



Some Techniques for Sodium Removal in CIAE

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Abstract

In this paper the experiment and application on sodium removal and sodium disposal are presented. Steam -nitrogen process was used in CIAE for cleaning cold traps, sodium vapor traps, a sodium tank. Atomized water-nitrogen process was used for cleaning dummy fuel assembly for CEFR and a sintered stainless steel filter. Sprinkle process was used for cleaning some tubes. Butylcellosolve was used for cleaning sintered stainless steel filter and sodium flow measurement device. Ethanol alcohol was used for cleaning electromagnetic pump. Paraffin, transformer-oil or their mixture was used for cleaning sodium valves, a sodium vapor trap and sodium-potassium alloy absorber. A small sintered stainless steel filter was distilled in vacuum. A simple sodium disposal device has been served for several years in CIAE. It can dispose about 10 Kg sodium each time and the disposal process is no-aerosol. It operates in open air for non-radioactive sodium. In recent years a small sodium cleaning plant has been built. It can use atomized water, steam or organic alcohol to removal of sodium. The LAVEL cleaning plant and SLAPSO cleaning plant were introduced from Italy. And CEFR preliminary design on sodium cleaning for spent fuel assembly and on sodium removal-decontamination for large reactor components is introduced. Vapour--nitrogen process is planned to use in them.

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1. Introduction

Some techniques for sodium removal have been developed as the fast reactor program goes on in CIAE (China Institute of Atomic Energy).

- *About removal process:*

Following processes have been used in our laboratory. They are:

- a. Water-based process:
 - 1). Mist-nitrogen process
 - 2). Steam-nitrogen process
 - 3). water spray or flushing process
- b. Alcohol process:
 - ethanol and butylcellosolve are used.
- c. Oil process: Paraffin, transformer oil or their mixture is used.
- d. Vacuum distillation: for cleaning small component
 - Among them, water fine spray (mist)-nitrogen process and steam-nitrogen process are widely used in removing sodium.

- *About sodium disposal:*

A small sodium disposal unit can dispose ~10 Kg of sodium each time.

- *About CEFR design on sodium removal and decontamination:*

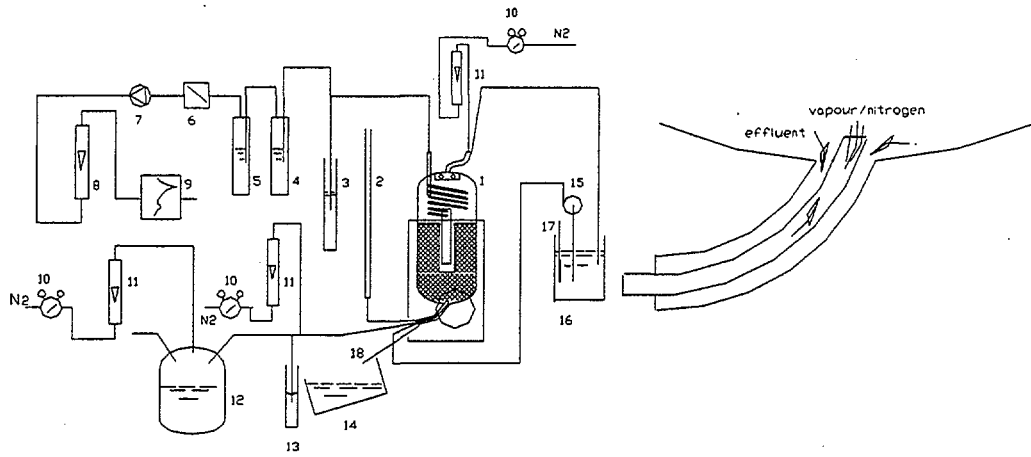
The preliminary design on sodium cleaning for spent fuel assembly and on sodium removal-decontamination for large reactor components has been done.

2. Description of sodium cleaning in CIAE

2.1. Steam -nitrogen process

Steam (wet saturated steam)-nitrogen process has been used in CIAE for cleaning of:

- a). cold traps. they are
 - Two CEDI cold traps, one ESPRESSO cold trap, two 401-cold traps . The simplified cleaning diagram are shown in fig.1. fig.2. fig.3. fig.4.



- | | | | | | |
|-----------------|----------------------|----------------|------------------------------|--------------------------|---------------------|
| 1 . cold trap | 4.5 . washing column | 8 . flowmeter | 11 . flowmeter | 14 . receptacle | 16 . tank |
| 2 . level | 6 . filter | 9 . recorder | 12 . steam generator | 15 . pump | 17 . monitor-sample |
| 3 . liquid seal | 7 . booster | 10 . regulator | 13 . water / vapor separator | 18 . stainless steel net | |

