distillation techniques).

II. Problems Arising from High Burnup (Fission Product Content)

(1) Removing insoluble fission products from the feed solution.

(2) Overcoming extraction failures due to radiolysis products.

(3) Reducing radiolysis by reducing contact times.

(4) Separation of accumulating radiolysis products in the solvent.

III. Problems Arising from Specific Power

(1) Cooling of discharged LMFBR elements during storage and transport.

(2) Removal of coolant sodium adhering to the fuel.

(3) Development and testing of a cooled disassembling and cutting system for LMFBR elements.

(4) Offgas retention and improvement in the dissolution behaviour of the fuel by oxidizing high temperature treatment (voloxidation).

C) Status of Development Work in the Federal Republic of Germany

I. Development Work to Cope with the High PuO_2 Content

(1) Dissolution experiments were carried out on

(a) unirradiated tempered mixed oxides and
(b) irradiated mixed crystals of UO_2/PuO_2.

(a) In unirradiated mixed oxides PuO_2 remains as an insoluble residue even in concentrated nitric acid. However, at temperatures above 1600°C soluble mixed crystal oxides are formed only after 4 hours of heating. These mixed crystals will completely dissolve in boiling nitric acid (50).

(b) Dissolution tests on 2.7 kg of UO_2/PuO_2 fuel specimens with burnup of 50 - 61 MWd/kg and 15 % of PuO_2 were completely dissolved in 8 M HNO_3 within 2 or 3 hours (23). A residue of 0.4 to 0.7 % contained mainly fission products in decreasing quantities in the sequence Mo, Ru, Zr, Pd, Sn. The contained plutonium and uranium fractions amounted to 0.04 % of the total fuel. The Pu:U ratio in the solution was constant throughout the whole dissolution step. As a consequence, it can be concluded that there is no preferred dissolution of the more easily soluble uranium oxide. For analysis the stainless steel cladding material was dissolved completely and its plutonium content determined. The plutonium content in the fuel element cladding was about 0.02 - 0.04 % of the fuel element.

(2) As a rule, extraction in the Purex process is operated at a TBP saturation level below 70 % to offset variations in concentration. At lower plutonium contents, down to approximately 1 %, which are encountered in LWR fuels, the excess of approximately 30 % of unsaturated TBP ensures complete extraction of the plutonium. In order to achieve a sufficiently high decontamination factor for the higher fission product contents of LMFBR elements, TBP saturation levels of 75 to 80 % will be required at higher burnup. For a

saturation level of more than 80 % in 20 % TBP-Alcane it was observed that the plutonium is already suppressed from the organic phase into the aqueous phase because of the excess uranium (Fig. 3) (51-52). The consequence is an increase in the plutonium concentration in the low-uranium extraction section of the contactor. Plutonium losses in the aqueous effluent might arise as a consequence and there may even be accumulation of the plutonium to critical levels. One precondition to the application of the Purex method to plutonium-rich fuels for this reason has been found to be the knowledge of distribution data of macroscopic quantities of plutonium in the presence of uranium (53-55). The interdependences in the distribution data of the elements plutonium and uranium and nitric acid were plotted and empirical functions were drawn up to extrapolate to other mixing areas. In the light of these data it was possible to develop an extraction flowsheet in the range of 1.5 - 3 N HNO_3 and for maximum TBP saturation which furnishes plutonium losses of less than 0.1 % and ensures safe concentration control of the plutonium.

(3) In the conventional Purex process the separation of plutonium from uranium is achieved by adding reducing chemicals (Fe(II), U(IV), H_2NOH etc.). This requires a 2- to 10-fold stoichiometric excess of the reducing agent. This considerable amount of chemicals, e.g. U(IV), almost duplicates the process volume in view of the high plutonium content of LMFBR fuel, with all the negative consequences for criticality safety and larger waste volumes this entails.

To overcome this problem an electrolytic multi-stage mixer-settler and an electrolytic extraction column working in a double function as extraction and electrolysis equipment have been developed (56-61). These units can be applied in plutonium/uranium separation without the addition of chemicals and, hence, without increasing the process volume. The separation results achieved are better than those attainable with chemical reducing agents (Fig. 4). In addition, this technique offers the advantage that the reducting capacity of each extraction stage can be controlled individually, which avoids instabilities that might result in local plutonium accumulation.

LMFBR fuel specimens have already been successfully separated by this technique in the MILLI facility (25).

One electrolytic extraction column with 175 kg of daily fuel throughput has been successfully tested in uranium operation. The installation of an electrolytic mixer-settler in WAK and of an electrolytic column in the SAP facility of Marcoule is being planned.

During our development work we were informed about a similar electrochemical development work at the AGNS-plant. At Barnwell however a diaphragm is used that is not needed in our devices (62).

(4) In the feed adjustment of the plutonium decontamination cycle electrolytic oxidation cells have proved to work satisfactorily for reoxidation of Pu(III) into the well extractable Pu(IV). These oxidizing cells avoid the addition of salt forming oxidants such as $NaNO_2$, NO_2 or N_2O_4.

