ENEAR&DPROGRAMMEON ENHANCED SEPARATION OF LONG LIVED RADIONUCLIDES FROM HLW IN VIEW OF THEIR SPECIFIC MANAGEMENT.

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1. Background.

In Italy, **a general public debate took place on the implications of the use of nuclear** energy after the Chernobyl accident (April 1986).

The debate culminated in a *referendum* vote on nuclear matter in November 1987: this vote was formally limited to **specific aspects of nuclear legislation, in force at that time, but was finally interpreted as a will to close all the existing nuclear plants.**

In 1988 a law was approved to close the 160-MWe gas-cooled reactor at Latina and to halt both the construction of the two boiling water reactors at Montalto di Castro (which **were 70 % completed) and the two new pressurized water reactors at the Trino site (on which construction was just starting).**

The Government, with Parliamentary approval, placed a 5-year *moratorium* on any new nuclear plant construction.

In June 1990, the Italian Parliament decided the final closure of the nuclear power plants with operating licenses but actually shut down (the 260-MWe pressurized water reactor at Trino and the 860-MWe boiling water reactor at Caorso).

A full revision of the National Energy Plan was made and it has been submitted by Government to Parliament. This plan calls for a re-orientation of Italian research in the nuclear fission field towards the exploration of new technical solutions which could allow the use of nuclear power in the future.

2. The 1990-1994 ENEA R&D Programme on Nuclear Fission Field.

On July 1990, following the guidelines of the aforementioned new National Energy Plan, The Board of Ministry for National Economy Plan (CIPE) approved the 1990-1994 Five Year Plan of ENEA (Italian Commission for R&D on Nuclear and

Alternative Energy Sources).

In this ENEA five year plan a new approach to safety is considered of major importance for the future of nuclear energy in Italy: the main objective is to improve safety and also tocontribute tothecomprehension of the nuclear problems both by politicians and by the general public.

The main objectives of the new ENEA R&D Programme, in the nuclear fission field , are the following :

- To develop a significant quality advance in reactor safety.
- To develop a significant quality advance in radwaste management and disposal safety.

As far as reactor safety is concerned (although the best existing nuclear technology is adequately safe) further improvements might still be possible by recourse to enhanced inherent and/or passive safety features which can also help in re-acquiring the acceptance from the general public. A major safety goal is to limit the environmental impact and the off-site radiological consequences in any accidental event in such a way that no specific evacuation plan shall be needed nor any significant long term land contamination could occur.

As far as radwaste management is concerned it is clear that there is a worldwide scientific consensus on the present management strategy (mainly based on geological storage of vitrified high level wastes) and there is no scientific or technical reason to believe that a satisfactory geologic repository cannot be built.

It is also true, however, that the implementation of such geologic repositories has been nowhere brought beyond a preliminary *in situ* laboratory stage and that any forecast about the date for the availability of any full scale final repository is at present unreliable, due **to** the **nearly unanimous refusal by the public to accept this concept as** a definitive solution for high level waste management.

In this scenario new studies on advanced HLW management schemes can be carried out to test the real possibilities of having an improved long term safety.

The ENEA R&D activities are briefly summarized in the following paragraphs of this presentation.

3. Guidelines of ENEA new R&D programme on radwaste management.

ENEA has developed in the past many R&D activities in the field of the back end of nuclear fuel cycle {reprocessing, radwaste treatment and conditioning): a large budget and a significant number of people were involved as well as experimental facilities such as the EUREX (Saluggia) and ITREC (Trisaia) hot reprocessing pilot plants and the Plutonium MOX fuel fabrication experimental facility (Casaccia).

Now, according to the political decisions, in the new 1990-1994 ENEA Five Year Plan, the human and financial resources have been drastically reduced and the mentioned facilities must be closed and decommissioned. A special Task Force has been set up for the decommissioning of these facilities and for the conditioning of the radioactive wastes stored there.

On the other hand, in the frame of the new safety approach of Italian research in the nuclear fission field, the importance of a convincing back end fuel cycle cannot be underestimated, in particular to re-assure public opinion.

Therefore, while many years, if not decades (considering the present Italian scenario), separate reactor operation from back end fuel cycle improvements, the latter should be addressed as long term research and development activities, better if in the framework of international cooperation.

In this connection, the major safety goals of such R&D Programme are the following:

- Re-examination of the reprocessing and waste management processes by considering the separation between high level waste (mainly fission products) and very long lived radionuclides (mainly actinides).
- Re-location of the high level waste in the environment, confined appropriately, so that the specific activity becomes, after a few centuries, less than that of natural minerals;
- Specific management of the isolated long lived radionuclides.

These objectives require in particular the development of techniques for enhanced separation of actinides (as well as some long lived fission products such as technetium) from fission products, leading to high level waste which is practically alpha-free and, in parallel, the study and evaluation of concepts for the specific management of the isolated Ions lived radionuclides, in particular by nuclear transmutation.

A small group of ENEA scientists has, therefore, begun to study these topics trying to

obtain the most from their previous expertise in radiochemistry, process chemistry and applied nuclear physics.