fig.1 flow process diagram for cleaning CEDI cold trap

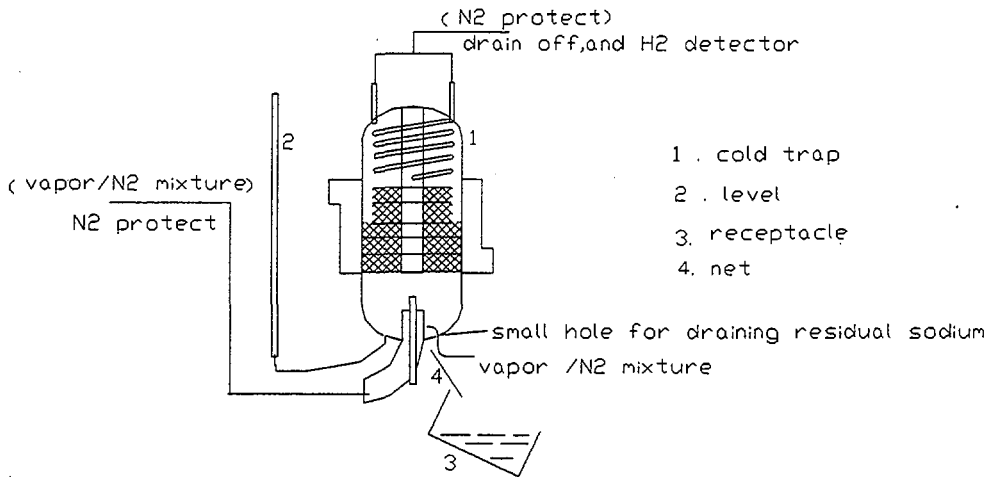
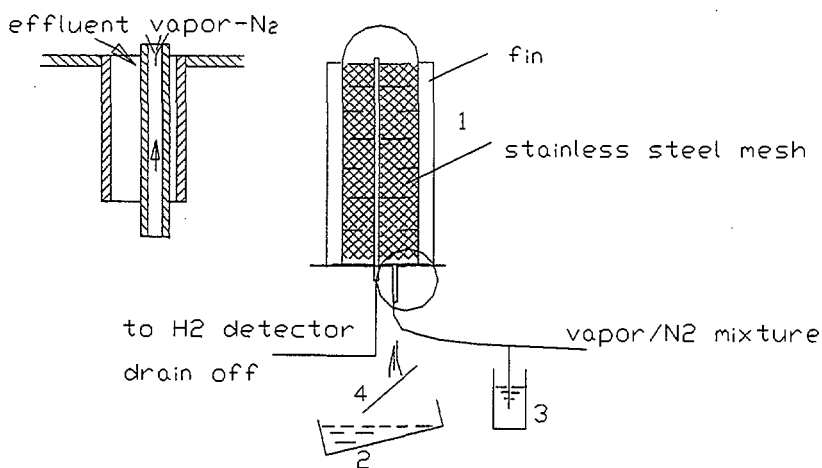


fig.2 schematic drawing for cleaning ESPRESSO cold trap



- | | | |
|-------------------------|----------------|---------------------------|
| 1 . cold trap | 2 . receptacle | 3 . vapor/water separator |
| 4 . stainless steel net | | |

fig.3 schematic drawing for cleaning cold trap-a of 401 corrosion loop

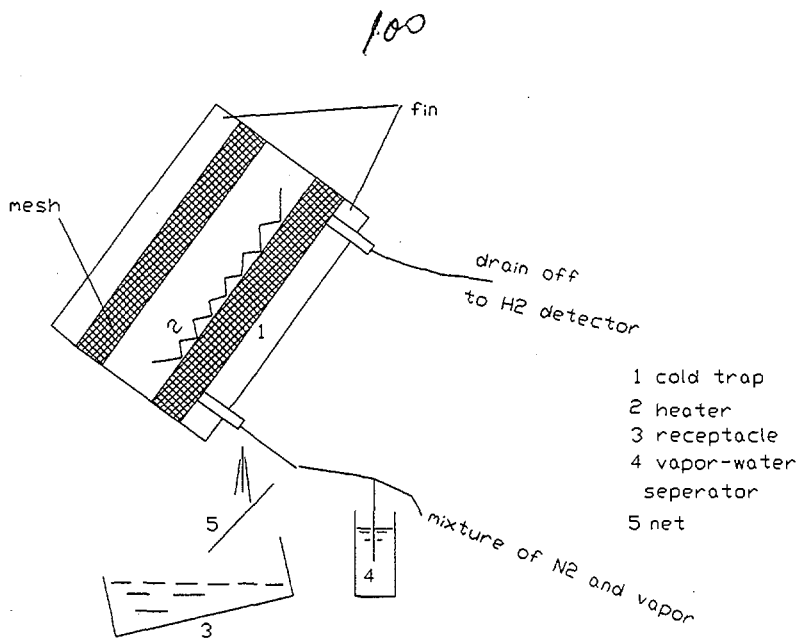


fig. 4 schematic drawing for cleaning cold trap-b of 401 corrosion test loop

-- The removal procedure mainly was :

- 1). To drain the sodium under the protection of nitrogen by heating.
- 2). To fill the mixture of steam-nitrogen under the protection of nitrogen and to make the reaction rate controllable by regulating the flow rate and content of the vapour in the mixture.
- 3). To fill water from the effluent outlet after the reaction finished (by detecting release of hydrogen). Then to recirculate the solution, to drain it repeatedly .

-- Two 401-a,b cold traps were cut for detection after cleaning. The results of the cleaning were in following:

- 1). A visual inspection of the mesh and stainless steel tank interior showed no evidence of unreacted sodium.
- 2). There were thin gray (black) powdery deposits on the stainless steel tank and on mesh surfaces. They can be partly rubbed with white cloth or paper.
- 3). The effluent liquid was mainly sodium hydroxide solution, sometimes it was like a gray-white paste accompanied with o'dious o'dour which can be smelled when calcium carbide reacts with water.
- 4). Typical curve of hydrogen content in nitrogen in cleaning process and hydrogen content in nitrogen before end of the cleaning process are shown in fig.5 and fig.6.
- 5). The summary of cleaning is shown in the following table.

table 1 the cleaning summary of cold traps

date	cold trap	volume of trap(L)	total time of cleaning(hr)	consume of steam(Kg)	amount of Na removed(Kg)
950329-0407	401-a	12	26	15	1.12
950707-07	401-b	7	5	3	0.2
950915-0917	CEDI-a	40	15	10	0.9
950919-1005	CEDI-b	40	21	16	2.4
951023-1030	ESPRESSO	220	33	45	6.33

b). sodium vapor trap

Two sodium vapor traps (for capture sodium vapor in argon cover gas) which was served in CEDI circuit (fig.7).

