CHEMICAL PROBLEMS ARISING IN THE OPERATION OF A SWIMMING POOL REACTOR AND USES OF THE REACTOR IN CHEMISTRY

PROBLEMAS QUÍMICOS ATINENTES À OPERAÇÃO DE UM REATOR NUCLEAR DO TIPO PISCINA E USOS DO REATOR EM QUÍMICA

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1. INTRODUCTION

The Brazilian Research Reactor (IEA-RI), a 5 Mw Swimming Pool type, first went critical on September 17, 1957.

Prior to that time, and owing to shortage of people, the installation of pieces of machinery, erection work, tile lining of the pool, etc. had to count, on quite a large scale, with the help of the small staff of scientific people themselves.

Buildings for laboratory facilities and laboratory work were ready only after the construction of the reactor building and reactor installation. In this way, the small group of five to eight people forming the scientific staff of the "Instituto de Energia Atômica" got hold of peculiar type of work as loading and installing ion-exchangers resin tanks, pool cleaning, erection work, soldering and so on, and there were times when no distinction could be made between a labor hand or a scientific one.

The first job assigned to the Division of Radiochemistry, whose staff by March 1957 was of only two men, one of them signing this paper, was to supervise the installation of the water treatment equipment. Quite soon after disembarkment of such equipment the supervisioning work turned out in actual labor work. Later on came loading the ion-exchangers tanks and filling the pool with water.

Cronologically, the work came out in the following order: filling the pool with demineralized water (about 270,000 liters) and maintaining its conditions in such a way that pH values would be kept around 6.5 and total soluble substances in the water around two parts per million. Later on, and after this quite simple problem but not quite so, had been solved and the fuel elements had been assembled, the Company that had designed and built the machine, advised their representative man at IEA, an Operator Engineer, to check the elements for possible corrosion spots, since some elements had shown corrosion in a similar type of reactor being built in another country.

Unfortunately, visual inspections (the elements were cold by that time) indicated that signs of corrosion were evident in, at least, five elements.

The elements were removed from the pool and given an anodizing protective treatment, as advised by the reactor's designers, under the supervision of their Engineer. The protection treatment was carried out, accordingly (1).

After treatment the fuel elements were reassembled and the Reactor' Physics people started their experiments on the operations of the machine while rigorous control was being made of water conditions concerning pH and conductivity as well as of the presence of fission products.

When the first indication arised that fission products were present in the water, radiochemical analysis were made on various points of the water system. These radiochemical analysis were described in detail in another paper (1).

By that time the presence of fission products in the water was suggested as being due to two main factors: surface contamination of the fuel plates with uranium during fabrication and/or plates with cladding defects in which corrosion had kept on going since the anodizing protective treatment had been given after corrosion had already started. Later tests (2) showed that the original elements in which corrosion had started before the anodizing treatment, were the ones in which fission products came out most frankly.

Removal of the defective fuel elements set, and substitution by another set in which the plates were protectively anodised before assembling the plates to form the elements, solved the problem of the presence of fission products in the water. These new elements had their plates assembled by riveting with aluminum rivets; no soldering of plates to form the element was made and no soldering flux was used in the new set. The first set had their fuel elements assembled by soldering the plates in place and using soldering flux in which fluorine was one of the components of the flux, in accordance with information from the designers. Poor removal of the soldering flux might have been the cause for the corrosion. Coriu (15) has, in fact, called attention for this type of corrosion cause, and suggested that assemblying of plates to form the elements should be made by riveting and not by brazing. Photographs of corroded plates showed that corrosion areas were along the places where soldering flux had been applied.

The new set of elements, assembled by rivets and not by brazing, has been in use for about two years and no fission products have been detected in the water or ion-exchanger recicling system.

After the solution of the problems in connection with water conditions on the pool, corrosion of fuel elements, identification and analysis of fission products, the reactor was put in routine operations and the radiochemistry group could make use of irradiation periods for activation analysis, radioisotopes production for tracer work at the Radiochemistry Division, for medical research and treatment at some hospitals (mostly iodine-131 and phosphorus-32) and for agricultural research work (phosphorus-32), at the Agriculture School of the University of São Paulo.