It is important to point out that this R&D Programme should be considered, in the present Italian scenario, in its appropriate perspective of long term fundamental research, and not aimed at supplying a short term available alternative to the current management strategy.

4. Previous experiences on separation of long lived radionuclides.

During 1986-1989 at ENEA, chemical processes were developed for the separation of long lived radionuclides from existing nuclear HLW stored at the pilot plants.

The liquid HLW (according to the Italian Regulatory Commission) stored in Italy are the following.

Reprocessina wastes: some 120 m^3 of aged (10-20 years) first cycle aqueous raffinates, coming from the experimental reprocessing campaigns carried out in the past, at the ENEA pilot facilities EUREX and ITREC, mainly with MTR-type fuels (some 85 m^3), with CANDU-type (some 25 m^3) and with ELK-RIVER thorium fuels (some 10 m^3).

The simplified composition of the MCE (MTR+CANDU+ELK RIVER) solution is shown in Table 1.

Transuranic Elements Wastes (TRU wastes): some 10 m^3 of aqueous solutions coming from the Casaccia Plutonium experimental fuel fabrication facility (based on oxalate precipitation as well as “sol gel”), including some 2 m^3 of relatively high plutonium content solutions, whose chemical composition is shown in Table 2.

Treatment of the MCE reprocessing waste.

The selective separation of actinides and long lived fission products (mainly strontium and cesium) from the liquid waste has been considered with the purpose of vitrifying only this radioactive fraction, keeping all the inert chemicals in the “declassified” liquid waste which, therefore, can be solidified by cementation.

In this way, the volume of vitrified product is drastically reduced: according to our evaluations, only some 1.5 m^3 of active glass would be produced instead of some 15 m^3 required by the direct vitrification.

In this connection, a “declassification” (selective separation) process has been developed and tested to separate the radioactive fraction (consisting essentially of CS-137, Sr-90 and transuranic actinides), from the liquid waste (containing a large amount of inert salts), see Figure 1 .

An extensive lab-scale experimental activity has been made in order to select the proper separation process.

The “alkaline flow sheet” (Figure 2) has been finally selected, with zeolite for the selective separation of cesium.

The results obtained in lab-scale with simulated solutions have been confirmed by subsequent experiments carried out with real HLW samples in the EUREX plant “29-1” hot cell, as well as by engineering scale tests carried out with simulated HLW in the SERSE (an Italian acronym which stands for “treatment of liquid wastes by selective separation”) cold pilot facility .

From both experiments it can be deduced that a very high Cs, Sr and actinides decontamination factors (DF) can be obtained.

The “reverse strike” alkalinization of the HLW solution (the acidic waste solution is added to the necessary amount of 10 M NaOH, and not *vice versa)* gave the best results in terms of volume reduction and physical properties of the precipitate.

The separation of the precipitate from the alkaline supernatant by centrifugation proved to be a successful step if sufficient residence time was allowed to the suspension (2 -3 minutes).

The concentrated slurry coming from the centrifugation, after a proper optimization of the solid ejecting system, entertained very small volume of supernatant, so that the subsequent washing of the precipitate can be avoided.

The zeolite columns performed very well, both from the chemical and the hydrodynamic point of view, and the data obtained in lab scale tests (Cs loading capacity, kinetics, temperature effect) were fully confirmed in the engineering scale tests.

Very good results have been also obtained using the sodium tetraphenylborate (NaTPB) instead of zeolites for the separation of Cs: however, the presence of this organic compound in the highly active fraction could interfere negatively in the subsequent vitrification step.

In any case, the high Sr, Cs and TRU elements overall decontamination factors obtained in all experiments :

DF (Sr)	>100
DF (Cs)	> 1000
DF (Actinides)	> 1000

can ensure a proper 'declassification" of the original HLW, by enhanced separation of the long lived radioisotopes (essentially actinides, cesium-137 and strontium-90).

Treatment of TRU wastes.

For the management of the liquid alpha bearing wastes stored at Casaccia Plutonium facility, a selective separation process of all alpha actinides through solvent extraction is used, so as to bring the quantity of radioactivity therein under the threshold of 370 Bq per gram, in order to be able to handle them as "non alpha" wastes.

The process, named TESEO (an Italian acronym which stands for "treatment of discharged effluents by extraction with organophosphorous compounds"), utilizes as actinides selective extracting agent a bidentate organophosphorous compound (the octyl phenyl N,N diisobutyl carbamoyl methyl phosphine oxide, CMPO) .

This process has been developed both with batch scale laboratory tests and with continuous countercurrent experiments using real alpha bearing wastes with the following main objectives:

To separate simultaneously the actinides (mainly U, Pu, Am) from liquid waste of different chemical composition, with a single extraction procedure, reducing the alpha activity in the aqueous raffinate to 370 Bq/g.

- To recover the actinides in a suitable form to allow their reuse.

To achieve the highest possible volume reduction of the original TRU wastes, and to minimize the production of secondary wastes.