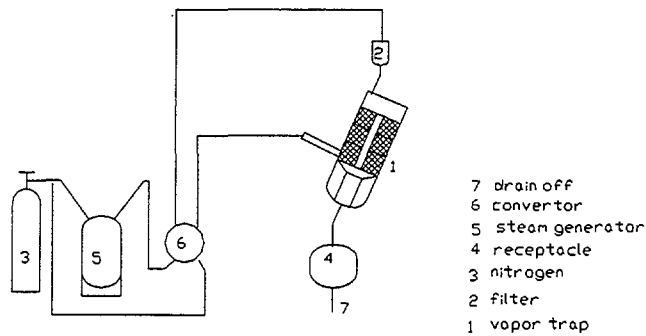


fig7 schematic diagram for cleaning sodium vapor trap

In the cleaning process the trap was suspended by chain-rope outdoor and the trap can be rotated (shaked) in order to drain the reaction products better.

For removing of sodium from this vapour trap, firstly to heat the trap under the protection of nitrogen, and about 6 Kg sodium was drained off. And secondly to use mixture of steam-nitrogen to clean it and 0.7 Kg sodium was cleaned (by analyzing the reaction products)

c). sodium tank

A sodium tank (which was used in 401-sodium corrosion test loop fig.8.).The inlet and outlet of the storage tank were clogged by sodium oxide and/or carbonate because it had been dismantled from their loop for several years.

After the blocking products were dug, the inlet and outlet were heated to drain the sodium off. Then it was cleaned by vapour -nitrogen in nitrogen atmosphere.

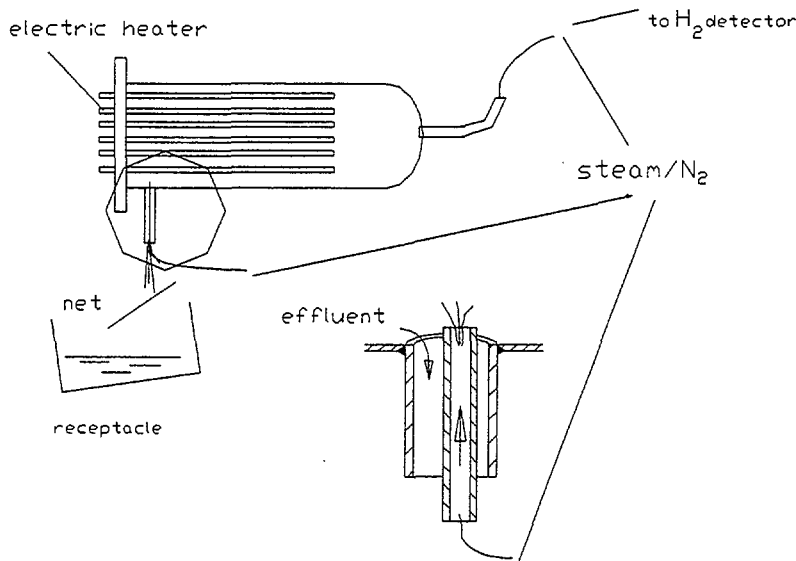


fig 8 sodium cleanig for a storage tank

2.2. Atomized water process

2.2.1. Atomized water -nitrogen process

Atomized water (which is generated by ultrasonic technique)-nitrogen process was used for cleaning of :

a). dummy fuel assembly

After the dummy fuel assembly for CEFBR (China Experimental Fast Reactor) was tested in sodium. It was cleaned in the Versatile Sodium Removal Plant.(fig.16)

The fuel assembly was tested at 400-430 °C in sodium and the sodium was drained

at 200°C (the fuel assembly was suspended for 20 hr.)

The sodium adhered to the fuel assembly was about 10µm in average by analyzing the effluent after the assembly was cleaned.

-- The cleaning procedure consists mainly of :

1) Making the atmosphere of the washing tank inert by vacuumizing and filling nitrogen;

2) Using atomized water-nitrogen mixture to react with the adhered sodium by circulating the mixture, (The constituent and flow rate can be regulated. The atomized water is generated by an ultrasonic mist generator. It is easy to control electrically). In the whole process a computer is used to measure the hydrogen content and to participate partly in controlling the cleaning process;

3) Rinsing -flushing

Deionized water was used to rinse the fuel assembly (generally, in this process no hydrogen was detected) after sodium -vapour reaction was finished, Then water was filled and it was circulated to flush the fuel assembly more than ten times .

-- The conductivity of the flushing water approached 20 µs-cm⁻¹ and the pH was near to neutral.

b) sintered filter

A sintered stainless steel filter (which was used in ESPRESSO sodium circuit). In the process the filter (laid in inclined position, and has very thin film of sodium oxide powder on the surface) was in flowing fog atmosphere. The sodium and its compounds absorbed water from fog and a film-solution on sodium surface is formed. The water in film-solution penetrated into microholes of the filter to react with sodium, and gradually past through the whole holes. More than three hours later the reaction finished. Then deionized water was used to spray it , then it was soaked in water and let water flow through the filter for cleaning it thoroughly. Several rusty spots were found on the surface of the sintered material several days later in air after it had been cleaned.