(5) Moreover, the electrolytic technique can be applied advantageously also in the plutonium decontamination cycles. Electrolysis in plutonium reextraction allows higher plutonium concentrations and throughputs while, at the same time, reducing the plutonium losses. For, in this type of reextraction the formation of Pu(III) increases the possibility of plutonium concentration in the aqueous product stream to about two to three times the original value.

II. Development Work to Overcome High Burnup

(1) Reprocessing of nuclear fuels with higher burnup in the Milli, WAK (63) and other facilities gave rise to insoluble fission product residues which, when entering the pipelines and contactors, can block the entire process by plugging up these units. Hence, the feed solution must be treated in technical plants. In the reprocessing campaigns so far carried out in MILLI it was sufficient to filter through a metal frit of <30 m/u pore size after previous sedimentation to produce a filtering layer on the filter.

Further studies of the chemical composition and grain size distribution of these residues are necessary and under way.

(2) The main radiolysis and hydrolysis product of TBP is dibutyl phosphate, HDBP. It binds plutonium in the organic phase (plutonium losses), reduces the decontamination factor by extracting zirconium and, because of its emulsifying action, gives rise to hydrodynamic failures in the extraction step.

Measurements of the rate of HDBP formation by α, β and γ-radiation of a 20 % TBP-alkane/HNO_3 system revealed a level of 30 mg of HDBP/Wh·l in the dose range of 10 Wh/l in single phase exposure and an amount of 150 mg of HDBP/Wh·l for mixed phase exposures (64).

When reprocessing real fuel specimens in the MILLI facility it appeared that the radiolysis problem is less grave than had been expected. Fuel of 33 MWd/kg burnup after 240 days of cooling time, when extracted with 30 % TBP-alkane in mixer-settlers with a contact time of 30 minutes, produced 20 - 30 mg of HDBP/l (59). This corresponds to approximately 0.2 Wh/l of radiation exposure of the organic solvent as a result of an activity of the feed solution of 200 β-γ-Ci/l each.

These results were confirmed also in reprocessing campaigns (28) of pure uranium solutions which had been exposed to approximately 1600 β-γ-Ci/l by the addition of Mn-56 activity and processed without any difficulties.

Studies of the solubility, rate of formation and dependence on acidity showed that the emulsifying action can be greatly curbed when keeping the acidity to 3 - 4 N HNO_3 (61) and maintaining the 30 % TBP saturation with uranium/plutonium in excess of 70 % (Fig. 5). So far, this fact has been confirmed regularly in a number of reprocessing campaigns in MILLI.

(3) The increased radiolysis in reprocessing LMFBR fuels may be counteracted by reducing the contact time in the extraction step, provided the extraction kinetics is sufficiently fast.

Measurements carried out to this effect of the mass transfer rates of plutonium and uranium in the Purex process (66-70) showed that the contact times of mixer-settlers and columns can be reduced to 1/100 of the original times without impairing the efficiency of the extraction.

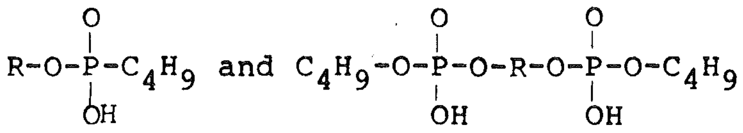
If the throughput is kept constant, a shortening of the contact time may be employed to reduce the contactor cross section and in this way achieve criticality safety by geometry.

A drum contactor with the critical safe dimension of 8 cm has been designed and tested in cold long term experiments in a series-connected 12-stage system (71-73). Using a centrifugal extractor with improvements in the construction of the drum and pump-mixer a maximum total throughput of 500 l/h was achieved. This corresponds to a fuel throughput of 1 t/d.

The practically 100 % efficiency of each stage of the fast contactors in addition results also in savings in the extraction stages compared with mixer-settlers.

(4) The re-usability of the Purex solvent is limited even if the hydrogen-dibutylphosphate (HDBP) is regularly removed by alkaline washing. In addition to HDBP, the ionizing radiation also gives rise to radiolysis products which cannot be removed by alkaline washing and for this reason accumulate. Their effect upon the Purex process resembles that of HDBP: plutonium losses due to complex-formation, reduced decontamination factors as a result of Zr-extraction, and hydrodynamic failures due to the formation of emulsions.

After distillation and column chromatographic enrichment these so far unknown radiolysis products have been identified as long-chain acid phosphate esters (74-78) with the basic formula



$$R{-}O{-}\underset{OH}{\overset{O}{\overset{\|}{P}}}{-}C_4H_9 \quad \text{and} \quad C_4H_9{-}O{-}\underset{OH}{\overset{O}{\overset{\|}{P}}}{-}O{-}R{-}O{-}\underset{OH}{\overset{O}{\overset{\|}{P}}}{-}O{-}C_4H_9$$

($R = C_5$ to C_{14}). They are the radiolytically induced reaction product of TBP with the diluent. These compounds can be removed from the Purex solvent by strongly oxidizing adsorbents like PbO_2/SiO_2.

