STUDIECENTRUM VOOR KERNENERGIE

CENTRE D'ETUDE DE L'ENERGIE NUCLEAIRE

ASSOCIATION S.C.K./C.E.N. - BELGONUCLEA RE

ANNUAL PROGRESS REPORT 1973

GAS PURIFICATION PROJECT

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Samenvatting Een overzicht van de konceptuele en eksperimentele studies over vangst- en retentiesystemen voor nukleaire afgassen wordt gegeven. Sorptietechnieken voor jodium en krypton en de katalytische oxydatie van waterstof als simulant voor tritium worden onderzocht op laboratoriumschaal. Een proefopstelling van 25 m^3h^{-1} voor het uittesten van de verschillende eenheidsstappen van gasreiniging bij opwerking van LMFBR splijtstoffen werd opgebouwd. Als eerste faze in de studies werden de prestaties van wastechnieken voor I_2 verwijdering bepaald.

De verdere konditionering van de gassen en de toepassing van de kryodestillatietechnieken voor kryptonvangst werd voorbereid. Voor de afgassen van LWR reaktoren wordt een ^{133}Xe vervalbed bestudeerd.

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Résumé Le rapport présente un résumé des résultats des études expérimentales et conceptuelles de systèmes de purification de gaz d'usines de retraitement et de réacteurs. L'adsorption d'iode sur tamis moléculaires, l'adsorption à basse température de gaz nobles sur charbon actif et l'oxydation catalytique de tritium, simulé par de l'hydrogène ont été étudiées à l'échelle du laboratoire. Une installation pilote pouvant traiter 25 m^3h^{-1} de gaz a été construite. Les résultats expérimentaux de la première phase du programme d'étude, concernant l'arrêt de l'iode par lavage caustique des gaz sont présentés. Une extension ultérieure du circuit a été préparée qui comprend le conditionnement des gaz en vue de la capture du krypton par distillation cryogénique.

Sont également mentionnés les résultats expérimentaux de l'étude de lits de rétention de xénon.

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Summary Conceptual and experimental studies on LMFBR reprocessing and reactor off-gas purification systems are summarized. Iodine sorption on zeolites, low-temperature adsorption of noble gases on charcoal and catalytic oxidation of hydrogen, simulating tritium, are being studied in laboratory set-ups. A pilot loop with 25 m^3h^{-1} throughput has been constructed. Results are quoted from the first phase of the iodine removal programme by scrubbing systems. Further extension of the test loop, comprising off-gases conditioning prior to removal of krypton in a cryodistillation unit, has been prepared.

Delay-bed studies on ^{133}Xe extraction from LWR off gases are reported.

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1. INTRODUCTION

The requirements of volatile fission product containment increase with the progressing development of nuclear energy production.

Higher activities of tritium, iodine and noble gases are generated in the fuel due to increasing burn up and specific power, and, specifically for fast reactor fuels, as a result of the considerable shortening of the post-irradiation decay times which are projected. Concentration of multiple reactor units in energy parks and size enlargement of the reprocessing plants, on the other hand, will restrict the possibilities of release at the stack because of local atmospheric dilution limitations.

The development of the required gas purification technology is consequently of primary importance for minimizing the environmental impact of the nuclear energy expansion. The association S.C.K./C.E.N.-BN has therefore projected a long-range programme which aims at the development of high efficiency fission gas retention systems for off-gases of fast reactor fuel reprocessing plants and nuclear power stations.

The following objectives have been stipulated :

- the conception of the required gas purification system for the reprocessing of SNR fuel with cooling times of 150 days or more ;
- the demonstration of efficient removal of ^{3}H (DF > 10^{2}) ^{85}Kr (DF > 10^{2}) and ^{131}I (DF > 10^{6}) in a composite gas purification system for reprocessing of short-cooled fast reactor fuel ;
- the development of reliable processes for the retention and disposal of ^{133}Xe and ^{85}Kr at nuclear power stations.

2. TREATMENT OF OFF-GASES IN REPROCESSING FAST REACTOR FUELS

During the past year the main efforts have been devoted to the following items :

- conceptual studies directed toward the establishment of flow sheets of off-gas treatment for the reprocessing of SNR fuel ;
- the construction and preliminary operation of the test loop for iodine trapping methods ;
- adsorption studies with commercial iodine sorbents.

2.1. Conceptual studies [1] [2]

A conceptual study has allowed the definition of the problems of gas purification connected with the adaptation of new types of head-ends in the reprocessing cycle of short-cooled fast breeder fuel. These head-ends have as their main function the

complete release of the fission gases contained in the fuel, namely tritium, noble gases, iodine.

Two methods have been investigated for the disaggregation of the fuel ; by voloxidation and by molten salts. The processes considered being at the lab-scale, the proposals of the two schemes (Fig. 1 and 2) give only the nature of the gas to be handled. Some gas processes and their estimated performances, connected with the different steps of the two proposed schemes, are listed.

All this information should lead to a better choice of the type of head-end and a better understanding of the gas treatment systems to be studied.

On the other hand the study of capacity increase of a "thermal" fuel reprocessing plant and, consequently, the updating of the existing off-gas circuit, has led to a better view of the gas purification problems. Among these must be noted the denitration of the gaseous effluents (and its relation with efficiency of iodine and active aerosols removal) and the importance of the choice of the trapping technique on the waste volume produced.

2.2. Test loop for iodine trapping methods

The general purpose test loop "GASTON" with a throughput capacity of 25 m^3h^{-1}, has been erected [3] (Fig. 3). During the first phase of operation it will be used for testing the most promising methods of iodine removal on a semi-pilot scale. Units for trapping interfering impurities will be added gradually in order to constitute a complete off-gas treatment system which allows the application of low-temperature techniques for noble gas retention.

During the past year the construction of this installation, comprising the containment, ventilation, shielding, and waste evacuation provisions and the distribution manifold for carrier- and contaminant gases, has been completed. Reception and working tests of the wet section of the loop, comprising the dissolver mock-up, condensors, venturi ejector scrubber, brink demister and iodine generator have been performed [4] [5].

The set-up allows the simulation of the multicomponent gas mixtures, which are to be expected in gaseous effluents from a fast reactor fuel reprocessing plant. These mixtures can be fed into the chemical (Venturi ejector scrubber) and physical (Brink demister) separation units, using nitrogen carrier gas, at flow rates varying between 5 and 35 Nm^3h^{-1}.

Loading with iodine is performed at a nominal rate of 2.5 g $I_2 h^{-1}$: the iodine is generated in a scaled-up iodine generator developed earlier for the determination of break-through curves in a 1 $m^3 h^{-1}$ loop.

After check-up of the control and measuring equipment, a number of cold runs has been performed in order to define the operating characteristics of the different units of the loop [6].