2.2.2. Sprinkle process

Sprinkle process was used for cleaning of tubes:

a). Some tubes after they were disassembled from their loop. Generally, there was some residual sodium on surface of tube. If the residual sodium film is not thick, it oxidizes in storage period. So there was not much metal sodium beneath the oxidation layer. In this case the spray or steam was used and the tube was laid in this way that the sodium was on the upper of the tube and the tube was inclined (fig.9) on its position when the cleaning process was going on;

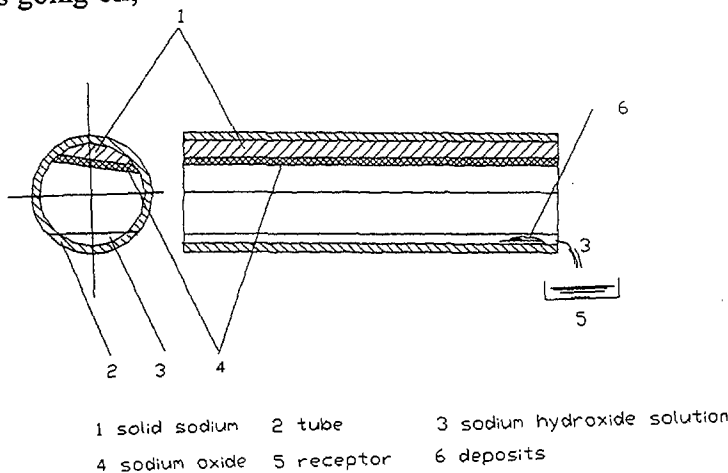


fig 9 sodium removing from tube

Under the flowing hydroxide solution, there was solid reaction products (mainly sodium hydroxide crystalline) especially in the downstream sometimes.

b). Another tubes. there were much more sodium in them--nearly half of the section of the tube was filled by sodium. In this case heating the tube to drain off the sodium, then spray or steam process can be used like above to clean them.

2.2.3. Water flushing process:

In cleaning of sodium from some tubes of the circuits ESPRESSO and CEDI it was noticed that the residual sodium on the internal surface of the tube had become:

- 1). white solid powder if the residual sodium was very thin film ;
- 2). wet hydroxide if the residual sodium was thicker and it absorbed water from air. The wet product caused corrosion of the tube, a thick brown (iron rusty crust) film like dirty grease was seen obviously. The crust consisted of a thick wet sodium hydroxide and a thin brown (iron rusty crust) film. In this situation water flushing process was used.

2.3. Alcohol process

2.3.1. Butylcellosolve process

Butylcellosolve was used for cleaning of:

a). sintered filter

Sintered stainless steel filter--one was used in multipurpose sodium purification loop(fig.10) and another was used in ESPRESSO sodium circuit .

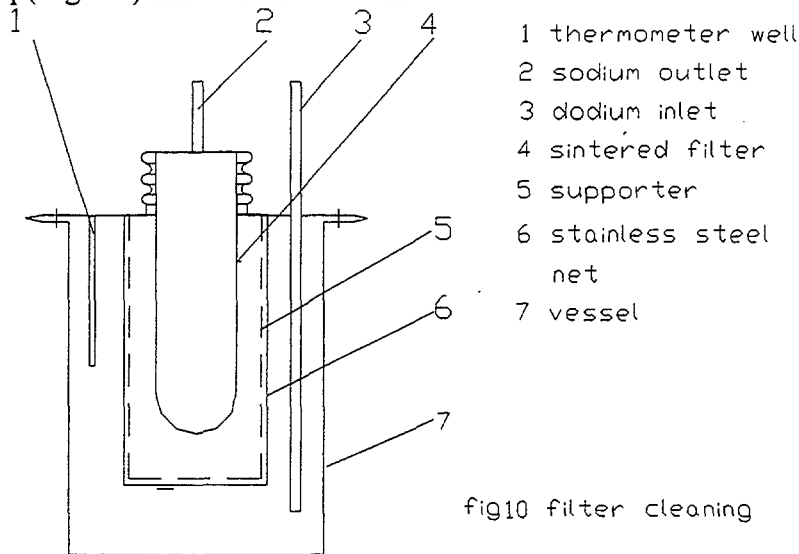


fig10 filter cleaning

-- The cleaning procedure was:

1). To cut the welding edge between the cover and the vessel, then to dismantle the cover (the sintered filter was welded on the cover).

2). To remove the sodium on the stainless steel net (for pre-filtering) using butylcellosolve . Then to flay the net.

3). To make butylcellosolve flow along exterior of the sintered filter to react with the sodium through soft polyethylene tubes. Then the solvent penetrated the microholes of the filter to react with the sodium staying in the holes. After that to pour the solvent from the outlet tube of sodium into the filter, and let the solvent pass through microholes to clean the residual sodium thoroughly.

4). Diluted acetic acid solution containing corrosion inhibitor was used to remove calcium oxide in microholes. For the filter($\phi 50 \times 3$, $h=160\text{mm}$), 2.3 g CaO was detected in acetic acid cleaning solution by analyzing .

At last , the deionized water was adopted to wash the filter.

-- The performance of the sintered filter was tested and no change was found after cleaning, comparing with the new one in penetrating rate of water.

b). submerged-in flowmeter

Sodium flow measurement device consisted of submerged-in flowmeter. It was cleaned using butylcellosolve (It is setup with oscillating in 3-dimensions when cleaning process was going on , fig.11)

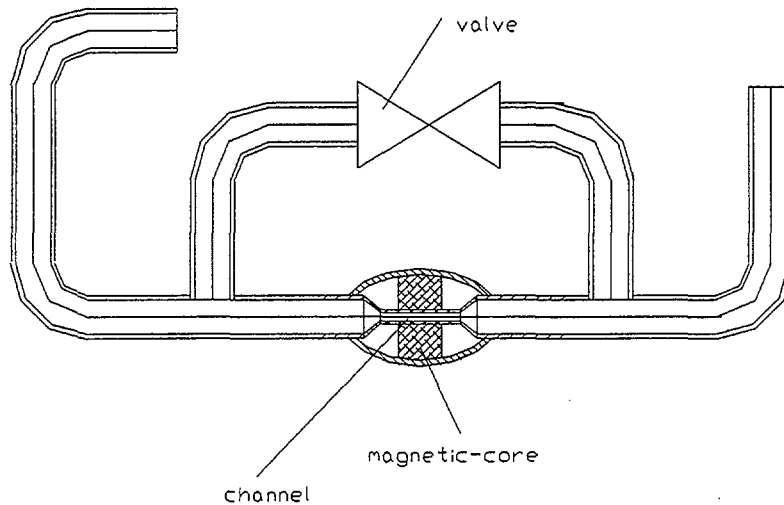
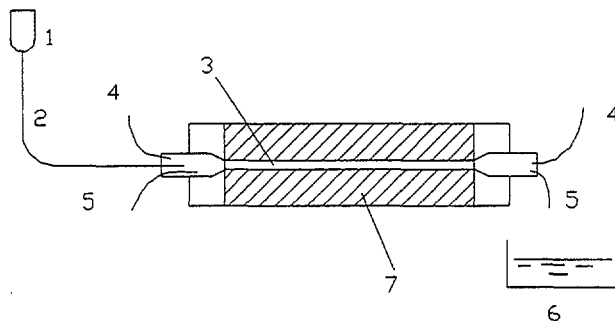