III. Development Work to Overcome the High Specific Power

As a result of the strategy of mixed core blanket reprocessing the problems arising from the high specific power of LMFBR fuels - beside fuel transportation and storage - are confined particularly to the head end step.

These problems are primarily not chemical but engineering ones and have to be solved in connection with the plant taken into consideration for adaptation to LMFBR fuels.

Accordingly at Karlsruhe a project study had been initiated within the framework of EBA (Entwicklungsprogramm Brennstoff-Aufarbeitung) in 1969 in order to define and clarify the open engineering problems associated with the construction of a head end cell at WAK (Brüter-Aufschluß-Stufe = BRAUSE).

This study dealt with all the process steps, from fuel transportation, unloading and transfer into the hot cell , removal of adhering sodium metal, testing for cladding defects, chopping the fuel bundles under cooled conditions and dissolution of the fuel.

(1) From the various possibilities for decay heat removal (e.g. cooling by gases, sodium metal, Bi-Pb-alloys, molten salts, water, organic substances) on the long term sodium is favoured as the coolant. These studies were carried out by Gesellschaft für Kernforschung Karlsruhe (GfK), Fa. Transnuklear, Hanau, and by CEN-SCK Mol.

(2) The removal of coolant sodium adhering to the fuel outsideas well as within the fuels elements should be be brought about by a water vapor treatment according to the procedure used at the KNK-Reactor in Karlsruhe.

(3) Development and testing of a sodium cooled disassembling device for LMFBR fuels was undertaken by Luxatom supported also by Euratom.

(4) After chopping the fuel rods with a cooled bundle shear, originally the voloxidation process was to be carried out with the purpose of releasing the gaseous fission products prior to the dissolution step by means of phase transformation $UO_2 \longrightarrow U_3O_8$ and at the same time facilitating the dissolution of the fuel.

Experimental investigations indicated however that soluble mixed crystal powders of plutonium and uranium oxide are maintained only at oxidation temperatures below 600°C (79). But within this range of temperature there is no complete release of the gaseous components; on the other hand above 650°C insoluble PuO_2 is formed.

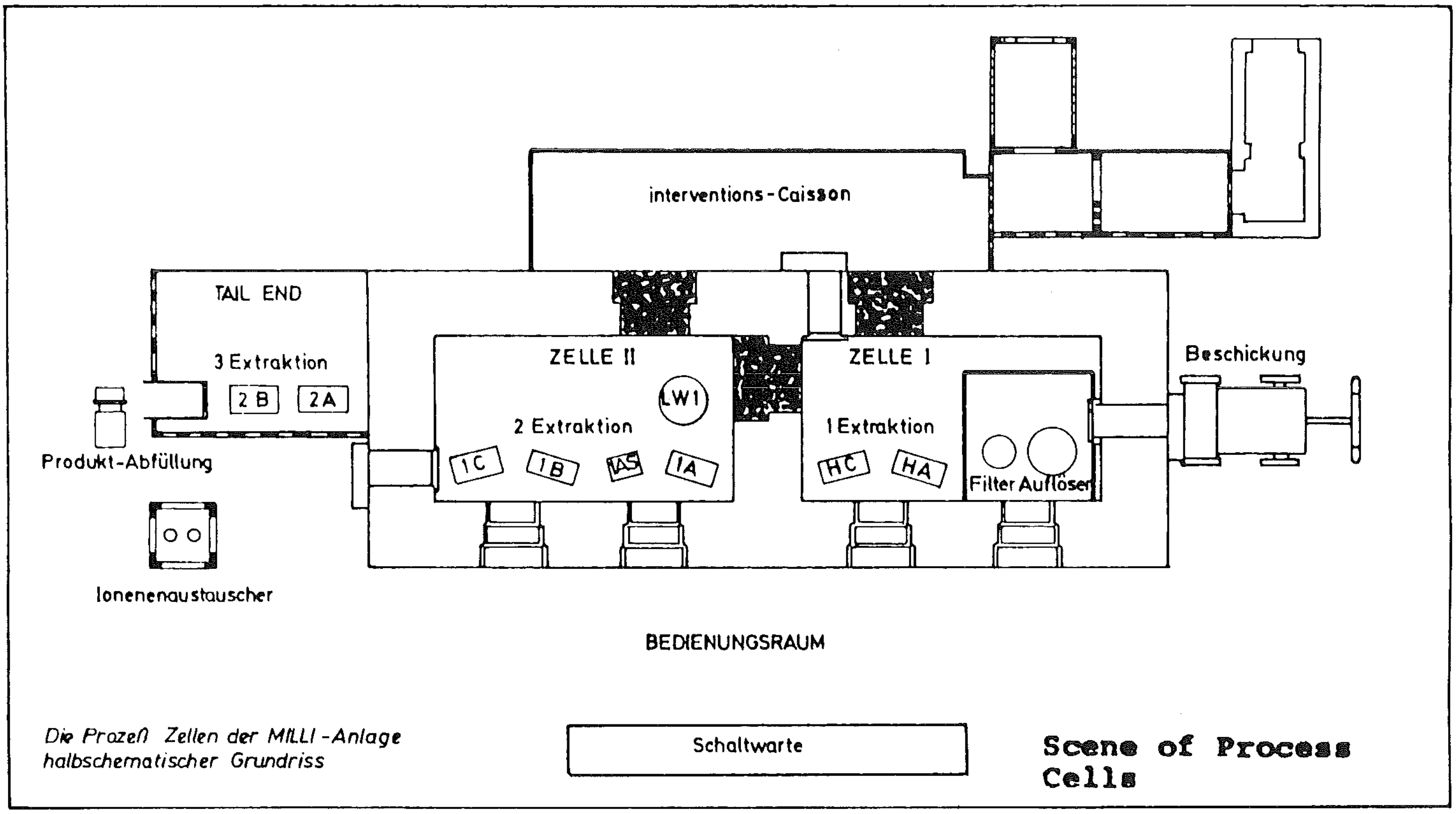
Furthermore, it was found that a PuO_2 content of 25 % in a plutonium-uranium oxide mixture phase transformation (80) no longer occurs followed by gas release in a voloxidation process. For all reasons voloxidation is no longer considered as a treatment step preceding dissolution of LMFBR elements.

