

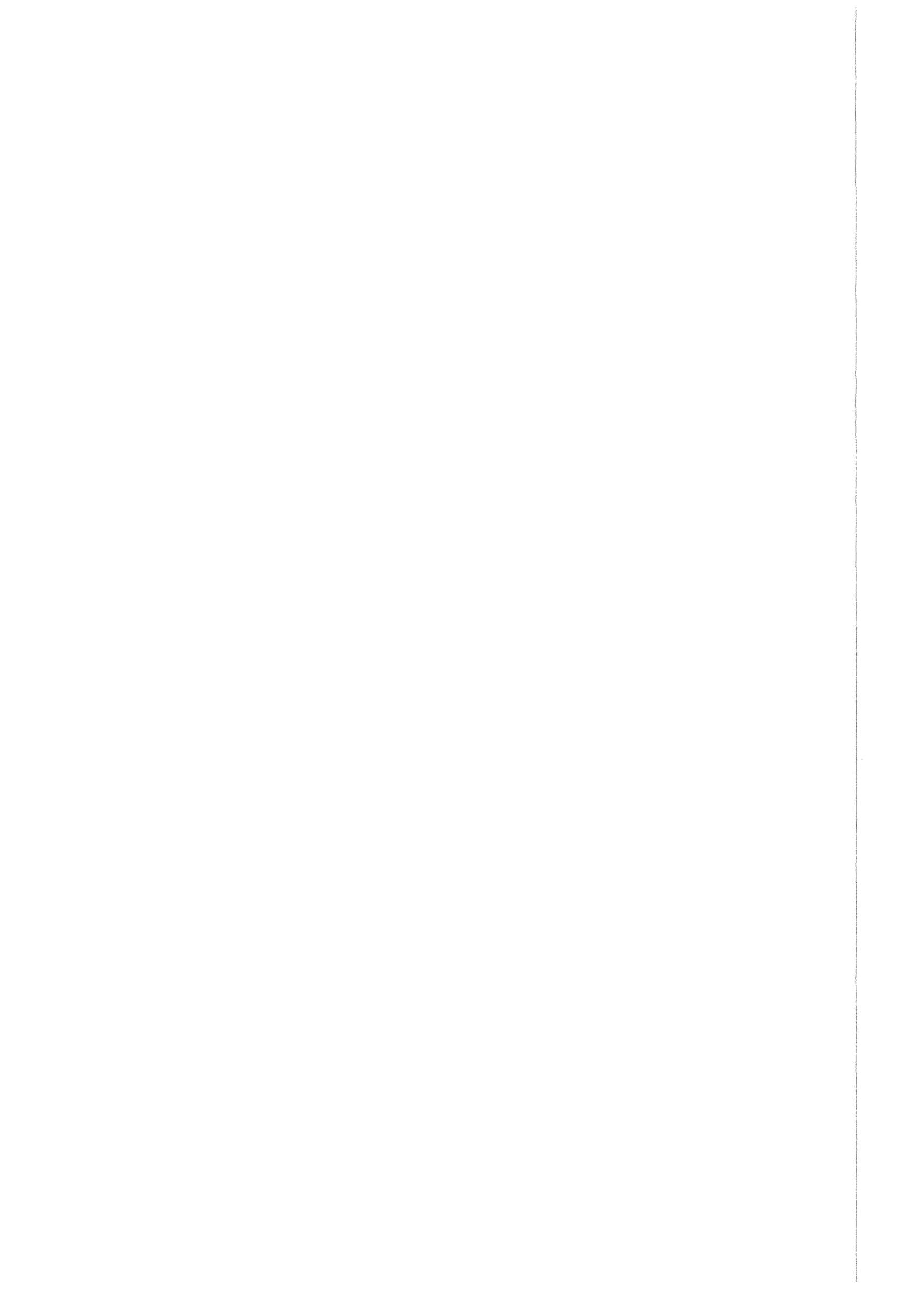
Forschungszentrum Karlsruhe
Technik und Umwelt

Wissenschaftliche Berichte
FZKA 6286

TRITEX
A Ferritic Steel Loop
with Pb-15.8Li
Facility and Operation

H. Feuerstein, S. Horn, G. Kieser
Hauptabteilung Ingenieurtechnik

Mai 1999



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*kiki Ingenieurgesellschaft GmbH, 76275 Ettlingen

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TRITEX

A Ferritic Steel Loop with Pb-15.8Li*

FACILITY AND OPERATION

Abstract

TRITEX was a pumped loop with Pb-15.8Li, fabricated from steel 1.4922. In contact with the molten eutectic were also molybdenum, vanadium and armco-iron. The loop was originally designed to investigate tritium extraction using solid getter metals. Over the years the goal changed to the study of metals, corrosion products and purification of the eutectic mixture. Therefore many modifications were done.

The first part of this report describes TRITEX. All parts were enclosed in thermoboxes for a homogeneous temperature. One test section was in an argon glove box and could be opened during operation. Other special equipment's were : permeation membranes and liquid-metal-covergas-interfaces to study the transport of H₂, D₂ and ³H, different purification devices, a quartz observation window to see the liquid metal surface, 4 different kinds of flow measurements, level indicators, freeze valves.

The second part describes loop operation. Between 1989 and 1996, the eutectic mixture was circulation for 13.003 hours in seven experimental phases. Temperatures in the main loop were mostly between 400 and 480°C, flow rates up to 2.8 l/min. The cold trap by-pass was operated between 240 and 270°C. Depending on goals for an experiment, parts of the loop were modified. After phase V and VII TRITEX was dismantled and all parts analyzed for impurities and deposits.

A third part shows photos of the facility and components.

* In the literature usually Pb-17Li is written. However the eutectic mixture contains only 15.8 at.% lithium.

TRITEX

Ein Pb-15.8Li* Kreislauf aus Ferritischem Stahl
ANLAGE UND BETRIEB

Zusammenfassung

TRITEX war ein Kreislauf mit Pb-15.8Li aus ferritischem Stahl. Molybdän, Vanadium und reines Eisen waren ebenfalls in Kontakt mit der eutektischen Mischung. Der Kreislauf war ursprünglich für die Extraktion von Tritium mit festen Getterstoffen ausgelegt. Im Laufe der Zeit verschoben sich die Aufgaben mehr zum Verhalten von Metallen und zur Abtrennung von Verunreinigungen und Korrosionsprodukten. Die Anlage wurde entsprechend den jeweiligen Versuchszielen modifiziert.

Im ersten Berichtsteil wird die Anlage beschrieben. Zur Erzielung einer gleichmäßigen Temperatur waren alle Anlagenteile in Thermoboxen eingeschlossen. Eine Versuchseinrichtung war in einer Argon-Handschuhbox und konnte während des Betriebes des Kreislaufs geöffnet werden. Andere spezielle Anlagenteile waren : Permeationsmembranen und Grenzflächen *Schutzgas-Flüssigmetall* zur Untersuchung des Verhaltens von H₂, D₂ und ³H, Kalt- und Magnetfallen zur Reinigung des eutektischen Gemisches, ein Fenster zur Beobachtung der Flüssigmetall-Oberfläche, 4 verschiedene Strömungsmesser, Niveau-Regler und Gefrierventile.

Im zweiten Berichtsteil werden die Versuchsphasen beschrieben. Insgesamt war TRITEX 13.003 Stunden in Betrieb. Die Temperaturen im Hauptkreislauf lagen in der Regel zwischen 400 und 480 °C, bei Strömungsgeschwindigkeiten bis zu 2.8 l/min. Der Kaltfallenkreislauf wurde zwischen 240 und 270 °C betrieben. Nach den Betriebsphasen V und VII wurde die Anlage vollständig zerlegt und analysiert.

