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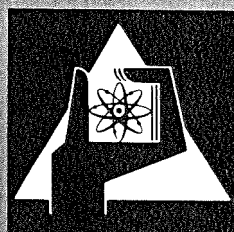
Institut für Heiße Chemie

The Highly Shielded Extraction Facility MILLI at KFK

W. Ochsenfeld, W. Diefenbacher, H. C. Leichsenring

**Equipment and Instrumentation of a Laboratory for
PUREX Process Analytical Chemistry**

D. Ertel



**GESELLSCHAFT
FÜR
KERNFORSCHUNG M.B.H.**

KARLSRUHE

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THE HIGHLY SHIELDED EXTRACTION FACILITY MILLI AT KERNFORSCHUNGSZENTRUM KARLSRUHE (KFK)

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Abstract

THE HIGHLY SHIELDED EXTRACTION FACILITY MILLI AT KERNFORSCHUNGSZENTRUM KARLSRUHE (KFK).

The miniature pilot plant MILLI was designed for experiments on dissolution and extraction of highly irradiated fast breeder fuels. The layout capacity is 1 kg fuel per day and per cycle. The facility is in hot operation since 1971 and enables experiments to be performed with highly active material and every plutonium concentration. One dissolver and filter, the first "co-decontamination" and the second partitioning cycle are located in two hot cells. The heavy concrete shielding of these cells allows the handling of 300 000-Ci γ -radioactive material with 1-MeV energy in each. The third extraction cycle is contained in a gas-tight glove-box. A description is given of the cells and auxiliary equipment. The extractors used are flat mixer-settlers, critically safe by geometry. The containers are 40- to 50-mm-thick slab-tanks or sandwich tanks. Over a period of five years different experiments with highly active materials have been achieved in MILLI.

INTRODUCTION

Work on reprocessing is done in the Research Centre Karlsruhe in the following consecutive steps:

- (1) Experiments with uranium and plutonium in alpha laboratories: For extraction studies, a counter-current extraction equipment with four mixer-settlers in gas-tight glove-boxes has been running for ten years in the Institut für Heiße Chemie (IHCH) of the Research Centre.
- (2) Experiments with highly radioactive materials, with real burnt fuels: The purpose of these examinations is to verify the flow-sheets elaborated under point (1), and to study the consequences of the high activity, the stability of the procedure, the failures, losses and decontamination factors (DF). For experiments in dissolution and extraction the design of a miniature pilot plant, MILLI, in the Institut für Heiße Chemie, was started in 1965. The layout capacity for the plant is 1 kg fuel per day and cycle, or one milliton, from which the name MILLI was derived. The facility has been in hot operation since 1971.
- (3) Development and test of the apparatus is done in the pilot test facility of the Institute in non-radioactive work or with uranium. A building with a 560-m² surface and a 13-m working height serves these tasks. For tests of 1:1 components of a commercial-scale reprocessing plant, a new building is now under construction in the KFZ.

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CONCEPT OF THE PLANT

The main aspects for the concept of the plant MILLI were:

- (1) The high shielding (90 cm of magnetic concrete) allows radioactive material equivalent to 300 000 Ci γ -radiation of 1 MeV energy to be handled in each of the two hot cells.
- (2) The equipment is designed by geometry to be critically safe. Transfers of solutions into waste storage containers, which are not safe by geometry are only allowed after duplicate analysis via a key-lock.
- (3) The volumes of the containers allow continuous operations over 50 h, without refilling the feed containers or emptying the product or waste containers. Without these restrictions continuous operations of the first (high-activity) extraction cycle over 200 h were achieved.
- (4) Mixer-settlers, generally with 16 stages, are used as extraction apparatus.
- (5) The equipment is welded and 100% X-ray checked.
- (6) The off-gas purification system for equipment and containers is separated from the cell ventilation.
- (7) Transfers of materials into and out of the cells are done with La Calhène-type double-cover devices of 270 mm diameter.
- (8) Transfers of liquids and gases into and out of the cells are made by double locks.
- (9) Analytical samples are taken from equipment and containers by specially designed sampling stations, and are transferred to the analytical hot cells by pneumatic post.
- (10) Eight gas-tight manipulators are used to handle samples and equipment. They are also used for maintenance and exchange of equipment parts.
- (11) One power manipulator is used to change air filters in the cells, for transportation in the cells or from one cell to the other, for moving the television camera, or for work on heavy parts of equipment.
- (12) In the case of severe failures, direct access by frogmen is possible after the exhaustive decontamination of equipment and cells.

DESCRIPTION OF MILLI

The plant is situated on a surface of about 600 m² on two floors. The analytical service of the plant needs in addition about half this area. The principal part of the plant are two hot cells with a 26.4-m² surface. Attached to them is the third extraction cycle incorporated in a gas-tight box on a 12.5-m² surface; 60% of the surface of the cells is 6.8 m high, the remaining 40% is 4.6 m. The lower inside part of the cells is lined with stainless steel.