Consequently, an offgas treatment still has to be developed reasonably following the LWR fuel offgas treatment which recently has also been submitted to most restrictive limitations.

In 1973 the necessity of a thorium fuel cycle following the promising development of the HTR reactor, forced the BRAUSE and PUDER project to be postponed as WAK was intended to become adapted for HTR fuel reprocessing. However, this question seems as yet not to be definitely decided.

As summary of the available results from MILLI campaigns on reprocessing nuclear fuels with high burnup and high plutonium content we conclude that LMFBR fuel reprocessing is quite feasible by an adjusted Purex process.

Experiments on mechanically mixed plutonium-uranium oxide fuels of high burnup will start this year with dissolution experiments in the GfK; experiments in MILLI will continue 1977 with fuel specimens of the LMFBR fuel irradiation program.



Die Prozeß Zellen der MILLI-Anlage
halbschematischer Grundriss

Scene of Process Cells

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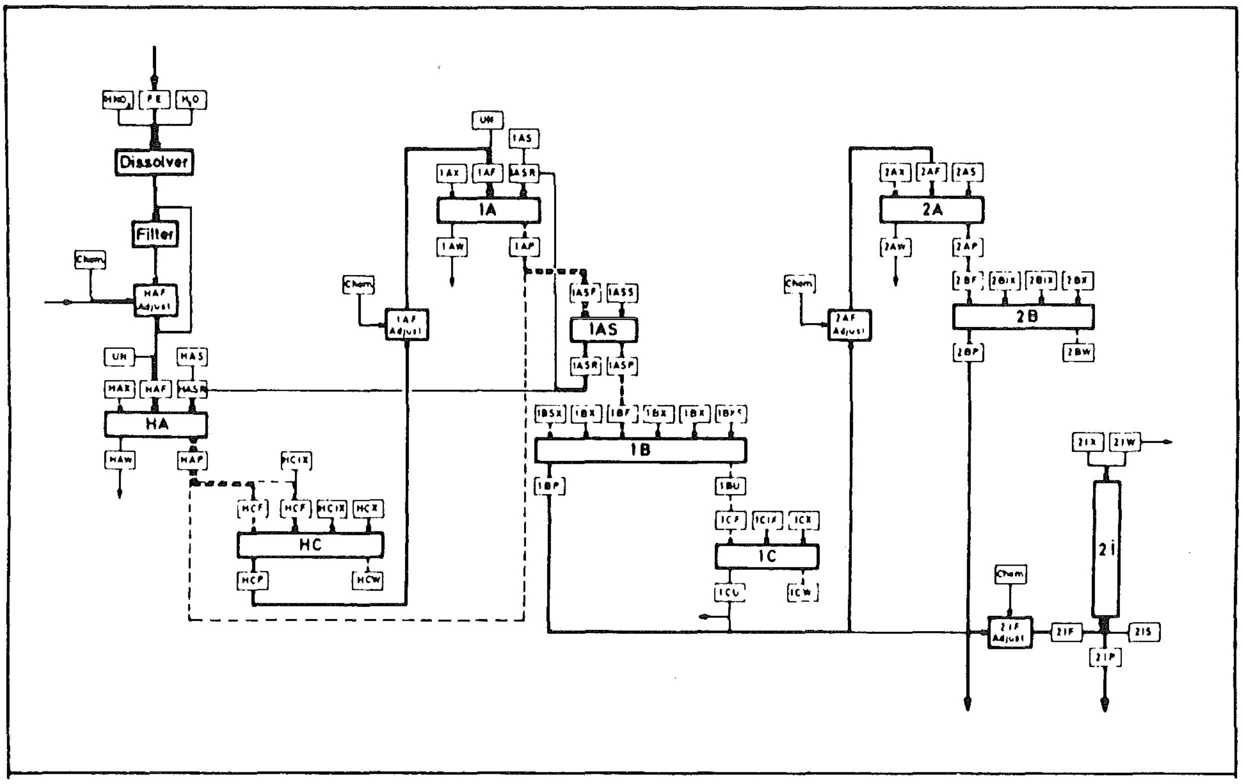
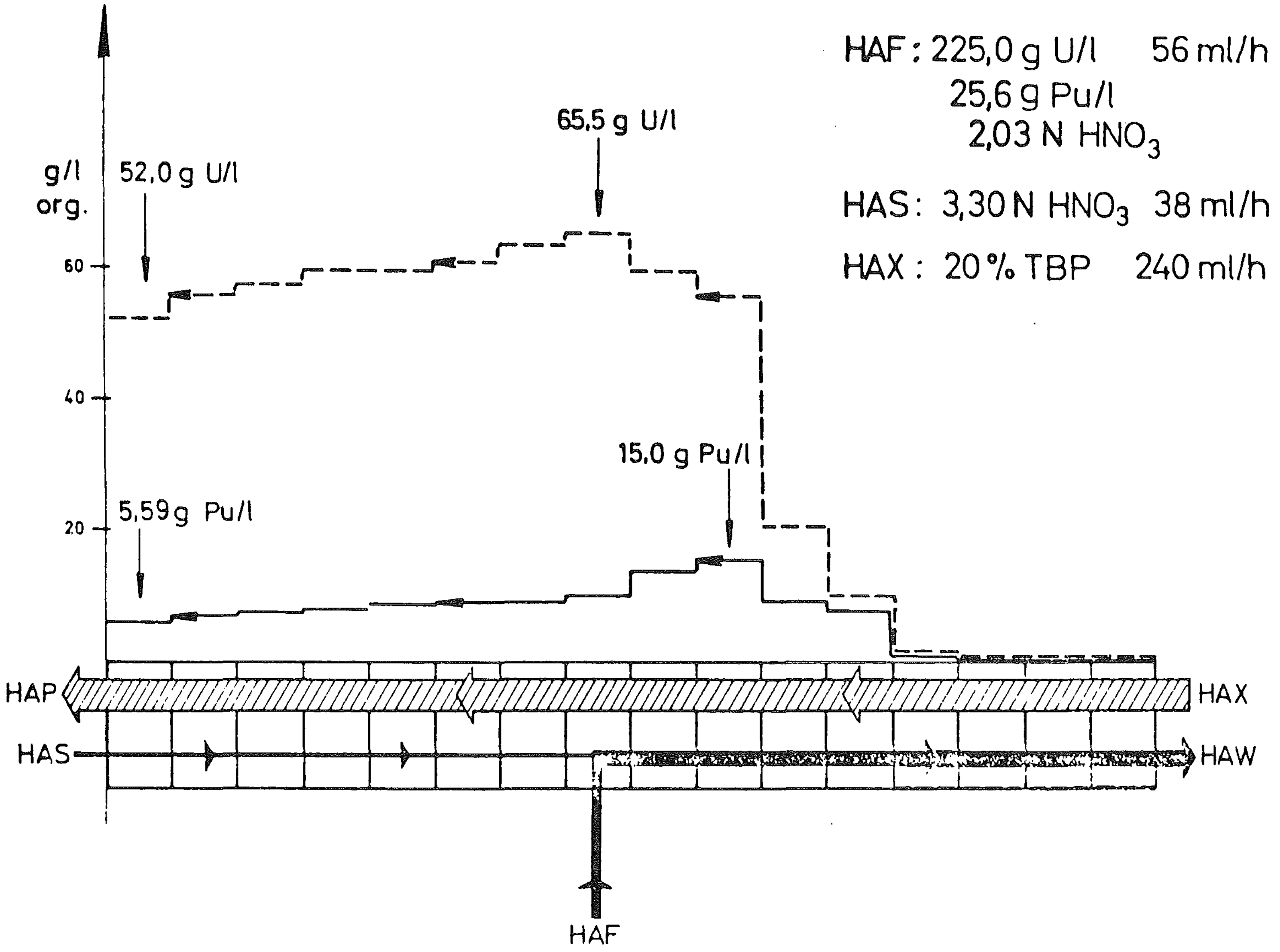
Fig. 1:



<u>Fig. 2:</u> Flowsheet of the first and second cycle in MILLI plant

Fig.3 : Plutonium and Uranium Distribution in HA-Extractor (TBP-saturation at Pu and U ~ 90 %)