fig11 cleaning for submerged-in flowmeter

2.3.2. Ethanol alcohol process

Ethanol alcohol was used for removing of electromagnetic pump (simplified diagram is in fig .12). In the removing process a soft polyethylene tube was extended into the pump channel progressively in order to fill the fresh solvent to the sodium surface.



1 alcohol 2 polyethylene tube 3 channel of pump
4 to H₂ detector 5 effluent 6 receptacle
7 electromagnetic iron

fig12 schematic diagram of cleaning electromagnetic pump

2.4. Paraffin-oil (or transformer-oil) process

Paraffin-oil (or transformer-oil) process for cleaning of :

a). sodium valves

The cleaning procedure was:

To put the bellows sodium valve in hot oil. After the sodium is melted, to move the valve-drive stem (open and close the valve) repeatedly and to rotate the valve in 3-dimensions to drain the sodium out. After this, to use flowing butylcellosolve to react with

residual sodium, and to disassemble it. At last, water or acetone is used to clean it.

b). sodium-potassium alloy absorber for trapping oxygen and moisture in argon.

The vessel was packed with stainless steel mesh. It contained about 0.5 Kg Na-K alloy and their oxides when it needed cleaning. The simplified removing diagram is shown in fig.13.

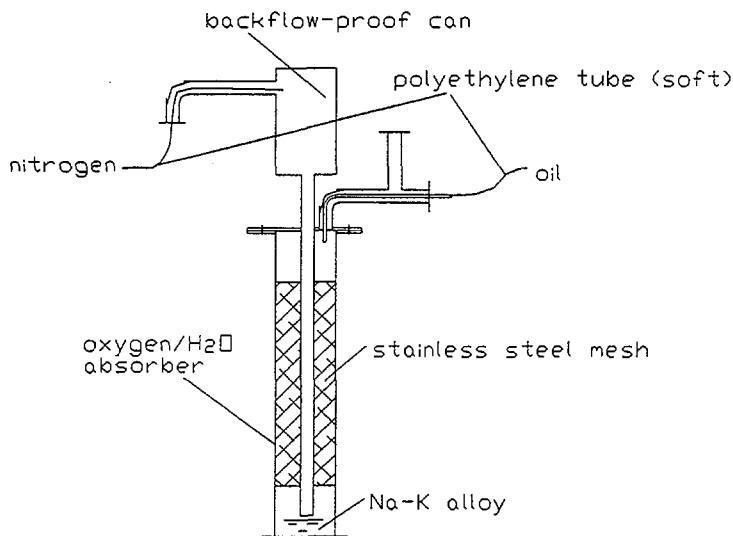


fig 13 removal of Na-K from absorber for oxygen/vapor

-- The removal procedure was in following:

several liters of paraffin oil was filled into the vessel through a long soft polyethylene tube under the protection of nitrogen. Then the mixture of alloy-their oxides-oil was drained off into a receptacle. After this the device was dismantled .

Then, the vessel and the central tube can be cleaned with mist-water because the residual alloy on the inner surface was very little; and the separated mesh was thrown into water tank if a very little Na-K alloy was on the mesh. In this way the mesh sank into water quickly and the residual sodium floated on the water surface.

-- No damage was found obviously on the surface of the mesh and the mesh can be reused.

c). sodium vapor trap

-- One of the sodium vapor trap which was served in ESPRESSO sodium circuit was packed with stainless steel mesh (fig.14.). The sodium removal procedure was in following:

To disassemble the trap because it had a long storage period after it was dismantled from its circuit , then to take out the stainless steel mesh, to sink the mesh which contains some sodium residual into hot oil and to shake it in order to make the residual sodium fall down into the oil. After this, the sodium was very little on the mesh .Then it was taken out from oil and sank into water. Subsequently it was cleaned with acetone for removing the residual oil. And the cylinder was cleaned with water sprinkling process.

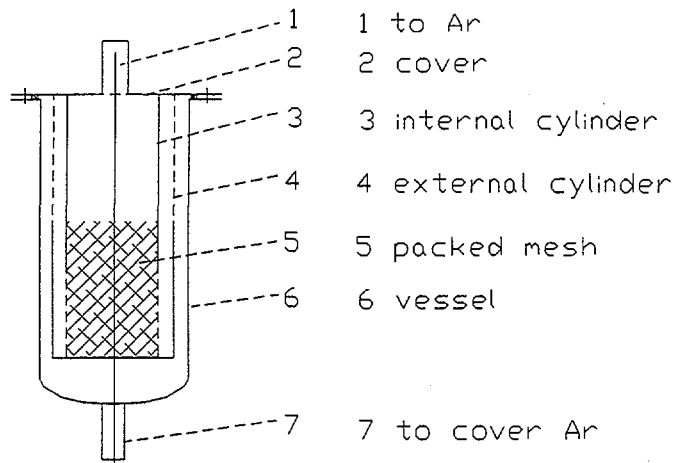


fig14 schematic diagram of sodium vapour trap

-- In another sodium vapor trap residual sodium became white powder (a very thin layer) on the packed mesh surface in storage period. It was easy to clean by wind-sweeping thoroughly. After sweeping, the surface of the mesh was bright like a new one.