Two lead glass windows (density 4.0 g/cm³) 480 × 800 mm in each cell offer a good view at sufficient illumination. Manipulations in the cell are performed with two gas-tight manipulators (Firma Wälischmiller) at each window. A General Mills heavy-duty manipulator allows handling of loads up to 250 kg. This manipulator can be moved in both cells which are connected by a rotating door. In the concrete walls are lead plugs for electrical cables and pipes for liquids, air, and gases.

The highly radioactive fuel is transported in 3.5- and 5-t shielded containers. In the receiving hall the container is transmitted on a track car and pushed to the front side of the cell. After opening the sliding protection shield on the cell and on the shielded container, the inner container is pushed through a tunnel in the cell wall and connected with the cover on the dissolver box. The double cover is opened and the fuel transmitted in the dissolver. In the first hot cell the dissolver caisson is located which is separated from the cell area.

Each cell is accessible from the rear through an intervention caisson (Fig.1). Entrance and exit of frogmen into the caisson is via separate locks. On leaving the caisson the frogman's

protective clothing is washed down under a shower in the first chamber, in the next he undresses. The man himself is showered in the next room. Frogmen are supplied with active charcoal cleaned compressed air. In the event of a failure this air pipe is connected with a battery of reserve compressed air bottles. Contaminated parts of equipment can be transferred over the intervention caisson in two working boxes. Analytical samples from the cells are pneumatically transported in the analytical cells [1].

The make-up of inactive solutions is in the basement. Solutions are pumped by means of metering pumps and rotameters into the cell. In the basement of the plant are different areas for the storage of highly active, medium and low active wastes. High activity waste (HAW) solutions are stored in three 600-litre tanks with additional shielding. These tanks can be cooled. The solutions from false operations are re-worked over separate tanks and pipelines, also located in the cellar.

High activity waste solutions for transportation are sucked into special containers of the "Centrillon" type with 70-litre capacities. The highly active waste produced up to now was vitrified in another cell facility. The medium and low active wastes are pumped over separate filling stations in tank waggons and conveyed to the decontamination services.

The main part of the plant is intended for the study of the Purex Process with high burnup fuels. The decontamination and recovery of uranium and plutonium is achieved by fuel dissolution and countercurrent extraction in three cycles.

In the dissolver of cell 1 cut pieces of fuel elements about 5 cm in length are dissolved in boiling nitric acid. The dissolver of 2 litre usable volume can be heated or cooled. The Nionel material of the dissolver is resistant to fluoride, which is sometimes needed for dissolution of PuO_2 . All other equipment and pipes are manufactured from stainless steel. Dissolution occurs in batches of 500 g in some hours.

The off-gases from dissolution containing oxides of nitrogen, volatile fission elements and aerosols diluted with the tank ventilation air are first washed into two absorption columns, filled with Raschig rings and, after preheating, cleaned on pre- and fine filters. After dilution with the Institute air and a renewed filtration on dry filters, the off-gas is blown via a 60-m-high chimney to the atmosphere.

The fuel solution is filtered over sintered metal filters from insoluble residues. The solution is conditioned for extraction in the feed tanks of the first cycle. The washed hulls are unloaded by means of the fuel-loading into shielded containers and transferred to the waste storage area.

The extraction in the Purex process is performed with tributylphosphate diluted with dodecane (Fig.2). The counter-current extraction occurs in multi-stage mixer-settlers. Generally, in an extraction cycle the extraction of uranium and plutonium is followed by back-extraction from the organic phase. In the first extraction cycle plutonium and uranium are extracted, scrubbed (contactor HA) and back-extracted (contactor HC) together ("co-decontamination cycle"). In cell 2, the partitioning cycle achieves further decontamination and partitioning of plutonium and uranium. After adjustment of the aqueous product solution of the first cycle, uranium and plutonium are co-extracted and the organic solution scrubbed in the contactors 1A and 1AS. The mixer-settler 1AS with eight stages can be heated. All other mixer-settlers of the facility have sixteen stages. Separation of uranium and plutonium is achieved by the backwashing of plutonium with U(IV) as a reducing agent in the third contactor 1B. The uranium remaining in the organic solution is backwashed with dilute nitric acid in the next contactor 1C.

Alternative cleaning of the plutonium or uranium products of the second cycle can be performed in the third extraction cycle. Extraction and scrubbing in the contactor 2A is followed by backwashing in 2B. Final cleaning of plutonium with anion exchange resins can be done in the tail-end. Nitrate product solutions are filled in bottles.

The third extraction cycle is located in an α -tight glove-box. The two mixer-settlers, the sampling stations, the pumps, the metering and controlling system of this cycle are visible and accessible. Only the feed and waste containers, and an evaporator tank are behind 10-cm-lead shielding.