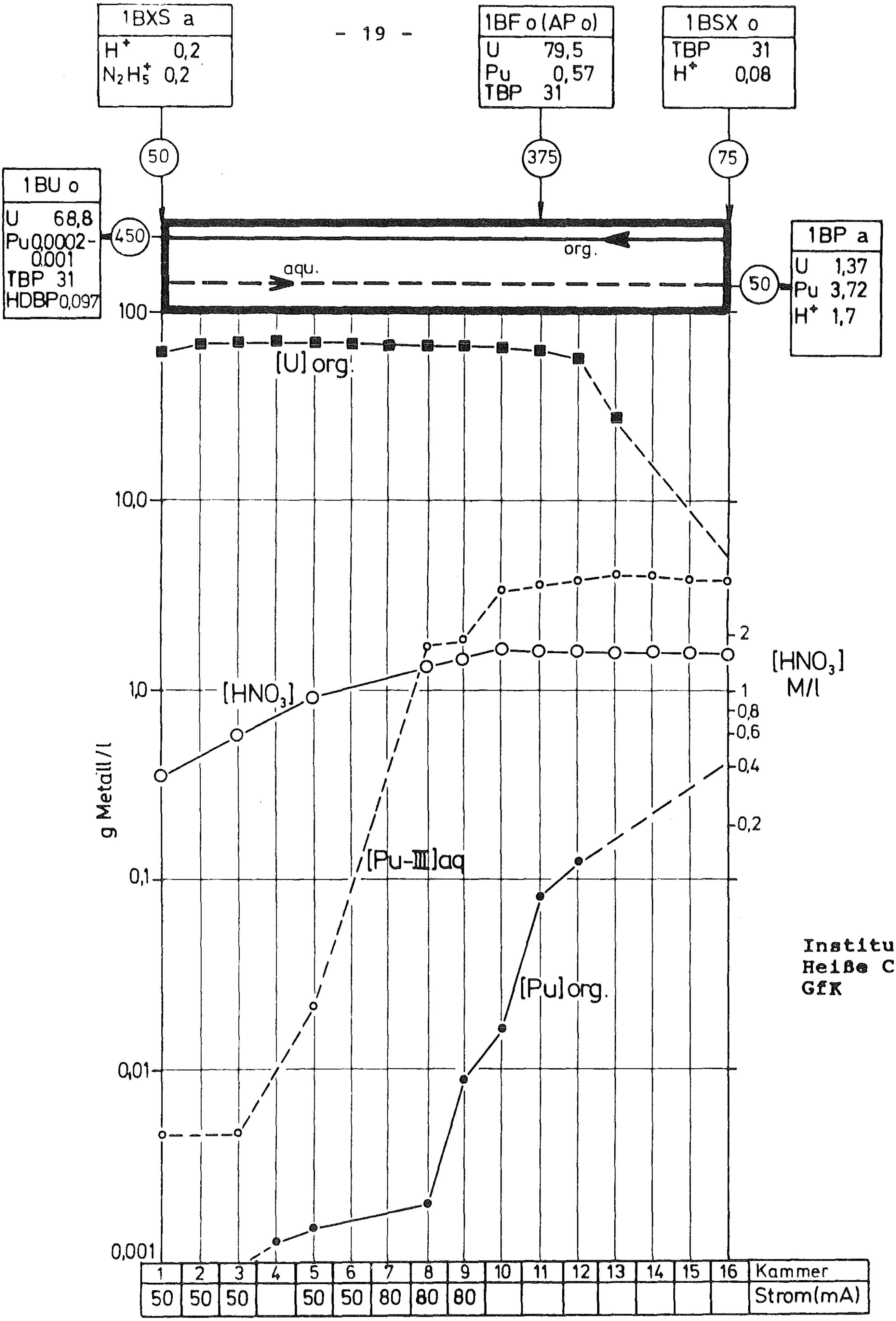


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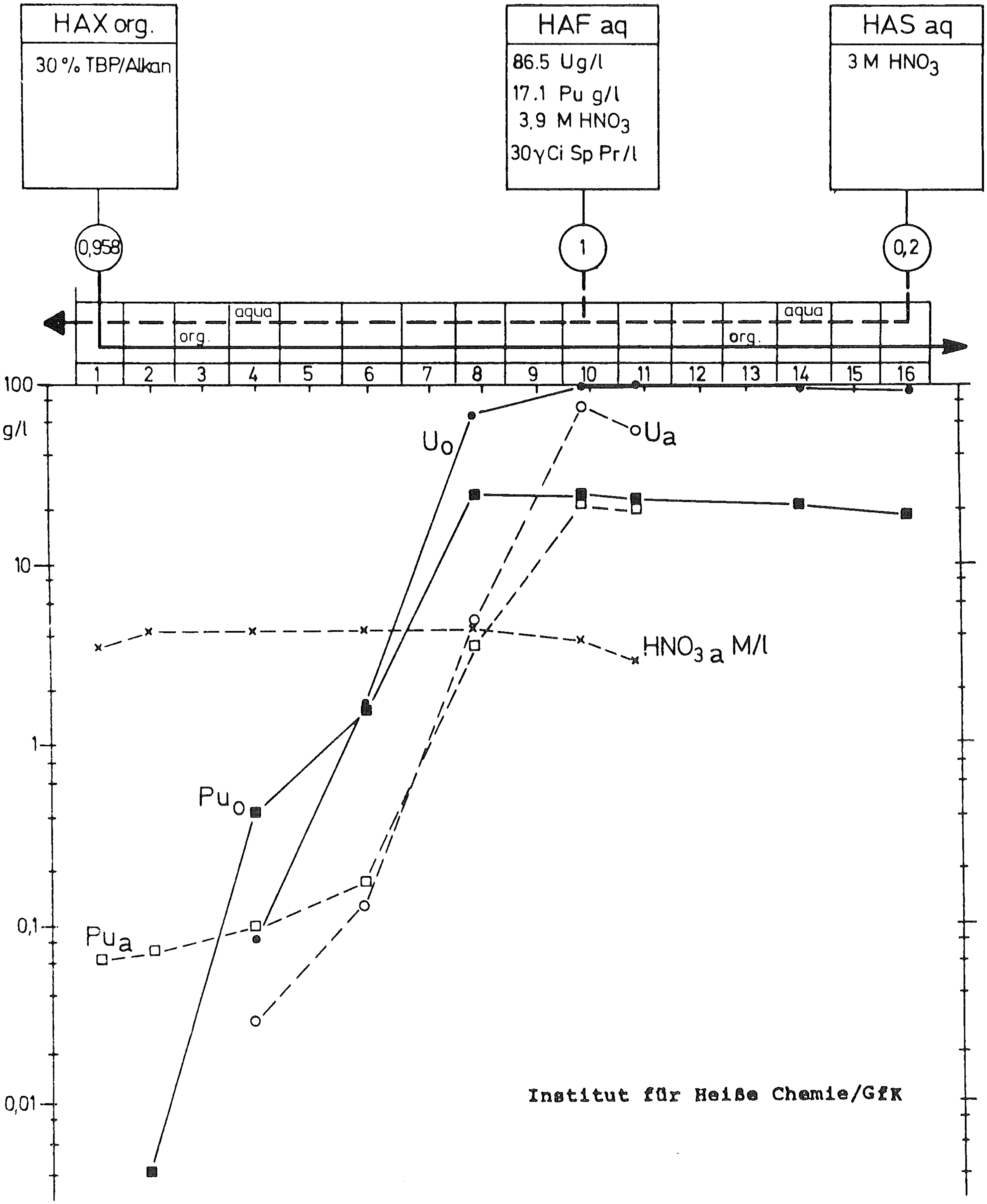
Fig. 4: Typical Concentration Profile on the electrolytic Mixer-Settler (MILLI-EMMA)