The reference solvent for this purpose has the following composition: 0.25 M CMPO + 1 M TBP in tetrachloroethylene or in n-dodecane.

According to a typical behaviour of the extraction procedure, the residual alpha activity in a typical liquid TRU waste coming from the Casaccia Plutonium Plant, after only few contacts (4-6) with the reference solvent, is of about 0.01 % with respect to the initial value (99,99 % extracted by the solvent). A higher separation of actinides can be obtained by increasing the number of extraction stages.

The reference TESEO process flow sheet is shown in Figure 3.

Results of continuous countercurrent tests with lab scale mixer settlers using this flowsheet have shown that :

- The alpha activity in the liquid wastes is reduced below to the analytical detectability with the usual alpha counting technique (16 extraction stages).
- The Am selective stripping section worked well increasing the back extraction stages to 8 (from the original 4).
- The recovered plutonium is concentrated by a factor of 25-30 with respect to its original concentration in the liquid waste, and further optimization of the flowsheet may reach higher concentration factors.
- The organic solvent at the end of the cycle is essentially free of impurities and of alpha contamination.

The real scale facility for the treatment of all the alpha contaminated wastes at present stored at Casaccia (some 10 m^3) has been designed and is now under commissioning tests; hot operation will start at the end of 1990.

These research activities have been performed in the frame of the 1985-1989 programme of the European Community “Management and Storage of Radioactive Wastes” - Shared Cost Actions.

5. The 1990-1994 programme on actinide and long lived FP partitioning.

The purpose of this programme for the short-middle term (1990-1994) is a comparative evaluation of chemical processes for the selective separation of long lived radionuclides from HLW, in view of the setting-up of an optimized partitioning flow sheet. It will be developed according to the following main phases.

Phase 1: “Review of the present state of the art on separation processes for actinides and long lived fission products”.

After the definition of the principal reference criteria (e. g.: reference HLW chemical and radiochemical composition, reference separation factor to be achieved for each radionuclide), a careful examination of the current literature on the separation techniques of actinides and fission products will be performed, with the purpose of collecting all the available data and in order to allow, after their evaluation, the selection of the more promising processes.

The long lived radionuclides to be considered for their pushed separation from HLW are: Np-237, Pu, Am-241, Am-243, Cm-244, Tc-99, Cs-137, Sr-90.

The reference separation processes are (in order of priority):

- Separation of actinides and lanthanides from HLW.
- Separation of actinides from lanthanides.
- Separation of Pu from other minor actinides.
- Separation of Tc, Sr, Cs from HLW.

The reference separation techniques are:

- Solvent extraction.
- Extraction chromatography.
- Sorption (mainly by inorganic compounds).
- Ion exchange.
- Selective precipitation.

The reference separation agents to be investigated are:

- Organo phosphorous compounds (mainly bidentate).
- Organo nitrogen compounds.
- Organo phosphorous-nitrogen compounds.
- Macrocyclic derivatives.
- Inorganic sorbers or exchangers.
- Soft donor ligands.

A preliminary list of compounds of interest in this connection is presented in Table 3.

Phase 2 : "Comparative evaluation (lab-scale) and modelling of the selected processes, and definition of partitioning flow sheets".

On the basis of the data collected in the previous Phase 1, a selection will be made of the more promising processes. For this selection, in addition to the intrinsic scientific value, other important parametres will be taken into account, such as, for example, the degree of maturity, the nuclear and/or conventional risks associated to the separation technique, the availability and cost of the separation agent and its stability.

An assessment of the selected processes will be performed in laboratory scale under the same experimental conditions to allow their comparative evaluation. In this laboratory scale step, additional thermodynamic data (essentially distribution coefficients and separation factors as a function of the most important parameters, with reference to the standard chemical and radiochemical composition of reference HLW)

will be collected to obtain further information not available from the literature for the process under investigation.
On the basis of the whole data collected (literature plus lab scale tests), the modelling of each selected process will be performed, in order to be able to set up a detailed chemical flow sheet for the verification of the process in more representative conditions (continuous tests with true HLW in minipilot hot facility).
The work under Phase 1 and Phase 2 will be partially funded by CEC in the framework of the 1990-1994 Shared Cost Action Programme on Management and Disposal of Radioactive Waste. It should be completed within the end of 1992.

Phase 3: "performance assessment of the selected process/es by continuous testings in minipilot hot facility with true HLW.
The chemical flow sheets set up at the end of the previuos phase will be tested in more representative conditions with true HLW to obtain more significant data on the validity and reliability of the process under investigation.
At present, the PETRA facility of CEC JRC-Ispra has been identified as the hot facility in which these investigation will be carried out. A special agreement cooperation between ENEA and JRC-Ispra, at present under evaluation, should allow the availability of that hot facility.
Phase 3 will cover the years 1993-1995.
At the end of this five year programme, the feasibility of the enhanced separation of actinides and some long lived fission products from HLW should be well understood.