2.5. Vacuum distillation:

A small sintered filter was heated in distillation vessel at 400°C under vacuum 5×10^{-4} mmHg for 4 hr to evaporate the sodium. After distillation the color of the sintered filter became somewhat yellow.

2.6. Summary of removal process

-- In these process the cleaning process should be made as controllable as possible and the reaction products should be made drain off as soon as possible.

-- It is recognized that organic alcohol is expensive and easy to burn; and wet vapour is better for cleaning sodium because the cleaning products have high solubility in water just condensed from vapour (higher temperature water), the sodium hydroxide solution flows out easily, it can avoid the solid products to deposit on the surface of residual sodium;

-- concerning the flow course of the wet vapour, it is better that vapour fill in from lower part of the vessel or from liquid effluent draitube (temporarily make the draitube sleeve i.e. the steam is forced to flow in internal tube into vessel, and the effluent drain off from the gap between internal tube and outer tube).

3. Sodium disposal

A simple sodium disposal unit has been built up in CIAE. It can dispose about ten Kg sodium each time and the disposal process is no-aerosol. It operates in open air for non-radioactive sodium.

3.1. Introduction

At present, the amount of sodium that needs to be disposed from our laboratory is not very much. Generally, the cheapest method of disposal is to adopt water. When sodium contacts with water, it reacts with water rapidly and violently, very soon it will explodes, burns in air and produces very large amount of white thick smoke. Considering of our circumstances (no free desert land and many resident population), a simple device of sodium disposal has been developed. It can be considered a no-aerosol digestion device for sodium.

3.2. The principle of disposal

The main disposal reaction is that sodium reacts with water which may be in different states, such as mist, fog, small water-droplet, sprinkle and vapor in the air. But the carbon dioxide (CO_2) in air which forms hard shell on the sodium will prevent sodium from disposal.

3.3. Device

A simple device has been built up . It consists of several layers of stainless steel net which have an inclined arrangement (fig.15).

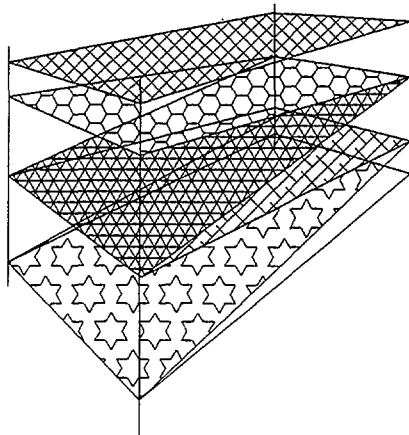


fig.15 disposal device for sodium

3.4. Phenomenon

a). Digestion in natural

-- In humid weather the sodium absorbs water in the air, and a liquid film forms on the sodium, and the liquid film absorbs water from air, a liquid drop forms and then it falls down leaving a new sodium surface for further reaction.

-- In dry day the sodium reacts mainly with oxygen and carbon dioxide in air and the oxide film of sodium stays on sodium surface, then it will obstruct further reaction of sodium. Meanwhile, the oxide of sodium reacts with carbon dioxide in the air to form sodium carbonate (which will form hard shell on the sodium surface) and the later prevents further reaction.

b). Disposal with atomized water generated by electro-ultrasonic fog (atomized water) generator

In autumn and in winter, the weather is very dry, a white thick film is formed on sodium surface. When atomized water is used it will react with sodium and sodium dioxide, and liquid hydroxide film on the sodium surface forms . Then the hydroxide absorbs water in fog continuously, a big drop of liquid hydroxide forms and it will descend from the sodium surface. At last, it leaves fresh sodium surface to react with water. So atomized water is recommended to accelerate the disposal process.

3.5. Summary of sodium disposal

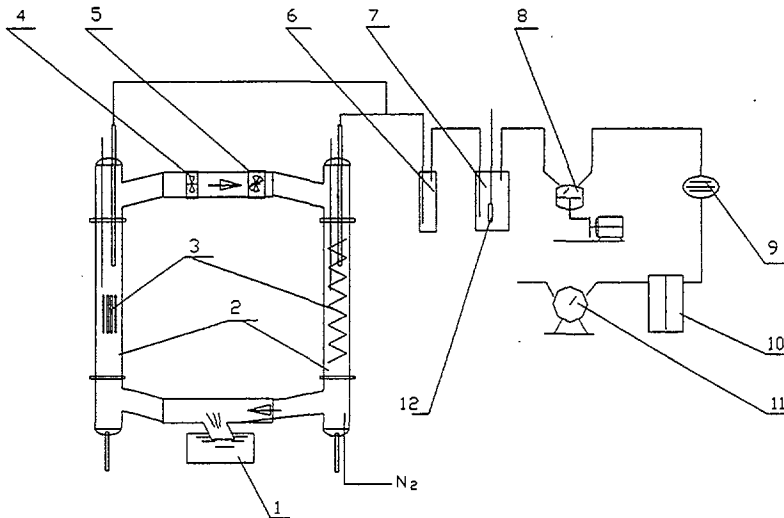
- 1). It can be considered a no-aerosol disposal device for sodium.
- 2). It has disposed tens Kg sodium from our laboratory safely for several years.
- 3). About ten Kg sodium can be put on it for disposing each time .
- 4). In the summer, ten Kg sodium can be disposed within a week. In autumn and winter, it will last longer and form hard shell. So using water- sprinkling or mist will accelerate the disposal process. The sprinkle day is the best day for sodium disposal.
- 5). Sodium-potassium alloy can be disposed on this device with help of liquid paraffin-oil , but large amount of water drop must avoid, and the alloy should be laid separately. Otherwise, the possibility of alloy's burning is bigger.