Fig.5 : Concentration Profiles of the Co-Decontamination Cycle (HA)
Brennstoff/Fuel: Dounreay (55000 MWd/t; 16,5 % Pu)



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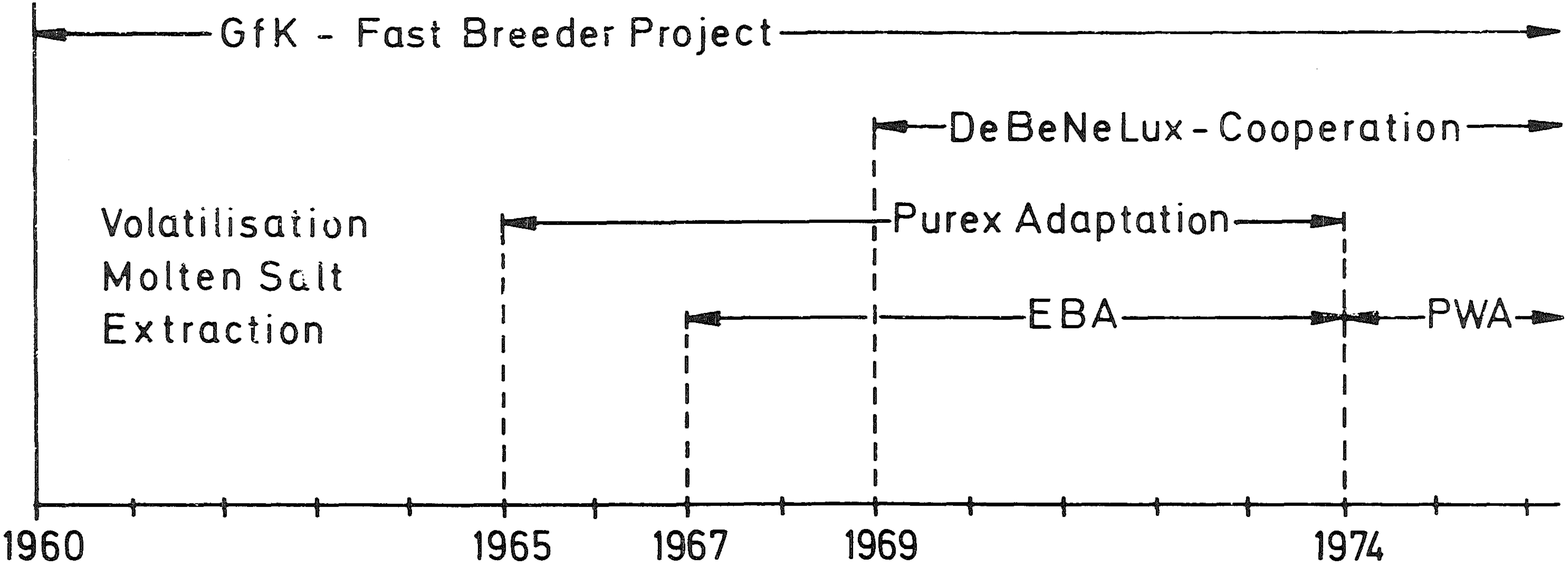