By this time the research laboratories and chemical buildings were already constructed and equipped with ordinary counting equipment (geigers and scintillators), scalers, single channell pulse analyser, as well as basic equipment for chemical work, comprising polarographs, pH meters, spectrophotometers, conductivity cells, thermo-balance etc..

The chemical staff had been increased from two people in 1957, to four-chemists and two technicians in 1958 and 1959, four chemists and four technicians in 1960 and seven chemists and four technicians in 1961.

The important aspects of the development of chemical work, by utilizing the reactor as mentioned before, will be briefly described as well as the chemical problems arised during the installation and initial operations of the reactor.

II. WATER TREATMENT, MAINTENANCE AND ANALYSIS

The water treatment system of the Brazilian Swimming Pool reactor is made up of two sets of purifying tanks. A primary system made up of the following components: a felt filter for gros filtration of suspendend particles in the water, followed by a softner tank 1,5 meters high by 75 centimeters in diameter containing 400 liters of cationic ion-exchanger (sulfonic acid type); after the softner tank there is an actived carbon filter one 1,5 meters high by 50 centimeters in diameter, followed by two ion exchangers mixed bed tanks 1,5 meters high by 50 centimeters in diameter each containing 60 liters of cationic and 60 liters of anionic ion-exchangers tanks. One is kept on operation and the other on reserve to be used when the resin in the first one is exhausted and needs regeneration.

The secondary system is made up of a tank of activated carbon filter 1,5 meters high by 1,5 meters in diameter followed by an ion-exchanger mixed bed tank 1,5 meters high by 60 centimeters in diameter containing 130 liters of cationic and 130 liters of anionic ion exchanger. Another pair of tanks identical to this one is kept in reserve; when the resin in the first ion-exchanger tank is exhausted, water circulation is deviated to the second pair of tanks and the first one is regenerated.

In the primary system tap water is first filtered from solid particles, softned, and then demineralised in the mixed-bed ion exchanger at a rate of about twenty liters per minute.

This treatment of the water in the primary system gives a water with about five parts per million of soluble substances. After passing through the primary systems the water is sent to the pool through stainless steel pipe-lines. Water from the pool is kept recirculating countinuously through another set of mixed bed ion-exchangers at a rate of about sixty liters per minute. In this way water is kept in the pool with a specific resistivity of about one megaohm x cm and around two parts per million of soluble substances.

If during regeneration procedures the anionic and cationic resins are throughly mixed as it is usual and recomended on water purification by ion-exchangers for general purposes, the pH will come out very nearly to 7.0. This value, however, is not convenient for aluminum-cladding fuel elements and aluminum frames and to keep corrosion within acceptable limits it is necessary that the pH be held to a value close to 6.5, in accordance with Breazeale (3). French researchers and engineers recomend that the pH value should be kept in between 5 and 7, in which range aluminum corrosion is the least (15). We could solve this problem by letting some cationic ion-exchanger to settle at the bottom of the tank without mixing it with the corresponding amount of anionic resin. This can be accomplished by diminishing slowlly the air stream used to mix the resins after regeneration, instead of cutting this stream suddenly when the two kind of resins would be perfectly mixed. The well-known phenomenon of cationic leaking (4), when the two kind of resins are not perfectly mixed and some cationic resins is at the bottom of the tank, will provide the required amount of hydrogen ions to low the pH to values close to 6.5.

The average four years performance of the ion-exchangers, expressed as volume of water treated between regenerations, is presented in Table I.

Table I

Volume of water treated
between regenerations.

	Volume treated
Softner	900,000 liters
Primary mixed-bed (non-radioactive)	30,000 liters
Secondary mixed-bed (radioactive)	1,000,000 liters

Any one of the mixed bed ion-exchanger is regenerated when the resistivity falls to 5 x 10^5 ohm x cm. It is usually recomended that resistivity be maintained above 4 x 10^5 ohm x cm (16). The regeneration intervals as presented on Table I have given water in a very good condition for our pool (ceramic tile lining) with pH kept around to 6.3 ± 0.1, crystal clear for the various operations of loading the reactor with fuel elements or samples to be irradiated and with low activity due to the eventual activation of substances still remaining in the water.