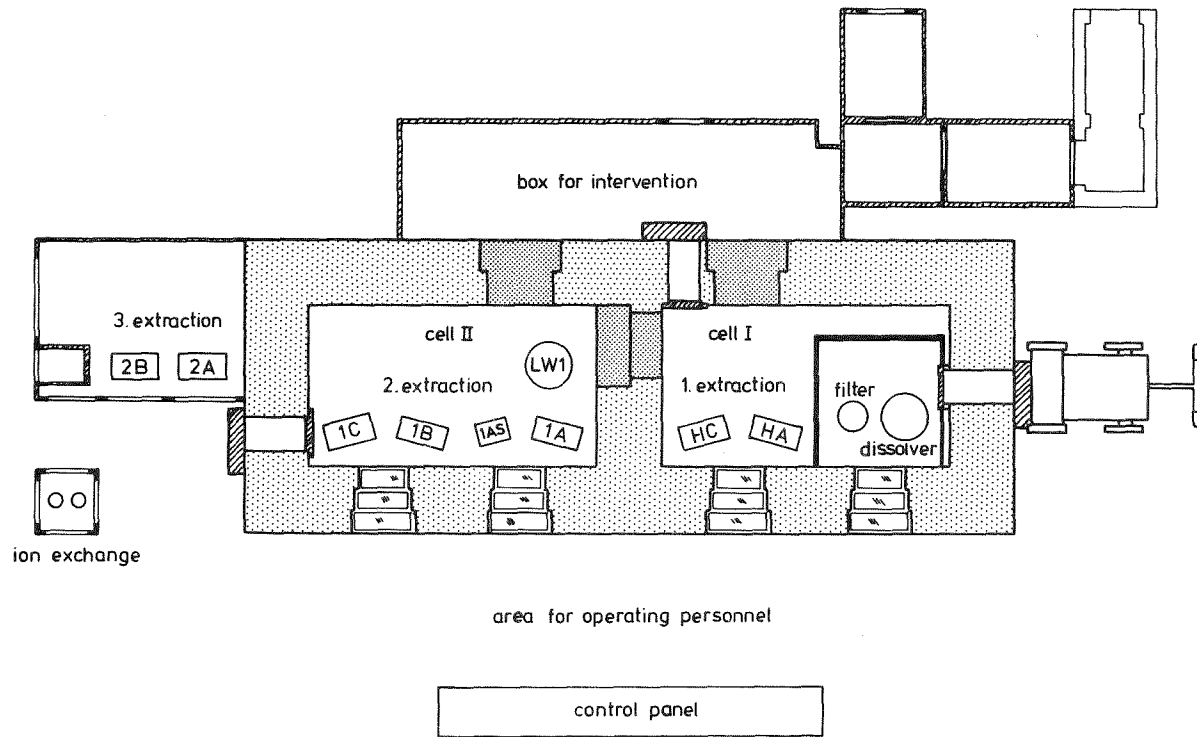


FIG.1. Outline of the MILLI facility.