6. Possible topics for the actinide transmutation studies.

In the past years (1 974-1982) ENEA developed a significant amount of research activities concerning the burning of actinides in reactors.
In particular, sensitivity methods were used to evaluate the errors in the actinide concentration due to the fluxes and cross section errors.
Another research field was the static analysis of safety.
The influence on the reactor parameters (e.g. : control rod worth, reactivity coefficients) of the actinides burning was also studied.
Most of there results, and the related references, are available in the IAEA Technical

Report Series N“ 214 “Evaluation of Actinide Partitioning and Transmutation”, under Appendix B, by L. Tondinelli (pages 55-61 of the Report).

After 1982, these studies were discontinued, and the R&D activities addressed mainly to the following items:

- Nuclear criticality safety analysis of fuel cycle plants.
- Shielding studies of fuel cycle facilities.
- Monte Carlo evaluation of nuclear reactor parameters (Keff, fluxes).

The use of calculation codes and cross section libraries (such as KENO IV, KSDRNPM, MCNP) was validated against several experimental benchmarks.

At present, the new 1990/1994 ENEA Programme on actinide transmutation is still under definition. Possible topics of this future programme could be:

- To use the existing technologies, with some improvements, to burn the actinides in thermal reactors, using fuel elements derived from MOX technology.
- To study machines especially designed for burning actinides.

As far as the actinide burning in thermal reactors is concerned, there are three possible options:

- Homogeneous core.
- Heterogeneous core:
  - special pins in the fuel elements;
  - special fuel elements.

(The options may become six if MOX fuel elements are considered.)

These options should be compared considering the following aspects:

- Reactor safety.
- Fuel fabrication dose.
- Overall liquid waste volume.
- “ cost.

The calculation codes and cross section libraries to be used in this connection are the following.

Calculation codes:

BURNY-BEVE, CITATION 2-3 VP2, ORIGENS, KENO IV, MCNP, XSDRNPM, MERCURE IV.

Cross section libraries:

ENEA 219 Group cross section derived from ENDF/B-IV.

ENEA 219 Group cross section derived from JEF-1.

BMCCS1.

Point cross section based on JEF-1.

As far as actinide burning in special burner reactors, reference will be made to the OMEGA Project (Japan) and to the Integral Fast Reactor (ANL, USA) concepts.

The ENEA contribution, to this research, could be related to the following aspects:

- Evaluation of the errors in the actinide concentration due to the errors in the fluxes, cross section etc.
- Taking into account the allowed errors in actinide concentration, determination of the maximum possible errors in fluxes, cross section, etc.
- Analysis of the burner reactors safety calculations:
  - Control rod worth.
  - Reactivity coefficient (void, Doppler).
  - Peak coefficients.
  - Reactor safety against a loss of coolant accident (re-criticality due to core melting).

In this connection, a sensitivity code to be chosen, together with the ORIGENS, CITATION 2-3 VP2 and KENO IV codes, and the already mentioned ENEA 219 Group cross section libraries will be used.

LIST OF THE MOST RECENT REFERENCES OF ENEA WORKS.

1. M. Casarci, G. M. Gasparini, G. Grossi, "Actinide recovery from radioactive liquid wastes produced by ENEA experimental fabrication and reprocessing plants by CMPO", Actinide-89, Tashkent, USSR, Proceedings of the International Conference.

2. A. Marrocchelli, L. Pietrelli, "Cesium adsorption with zeolites from nuclear high salt content alkaline wastes", Solvent Extraction and Ion Exchange, 7(1),1 59-172, (1 989).

3. P. Venditti, G. Grossi, "The Italian R&D activities in the field of treatment and conditioning of third category (high level) liquid radioactive wastes", Joint International Waste Management Conference, 1989, Kyoto, Japan.

4. L. Pietrelli, G. Grossi, G. Torri, A. Donato, "Process selection study for the decontamination of liquid HLW produced by EUREX pilot plant", International Conference SPECTRUM 86, Niagara Falls, N. Y..

5. L. Pietrelli, G. Grossi, F. Troiani, "Selective separation of actinides and long lived fission products from liquid waste produced by EUREX plant", IAEA/CEC Proceedings of International Conference on Management of Radioactive Waste, Stockholm, 1988.

6. M. Casarci, G. Grossi, G. M. Gasparini, G. Valeriani, "Separation and recovery of TRU elements from liquid wastes produced by the Casaccia Plutonium Plant, *ibidem*.

7. M. Casarci, G. M. Gasparini, G. Grossi, L. Pietrelli, "Actinide recovery from radioactive liquid wastes by CMPO, Journal of Less Common Metals, 149, (1989), 297-303.

8. L. Pietrelli, A. Salluzzo, F. Troiani, "Sorption of Europium and Actinides by means of CMPO loaded in silica", Journal of Radioanalytical and Nuclear Chemistry, 141 (1990), 107-115.

9. L. Tondinelli, "Transmutation or geologic disposal of actinides", IAEA Technical Report Series N" 214, p.55-61.

10. Various Italian Authors, 1974-1982 literature on Actinide Transmutation, in the reference list of previous Reference 9.

Table 1

Simplified chemical composition of the MCE(*) solution.