Typical analysis of the pool water is presented in Table II. Table III gives the maximum and minimum values for resistivity and pH for the year of 1960.

Table II

Analysis of Pool Water
for the Brazilian
Swimming Pool Reactor
(pool with ceramic
tile lining).

Al	0,1 ppm
Cl	0,02 ppm
SO_4	-
SiO_2	1 ppm
Fe	0,003 ppm
Na	0,4 ppm
Ca+Mg	0,2 ppm
Resistivity	8x10^5 ohmxcm
pH	6,5

Table III

Maximum and minimum values for resistivity and pH for the water in the pool during the year of 1960.

	Resistivity (ohm x cm)		pH	
	max	min	max	min
January	9.1×10^5	4.8×10^5	6.2	6.1
February	9.1	7.1	6.3	6.2
March	8.1	7.3	6.3	6.2
April	5.9	4.9	6.3	6.2
May	12.1	4.1	6.3	6.2
June	13.7	5.0	6.2	6.2
July	12.1	9.4	6.3	6.3
August	9.7	7.3	6.2	6.2
September	7.6	5.5	6.3	6.1
October	10.2	8.1	6.4	6.2
November	11.8	7.6	6.4	6.2
December	6.8	4.9	6.4	6.3

After about four years of work with regeneration intervals as described in Table I, there is indication of silica leakage on the primary system as well as loss of capacity of the ion-exchangers, which are to be replaced by a new charge of resins.

III. CORROSION OF FUEL ELEMENTS

As mentioned on the Introduction, after the first visual indications that the fuel elements might be corroded they were removed from the pool and given the anodising protective treatment as described in (1).

The elements were then returned to the pool, the core was reassembled and criticallity experiments were carried out at low power level, mostly zero megawatts. Before criticallity, samples of water were taken to check for the presence of radioactivity. The results were negative.

After about one to two hours runs at zero power level, samples of water were collected by passing through small ion-exchangers columns placed at convenient places. Samples were taken from the primary refrigerating system (capacity flow of about 11,400 liters per minute), from the pool itself, and the inlet of the ion-exchangers recirculation system.

The collected activity was very small. However, at 10 kilowatt power level the collected activity was of some significance. Most of the samples were taken at a by-pass made in the primary refrigerating system since water at this point would have passed directly through the reactor core resulting in an entrainment of fission products if they were being released by the fuel elements.

A systematic plan of study was made in order to identify the radionuclides collected on the small ion-exchangers columns used for water sampling. Chemical separation schemes for fission products radioisotopes were developed and also well known separations methods were applied; the ones developed by our group have already been described in detail in another papers, (1) and (5). Some comments on this chemical work will be made later.

Fission products activities were definitely identified and this indicated that every fuel element should be checked for failure. Evidently this could not be done by direct inspection since the elements were sufficiently hot by that time.

The elements were checked one at a time by the method devised by Toledo, Souza Santos, Brenner and Penteado (2) and 10 elements out of 35 were found as definitely bad.

As mentioned in the introduction the assemblying of the plates to form a fuel element had been made by brazing the uranium aluminum alloy plates into aluminum side plates which formed a container. When the elements were visually examined, while cold, but having been in demineralised water for some time, it was noticed that corrosion areas extended along the brazed region.

The presence of fission products in the water and the corrosion along the brazed region raised the question that pit corrosion might had gone deep enough to allow fission products, specially fission gases, to escape through the cladding into the water. Consequently the bad set of elements was removed from the pool and water was kept recirculating continuously through the demineralized system for about three months, while checked periodically for fission products that might have deposited on the aluminum frames or the walls of the pool; no significant activities were found.

The new elements which were used to reload the reactor had their uranium-aluminum alloy plates revited, with aluminum rivets, into the aluminum side plates, instead of brazed. Also, the plates were given the anodized protective coating prior to the assemblage into elements. These new elements have been in use for about two years, with the reactor operating at various power levels, mostly at two megawatts, without any leakage of fission products. Inspection of the fuel elements, made by bringing them at about two meters from the surface of the water and examining with a telescopic device did not show any visual signs of corrosion.