In einem dritten Berichtsteil werden Fotos der Anlage und von Komponenten gezeigt.

* In der Literatur wird Pb-17Li geschrieben. Die eutektische Mischung enthält jedoch nur 15.8 at.% Lithium.

Table of Contents

Abstract

Zusammenfassung

Used abbreviations

List of tables and Figures

List of photos

1.	Introduction	1
2.	Facility	2
	2.1 Loop TRITEX	
	2.1.1 Electro magnetic pump	
	2.1.2 Mechanical Valves	
	2.1.3 Freeze valves	
	2.1.4 Drain tank	
2.2	Covergas system	10
	covergas transport	
2.3	Test facilities	11
	2.3.1 Expansion tank	
	2.3.2 Sampling station	
	2.3.3 Observation window	
	2.3.4 Test section	
	2.3.5 Cold traps 1 and 2	
	2.3.6 Cold trap 3	
	2.3.7 Magnetic Traps	
	2.3.8 Corrosion samples	
2.4	Measurements and Control	17
	2.4.1 Data registration, temperature and gas pressure	
	2.4.2 Flow measurements	
	Electro magnetic and mass flow meters, gas pressure	
	2.4.3 Liquid metal level indicators	
2.5	Safety	19
2.6.	Operation	21
	2.6.1 Filling of drain tank	
	2.6.2 Loading the loop	
	2.6.2 Drain procedure	
3.	Operation history of TRITEX	25
3.1	Pre-phase	
3.2	Phase I, 960 hours	
3.3	Phase II, 1866 hours	
3.4	Phase III, 483 hours	
3.5	Phase IV, 3143 hours	
3.6	Phase V, 3431 hours	
3.7	Phase VI, 2398 hours	
3.8	Phase VII, 722 hours	
4.	Photographs of TRITEX	35
	ACKNOWLEDGMENT	51
	References	52

Used abbreviations

Pb-15.8Li	eutectic mixture
Pb-17Li	as received mixture
LM	liquid metal
EMP	electro magnetic pump
FM	electro magnetic flow meter
MFM	mass flow meter
CT	cold trap
MT	magnetic trap
MH	main heater
ET	expansion tank
SS	sampling station
TS	test section
V	mechanical valve
FV	freeze valve
DT	drain tank
LI	LM level indicator
CS	corrosion sample

List of Tables and Figures

Table 1 : Facility TRITEX

Table 2 : Used materials

- Fig.1 :** Pb-Li phase diagram
Fig.2 : Flow sheet of TRITEX,1996
Fig.3 : Leak of a pipe with trace heating, 1988
Fig.4 : Ballooning and broken pipe of Fig.4, 1988
Fig.5 : First EMP after removing the pump duct, 1988
Fig.6 : Crack at the TZM tube, 1988
Fig.7 : EMP with TZM liner, 1989
Fig.8 : Valve 5 after phase V, 1993
Fig.9 : Freeze valve
Fig.10 : 1036 cm² permatation membrane in expansion tank, 1990
Fig.11 : 100 cm² permatation membrane in TS1 and TS3, 1989
Fig.12 : Operation phase VII, TS2 with vanadium filling, 1996
Fig.13: Temperature profile in cold trap 1 (cooler), 1990
Fig.14 : Wire mesh packing in cold trap 2, operational phase VI, 1994
Fig.15 : Cold trap 2 in phase VII, closed with heater and cut open, 1996
Fig.16 : Temperature profile in cold trap 3, 1996
Fig.17 : Magnetic trap 1, strength of the magnetic field
Fig.18 : Holder with corrosion samples
Fig.19 : Temperature distribution in TRITEX. Phase VI, 1994
Fig.20 : Gas pressure differences V4 and SS2 against inert gas manifold
Fig.21 : Five point level indicator, 1989
Fig.22 : Filling equipment for pre-phase, 1988
Fig.23 : Pb-17Li bars in DT before closing, 1993
Fig.24 : Heating an cooling of 120 kg eutectic mixture in DT, 1991
Fig.25 : Filling of TRITEX in phase VI
Fig.26 : 10 years operation of TRITEX
Fig.27 : Operation phase I
Fig.28 : Operation phase II
Fig.29 : Operation phase III
Fig.30 : Operation phase IV
Fig.31 : Operation phase V
Fig.32 : Pb-17Li flow rates in phase V
Fig.33 : Operation phase VI
Fig.34 : Operation phase VI, flow rate of CT bypass
Fig.35 : Operation phase VII

List of Photos

- Photo 1 :** 1989. TRITEX facility with thermal isolation
- Photo 2 :** 1988. Control board with ELZET monitor
- Photo 3 :** 1988. Display at ELZET monitor
- Photo 4 :** 1991. Control board, T-controller and flow diagram
- Photo 5 :** 1991. Philips data-logger and PC
- Photo 6 :** 1992. Gas control panel; at right ampère meters for EMP and box heater
- Photo 7 :** 1989. Upper box, rear side: glove box, box heater, valves, MH, CT
- Photo 8 :** 1991. Upper box, front side: box heaters, valves, main heater, glove box
- Photo 9 :** 1989. Upper box rear side : main heater, cold traps, motor-valves
- Photo 10 :** 1989. Upper box rear side : box heater, SS1, V7, ET, MH
- Photo 11 :** 1989. Upper box rear side : main heater, valves 4, 3, 5, box heater
- Photo 12 :** 1989. Upper box rear side : main heater, valves, box heater, cold trap
- Photo 13 :** 1994. Upper box rear side : magnetic trap 2, cold traps
- Photo 14 :** 1994. Upper box front side : box heater, motor-valve 3, V4, main heater
- Photo 15 :** 1994. Upper box front side : main heater, ET, V4, box heater
- Photo 16 :** 1996. Test section in the Ar-glove box
- Photo 17 :** 1990. Lower box rear side : first MT1
- Photo 18 :** 1994. Lower box rear side : last MT1
- Photo 19 :** 1994. Lower box rear side : mechanical filter, freeze valve, drain tank
- Photo 20 :** 1992 : Lower box front side: flow meter 1
- Photo 21 :** 1988 : Mrs. Neumann, NTG in lower box during installation
- Photo 22 :** 1988 : First filling, Mrs. Neumann and Mr. Bott, NTG on top of TRITEX, looking through the observation window
- Photo 23 :** 1996 : First filling, Mr. Kieser, kiki company, on top of TRITEX looking through the observation window
- Photo 24 :** 1996 : Mr. Kieser, kiki company; last filling. 'The loop is filled.'
- Photo 25 :** 1996 : Mr Horn, right and main author, FZK, last shut down of TRITEX. 'Happy?'

1. Introduction

An eutectic mixture of lead and lithium will be used for fusion reactor blankets. The Li concentration has to be controlled. During operation lithium will be consumed by transmutation to tritium, evaporation and reaction with air. On the other hand Bi, Po-210, Tl-202,204 and Hg-203, important for safety considerations, are formed from lead [1].

The Pb-Li phase diagram [2] shows several intermetallic compounds and eutectic compositions, Fig.1. At the melting point of the interesting mixture, 234°C, a liquid phase with 15.8 at.% lithium in lead is in equilibrium with a solid phase, consisting of LiPb and Pb. In a non-isothermal Pb-Li system including a solid phase (lowest temperature 234°C), the Li concentration in the molten phase will be always 15.8 at.%. Any excess Li will deposit as LiPb at the phase boundary. With an initially hypo-eutectic concentration a lead phase will be formed. Either lead is deposited, or Li dissolves from the solid phase [3] [4].