2.2.1. Removal of iodine in aqueous systems

The first experimental campaign in GASTON was devoted to the study of the efficiency of the venturi scrubber (with a 10 % KOH scrub solution), of a primary impaction demister and of the brink demister as final element of I_2 removal from N_2-I_2 mixtures. The venturi scrubber was operated with liquid flow rates between 300 and 750 $\ell\ h^{-1}$, whereas the total gas flow varied between 7.2 and 34 $Nm^3 h^{-1}$. [7]

Depending upon the I_2 mass flow rate, typical results for the iodine trapping efficiency of the systems investigated are :

- for the venturi scrubber : > 97-99.5
- for the venturi scrubber+primary demister : > 99-99.5
- for the venturi scrubber+primary+brink demister : > 99.9

Quantitative evaluation of the results as a function of the operating parameters appeared to be difficult, due to liquid particle entrainment from the scrub solution in the gas stream. Nevertheless, it became clear that the rather high liquid-to-gas molar ratios (10^4 - 10^6), at which the venturi scrubber was operated, ensured almost constant and high I_2 trapping efficiencies even for extreme variation of scrub solution and I_2 mass flow rate (Fig. 4) operating conditions.

In a second phase of the operation of GASTON, for testing wet purification systems for I_2-trapping, the venturi scrubber was replaced by a gas atomizing aerosol generator. The latter produces diluted KOH liquid particles of 0.5 µm diameter at flow rates between 10 and 40 g h^{-1}. These particles were entrained with the main gas stream as to capture part of the gaseous iodine. For the same gas and I_2 flow rates as above, the concentration of the residual gaseous I_2 before the final brink demister, showed efficiencies of this system between 32 and 99.9 %, depending upon KOH molarity and solution flow rate. The results obtained with this aerosol system were analysed as a function of the molar I_2 concentration, KOH normality, aerosol concentration and contact time, yielding the plot shown by Fig. 5, which demonstrates that, even at stoichiometric KOH/I_2 conditions, 70 % of gaseous iodine is removed.

2.2.2. Extension of the test loop [8] [9]

In order to increase the experimental possibilities of the CASTON loop, a study of the extension of the wet section has been performed. Two goals were aimed at for a new scrub installation :

- behaviour of iodine in gaseous effluents with high content of nitrous vapours ;
- study of the trapping of I-compounds by aqueous scrubbing.

To determine the size of this scrub column, an evaluation of the composition of the main compounds of the gaseous waste from a reprocessing plant has been made. The corresponding inlet flow rates for the CASTON loop are the following :

nitrous vapours	: 8.5 m^3h^{-1}
O_2	: 5.3 m^3h^{-1}
N_2	:21.2 m^3h^{-1}
(I compounds)	trace
total flow	35 m^3h^{-1}

If an absorption efficiency of the nitrous vapours of 90 % is assumed (as in industrial conditions), the outlet flow is 25 m^3h^{-1}.

Design studies for two types of column (trays and packing) have shown the difficulty of putting this new wash installation in the existing hood. For the above absorption conditions, the kinetics of nitrous vapours removal are led by the oxidation of NO to NO_2 in gaseous phase which involves a long residence time of the gas in the column. In this case, the trays column is the best choice. However, the preliminary information obtained concerning the trapping mechanism of iodine by scrubbing (main reaction in liquid phase) and the fact that the packed column is better adapted to experimental work, have led to the choice of a packed column.

The available space in the existing hoods, together with a market research led to the following specification for this scrub column :

diameter	: 150 mm
packing height	: 1800 mm

An experimental programme has been written which considers the choice of this column and the information to be collected on gas purification by wet methods. This programme concerns both scrubbers (venturi and packed column) and includes four working conditions for the new scrubbers.

	Flow rate	Inlet composition	Scrub solution
Option 1	25 m^3h^{-1}	3.5 % NO_x	NaOH
Option 2	1.6 m^3h^{-1}	25 % NO_x	HNO_3 5 N
Option 3	25 m^3h^{-1}	5-10 % NO_x	HNO_3 5 N
Option 4	3.5 m^3h^{-1}	3.5 % NO_x	HNO_3 16 M (iodex process)

From these operational conditions, the different elements of the new scrub section have been analysed and specified (cooler, pump, waste, buffer tanks).

The connection to the GASTON loop has been studied and the process flow sheet, including the instrumentation, has been drawn.

The detailed design of this wet section has been started and the mounting of the column is scheduled in the first months of 1974.

2.3. Iodine adsorption studies

2.3.1. Preparatory work

In view of the study of the removal of ^{131}I labelled organic iodine compounds, a methyl iodide generator has been developed which is based on diffusion through a capillary tube connected to a temperature-controlled CH_3I vessel in which complete isotopic exchange has been achieved. Although this system gives good results during short operation times, it is necessary to control the operating conditions very strictly.

Iodine adsorption has been investigated [10] in a silver-coated copper tube (diameter 3 mm, length 1 m and 15 m. The removal efficiency was 99.99 % ; scanning measurements indicated that the distribution of ^{131}I was exponential.

The pressure drop characteristics of sorbent beds were determined [11] in a column of 3.4 mm diameter.

A literature review and evaluation study of sorbents for applications in humid NO_x atmospheres was made [12].

2.3.2. Adsorption isotherms for iodine on impregnated sorbents

Impregnated zeolites have been considered for high efficiency trapping of iodine and its compounds, in the presence of disturbing agents such as water vapour and nitrous oxides. The iodine capacity of copper, lead and silver zeolites was measured by trapping ^{131}I traced iodine on a shallow bed of sorbent in a well-type scintillation crystal. It was found that the capacity of these impregnated zeolites is smaller

than the capacity of the non-impregnated ones. Silver is the best impregnant, leading to a capacity of silver zeolites of 5 wt. % which was not influenced by a temperature change from 30 to 100°C, the latter temperature being of interest for obtaining very high iodine retention efficiencies.

Based on these promising results for silver zeolites, a parametric study was started on the silver impregnation of large samples of 500 g molecular sieves Union-Carbide type 13-X using the through-flow column investigated last year. The best results were obtained with a small excess (10 %) of a molar solution of silver nitrate giving a 92 % cation exchange efficiency for the impregnation process.

2.3.3. Dynamic adsorption of iodine on molecular sieves

Based on capacity measurements on various sorbents, Union Carbide 13-X molecular sieves were selected for parametric investigation of the dynamic behaviour of sorbents for trapping iodine from dry gases. From measurements of break-through curves performed in the 1 m^3h^{-1} loop, the following conclusions could be drawn :

- the residual sorbent capacity influences the kinetics according to a second order law, as was deduced earlier from static shallow bed measurements ;
- the sorbent capacity is independent of the inlet iodine concentration in the range 70 to 400 mg $I_2m^{-3}N_2$.
- regeneration at 200°C has no subsequent effect on the dynamical behaviour of the sorbent : after five adsorption-desorption cycles, each involving more than 200 h at 200°C, neither the capacity nor the shape of the break-through curve are significantly affected.