Fig. 6

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R&D LMFBR-Reprocessing GFK-Karlsruhe

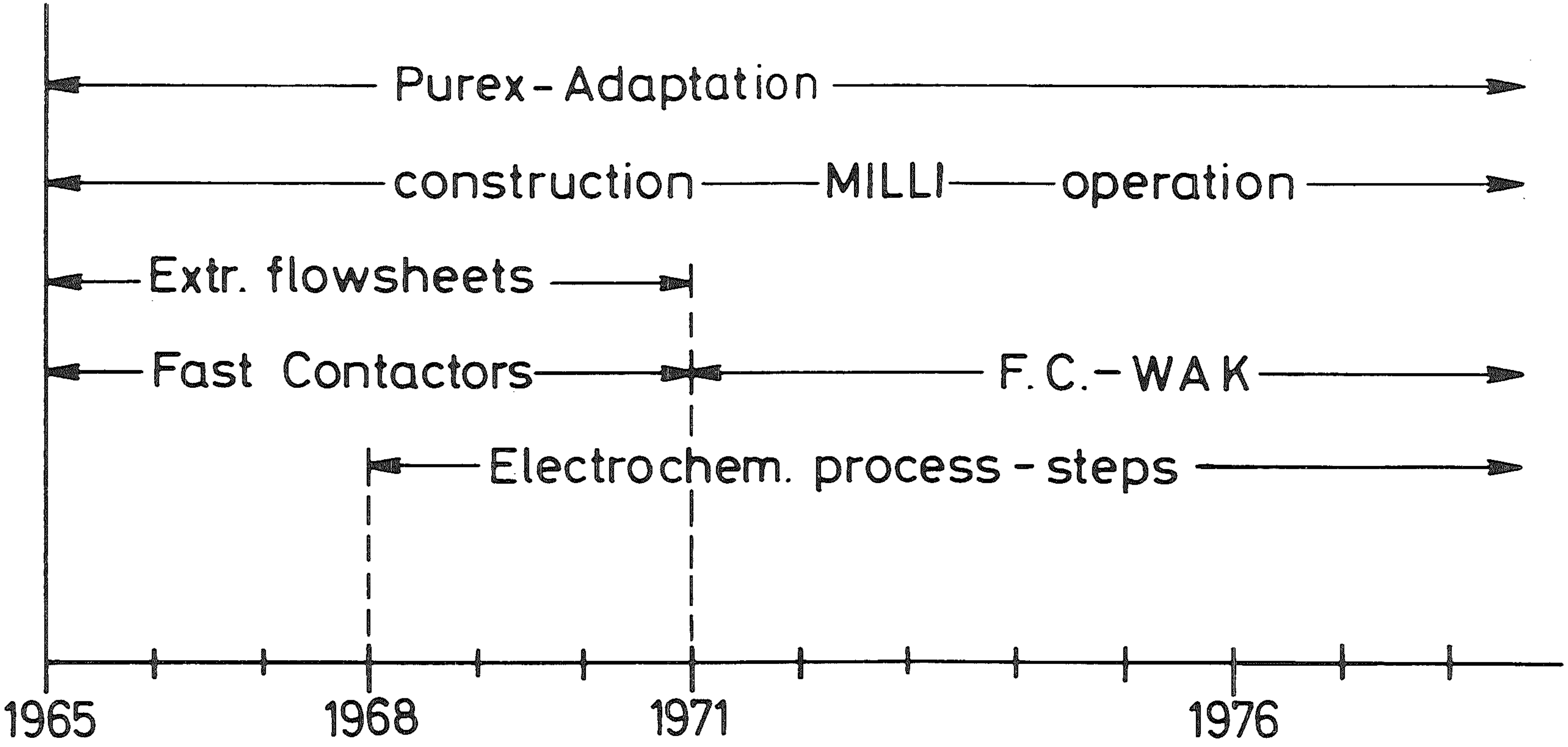

Fig. 7

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