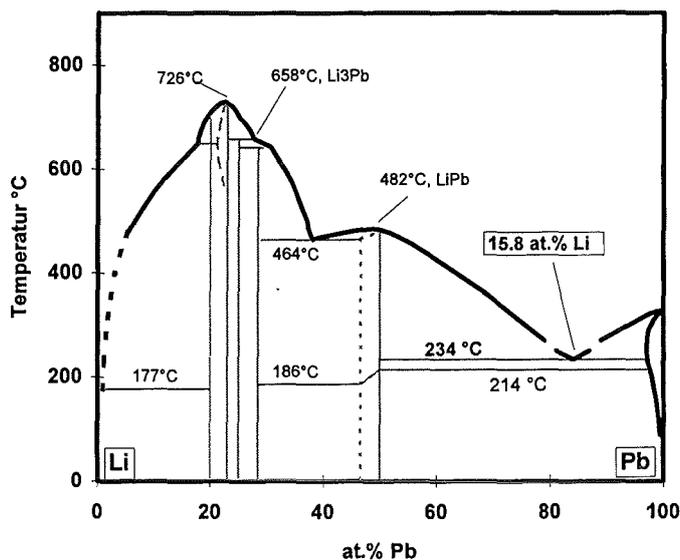


Fig.1 : Pb-Li phase diagram [2]

At start of the experimental program, always $\text{Li}_{17}\text{Pb}_{83}$ was written. However the mixture is no chemical compound. At an international liquid metal workshop in Karlsruhe in 1986, experts agreed to write Pb-17Li. In the mean time this is widely used. Actually however the eutectic mixture contains 15.8 at.% Li [2][3]. In non-isothermal systems like TRITEX with a solid phase for example in freeze valves, always Pb-15.8Li is circulating. Therefore **in this paper Pb-15.8Li is used**. The as-received material contained as ordered 17 at.%Li [3], only for this material Pb-17Li is written.

The loop TRITEX [5] was designed to develop methods for tritium extraction from molten Pb-15.8Li. The goals changed over the years to purification of the eutectic mixture and the study of corrosion products. It was ordered in December 1985 and delivered in summer 1987. TRITEX was operated between 1989 and 1996 in seven operation phases for 13.003 hours. Before a first operation with Pb-15.8Li many modifications were required. Between experiments the loop was modified depending on requirements. During and after a phase samples were taken from different positions and analyzed. After phase V, the loop was completely dismantled and investigated.

The Pb-15.8Li program in FZK ended by decision of the management after phase VII in 1996. The loop was again completely dismantled and all parts analyzed, especially for corrosion products. This report describes facility and operation of TRITEX. Results obtained with TRITEX are described separately [6].

2. Facility

2.1 Loop TRITEX

Chapter 4 shows photos of the facility and components.

TRITEX was a forced convection loop with 100 kg Pb-15.8Li circulating and about 20 kg in the drain tank. The loop was fabricated from ferritic steel 1.4922, last annealing of welds was done in the facility by heating to 550°C for one week. Connections of pipes were of VCR type with soft-iron gaskets. Larger flanges were not in contact with the molten mixture. Tubes of EMP, FM and MT were from Mo respectively TZM or from vanadium 99.5. Permeation membranes were from armco iron. Table 1 shows main data of the loop, Table 2 used materials, Fig.2 a flow sheet of TRITEX in operation phase VII.

Table 1 : Facility TRITEX

<u>Materials</u>	
main loop	steel 1.4922
some parts	TZM, V, armco iron
circulating Pb-15.8Li	80 to 100 kg
total inventory	ca. 120 kg
covergas	argon-6.0
heating concept	thermo boxes
temperature range	250 to 550 °C
flow rates	0.02 to 3.0 l/min (main pipes 0.2 to 30 cm/s)
inner diameter of main pipes	15 mm
length of main pipes	10 to 11 m
Pb-15.8Li wetted surface	1.2 to 1.5 m ²

Table 2 : Used materials

Structural materials in contact with the gas phase : mainly austenitic steel 1.4301, a few parts armco iron

Structural materials in contact with Pb-15.8Li

1.4922 : (different companies) About 1.2 m² wetted surface were from this steel. Own analysis in wt.% : Cr-12.1%, Ni-0.31%, Mn-0.53 %, Mo-0.54%, V-0.30%, Cu-0.07% and Si-0.37%. Last annealing of welds was done in the facility by heating to 550°C for one week.

1.6770 : 0.13 m², core of the EMP, similar to 1.4922, but only 2% Cr

1.4104 : 1.7 m², wire mesh of CT2 in Phase VI, similar to 1.4922*

vanadium 99.5 : Less than 160 cm² wetted surface. Mainly used in MT's and FM's, in phase IV and VII also in TS2. Analysis as specified, Kelpin or GoodFellow company

TZM : 0.13 m² wetted surface (EMP). It was used as tubes for EMP, MT, and FM's. TZM is an alloy with Mo-99.5%, Ti-0.5%, Zr-0.08%, Cr-0.02%. Chemically similar to pure Mo, but with a better workability. It was always used instead of pure Mo. Analysis as specified, PLANSEE, Reuthe, Austria

Fe-99.6 (armco-iron) : 0.19 m² wetted surface (TS). It was used for the test sections, which were heated under Argon, and for permeation membranes. Analysis as specified, GoodFellow and others.

Pb-15.8Li : The first Pb-17Li, used only in phases I and II, was obtained from Metallgesellschaft. It was not very clean. All other Pb-17Li was delivered by Metaux Speciaux company, together with material for the loop PICCOLO [7] about 600 kg. The own analysis of this material was : average 0.68 w.% Li (17 at.%) [3]; the elements Sb, Ag, Zn, Cd, Fe, Cu, Ni were all below 10 wppm, Bismuth average 35 wppm [3] [6].

Covergas : Ar-6.0, at inlet to TRITEX purified by OXISORB^R, Messer Griesheim company. The specified purity of <0.1 vpm O₂ and <0.5 vpm H₂O was confirmed in our laboratory.

* Used only at low temperature.

The concept of thermo boxes in two levels was used for heating. Nearly all components were in the upper level, the lower level thermo box contained only magnetic trap 1, flow meters 1 and 2, and the drain tank. Test sections and cold traps were in separate boxes. Test sections were located in an argon glove box and could be opened without draining the loop. EMP and freeze valves were outside of thermo boxes.

Originally there were only thermo boxes for the upper part, photo 1. Trace heating and thermal insulation in the lower part was not sufficient for heating, an additional lower level thermo box was installed. Thermo boxes have the advantage of homogeneous temperatures in the loop and easy replacement of parts. A disadvantage is that in case of an Pb-15.8Li leak all the eutectic mixture may flow out, while in case of trace heating the molten mixture will solidify in the thermal insulation and often seal the leak even if the pipe is broken, Fig.3, Fig.4. For safety reasons, therefore, a large pan was below the loop to collect any leaking Pb-15.8Li.