The kinetics of iodine adsorption on 13-X-sorbent follows a mathematical model which allows the prediction of the outlet iodine concentration as a function of gas flow rate and bed height. Fig. 6 shows the fitting of experimental data to the mathematical model.

The effect of humidity in the carrier gas is disastrous for the iodine retention on this type of molecular sieve : a bed saturated with iodine instantaneously releases the iodine as soon as water vapour is present in the carrier gas. Similarly, molecular sieves (such as Norton Zeolon 900 and CECA NK 20), adsorb only 0.5 wt. % when they have been previously saturated with humidity. Consequently, the sorption of iodine from a humid gas on these molecular sieves requires either predrying of the humid carrier gas or metallic impregnation of the molecular sieve.

With a UC 13-X sorbent, predrying and adsorption can possibly be combined in one column, the iodine captured on the sorbent is displaced by water without affecting the initial iodine decontamination factor as long as the iodine front does not reach the bed end.

The behaviour of organic iodine compounds under dynamic operating conditions remains to be determined.

2.4. Removal of tritium from dry head-end off-gases

If complete release of tritium from the fuel can be obtained in the dry head-end processes under development, the proposed treatment for removal and disposal of the nuclide consists of catalytic oxidation on noble metals or metal oxides and subsequent adsorption of tritiated water on molecular sieves.

The catalytic oxidation of tritium and in-situ retention of water on the substrate of the CuO catalyst is investigated in a test loop of 5 m^3h^{-1} where tritium is simulated by hydrogen at a maximum flow rate of 3 l h^{-1}. The first run indicated the necessity for strict control of the gas preheater and the catalytic bed and the need for discrete chromatographic gas analysis. A 5 l bed of Cat-Chem C54-8 catalyst working for 150 h at 160°C and using 5 m^3h^{-1} carrier gas, showed a hydrogen decontamination factor greater than 250.

2.5. Noble gases recovery

BELGONUCLEAIRE has updated the scheme and the working conditions of a pilot plant for the recovery of noble gases from gaseous wastes. Inquiry has been made into the composition of the gaseous effluents to be handled in reprocessing plants and light water power stations (PWR and BWR). The results of this inquiry have allowed the definition of the optimum service conditions of a pilot plant and a range of values in which the performances of the pilot must be investigated [13].

The inlet composition of the off-gas was :

N_2 : 80 %
O_2 : 20 %
Kr : 150 vpm
Xe : 1500 vpm

The performance required was 99 % recovery of Kr and Xe, with a concentration factor of 10^4 for the noble gases recovered.

2.5.1. *Adsorption of noble gases on charcoal*

The static adsorption capacity of activated carbons for Kr and Xe has been determined at different temperatures [14]. The adsorption isotherms of Kr on various activated carbons (RBL-3, Norit Extra, Merck Carbon and CECA NC 30) with specific surface areas ranging from 1300 to 2000 m^2g^{-1}, were found to follow Freundlich's law in the temperature range -198 to 25°C. The adsorption of Xe on the same carbons has been studied at room temperature (for delay bed operation) and at +78°C (for chromatographical separation from Kr). The Xe isotherms derived showed an uncertain

dependence on partial pressure, which can be approximated by a linear relationship. During adsorption tests of Xe and Kr in a loop constructed to perform dynamic tests on chromatographic separation of Xe and Kr at -76°C, it was found that Xe was absorbed in a rather small part of the bed whereas the Kr was spread over the whole bed.

An evaluation was made of a low temperature adsorption system for the removal of ^{85}Kr from reprocessing off-gases, [15]. It followed from the conceptual study that, compared to cryogenic distillation, this system is more complicated ; the former was consequently chosen for construction and study at the S.C.K./C.E.N.

2.5.2. Cryogenic distillation

Concerning the cryodistillation unit retained as a pilot plant (Fig. 7), the entire scheme, including instrumentation, has been reviewed.
The preliminary design of each equipment has begun, and two types of low temperature heat exchanger have been studied and compared: packed regenerators and plate-fin heat exchangers. The latter are nowadays widely used in large cryogenic gas separation plants and they offer several advantages :

- high performance ;
- no contamination of purified gas by the incoming stream ;
- simpler set-up (no valves for flow inversion).

However, this last advantage is lost when the incoming gas contains impurities such as H_2O, CO_2, etc. having relatively high condensation and freezing temperatures ; in this case, plate-fin exchangers must be operated in reversing mode.

In addition, their length must be sufficiently great to limit heat leakage by axial conduction, which leads, for small units to an awkward design. The regenerators therefore seem to be preferable for the pilot plant.

For the sizing and the choice of the absorption and distillation columns, some information has been collected and computation done. Theoretical methods for predictions of phase equilibria have been used for $Kr-Xe-O_2-N_2$ systems. The existing methods have not given better results than Raoult's law [16] [17].

The advantages and disadvantages of packed and tray-columns have been discussed for the pilot plant. A packed column has been chosen because it gives greater experimental possibilities than trays ; the height and type of packing can be changed. After a literature review and a market inquiry small metallic mesh rings are considered the best choice.

To speed up the installation of this pilot plant, a global offer for the cryodistillation unit has been written and sent to potential manufacturers.

This preliminary specification [18] gives the conceptual design, the characteristics of the effluent to be processed (see above, Inlet Composition of the Off-gas), the requirements concerning operation and the performances demanded.

3. REACTOR OFF-GAS [19]

The study of an alternative to the conventional decay tanks to process the off-gas from PWR has been pursued. The flow sheet of an installation based on delay beds has been defined (Fig. 8), and gas composition and flow rate have been estimated for a 1000 MWe PWE. The gas decontamination factor retained for the ^{133}Xe is 10^3.

Two types of operation of the delay line have been proposed and studied : delay beds working at atmospheric pressure in the first case, at 5 ata in the second one. The sizing and the preliminary design of equipment corresponding to these two working conditions have been carried out, namely buffer tanks, dryer unit and delay beds.

Increase of pressure allows an increase in the quantity of gaseous waste processed with the same gas facility, or a size reduction of the delay beds for the same flow rate. Nevertheless, the work at pressure requires an accurate regulation of the pressure and increases the risk of leakage. The final choice between these two working conditions will be a function of both safety and economical criteria. Thus the preliminary design of the two off-gas systems based on theoretical data has to be checked with the help of an experimental programme. For this purpose, a column of 15 cm diameter using various lengths of active charcoal has been built at S.C.K./C.E.N. Mol. The dynamic runs of adsorption will be started in the beginning of next year. In all cases the study has shown that the delay line allowing instantaneous handling of contaminated off-gas gives more reliability and safety than the decay tank system.