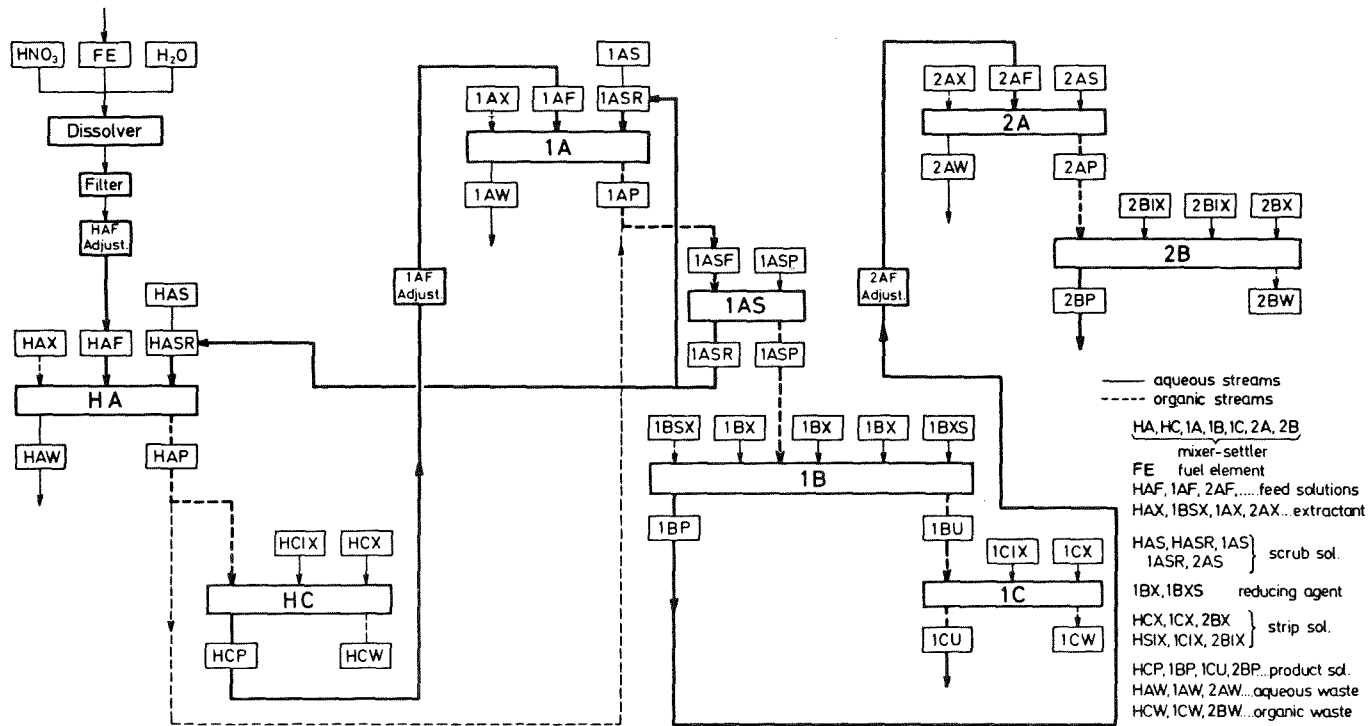


FIG.2. Basic flow-sheet of the MILLI plant.

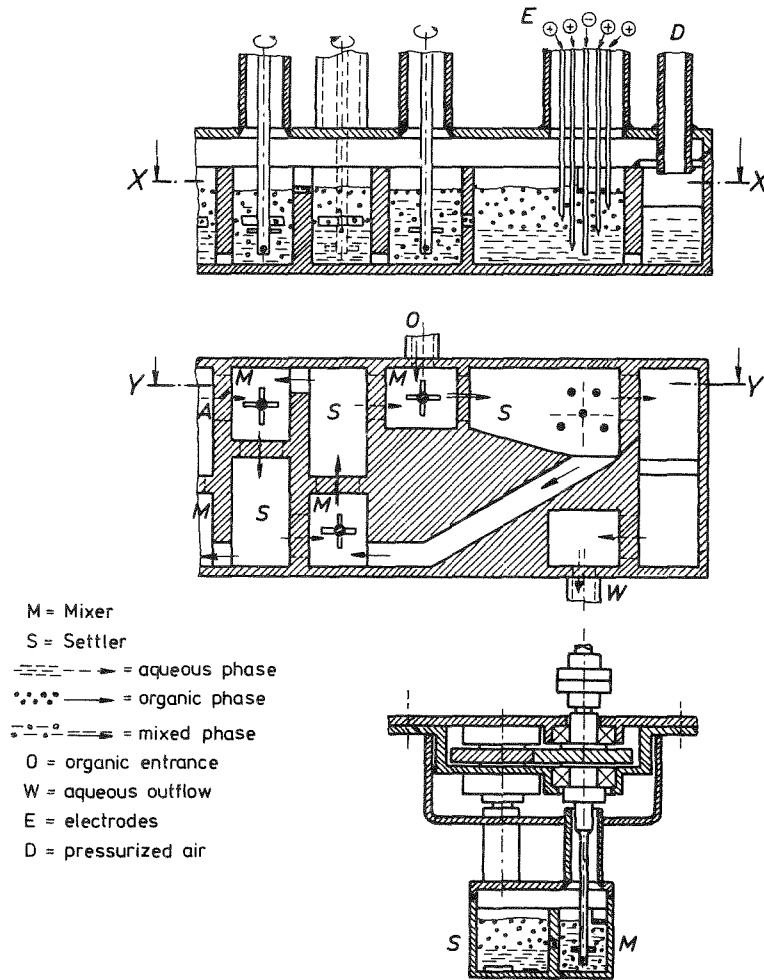


FIG.3. Mixer-settler of MILLI.

The mixer-settlers are flat and critically safe by geometry. The level of liquid is held below 40 mm by overflow. The interface between aqueous and organic phases on the outlet is measured by electrodes and regulated pneumatically. Each mixer-settler has one driving motor. The motor, the gear box and the mixing paddles can be exchanged remotely.

The tanks are slab tanks 40–50 mm thick. They are either positioned in one plane or sandwiched. In sandwich tanks 300-mm polyethylene and a thin cadmium sheet are contained on each side of the tank. For all tanks, apparatus and pipelines the reciprocal effect of neutrons was calculated. Final waste storage tanks with capacities of over some hundred litres are not critically safe by geometry (Fig.3). Transfers of solutions in these tanks are only allowed after duplicate analysis via a key-lock.

Transfers of liquids within the cells are achieved by air-lifts. Radioactive feed solutions are pumped with remote head membrane pumps. Flow-rates of feed solutions are measured with a burette with two electrodes.

All apparatus and tanks have a separate ventilation system. The off-gas is joined with the dissolver off-gas before the wet scrubbing columns. Filters for the cell ventilation are situated in the cells.

Over a period of five years different experiments with highly active materials were carried out in MILLI. Examples are the isolation of ^{238}Pu from irradiated ^{237}Np [2,3] reprocessing experiments with fast breeder fuel irradiated up to 60 000 MWd/t [4] and with different LWR-fuels irradiated up to 40 000 MWd/t [5].