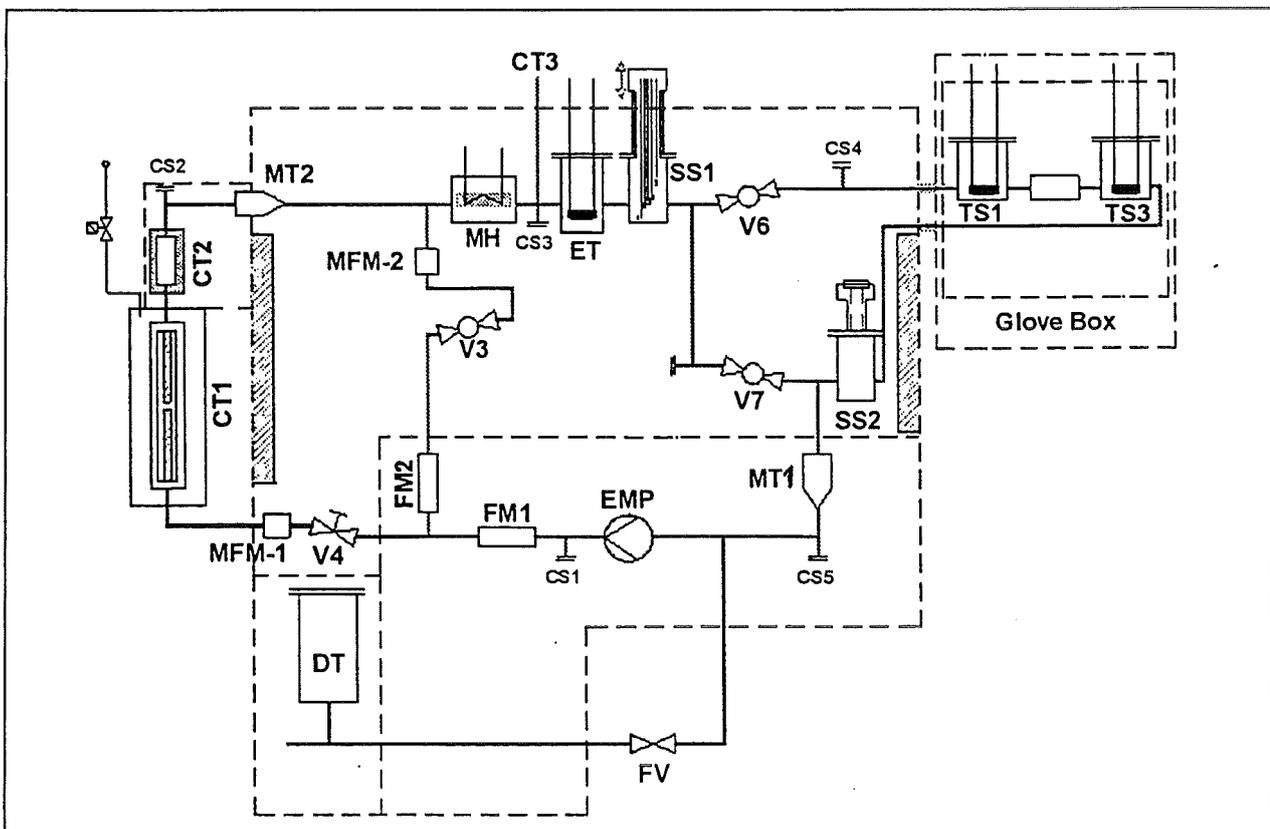


Fig.2 : Flow sheet of TRITEX, 1996

Thermo boxes got heated by two box heaters and the main heater, each 3000 Watt. Additional smaller heaters were at TS, CT's and DT. The facility was located in a radioactive controlled laboratory with five times exchange of room air per hour. All heat, therefore could be dispatched to the room. Especially on hot summer days the room temperature rose to 40°C and some special precautions were needed for the electronics. Also some parts outside of thermo boxes had to be cooled by ventilators, photos 1 and 16.

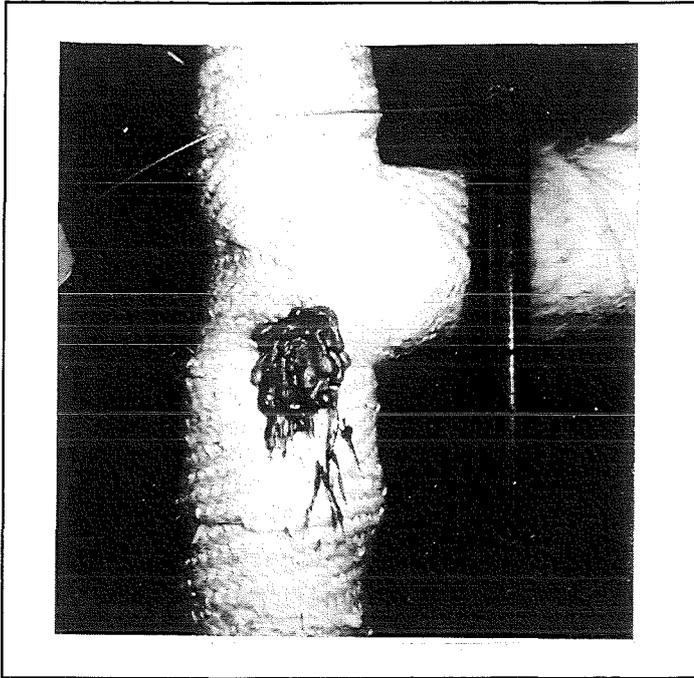


Fig.3 : Leak of a pipe with trace heating, CT bypass, April 4, 1988

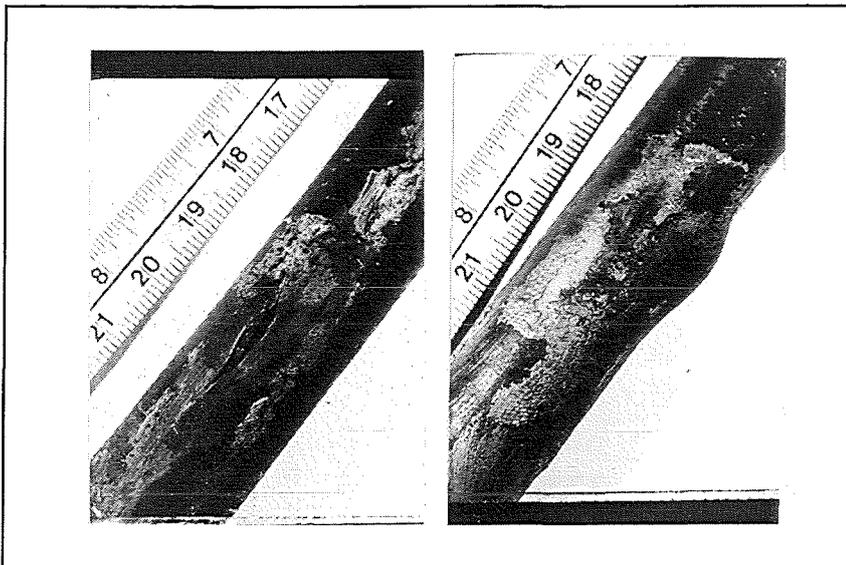


Fig.4 : Ballooning and broken pipe of Fig.3, CT bypass, April 4, 1988

In the main circuit the eutectic mixture was flowing

EMP - FM1 - FM2 - V3 - MFM2 - MH- ET - SS1 - V6 - TS - SS2 - MT1 - EMP.

Between main heater and expansion tank was cold trap-3. Valve-7 was required for a complete drain. In the cold trap bypass the mixture flowed from behind FM1 to

V4, MFM1, CT1 and 2, MT2

and back to the main loop. The cold trap bypass was controlled with V3 and V4. Different other flows were also possible, for example :

The shortest circuit was

EMP - FM1 - FM2 - V3 - MFM2 - MH- ET - SS1 - V7 - MT1 - EMP.

The longest circuit was

EMP - FM1 - V4 - MFM1 - CT1,2 - MT2 - MH - ET - SS1 - V6 - TS - SS2 - MT1 - EMP.

Operation without TS and SS2 was possible by closing V6 and opening V7.