VI- RADIOCHEMICAL IDENTIFICATION OF FISSION PRODUCTS IN THE WATER

When the activity collected on the small ion-exchanger columns placed in the by-pass of the primary water refrigerating system was high enough to raise suspicion of fission products leakage through the elements, chemical separation schemes were devised to identify the radionuclides.

Detailed description of these separations has already been published, (1) and (5). A brief account of this work will be made.

The radionuclides identified were Ba-140, La-140, Y-91, I-131, Sr-89, Ce-141 and Mo-99. All radionuclides found have precursors that are formed as gases in fission, with the exception of Mo-99.

Ba-140 and La-140 are descendants of Xe-140 whose fission yield is 6.1%; Y-91 is descendant of Kr-91 with a fission yield of 5.9 %; Sr-89 descends from Kr-89 whose fission yield is 4.6%, Ce-141 descends from Xe-141 whose fission yield is 5.7%.

By the time paper mentioned on reference (1) was published, no definite evidence for radionuclides formed directly on fission had yet been found, with the exception of Mo-99. This radionuclide was first thought as comming from a (n,gamma) reaction on possible corrosion products from stainless steel pipes;

however, by checking the composition of the steel alloy it was verified that molybdenum was not a component of the alloy.

Later on Nd-147, Zr-96 and Te-132 were identified, although in lesser amounts than the radionuclides forming as desintegration products of fission gases, even taking into account the smaller fission yields of the radionuclides Nd-147, Zr-96, and Te-131 as compared tothe fission yields of the gases mentioned.

The presence of the radionuclides formed directly on fission or as desintegration products of fission gases was a good evidence that fuel element failure had occured since the anodising cleaning procedure would have cleaned the elements from any external uranium contamination. The steps taken to identify which elements had failures have already been mentioned in III and described in detail in reference (2).

During the critical experiments in which fission products were released from the fuel elements, the fission radionuclides were retained by the ion-exchangers in the repurifying system. Since the reactor had not worked at high power level and even so, for short periods of time of about one to two hours, the amount of radioactivity acummulated in the ion-exchangers was not high enough to justify disposal of the resins and tanks.

Instead, this waste disposal problem was even utilized as an education and training expedient.

After a cooling period of about three months, while the new set of elements were being finished to substitute the old one, the ion exchanger which had collected the fission products was regenerated as usual. However, efluent liquids coming from the regeneration operation were not sent to the retention tanks as with ordinary regeneration solutions, when only radioactive species formed by activation of substances dissolved in the water would be present. The efluents were collected in large steel drums and the radioactive species were removed by coprecipitation with aluminum hydroxide, followed by another coprecipitation treatment with ferric hydroxide.

Practically, all radioactive nuclides are entrained with the precipitates of aluminum hydroxide and ferric hydroxide. Cesium, however, is not coprecipitated and remains in solution.

The liquid is decanted and removed to another steel drum where a ammonium alum precipitation in alcoholic medium is carried out in order to remove cesium-137. Details of this operation is described in reference (5).

Instead of just disposing the hot precipitates of aluminum hydroxide, ferric hydroxide and ammonium alum which had entrained the radioactive species collected on the ion-exchangers, they were used, in small portions, for laboratory work in radiochemistry courses. Quite a good training can be obtained by working this material and various separations schemes for group of radioelements or individual ones can be devised, as well as identification of the separated radionuclides by half-life determinations, aluminum or lead absorption curves for the determinations of energy of particles or radiation, gamma ray espectrometry, etc.

We have noticed that students become enthusiastic with this type of actual work when frequently not even the instructor knows the complete composition of the samples to be analysed, and where various classical chemical precipitation techniques have to be applied as well as ion-exchanger chromatography, solvent extraction, carrier techniques, etc. Obviously, owing to the type of material, very good Health Physics procedures in teaching laboratories have to be followed as well as close control of contamination of working tables, rubber gloves, laboratories garments,etc. Up to now this has not constituted any serious trouble.

V-USE OF THE REACTOR FOR RADIOISOTOPES PRODUCTION FOR MEDICAL APPLICATIONS AND CHEMICAL RESEARCH.

Two of the most used radioisotopes in medicine have short half-lives, meaning iodine-131 and phosphorus-32.