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DISCUSSION

I.A. SHUJA: What are the main problems that you have had to face in the separation of uranium and plutonium, and what sort of efficiency have you attained in the recovery of plutonium?

Secondly, on what basis do you claim that the Purex method is better than the ion-exchange technique?

W. OCHSENFELD: Our main problems with highly irradiated fuels concern the first extraction cycle and result from the radiolysis of TBP (tributylphosphate). Degradation products from TBP react with certain fission products, more especially zirconium, resulting in the formation of emulsions and crud. We have managed to overcome these difficulties by suitably adapting the Purex process conditions.

As regards plutonium recovery, the losses in the high-level waste solutions have been lower than 0.1%. We have observed that the separation of U-Pu with U (IV) from highly irradiated fuels has not always been stable.

To answer your second question, solvent extraction is a continuous method, while ion exchange is usually not so. The development of continuous ion-exchange columns is, however, under way.

The Purex extractant can be continuously washed free of degradation products, whereas ion-exchange residues have to be discarded.

The ion exchanger works with a high nitric acid concentration of 7-8M. This results in higher amounts of waste or requires further acid recycling.

A further point is that as a result of bad experience with fire and explosion hazards at different sites in the United States of America and France some ten years ago, interest in ion-exchange resin reprocessing has greatly fallen off.

K. KABONZA: How do you monitor the liquid level in the storage tanks?

W. OCHSENFELD: The level of the solutions in the tanks is measured by the conventional dip-tube method. Both the weight and the specific gravity of the liquid are measured by the pressure at the outlets of tubes fixed in the tanks.

C. CESARANO: Do you intend to try out a centrifugal extraction system instead of the mixer-settler?

W. OCHSENFELD: No, we have no plans to test centrifugal extractors in the MILLI plant. The limited size of the plant containers (the geometry of which makes them safe against criticality) would not provide the volume required for centrifuges with their high throughput.

At the Hot Chemistry Institute we have developed and tested centrifugal extractors with a throughput of 200 kg/day. These have now been installed at the WAK reprocessing plant in Karlsruhe. We hope that, after the long period of cold testing, they will soon be licensed for hot operation.



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EQUIPMENT AND INSTRUMENTATION OF A LABORATORY FOR PUREX PROCESS ANALYTICAL CHEMISTRY

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Abstract

EQUIPMENT AND INSTRUMENTATION OF A LABORATORY FOR PUREX PROCESS ANALYTICAL CHEMISTRY.

By the example of the process analytical service for the small-scale MILLI reprocessing facility a description is given of the underlying prerequisites such as laboratory equipment, working conditions and safety precautions. In addition, the fundamentals of analytical instrumentation and associated problems are discussed and finally some facts are presented which result from several years of practical experience.

1. INTRODUCTION

At present spent reactor fuel elements are almost exclusively reprocessed according to the proved Purex process. Starting from nitric acid solutions uranium and plutonium are first separated from the fission products by multi-stage counter-current extraction using tributylphosphate and subsequently separated from each other. The finely purified uranium and plutonium end-products are recycled while the fission products are normally considered as wastes.

On account of the intensive β - γ -radiation of many fission products, reprocessing calls for thick shielding and remote handling of the equipment. Moreover, the enormous radiotoxicity of plutonium and other α -emitters requires an α -tight containment. These indispensable conditions confer a unique and characteristic status to the Purex process. Besides complicating plant operation they also make the process control quite difficult and have led to the development of special working techniques.

It is the task of process analytical chemistry to provide information about the respective operation conditions of the reprocessing plant. Generally, this can be done by chemical-analytical investigations of samples taken from the process. Furthermore, to guarantee up-to-date information, the analyses must be made quickly. It is quite evident that the conditions allowing this task to be fulfilled must be similar to that of plant operation proper.

At the Hot Chemistry Institute of the Gesellschaft für Kernforschung Karlsruhe the MILLI pilot facility [1] has been operating since 1970. It has been used in experimental investigations of the Purex process, e.g. systematic flow-sheet and parameter studies for optimization of reprocessing plant operation, for testing modified contactors and for dealing with specific problems (reprocessing of fast breeder fuels, crud formation, etc.). Guided by the analytical service for this facility a description of our analytical laboratories and the analytical instrumentation is given.

2. EQUIPMENT AND ORGANIZATION

The numerous process analytical tasks first call for adequate locations allowing the handling of process samples, part of which are highly radioactive. Accordingly, the following working places are available to our analytical staff:

- 3 concrete cells: Shielded on all sides by 90 cm concrete, equipped with master-slave manipulators and lead-glass windows, approved for 300 Ci (γ); one cell is used as a sample storage place and rabbit station; two cells are working cells.
- 1 lead cell: Glove-box type, shielded on its front side by 10 cm lead and freely accessible on its rear side, equipped with small-scale manipulators and lead-glass windows, approved for 1 Ci (γ); 3 working places.
- 1 laboratory for routine analyses, equipped with glove-boxes with 16 working places.
- 1 laboratory for nuclear radiation measurement, equipped with glove-boxes and open hoods; 4 working places.
- 1 laboratory for trace analyses.