2.1.1 Electro magnetic pump

The electrical part of the EMP from INTERATOM company was designed for sodium. This pump was actually too big for TRITEX. Because of a large duct diameter the liquid metal had to be pumped in an annular gap. The core from steel 1.6770 was 775 mm long with a diameter of 52 mm. This steel is similar to the loop steel. The Pb-15.8Li gap was only 2 mm wide. The outer tube was from TZM, fabricated by PLANSEE company and welded on both ends to steel 1.4922. This pump failed during startup. The TZM weld cracked over the whole length of the tube, Pb-15.8Li flowed out, Fig.5, Fig.6.

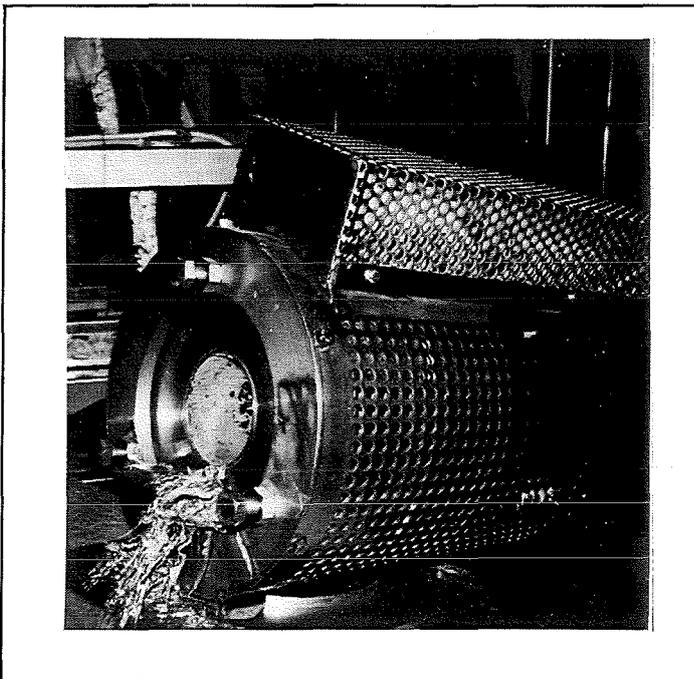


Fig.5: EMP after removing the pump duct, April 19, 1988

A new EMP tube was fabricated by PLANSEE company. It consisted of an austenitic tube, welded on both sides to steel 1.4922. Inside was a TZM liner with labyrinth seals to 1.4922, Fig.7. This pump was cut open after 9860 hours of operation (phase V) and investigated [6]. Then a pump with a gap of 5 mm was installed to reduce pressure drop.

Except for the freeze valves, EMP was the only part outside of any thermo box. Before filling of the loop it was heated by pump power. During operation the electro magnets got as hot as 400°C. It was amazing that all electrical parts withstand 13000 hours operation time.

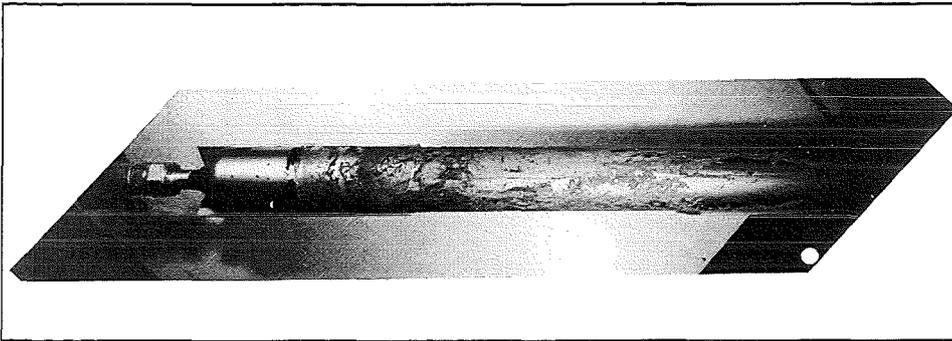


Fig.6 : Crack at the TZM tube, April 19, 1988

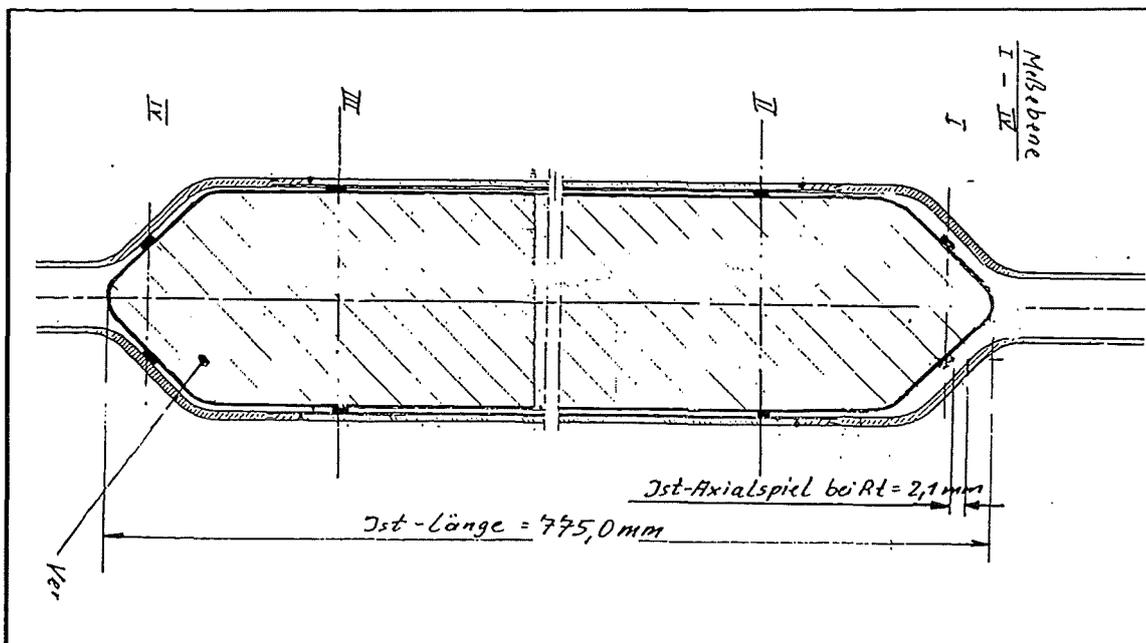


Fig.7 : EMP with TZM liner, 1989

2.1.2 Mechanical Valves

There were seven mechanical liquid metal valves in the loop as ordered. V2, V3, V6 and V7 were motor valves. Valve 1 belonged to the first filling equipment and was removed 1989. Valve 2 between loop and drain tank was replaced 1991 by freeze valves. In 1992 also valve 5 was removed. Only four valves were sufficient to control flow rates in main loop and cold trap bypass; from these valve 7 was needed for a complete drain.