4 Recent aspect in sodium cleaning

4.1 Experiment plant

a). A small device using atomized water was adopted to test mist-nitrogen process for removing sodium. The schematic diagram is show in fig.16. It consists of polyethylene tube, and the material of the reaction tube ($\Phi 70\text{mm} \times 500\text{mm}$) is organic glass.

fig 8 sodium cleanig for a storage tank



- | | | |
|----------------------------|-----------------|----------------------|
| 1 atomized water generator | 2 reaction tube | 3 sample |
| 4 fan | 5 flowmeter | 6,7 absorbor |
| 8 air-boost compressor | 9 filter | 10 hydrogen detector |
| 11 flowmeter | 12 humidimeter | |

fig16 test device for mist-sodium reaction

Some characteristic parameters are:

- | | |
|----------------------------|---------------------------|
| --- content of mixture | 0.02 g H ₂ O/l |
| --- flow of mixture | 0.5 m/s |
| --- size of atomized water | 2-5 μm |

b). A Versatile Sodium Removal Plant has built in CIAE (fig.17).

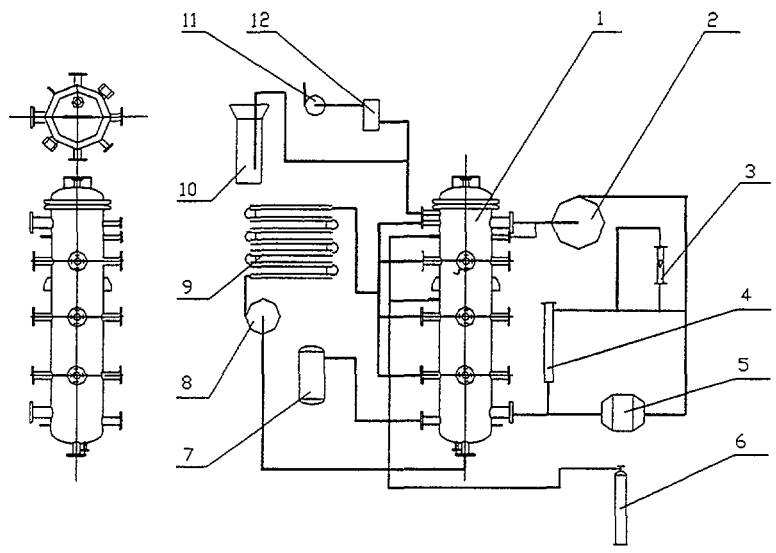
It is used to clean some small components like a dummy fuel assembly, a lower part of control bar, etc.

It consists of a washing tank, a mist generating-circulating system, steam generation system, a water (or organic alcohol) circulating-flushing system, a vacuum-exhaust system, water (or organic alcohol mixture) supply system, nitrogen and carbon dioxide supply system, etc. The washing tank is a cylinder ($\phi 400\text{mm} \times 2850\text{mm}$) with 3 layer manifold for spraying

Some characteristic parameters are:

- | | |
|---|------------------------------|
| --- yield of atomized water | 5 Kg/hr |
| --- flow of circulating of the nitrogen | 160, 250 m ³ /hr |
| --- flow rate of water | 3 T/hr x 2 |
| --- yield of steam | 4 Kg/hr, 15 Kg/hr (t=138 °C) |
| --- vacuum | ~ -0.1 Mpa |
| --- pressure of nitrogen | 0.3 MPa |

So, it can use atomized water, steam, organic solvent as cleaning medium and it can operate in pressure or vacuum and it can use nitrogen or carbon dioxide as protecting gas in design.



1 washing tank 2 circulating blower 3 flowmeter 4 heater
 5 atomized water generator 6 nitrogen 7 steam generator
 8 pump 9 heat exchanger 10 water seal 11 vacuum pump
 12 separator

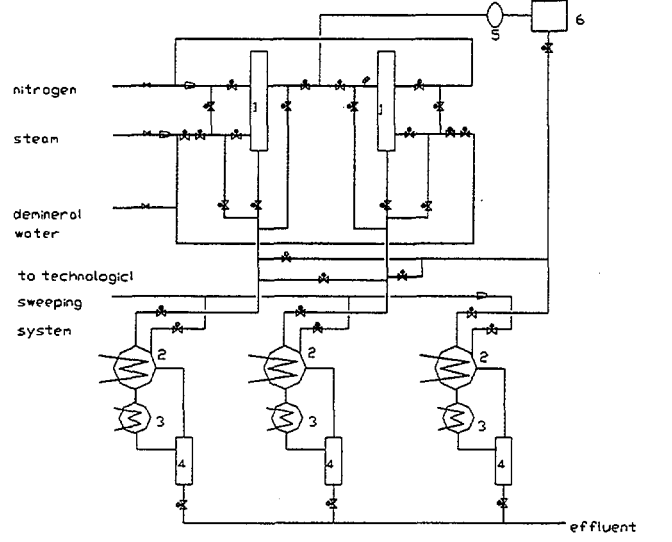
fig.17 schematic diagram of versatile sodium removal plant

c). In recent years some experimental devices were introduced from Italy, among them the LAVEL cleaning plant and SLAPSO cleaning plant have been rebuilt.

4.2 CEFR (China experimental fast reactor) cleaning and decontamination plant design

a). Spent Fuel Assembly Cleaning System

According to CEFR's design demands the spent fuel assemblies should be cleaned after they are discharged from reactor in order to keep them in water pool for a long time. The vapour - nitrogen process is demanded. The simple flow sheet of the Spent Fuel Assembly Cleaning System is in following. (fig.18).