For the BWR, the study of the off-gas system has been started. Process flow sheets with different options have been established (Fig. 9). Each flow sheet includes the recombination of radiolytic H_2 and O_2 and a delay line for the ^{133}Xe. Further work will be devoted to the analysis of the options, the elaboration of an engineering flow sheet and the preliminary design of equipment.

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FIG. 1 REPROCESSING OF FAST BREEDER FUEL (SHORT COOLING TIME) OFF-GAS TREATMENT

Option : "Disaggregation by Voloxidation"

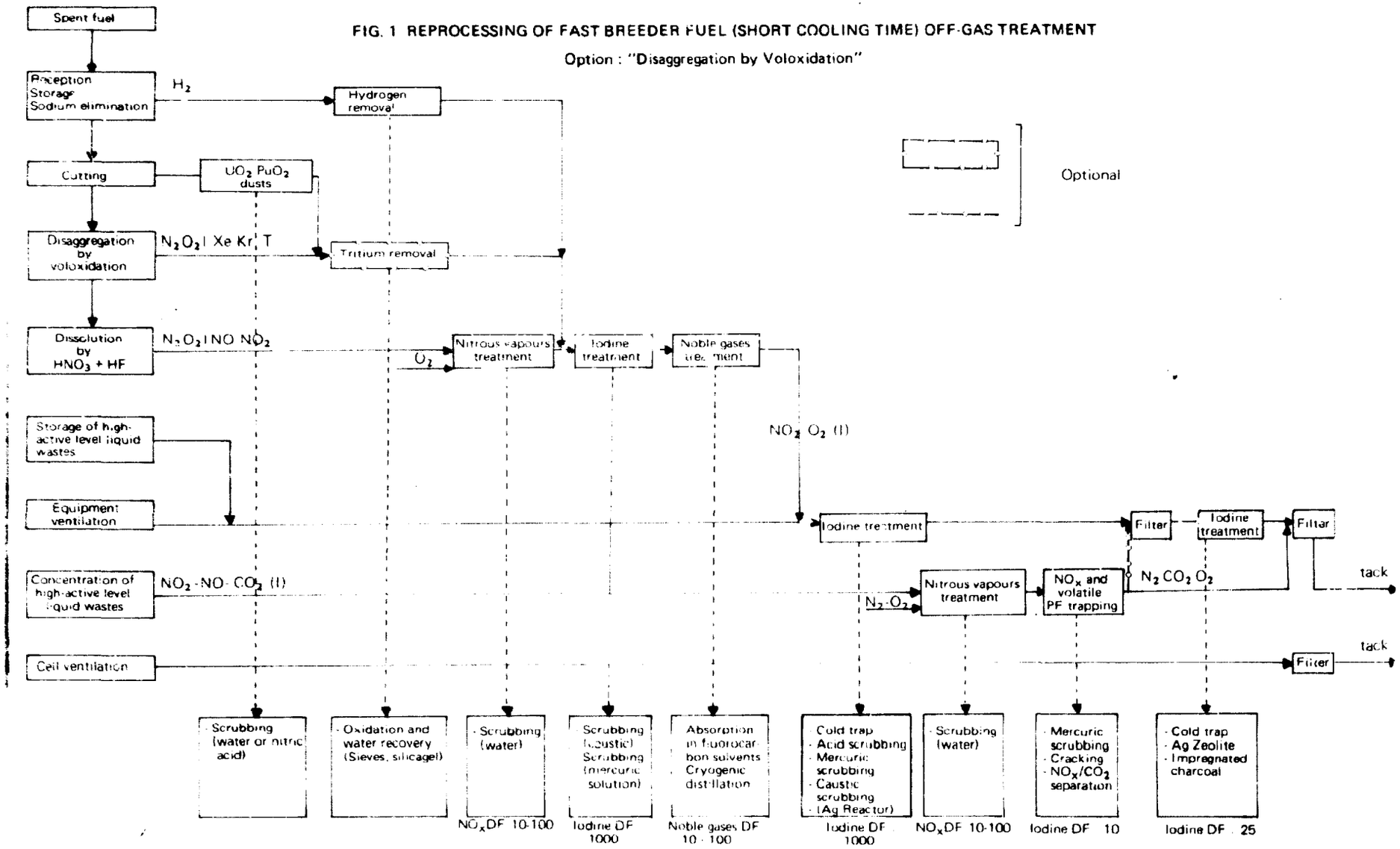


FIG. 2 REPROCESSING OF FAST BREEDER REACTOR FUEL (SHORT COOLING TIME) OFF-GAS TREATMENT

Option : "Disaggregation by molten salts"