Al	20.4	g/l
H^+	1.34	M
Hg	0.97	g/l
Fe	0.57	g/1
u	5 "t	mg / l
Cs	20	mg/l
Sr	8	mg / 1
Pu	3	mg / l
NO_3^-	3.6	M
Activity	1.3 E11.1. (3.5	Bq/l cm.)
Total volume	120	m^3

(*) MCE: (MTR+CANDU+ELK RIVER): mixture of the HLW produced by different reprocessing campaigns at EUREX and ITREC pilot plants.

‘1’able! 2

Chemical composit ion o f “high plutonium content” waste solutions proci~.iced by the Casacci a Plutoni um Plant . Total volume ~ 2 m^3.

Acidi c Waste.		Oxalic waste.	
HNO_3	: 3-7 M	HNO_3	: 1 M
Pu	: 244 g	Pu	: 50 g
u	: 43 g	u	: 9 g
volume	: 78%	Oxalic acid	: 0.6 M
		Volume	: 12 %

Alkaline waste		Analyt ical waste	
NH_4OH	: 11 M	Pu	: 33 g
NH_4NO_3	: 0.5 M	u	: 1830 g
THFA (*)	: 0.5 M	Acids	: (a)
Pu	: 14 g	Other Chemicals	: (b)
Volume	: 2 %	Volume	: 8 %

(*)tetrahydrofu rfurilic alcohol. .
(a) H_2SO_4 , H_3PO_4, HNO_3 .
(b) KSCN, $AgNO_3$, $Fe_2(SO_4)_3$, others .

TABLE 3

PRELIMINARY LIST OF SELECTIVE SEPARATION PROCESSES TO BE CONSIDERED FOR ACTINIDES AND LONG LIVED FP PARTITIONING.

SELECTIVE SEPARATION OF TRU ACTINIDES

- **selective separation agents**
  - *CMPO and homologues* (ANL,USA; ENEA Italy)
  - *DIDPA* (JAERI, Japan)
  - *DHDECMP* (LANL, USA)
  - *tetra aryl methylene di phosphine oxide* (All Union Inst.in.Mat.,URSS)
  - *bis m-(diaryl phosphinyl methyl) benzene* (Vernadski Inst., URSS)
  - *carbamoyl bidentate organophosphorous extractants* (idem)
  - *poly(diphenylphosphinylmethyl)arenes* (Vernadski Inst., U RSS)
  - *amines + heteropolyanions* (Vernadski Inst., URSS; CEA,France)
  - *D2EHPA + PODTPA or DOIDA* (Kurchatov Inst + All Union Inst., URSS)
  - *DIOMP* (Inst. Phys. Chem. Acad. Sci. URSS)
  - *subst. pyrocatechols* (Vernadski Inst., URSS)
  - *TRPO and similar* (China, CEC JRC-TUI)
  - *amides, diamides* (CEA, France; ENEA,Italy; China)
  - *phosphoramides* (Ins. Chem. Technol. URSS; CEA,France; ENEA, Italy)
  - *Reillex HPQ* (LANL, USA)
  - *Phosphinic resins* (Univ. Tennessee, USA)
  - *covalently immobilized organophospor. ligands* (Un. NewMexico,USA)
  - *inorganic and complex forming absorbers* (many)
  - *oxalates (OXAL process)* (CEC JRC-Ispra)

SELECTIVE SEPARATION OF TRIVALENT TRU ELEMENTS FROM TRIVALENT RE FISSION PRODUCTS

- **selective separation agents**

- *phosphino-pyridine N,P dioxides* (Univ. New Mexico-LANL, USA)
- *mixtures of pyrazolthiones (or pyrazolones) and organophosphorous (or substituted phenantrolines)* (LANL, USA)
- *mixtures of dithiophosphoric acids and tributylphosphate* (CEA, France)
- *mixtures of organophilic acid and pyridiltriazines* (CEA, France)
- *polydentate, alkyl substituted Schiff bases* (CNR Padova, Italy)
- *azamacrocyclic compounds with additional S donor groups* (Vernadski Inst., U RSS)
- *carboranes* (U RSS, Czechoslovakia)

SELECTIVE SEPARATION OF FISSION PRODUCTS

- **selective separation agents**

- *long chain chetones (Tc-99)* (Rockwell Hanford, USA)
- *quaternary ammonium compounds (Tc-99)* (many)
- *cobs// dicarbolyde (Cs, Sr)* (Khlopin Inst., U RSS)
- *crown ethers (Sr, Cs)* (many)
- *polyaza crown, cyclams (Sr)* (Brigham Univ.,USA)
- *calixarenes (Sr, Cs, Ln)* (Univ. Parma, Belfast, Mainz, Barcelona)
- *metal hexacyano ferroates (Cs)* (Univ. Loughborough, UK)
- *inorganic absorbers (Tc, Sr, Cs)* (many)