The analytical laboratories adjoin each other and are located in the centre of the control area. Within the control area there is a negative pressure of -3 to -5 mm water height as against the outer atmosphere. A pressure of about -25 mm with respect to the laboratory atmosphere prevails in the hot cells and in the glove-boxes. The ventilation system of the institute is divided into the laboratory and box exhaust branches.

The whole air feed consists of fresh air which has passed a filter (98%, 0.4 μm). The laboratory exhaust air passes an absolute filter (99.95%, 0.4 μm). The box exhaust air passes a prefilter and an absolute filter immediately at the working place; a second absolute filter has been installed in front of the ventilator blowing off the box exhaust air via a 60-m-high stack. The negative pressure in the box exhaust channel amounts to -180 mm water height. In the chemical laboratories the air is exchanged 20 times per hour; the same applies to the cells and the boxes. If a leakage occurs, the rate of air exchange in both the cells and the boxes is automatically increased to 50 so that a linear air velocity of about 0.5 m/s at the leak points can be guaranteed. The maximum admissible leak rate of the hot cells is 1% of the cell volume per hour at -25 mm, that of glove-boxes is 0.5% of the box volume per hour at -100 mm.

The analytical samples are conveyed by a pneumatic rabbit system, movable double-lid locks and/or bag technique. The concrete shielded cells are interconnected by a conveyor system equipped with an autostop device.

The analytical staff is divided into three subgroups: (1) routine analytics; (2) nuclear radiation spectrometry, and (3) special analytical methods and development. At present, 14 persons are employed who make 20000 analyses per year. About 30% of the work is done in shift operation around the clock.

The operation sequence is fixed by a central office where the orders for analyses are accepted, operator instructions are issued, analyses are evaluated or critically supervised and, finally, the results are transmitted to the customers. Before each extended process campaign an analytical schedule is set up after consultations between the process management and the analytics central office, indicating the place and date of sampling as well as the objects to be analysed. On the one hand, this schedule serves to estimate the required analytical capacity and, on the other hand, it guarantees that the process management receives up-to-date information on all process parameters which are significant for operation.

3. SAFETY ASPECTS

All facilities and equipment of the institute comply with the regulations issued under the Federal German Radiation Protection Regulation (which in turn largely conforms with the IAEA standards); they are controlled by supervising authorities.

The control area includes all localities and facilities in which radioactive substances are handled. It is surveyed on a routine basis by the health physics staff. Working within this area

requires special clothing and personnel dose meters. Fixed hand and foot monitors for α, β, γ -measurements are installed in the laboratories and at strategic points of the control area. Every person working in the control area must undergo a regular medical examination (thorough medical inspections, body-counter measurements, urine tests); medical objections may lead to a restriction or even prohibition of work within the control area. Similar restrictions apply in the case of excessive radiation exposure.

All activities carried out inside the control area are subject to prior authorization and are supervised by the director of the institute or his representatives. Instructions on radiation protection measures and safety precautions are given every six months and the attendance of such courses is compulsory. Work implying an increased risk can be done only after an accurate analysis of the given situation and if a written order has been obtained.

Overshoes must be worn in the plutonium laboratories in order to avoid the spreading of α -activity in the event of contamination. In these laboratories air dust measurements for activity control are performed regularly, part of them continuously; routine wiping tests at the working places and on the floor serve the same purpose.

As a safety precaution against criticality the amounts of plutonium available in the laboratories are portioned into batches for which upper quantitative limits and clearly defined personal responsibilities are specified. The actual amounts of plutonium contained in such batches must be recorded by accountancy procedures. The transport of plutonium and of other radioactive materials is subject to administrative limitations and controls.

In the central alarm station of the institute all significant parameters of operations carried out are permanently surveyed and recorded. The negative pressure in the ventilation systems and the radioactivity of the exhaust air are measured and registered continuously; as soon as a preset limit value is exceeded, an alarm is given automatically. Contamination alarm devices provided at each individual working place, as well as automatic fume and radiation warning devices installed at strategic points in the control area, complete the surveillance system. By an intercommunication system important information can be quickly and simultaneously transmitted from the central alarm station to all persons working in the control area.

In contamination incidents the health physics staff summoned give first-aid treatment and decide upon measures to be taken afterwards. Contamination of persons is normally treated under the supervision of a physician.

In the case of fire and radiation alarm the control area is evacuated immediately; all persons gather and wait for the instructions of the emergency brigade of the Karlsruhe Nuclear Research Center which had been alerted simultaneously.