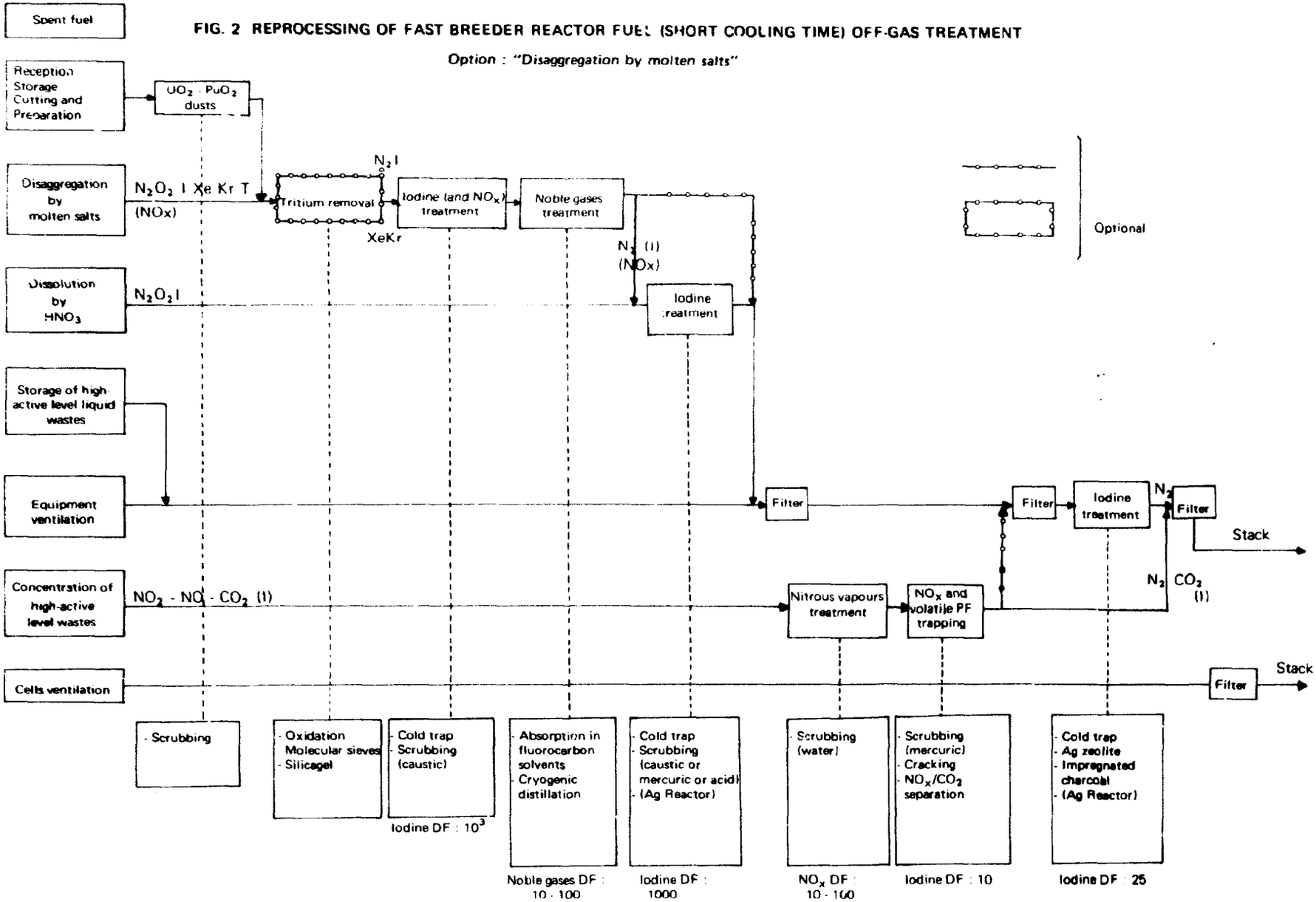
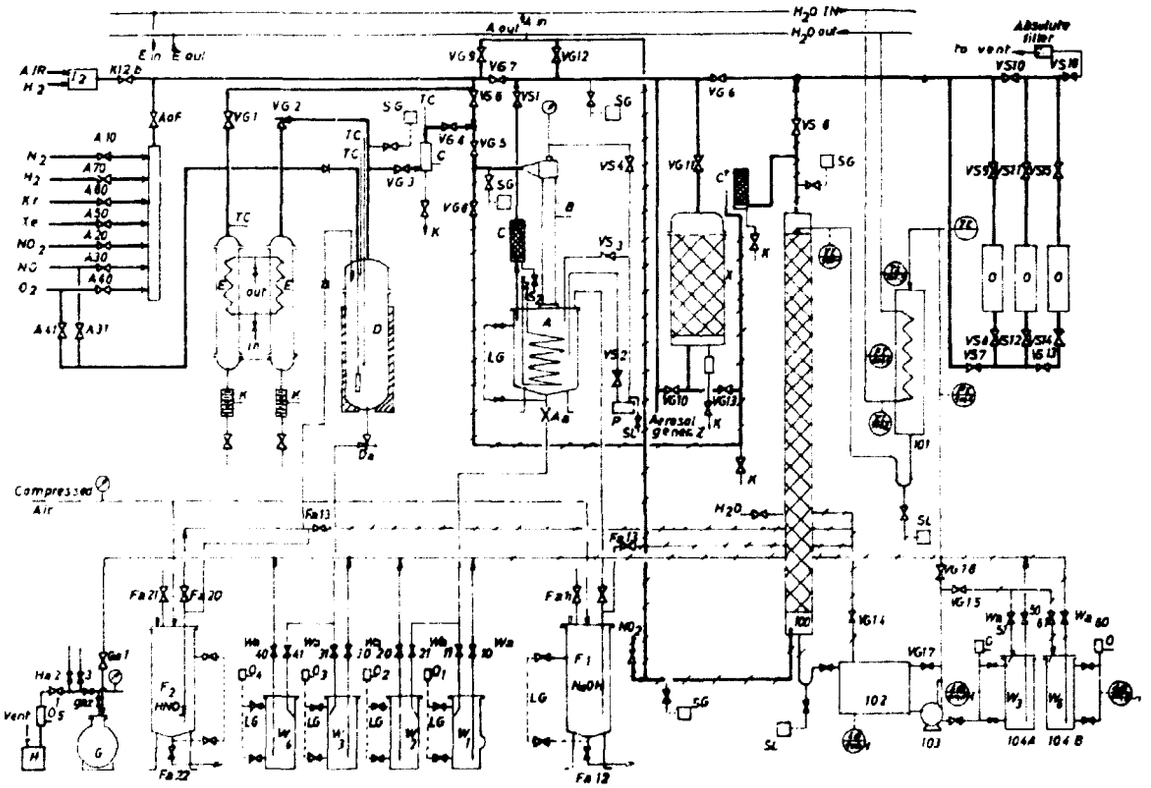


FIG 3 TEST LOOP FOR IODINE TRAPPING



A	Scrubbing liquid	100	Column
B	Venturi scrubber	101	Cooler
C	Demister	102	Recycle vessel
D	Dissolver	103	Pump
E	Condensors	104 A/B	Waste solution storage
F_1	Storage of basic solution		New connections
F_2	Storage of acid solution		New lines
G	Waste transport		Gas circuit
H	Vacuum group		Liquid circuit
I_2	Iodine generator		
K	Condensed liquid		
O	Charcoal filter		
P	Pump		
T.C.	Thermocouple	FI	: Flow indicator
W	Storage of waste solution	PI	: Pressure indicator
X	Brink demister	TI	: Temperature indicator
Y	Dry section	LG	: Level gauge
S_G	Gas sampling		
S_L	Liquid sampling		