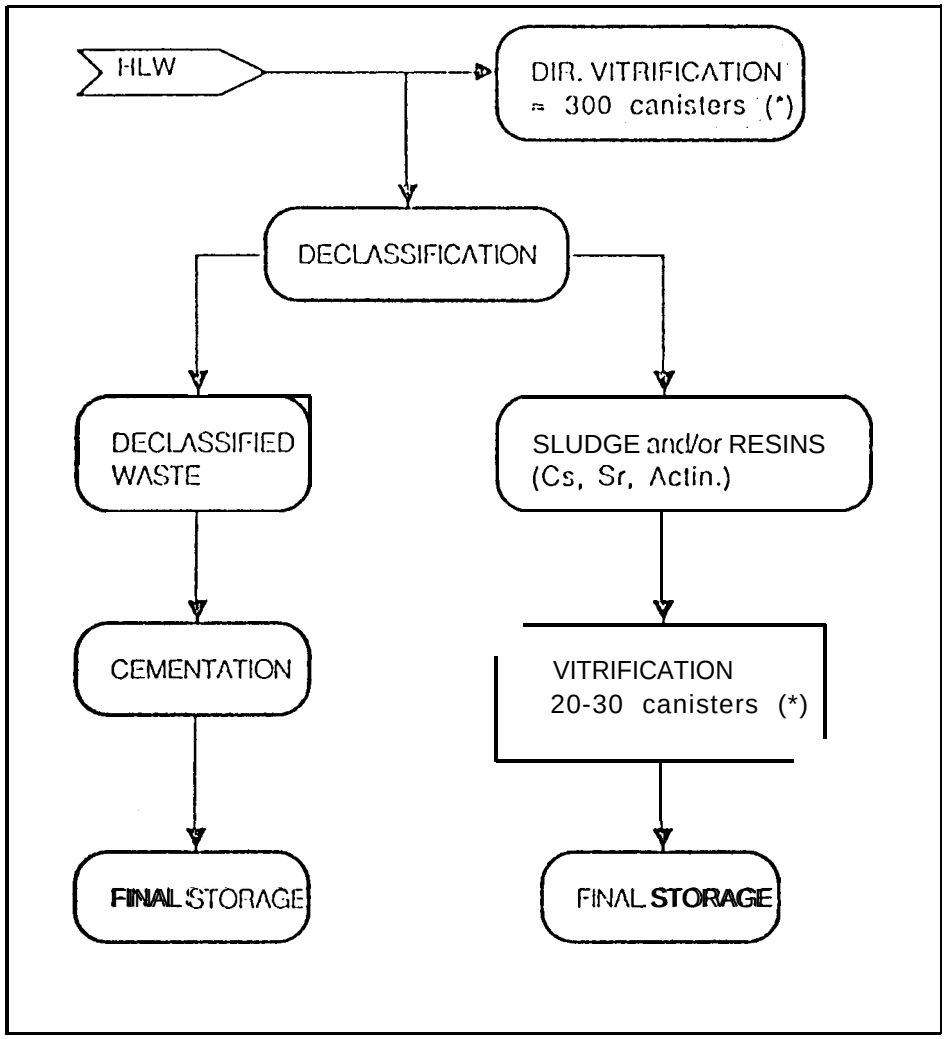

Fig. 1. Conceptual flowsheet for the management of "third category" liquid wastes at present stored at EUREX Plant.
(*) IVET canister (Volume 50 1).