This constitutes a serious handicap in the development and spreading of new techniques for diagnosis and treatment, what is specially true in the case of iodine-131 with a half-life of about only 8 days. In Brazil it is usually necessary to import the double of the doses required for an application.

For thyroid diagnosis the doses is usually under one millicurie per subject. This doses goes up to ten millicuries per subject for treatment of hyperthyrodian disturbs and to a hundred to three hundred for thyrodian cancers.

Studies carried out at this Instituto have shown that by making judicious uses of irradiation periods during critical experiments carried out for Reactor Physics and Nuclear Physics Research, Radiobiologic Effects of Radiation, training of operating crews etc.. it is possible to produce carrier free iodine-131 and phosphorus-32 that will supply a good part of the medical requirements for those two radioisotopes, at least for public hospitals.

At the Instituto de Energia Atomica it has been possible to produce about 50 millicuries of carrier free iodine-131 and 40 millicuries of carrier free phosphorus-32 per eight hours operation of the reactor, at two megawatts, with the operator and chemical staff presently available at the Instituto.

This work is being intensified presently in order to increase the production up to 300 millicuries of iodine-131 per eight hours of reactor operations. This should give about 800 to 1,200 millicuries per month. The actual medical demand is about 500 millicuries per month.

Phosphorus-32 demand is, by now, smaller than iodine-131 and it is of the order of 120 millicuries per month. The present production at IEA is 160 millicuries per month and it should go to about 400 millicuries per month in the near future.

Iodine-131 is produced by irradiation of telluric acid and at thermal neutron fluxes of about 10^{12} neutrons/seg. cm^2.

Phosphorus-32 is produced by irradiation of water soluble sulphates instead of elemental sulphur. We have used magnesium sulphate and by now we are trying ammonium sulphate to avoid the inconvenience of sodium-24 formation by a (n,p) reaction with magnesium-24. We are faced with some radiation chemistry on the ammonium sulphate owing to the formation of sulphur gases, what had not been observed with magnesium sulphate.

The chemical process for the production of phosphorus-32 from the irradiation of magnesium sulphate have been described in another paper (6).

A preliminary estimation of cost of production of iodine-131 and phosphorus-32 have been made (7). This estimation takes into account combinations of time of irradiation and duplication of samples to be chemically processed, for monthly values up to

1,000 millicuries of iodine-131 and 400 millicuries of phosphorus-32. The results indicate that the production might be economical and in fact competitive with importation in the case of short half-lives radioisotopes.

All radioiodine produced at the IEA up to now have been used for thyroids diagnosis and treatment at Universities hospitals; radiophosphorus has been used for agricultural and medical research at the University of São Paulo.

The production of other radioisotopes, usually by (n,gamma) reactions, for tracer work, has not constituted any problem. A large variety of these isotopes have been produced for research at the Instituto itself or other places. Some of the radioisotopes produced are Na-24, Hf-181, Eu-152, Nd-147, Cu-64, Sr.89-90, fission products radioisotopes, Np-239, Au-198, Zr-95, Co-60.

A brief survey of chemical research work in which these radioisotopes have been used as labelling material will be made. Detailed description of the work can be found on the papers mentioned on the references.

VI- CHEMICAL PROBLEMS SOLVED BY USING RADIOISOTOPES PRODUCED AT IEA-RI AS LABELLING MATERIAL.

VI-1 - Separation of Zirconium from Hafnium-Ref. (8)

The problem of complexes formation of zirconium and hafnium with sodium triphosphate was of interest to the Chemistry Department of "Faculdade de Filosofia, Ciências e Lêtras" of the University of São Paulo. Previous work by that group, and others, showed that a separation of those two elements by sodium triphosphate might by achieved. Dificulties to carry on ordinary chemical analysis owing to similarity of behaviour of hafnium and zirconium were solved by a joint work between that Department and the Radiochemistry Division of IEA. Zirconium and hafnium oxides were irradiated at IEA-RI for periods from three to six hours in thermal neutron fluxes of the order of 10^{12} neutrons/seg. cm^2.