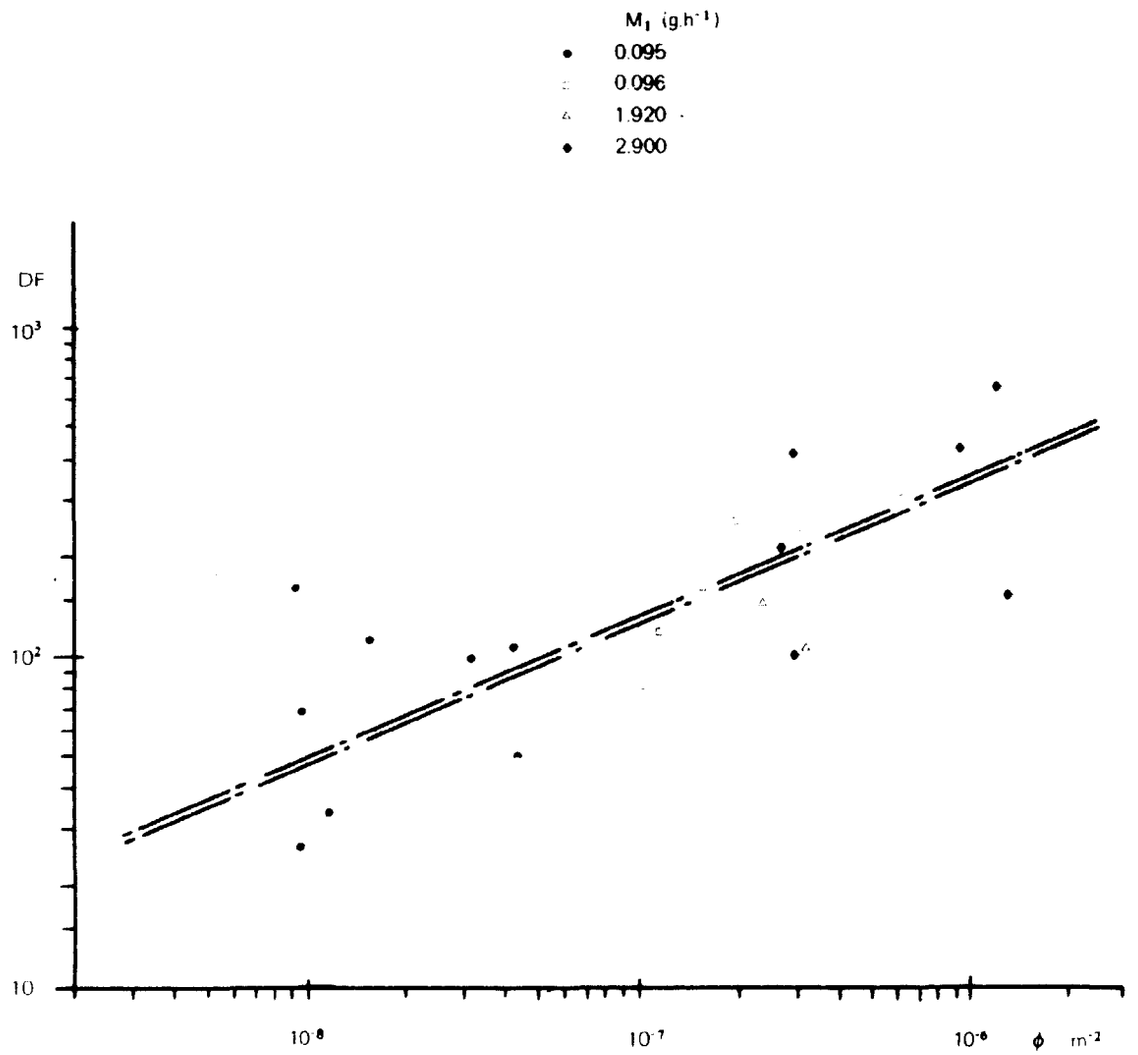


FIG 4 IODINE TRAPPING IN VENTURI SCRUBBER

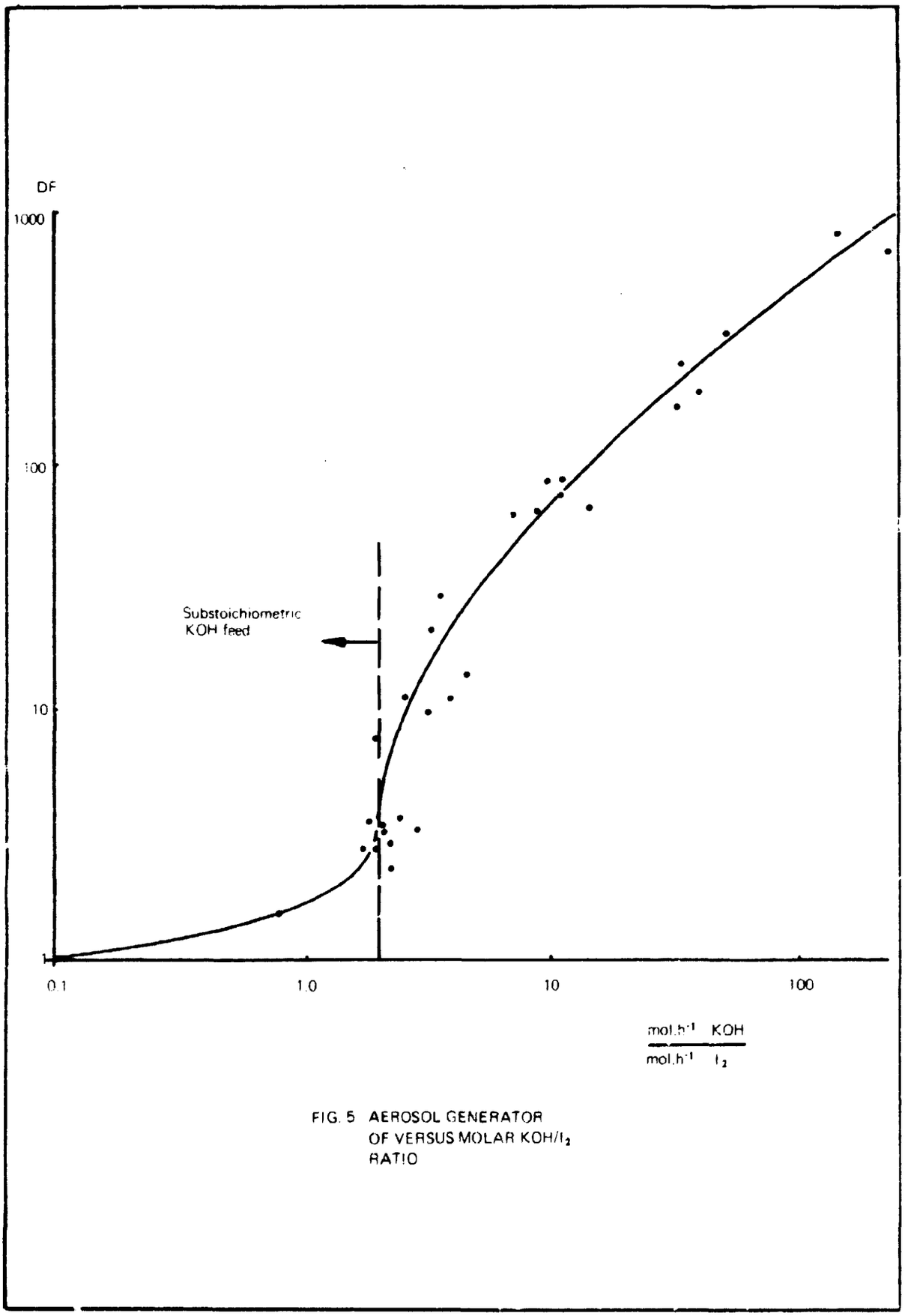
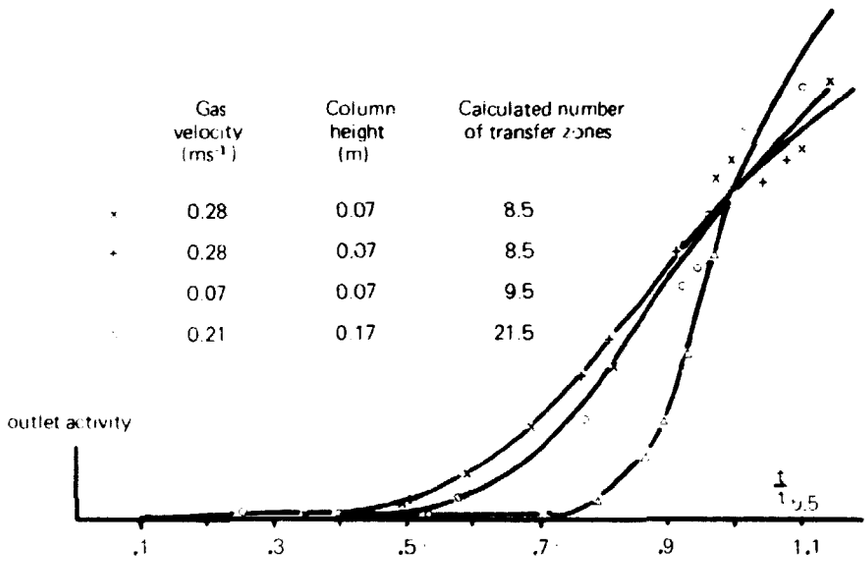