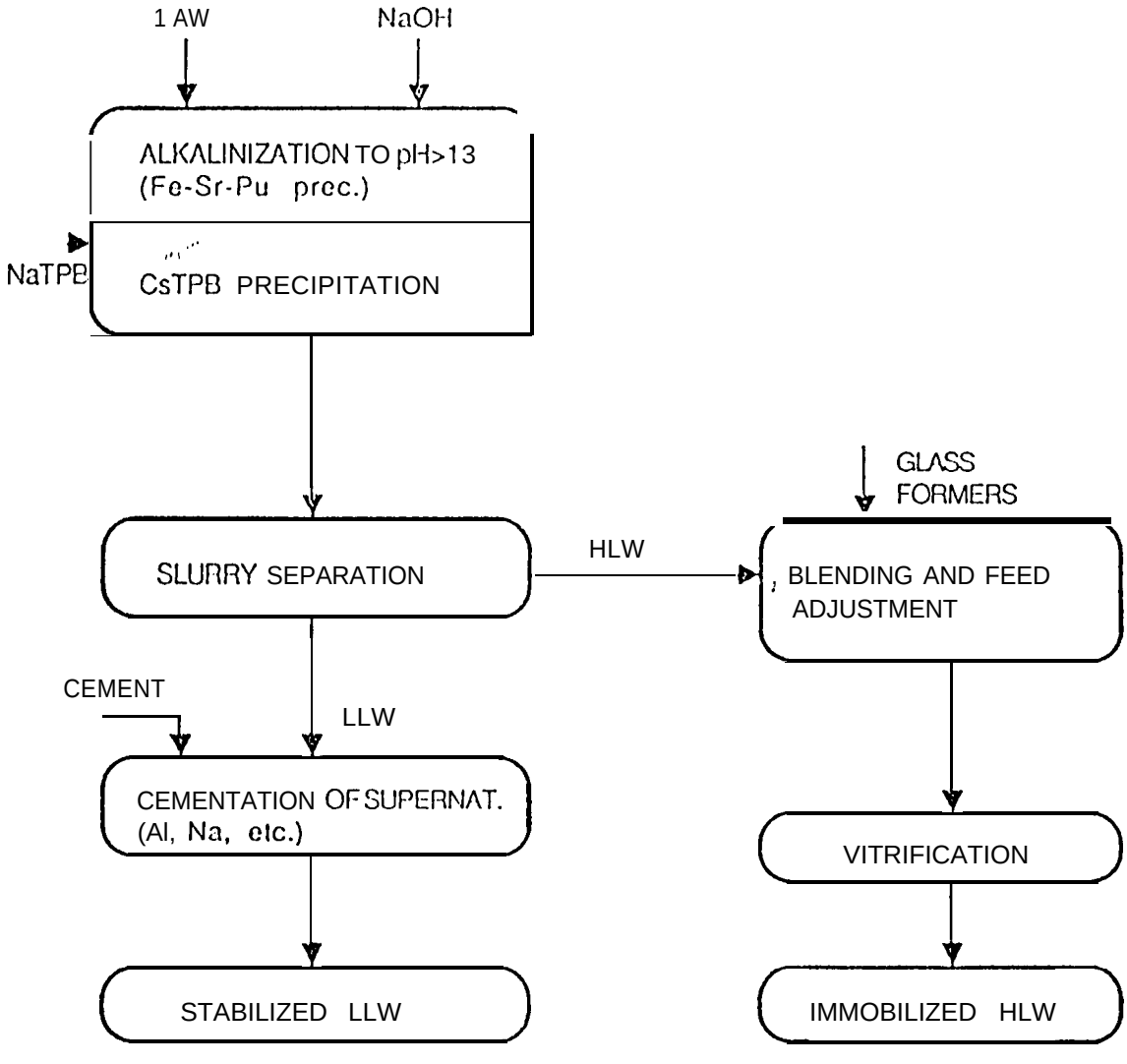

Fig. 2a -The "Alkaline" flow-sheet : Cesium precipitation by NaTPB

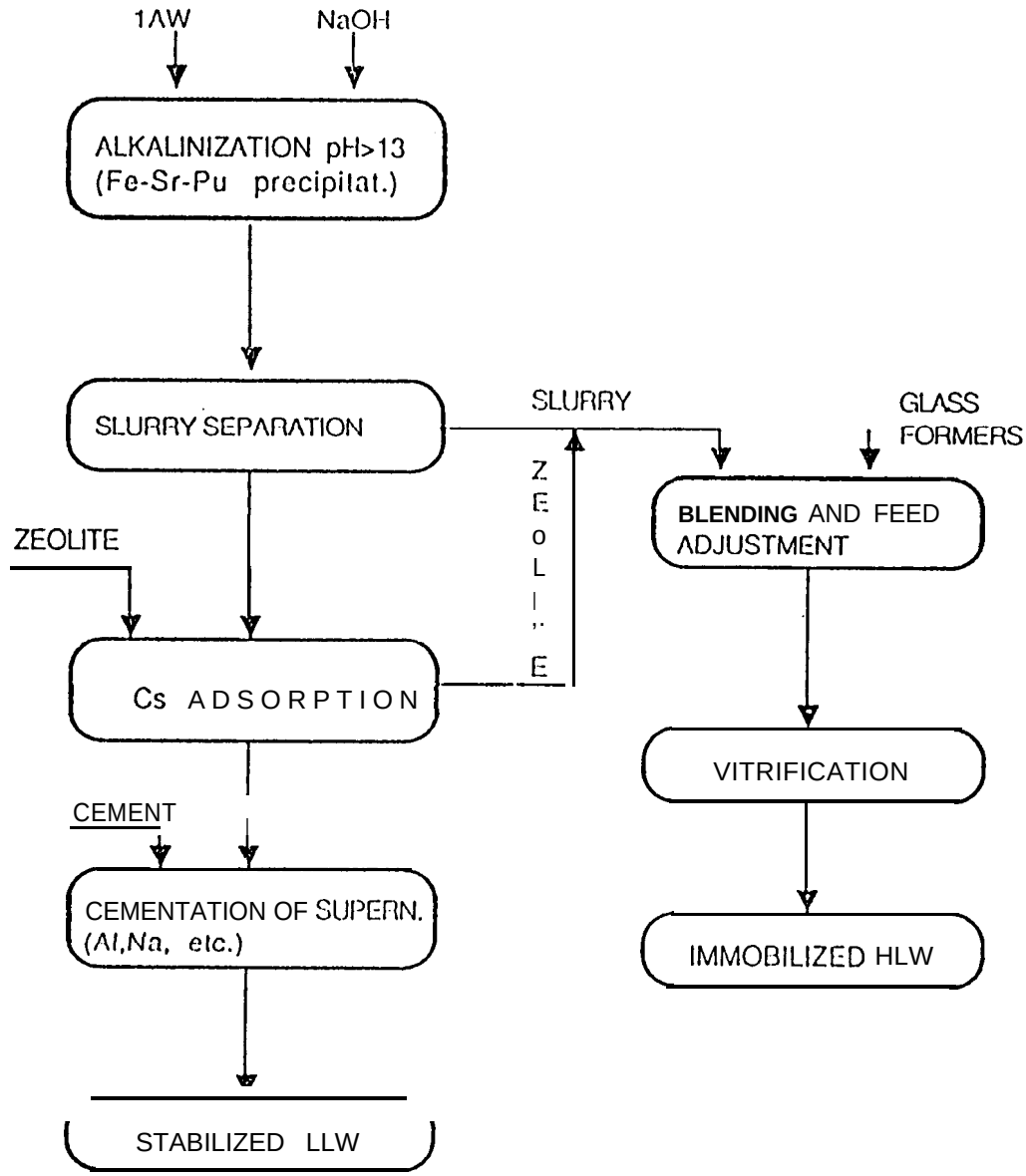