4. ANALYTICAL INSTRUMENTATION

The requirements for the analytical group, which are imposed by reprocessing and its special features, determine the equipment and analytical instrumentation used. On account of the distinctly experimental character of the MILLI facility operation this potential must, moreover, be so flexible that all analytical requests can be satisfied. So, the following aspects served as a guideline in the development of process analytics:

- (1) The analytical methods to be selected should be as versatile as possible. They should be characterized by specific measuring signals for the substances or for the elements to be analysed so that interfering influences by accompanying substances, which might call for chemical separations, can be largely avoided. Regarding implementation, the analytical methods should be uncomplicated (remote handling, time expenditure) and give sufficiently accurate results; they should be highly sensitive.

- (2) The analytical instruments to be selected should be highly reliable in operation and easy to handle. The measurement signals should be suitable for computer processing. The apparatuses should be so conceived that they comply with the technical requirement of adaptability to both cells and boxes. In the ideal case only the measurement position of the analytical instruments should come into direct contact with the radioactive material; all the other parts should remain free from contamination.

The most frequent analytical tasks are the determination of uranium, plutonium, nitric acid, fission products, reduction and oxidation agents and tributylphosphate decay products, both in aqueous and in organic process solutions. Therefore, we have chosen as basic analytical methods first X-ray fluorescence analysis for (simultaneous) determination of heavy elements and, second, high-resolution nuclear radiation spectrometry with solid-state detectors for the analysis of α -active trace elements and γ -emitting fission products.

About 60% of all process analyses are performed with these key methods; they are supplemented by potentiometric, spectrophotometric and gas chromatographic analytical methods and by systems for remote aliquotation and dilution, density determination and weighing.

Apart from some exceptions only such work is performed in the hot cells by which the β - γ -activities of the analytical samples are reduced. The analytical determinations proper are subsequently performed in non-shielded glove-boxes under much more favourable working conditions.

When providing the hot cells with analytical instrumentation we have made it a rule that within the cells only the indispensable equipment is installed while, for example, electronic instruments, automatic titration apparatuses, reagent flasks, etc. are installed outside the shielding and are freely accessible. Generally, reagent solutions are carried into the cells through stainless-steel or plastic tubes and metered from outside. Figure 1 illustrates this procedure by the example of a remotely handled pipetting device conceived by us. The pipette is connected to a small expansion vessel by a stainless-steel capillary tube which in its turn is coupled with the microburette placed outside the cell by a Teflon tubing. The burette, the plastic tubing and the

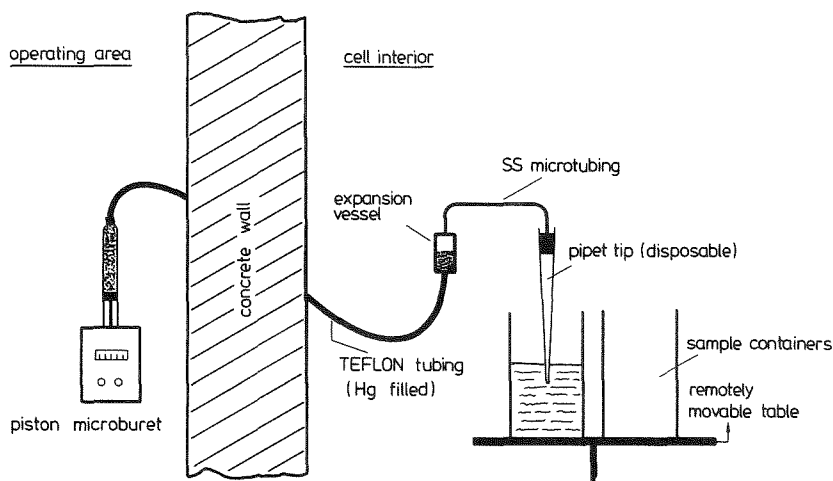


FIG.1. Remotely operated aliquotation device.

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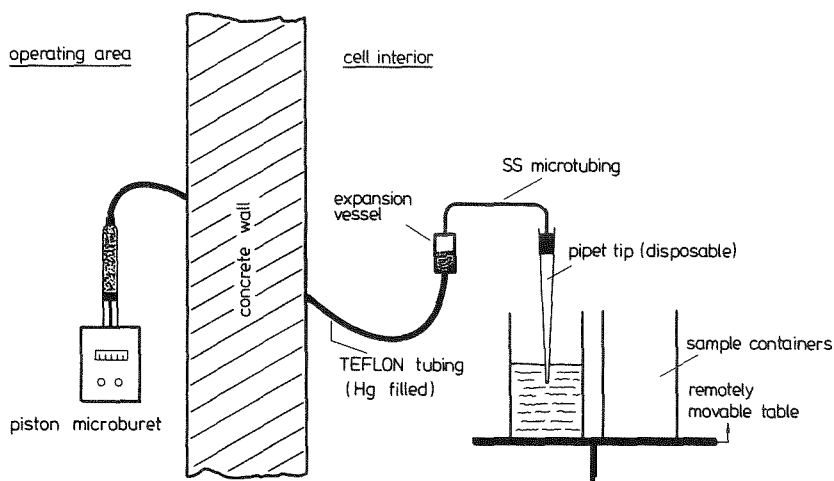


FIG.1. Remotely operated aliquotation device.

expansion vessel are filled with mercury so that lifting movements of the burette piston are hydraulically transferred into the expansion vessel. The flask from which the sample must be taken is conveyed by remote handling to a small motor-operated table, movable both in the horizontal and in the vertical directions, and is brought under the fixed pipette. In this way, volumes of solutions between 50 and 1000 μl can be reliably metered and transferred by remote handling. Cross-contamination effects are eliminated by the use of disposable pipette tips made of plastic material.