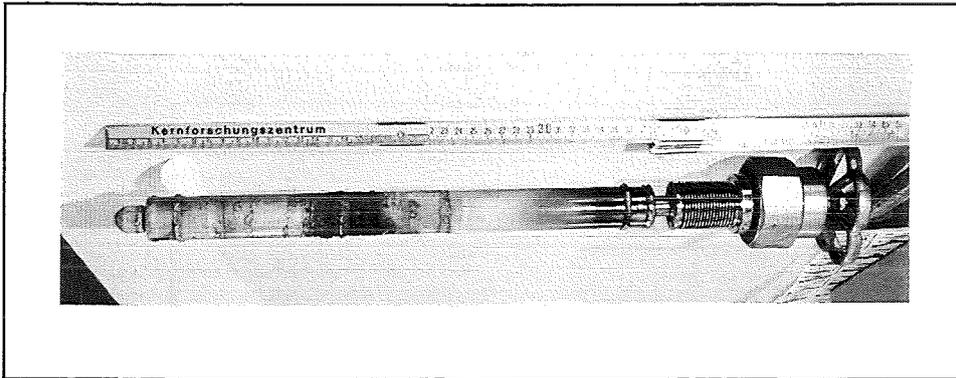


Fig.8 : Valve 5 after phase V, it was located behind CT2, 1993

Mechanical valves were engineering standards from Stöhr company. With the exception of the valve cones, all parts in contact with the liquid metal were made from steel 1.4922. Cones were from TZM. The space above Pb-15.8Li was flushed with argon-6.0. Sealing to the atmosphere was out side of the thermo box with austenitic steel metal bellows. The drive mechanism was also outside, the longest stem and tube were 1.2 meter for V2. Fig.8 shows the photo, valves can also be seen at several photos in chapter 4.

2.1.3 Freeze valves

Because of oxide and or corrosion product particles, mechanical liquid metal valves were not completely tight. This was no problem except for valve 2. It was leaking with about 1.4 kg/day to the drain tank, equivalent to a liquid metal level drop of 6 mm/day. During phases I to IV the loss was compensated by controlling the gas pressure in the drain tank. In 1991 valve 2 was replaced by freeze valves, two in parallel. These were simple pieces of a pipe with a solid plug of Pb-15.8Li in the center. Compared to mechanical valves, freeze valves were slow. Depending on loop temperatures 15 to 40 minutes were needed for opening, closing needed less than 10 minutes.

Because of a large volume change of Pb-15.8Li during freezing or melting, special measures were required to avoid stress. The freeze valve center part had forced air cooling to start freezing there and proceed to outer sides. More complicated was remelting. Both ends of a freeze valve contained molten eutectic, one side heated from the loop, the other side from drain tank. The lowest temperature was in the center. A wedge shaped block of stainless steel, heated only from the ends, moved the molten zone slowly to the center. Fig.9 shows a freeze valve.

While the melting point of the eutectic is 234°C, freeze valves must have a possibility to be heated at least to 482°C, the melting point of LiPb. As described with lithium [4][6] LiPb get deposited at the liquid-solid phase boundary. In TRITEX it was usually dissolved in the molten mixture from both ends at lower temperature. If the deposited LiPb plug, however, would be too long, 482°C may be required for remelting.

2.1.4 Drain tank

The drain tank was in the lower level thermo box, photo 19. For a faster drain and to avoid thermal shock DT was heated during operation of the loop to 350°C. Liquid metal samples were taken from DT in all operation phases through a long experimental plug under argon.

The covergas supply was independent from that of the loop. Similar to the first EMP tube, a level meter of TZM in DT failed in the pre-phase, causing heavy oxidation of the molten mixture.

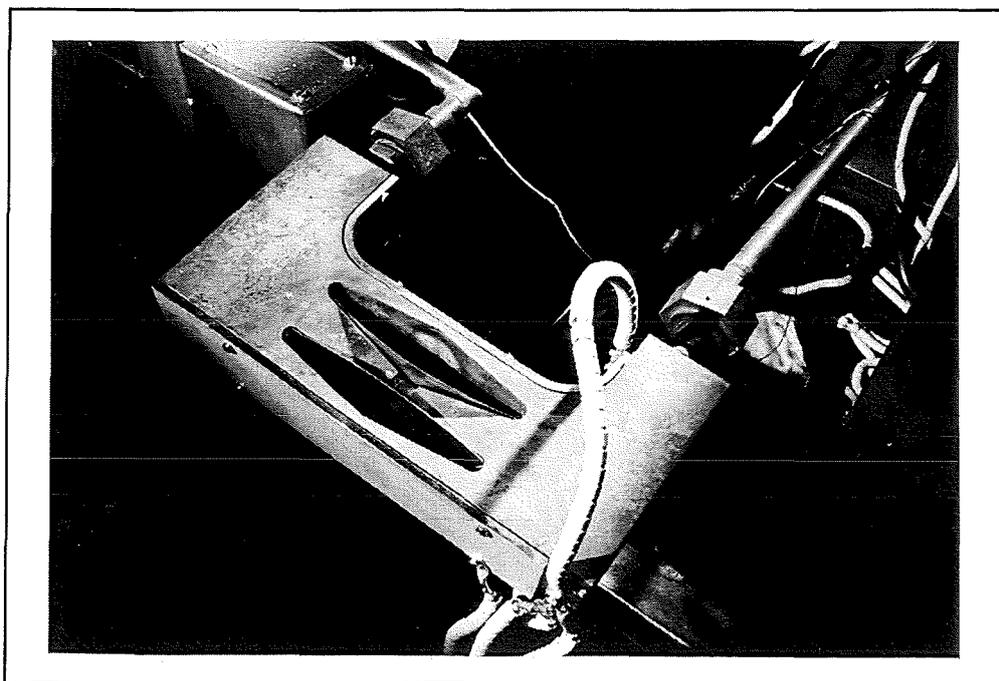


Fig.9 : Freeze valve

2.2 Covergas system

Liquid metal-covergas interfaces were at 10 positions in the Fig.2 loop, four of these in valves. Sintered metal filters were at inlet and outlet of a component. All covergas spaces were interconnected, but with the possibility to control flow rate and pressure for each separately. No gas flow was in valves. Mostly covergas spaces were closed and kept at a pressure of 1.3 to 1.5 bar. Different flow resistances caused different pressures and LM levels in each covergas volume. To check flow resistance's, pressure differences between a recipient and covergas volumes were measured (chapter 2.4.2).

covergas transport

Except in some experiments, covergas spaces were closed. There was clearly a transport of argon covergas between different covergas volumes, causing pressure changes there. Because of low Ar solubility [8] probably small bubbles are transported. From time to time the EMP had to be stopped and all gas spaces shortly connected to each other for pressure equilibration. The gas transport was not constant. Pressure equilibration had to be done after flow or temperature changes within a few hours, at constant operating conditions however usually after several days.

TRITEX was build for tritium extraction. Mainly H_2 or D_2 were used in experiments. Loading the liquid metal with these gases was done over the gas phase in ET, requiring additional gas supply possibilities. In 1990, a 1036 cm^2 permeation membrane was installed in ET, Fig.10. It was a 6.6 meter long armco iron tube, 5mm diameter with 0.5 mm wall. Unfortunately a part of the membrane was in the gas phase. With pure D_2 in the membrane the permeation rate was so high that the ET covergas volume had to be opened and operated with flow-through. Otherwise the gas pressure was rising, pressing the eutectic out of ET. At 383°C the pressure rise in ET was 4 mbar/h. Except for some tests, Pb-15.8Li was loaded with hydrogen isotopes through this membrane.