1 cleaning cylinder 2 heat exchanger 3 cooler
 4 receptacle 5 filter 6 detector for rupture assembly

fig.18 cleaning flowsheet for spent fuel assembly

The main parameters are:

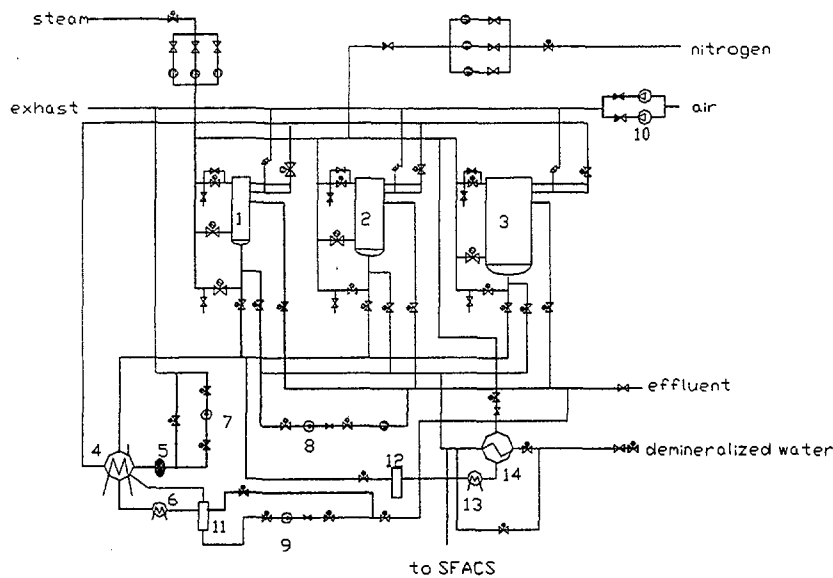
- yield of the steam 300 Kg/hr (t=138 °C)
- pressure of nitrogen 0.6Mpa
- flow of nitrogen 100 NM³/hr
- volume of the cleaning cylinder 0.03 M³

b). Component Cleaning and Decontamination System

Large reactor components, for example, intermediate heat exchanger, pump, fuel charging machine, etc. will be cleaned in the Component Cleaning and Decontamination System which also use vapour-nitrogen mixture to clean. Decontamination solution will adopt sodium hydroxide / potassium permanganate solution mixture and oxalic acid solution. The simple flow sheet is in fig.19.

The main parameters are:

--- yield of the steam	1000 Kg/hr (t=138 °C)
--- pressure of nitrogen	0.6 Mpa
--- flow of nitrogen	1000 NM ³ /hr
--- size of washing tanks	φ1500 × 9000mm
	φ600 × 11600mm
	φ400 × 13600mm



1,2,3 washing tank 4 heat exchanger-condenser 5 filter 6 watercooler
7 rotary water-sealed vacuum pump 8 drainage pump 10 ventilator
11,12 tank 13 water-cooler 14 heat exchanger
fig19 cleaning flowsheet for component

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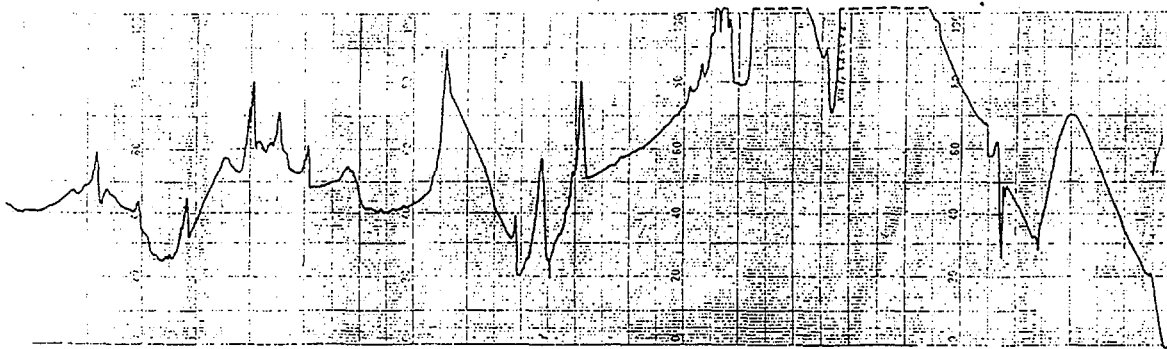


fig.5 typical curve of hydrogen content in nitrogen in cleaning process

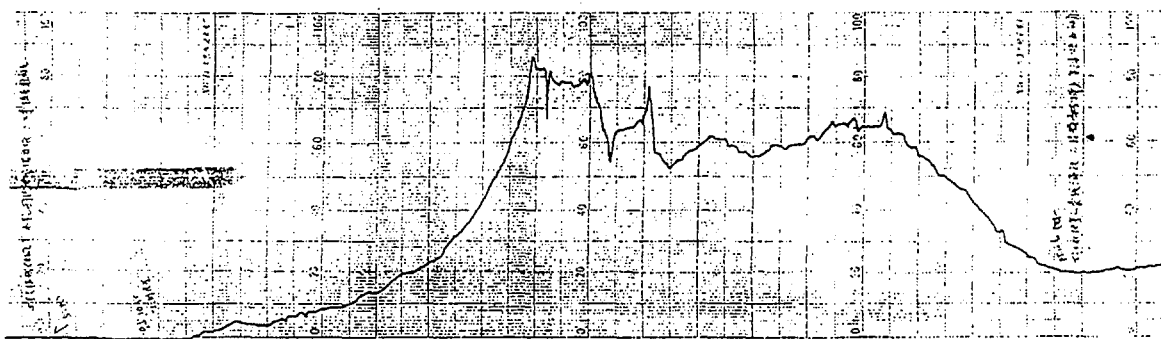


fig.6. hydrogen content in nitrogen before end of the cleaning process