FIG. 5 AEROSOL GENERATOR
OF VERSUS MOLAR KOH/I_2
RATIO

FIG. 6 FITTING OF EXPERIMENTAL BREAK-THROUGH CURVES



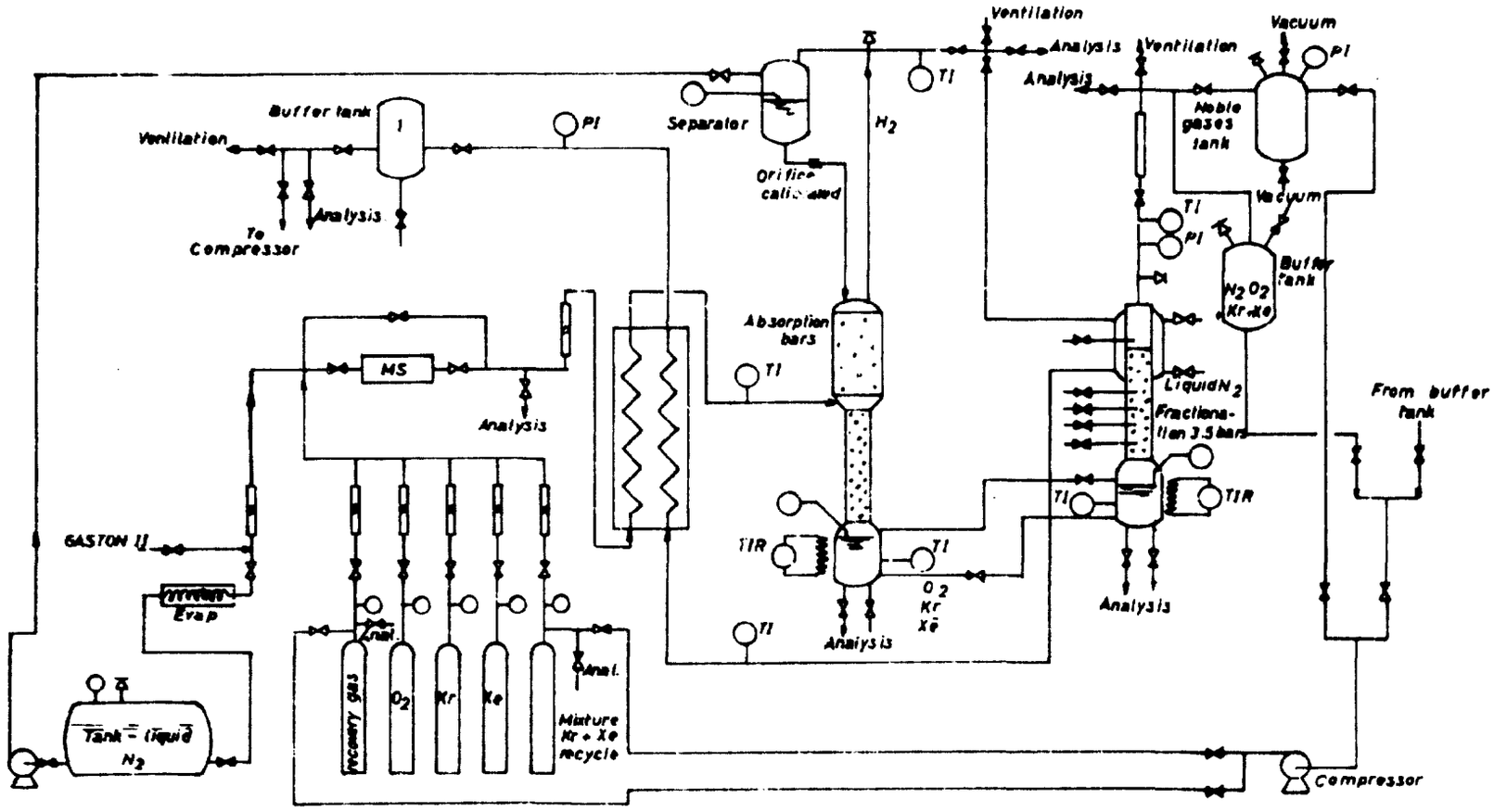
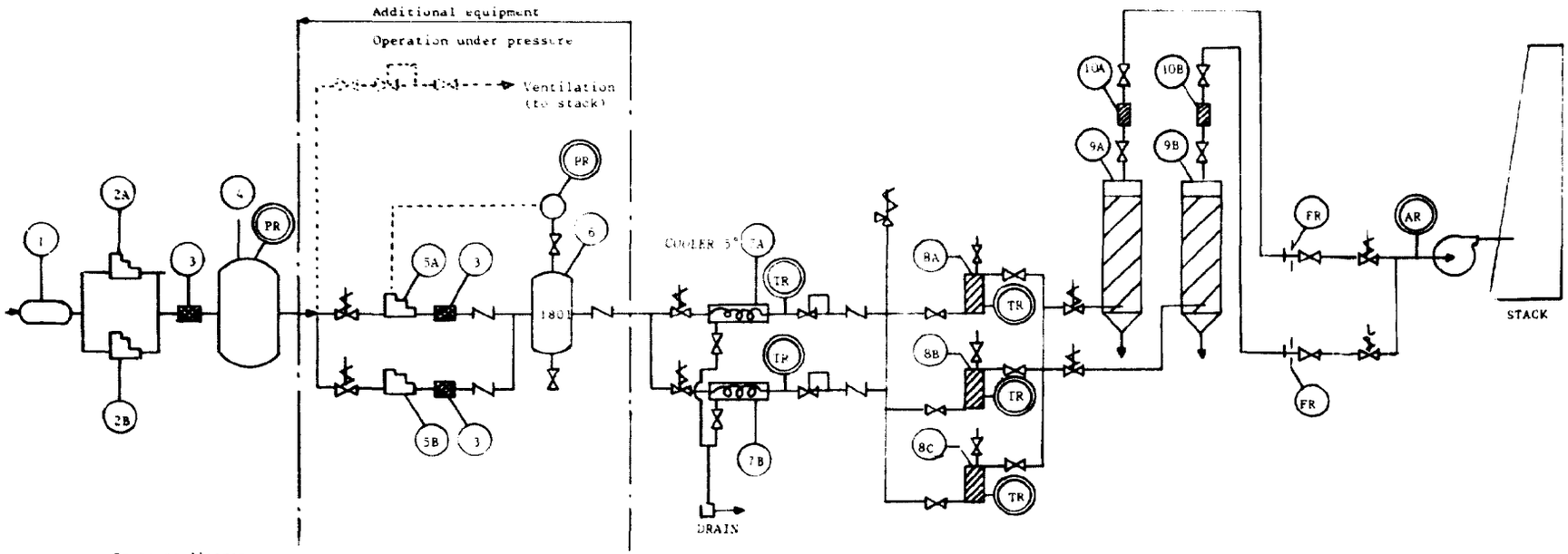
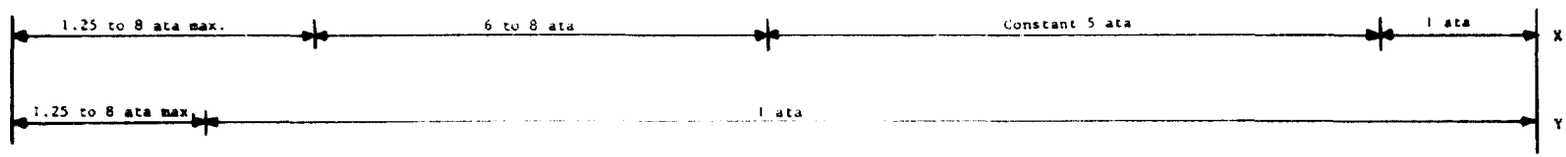