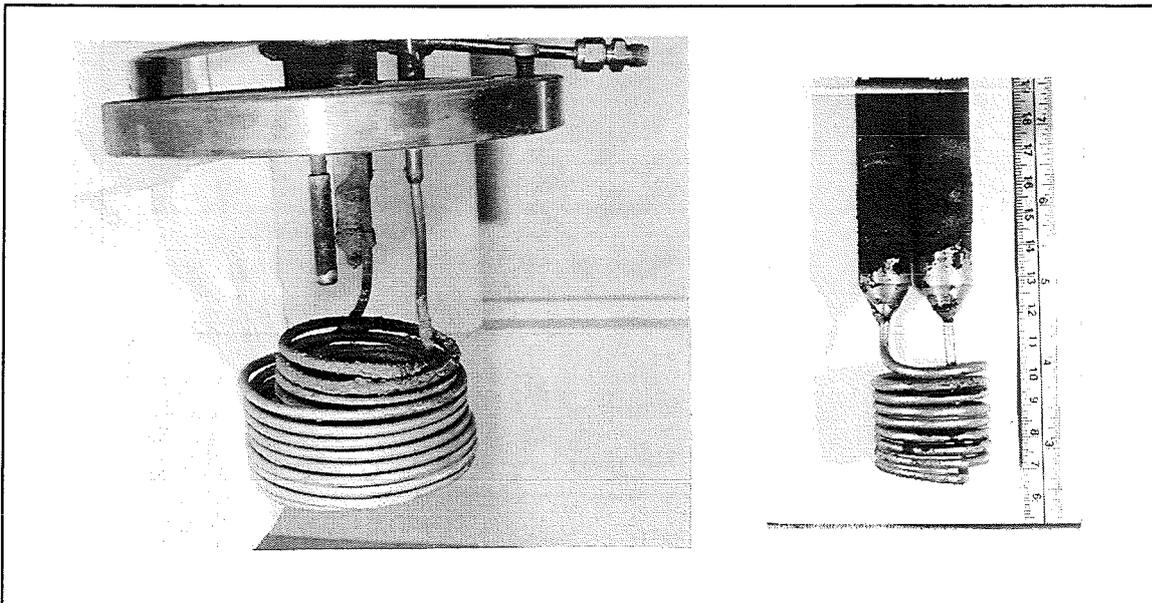


Fig.10 : 1036 cm^2 permeation membrane of armco iron in ET, 1990

Fig.11 : 100 cm^2 permeation membrane of armco iron in TS1 and TS3, 1989

2.3 Test facilities

Most test facilities can be seen in chapter 4, more details are given in [6].

2.3.1 Expansion tank

ET was originally a tank without installation with a long 40mm diameter tube to the outside of the thermo box. Because of many liquid metal-covergas interfaces, actually no expansion tank was needed. ET added just one more experimental device to TRITEX. The 1036 cm² permeation membrane was installed in ET, Fig.10.

2.3.2 Sampling station

Two identical sampling stations, smaller tanks than ET, were located before and after the test section. SS1 was used for level indicators, an observation window was on SS2. In each operation phase Pb-15.8Li samples were taken, mainly from SS2. This was done under argon to avoid oxidation of the molten metal.

2.3.3 Observation window

To see the liquid metal surface, a quartz observation window was on top of SS2. It was impressive to see a surface like mercury. Sometimes also oxides were seen, disappearing usually to crusts at the wall. But also sparkling crystals could be seen at the surface. An attempt to sample these failed. Probably LiPb or corrosion product particles were floating at the surface. But mostly mirror, oxides and crystals were together at the surface, typically 50% mirror, 25% oxides and 25% crystals. Larger flow rates could be seen, in this case a mirror like surface was visible.

2.3.4 Test section

Test sections were located inside of an argon glove box. This allowed modifications without drain and cooling down the loop. A TS was similar to a sampling station. At the beginning there was one empty TS. In operation phase IV, three identical TS were installed. Deuterium partial pressures were measured before and after a getter bed. The first and the third TS contained each an 100 cm² permeation membrane, Fig.11. In the middle a basket filled with vanadium wire was inserted. In phase VII TS2 was replaced against an helical chamber, filled with vanadium with a large surface. Fig.12 shows a photo of a part of TS2 before welding. This arrangement is included in the loop of Fig.2 and shown in photo 16.

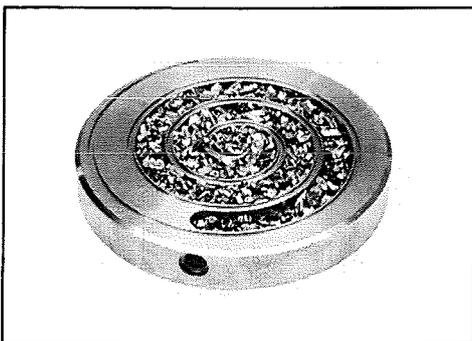


Fig.12 : Operation phase VII, TS2 with vanadium filling

2.3.5 Cold traps 1 and 2

Cold traps are standard equipment's to control oxygen concentrations in sodium systems [9]. All or a part of the main flow is passing through an area with low temperature, excess oxygen is deposited. The oxygen solubility in Pb-15.8Li is extremely low. But cold traps can also be used to deposit other impurities and corrosion products. During corrosion elements from the metal surface get dissolved in the molten metal. If the solubility of an element is exceeded, it will precipitate. While corrosion takes place mainly in areas with high temperature, deposition will occur in colder parts, that means in cold traps. The efficiency of a cold trap depends on several parameters. Solubility functions and super-saturation are different for each element or compound¹, surfaces for deposition and fluid dynamic parameters are very important, a longer delay time for deposition is required. Also, often cold traps act as filters for particles.

To keep the molten eutectic as clean as possible, cold traps were installed in TRITEX from the beginning. Different kinds of cold traps were used in different operation phases. All consisted of two parts. CT1 was the cooler, CT2 was at the lowest temperature for deposition. In addition a diffusion type cold trap with a solid phase was tested.

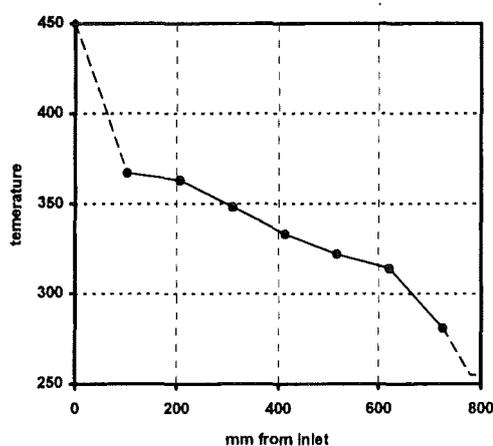


Fig.13: Temperature profile in CT1 (cooler), 1990

Operation phases I to V

The cooler was an 0.72 meter long 8 mm wide annular gap, cooled at cooling fins by air. Heat was dispatched to the room. Heating was also from outside. About 5000 cm² steel 1.4422 were in contact with molten Pb-15.8Li. The temperature profile is shown in Fig.13 and photos 7 and 9 in chapter 4. The flow velocity in the cooler was 13 times lower than in main pipes.

¹ Mostly compounds or alloys will precipitate, [6]

Behind the cooler was CT2 for deposition, also cooled via cooling fins. Pb-15.8Li was flowing from top to bottom through a basket with metallic rings. In phase I and II these were from TZM, later from steel 1.4922. The deposition surface was about 1500 cm². Because of a large cross section the flow velocity in the basket was 40 times lower than in main pipes, the delay time hence long.

Operation phase VI

New cold traps-1 and -2 were installed for phase VI, photo 13. The cooler CT1 consisted again of the annular gap, but had also 6 chambers filled with wire mesh of ferritic steel [6]. In each chamber was a thermo couple, allowing to correlate deposits and temperatures. Size and temperature profile was comparable to CT-1 described before. The new CT1 was heated by an heat rod in the center and cooled by air from outside. No cooling fins were needed.

A special cold trap 2 was requested by FZK institute IATF for operation phase VI. To check a model for corrosion product deposition, about 1.7 m² deposition surface at low temperature were provided behind CT1 in form of wire mesh. The material was steel 1.4104, similar to that of the loop. The difference between in- and outlet temperature was less than 1°C. Heating was done from outside, no cooling was required. In the center was a thimble with 10 thermo couples. Fig.14 shows the wire mesh packing.