Separations procedures were applied, and the various fractions were analysed quantitatively by gamma-ray spectrometry measuring the photopeak areas for Hf-181 (0.133 Mev) and Zr-95 (0.72 Mev). Conclusions were that fractional precipitations of zirconium and hafnium triphosphate would enrich the precipitate fractions in hafnium, and the filtrate in zirconium.

VI.2 Isotopic Dilution Analysis of Phosphorus in Atomically Pure Uranium Compounds. Ref. (9).-

Phosphorus analysis in uranium compounds is usually made by optical spectrography by using 50 grams samples of uranium compound.

Necessity to control amounts of phosphorus present on samples of uranium obtained at a IEA Pilot Plant, and lack of optical equipment by that time, enforced the Radiochemistry Division to develop a method of analysis by using phosphorus-32, prepared at IEA, as tagging element.

Samples of uranium containing as low as 10 parts per million of phosphorus can be analysed in eight hours work by using 10 grams samples of uranium compounds. Standard deviation of the analysis is of the order of 10%.

VI.3 -Control of Purification Procedures for Uranium Compounds - Ref. (10).-

Purification methods of uranium can be controlled by spectrographic analysis.

However, the best way to follow the fate of an impurity on the various purification steps, seems to be by tagging the element that constitutes the impurity and check its presence by the various ways of detecting and measuring radioactivity.

On the process studied at IEA to purify uranium,samples of this element were tagged with radioisotopes of various possible impurities, such as rare earths elements, thorium, sodium, strontium, phosphorus, copper, bismuth, lead, zirconium, hafnium, and the purification procedures carried out. Monitoring of the various apparatus where purification were being carried on gave qualitative indication of the behaviour of the impurity. Samples taken at the various points of the equipment allowed quantitative analysis by gamma-ray spectrometry or Geiger counting, making the determination of decontamination factors very easy. Also, radioautography of samples of ion-exchangers used on the process indicated the distribution of the impurities along the columns.

VI.4 Activation Analysis of Sodium in the Water of the Pool - Ref. (11) .

In order to verify what amount of sodium ion would remain in the demineralized water, 250 milliliters samples of water were taken, evaporated to dryness and the residue irradiated in thermal neutron fluxes of 10^{11} to 10^{12} neutrons/seg. cm^2, for irradiation periods in between one to four hours. Standards samples of sodium were also irradiated at the same geometry and for the same lenght of time.

After irradiation standards and water residue were removed to counting vials and counted by gamma-ray spectrometry. Amounts around 0.05 parts per million of sodium could be measured.

VI-5. Activation Analysis of Hafnium in Zirconium Ref. (12).

In order to check purity of samples of zirconyl chloride for their hafnium content, those samples were irradiated in the same conditions as for the ones described for sodium. Standards of hafnium oxide were also irradiated for the same lenght of time and at same geometry. Standards and samples were counted, without previous chemical separation, by scanning in gamma spectrometer and comparing the areas for hafnium-181 photopeak at 0,133 Mev. **Complete** analysis could be made in from two to four hours, depending on the irradiation time. Samples of zirconium with a hafnium content from 0,1 up to 5% were easily analised.

VI-6. Activation Analysis of Sodium in Graphyte Ref.(12)

Possible utilization of national graphyte as moderator for nuclear reactors required the determination of their purity grade.

Besides of boron determination (14), activation analysis of sodium were also carried out with the same general technique as the one for sodium analysis in pool water. Amounts of sodium corresponding to about 35 parts per million could be analysed in about two to four hours and using 0,1 grams sample of graphyte, with a reproducibility of 3%.

VI- 7. Simultaneous Activation Analysis of Gold and Uranium in Mineral Ref.(13)

Minerals of gold from operating brazilian gold mines were analysed simultaneous for uranium and gold. Uranium was analysed by the amount of neptunium-239 formed by irradiation,and gold by the isotope gold-198. The general technique of standards irradiation was also followed in this case. Radiochemical separations were required in this case since various activities were induced in the ore samples. Gold-198 and neptunium-239 were scanned in separate fractions and their amounts calculated by comparing areas in the corresponding photopeaks with the ones obtained with standards. Precision of the analysis is from 1 to 10% expressed as standard deviation, for ore samples containing 20 parts per million of gold and 0.1 % of U_3O_8.

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