FIG 7 CRYODISTILLATION SCHEME OF THE PILOT INSTALLATION



Pressure diagram



1 : Buffer tank
2A - 2B : Compressors
3 : Filter
4 : Buffer tank
5A - 5B : Compressors
6 : Buffer tank
7A - 7B : Coolers
8A - 8B - 8C : Silica-gel dryers
9A - 9B : Charcoal beds
10A - 10B : Filters

PR : Pressure Recorder
TR : Temperature Recorder
FR : Flow Recorder

X : Charcoal bed operating pressure : 5 ata
Y : Charcoal bed operating pressure : 1 ata

Fig. 8

PRESSURIZED WATER REACTOR 1000 MWe
OFF-GAS TREATMENT
GENERAL FLOW-SHEET

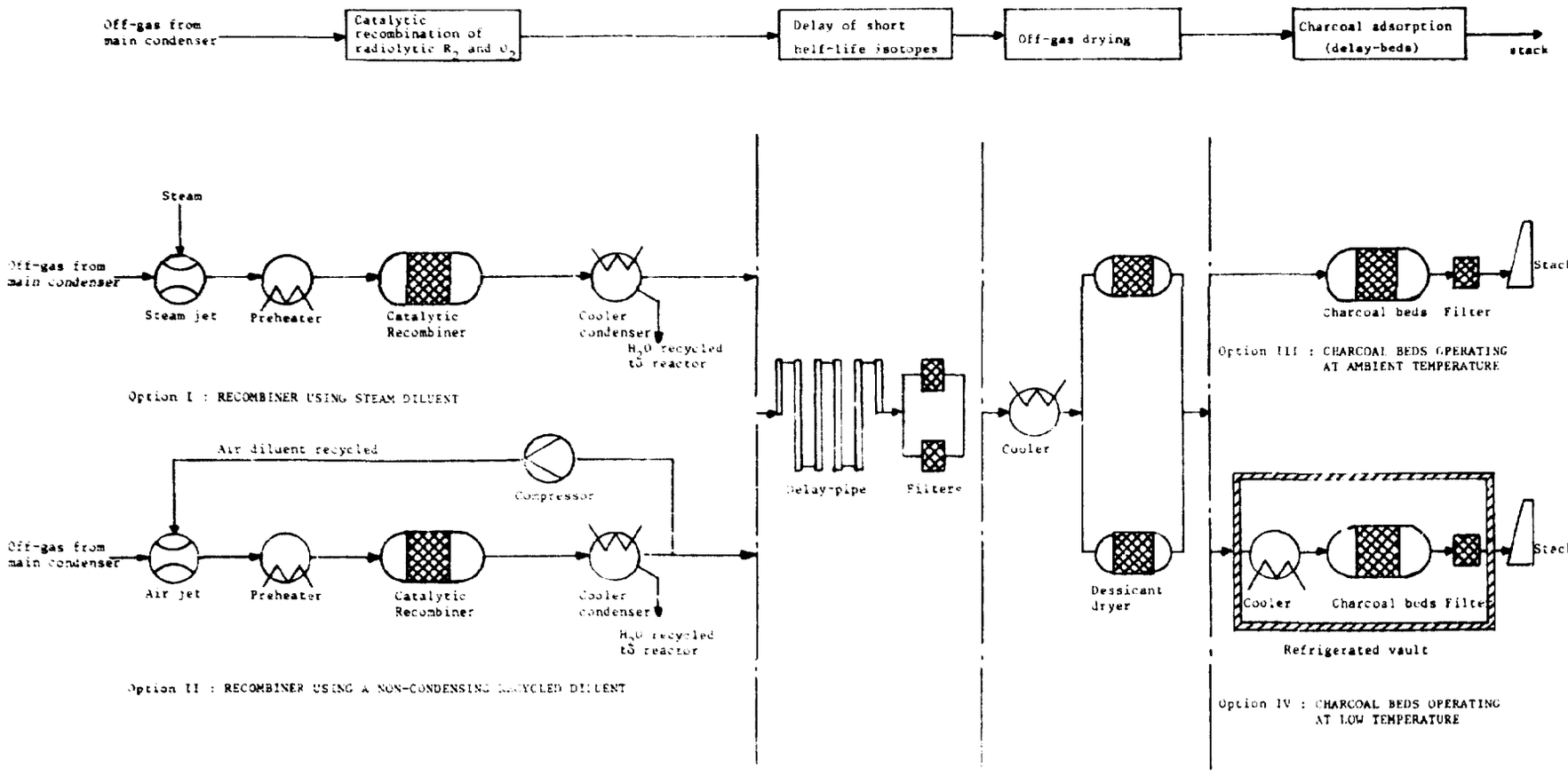


Fig. 9

BOILING WATER REACTOR 1000 MWe
OFF-GAS TREATMENT
PROCESS FLOW-SHEETS