Regarding the installation of hot cells, similar statements can be made for the instrumentation of glove-boxes. However, since shieldings are commonly not required, even more complicated analytical instruments can be directly adapted. For example, to perform spectrophotometric investigations we integrated into the box only the cuvette part of the Zeiss PMQ spectral photometer while the monochromator, the detector, the light source, the electronic components, etc. remained freely accessible outside the box [2].

Another example is the Siemens MRS multi-channel X-ray spectrometer where, by introducing a thin beryllium sheet into the beam path, we achieved the situation whereby only the measuring position of the spectrometer lies inside the box [3].

By the use of highly sensitive, element-specific analytical methods the amount of substances needed and consequently the amount of activities handled can be reduced to such an extent that even highly radioactive solutions can be analysed in the absence of specific shielding precautions after preparation of corresponding low-level samples by remote aliquotation or dilution. For instance, uranium and plutonium analyses can be performed simultaneously by X-ray spectrometry in process solutions with specific activities up to 1000 Ci/l by dropping only about 10 μl of solution on to a prepared filter paper as the carrier material [4]. In spectrophotometric and gas chromatographic analyses, rarely more than a few micrograms of the substance to be analysed are required, so that also for these analyses shieldings are normally not necessary. Finally, the currently used high-resolution α - γ -analysers equipped with solid-state detectors require hardly more than 1 μCi activity to determine even complex mixtures without the necessity of preliminary chemical separations.

Both the lack of personnel and the rapidity imposed in process analyses gave rise to partial automation of some analytical procedures. For example, potentiometric titrations are mainly performed using automatic titration apparatuses and the X-ray spectrometric U, Pu data are evaluated by a small computer. Also the γ -spectra are measured and evaluated by computer control in a fully automatic way by connecting our spectrometer system to the central data processing station of the Karlsruhe Nuclear Research Center [5].

5. OUTLOOK

After several years of operating experience with the equipment and working methods described we can state in summary that the basic concept of analytical methods and laboratory equipment used, as well as the organization of work, have proved their worth. Owing to the comprehensive safety measures and their strict enforcement the analytical staff has been spared accidents or other noteworthy consequences, also in critical situations.

For the most important analyses a high degree of operating reliability was achieved by the redundant design of analytical working places. By centralizing the analytical laboratories of the institute short transport paths have been achieved and both operation and supervision of the analytical work are facilitated. The transport of analytical samples via double-lid locks proved to be a bottle-neck in our work because these locks are susceptible to mechanical failures and frequent contamination. Therefore, we are at present replacing these locks by a pneumatic rabbit system. The initially feared corrosion problems in the hot cells and in the boxes have been kept

within reasonable limits. This is probably to a large extent attributable to the elimination of halogens and hydrohalogenic acids in process analytical applications.

At times, appropriate commercial instruments or apparatuses could not be acquired for special applications. In many such cases satisfying solutions for the actual analytical problems were found by modifying commercial instruments or by our own constructions.

In the given case extensive automation of analytical methods and procedures is counteracted by the experimental situation which is characterized by a frequent change of analytical problems. Therefore, we concentrated first on the use of computers for evaluating measured data. Nevertheless, efforts are under way to continue the automation of the basic methods of X-ray fluorescence analysis and nuclear radiation spectrometry beyond the presently attained level.

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DISCUSSION

E. LILLYMAN: What work is being done on in-line instrumentation in the control of solvent extraction plants for irradiated fuel reprocessing?

I do not think that the control of the plant by retrospective analysis of spot samples is a satisfactory method to use.

D. ERTEL: At our Institute there are some people working on the development of analytical in-line instrumentation and/or automation of routine analytical work. This was described by Mr. Groll in his paper¹. The analytical in-line instrumentation being studied is based on measurement of nuclear radiation effects, density, conductivity and electropotential properties of process solutions, together with light absorption capacity. Some of the instruments developed have been successfully tested in simulated circuits and will shortly be installed at a variety of plants for trials under actual conditions.

With regard to the retrospective analysis of process samples, so far this method has proved indispensable for controlling production plants, and I believe the same will apply in the near future. In the case of experimental facilities like MILLI, it is the only possible way of control. We have had satisfactory experience with the special type of spot analysis I describe in my paper. The simultaneous determination of uranium and plutonium in highly active process solutions only takes about 30 minutes and produces accurate results.

¹ IAEA-SM-209/24, these Proceedings.

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