Fig.2b The "Alkaline" flow-sheet : Cesium adsorption by zeolites

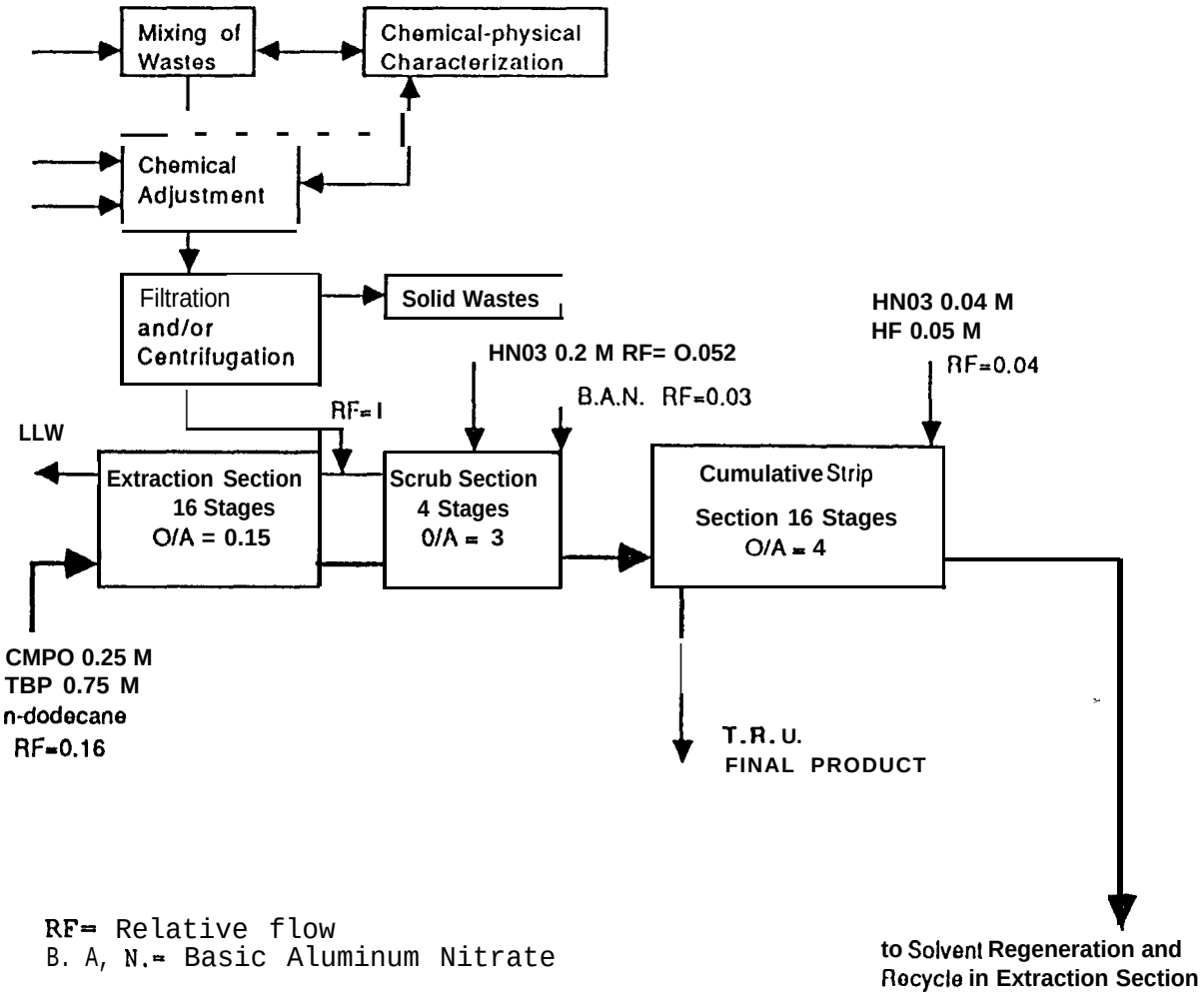

FIGURE 3 - The TESEO plant flow sheet.