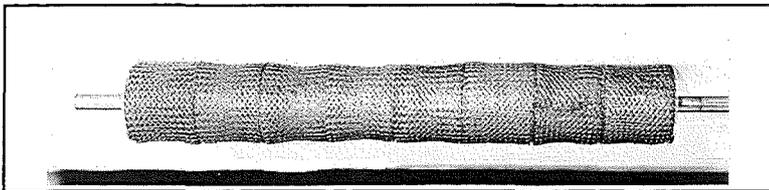


Fig.14 : Wire mesh packing in cold trap 2, phase VI, 1994

Operation phase VII

No deposits were found in the new cold traps after phase VI, but always a large fraction of corrosion products were at boundaries liquid metal-steel-covergas. Cold trap-1 was identical to that in phase VI. A new CT2 with a large boundary was installed in phase VII, operated isothermally as before. The liquid metal-steel-covergas line was 750 cm, compared to CT2 in phase I to V of only 50 cm, and that the main loop of 170 cm. Fig.15 shows this CT2. Unfortunately phase VII was short and the whole program ended.

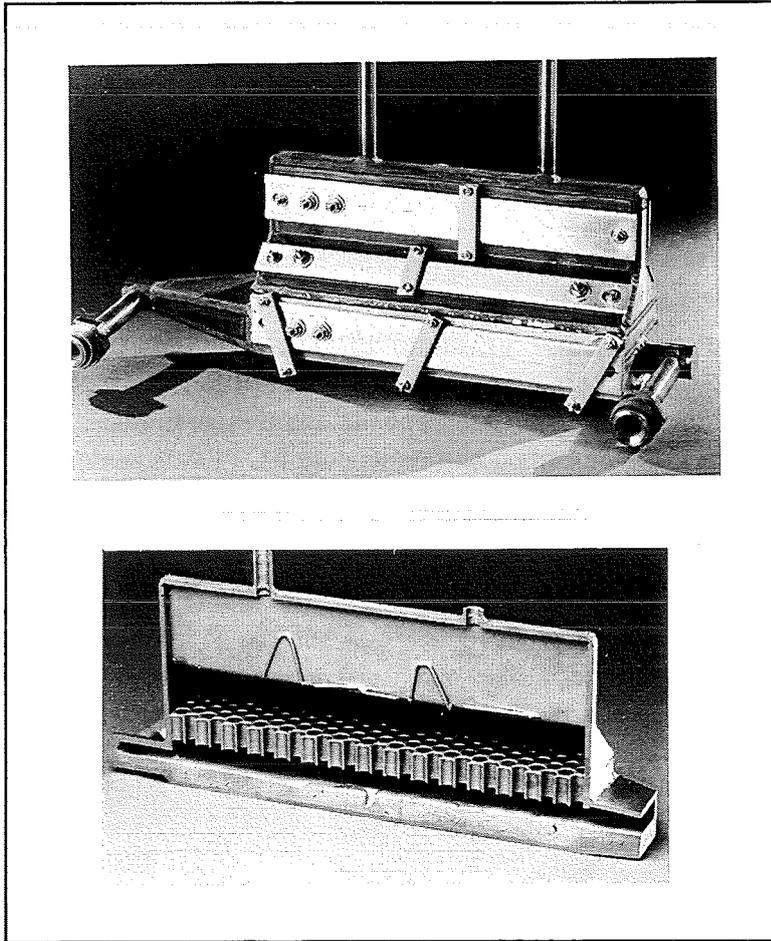


Fig.15 : Cold trap 2 in phase VII, closed with heater and cut open, 1996

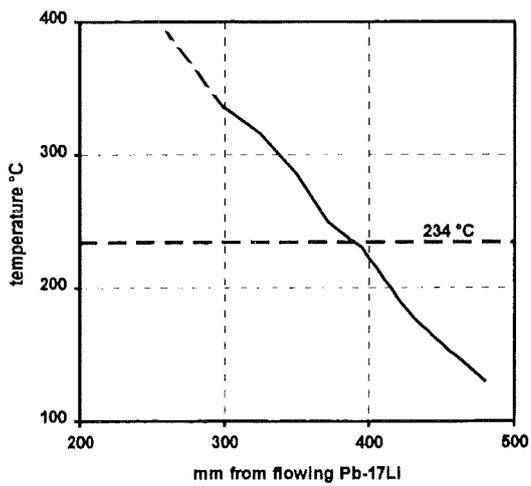


Fig.16 : Temperature profile in cold trap 3

2.3.6 Cold trap 3

CT3 was installed in operation phases VI and VII. It was a diffusion type cold trap, simply a tube behind the main heater going out of the thermo box. As in freeze valves there was a liquid/solid Pb-15.8Li boundary at 234°C, the lowest possible temperature in a molten Pb-15.8Li system. Deposition of excess lithium, but also of impurity elements like Bi was expected at this position [4][12]. The temperature profile is shown in Fig.16.

Actually there were always a kind of CT-3 in TRITEX. A long 'residue drain' tube below the drain tank with a solid phase was installed until phase V. Then freeze valves acted as CT3. Freeze valves however could not be analyzed, they were drained to the drain tank.

2.3.7 Magnetic Traps

A fraction of formed corrosion product particles is ferritic and can be deposited in magnetic fields. There was one magnetic trap, MT1 in the loop as delivered, located before EMP. A TZM tube of 15 mm outer diameter, welded on both sides to steel 1.4922, was fixed between the tips of a magnet with 65 mTesla (650 Gauss). Fig.17 shows the magnetic field intensity, measured with a Hall meter. The temperature influence was small. However flow meters were of similar shape, acting also a magnetic traps. And finally a strong magnetic field was in the electro magnetic pump.

Because of the problem with the TZM tube of EMP, also for MT1 an austenitic steel tube with TZM-liner was used in phases I and II. In phases III to V, a vanadium tube was used for the MT. The first magnetic traps were not effective. In phase V, a flat vanadium capsule with a ferritic core was used, in phase VI and VII the same kind of capsule with a larger ferritic core. The vanadium part was 100 mm long, 35 mm high and 18 mm in depth. The magnet was located near the inlet. Photos 17 and 18.

In phase VI and VII, a second magnetic trap was installed at the outlet of the CT2. An empty flat vanadium capsule of 80x60x18 mm was used, a flat magnet of about 100 mTesla was fixed on top of this capsule, photo 13.

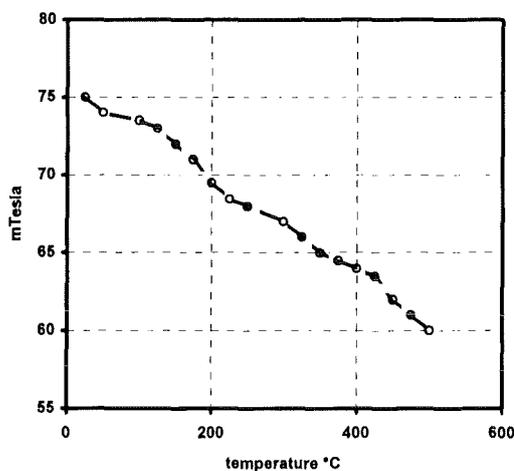


Fig.17 : MT1, magnetic field intensity, measured with a Hall meter, 1993.

2.3.8 Corrosion samples

Corrosion samples were placed into the flowing eutectic at 5 positions of the loop, marked in Fig.2 by 'CS'. Only loop materials were used, three samples were fixed to one holder, Fig.18. Always a thermo couple was placed near the samples. They were not for corrosion studies, but merely to learn about the condition of loop materials. For this some were replaced after phase III and V.

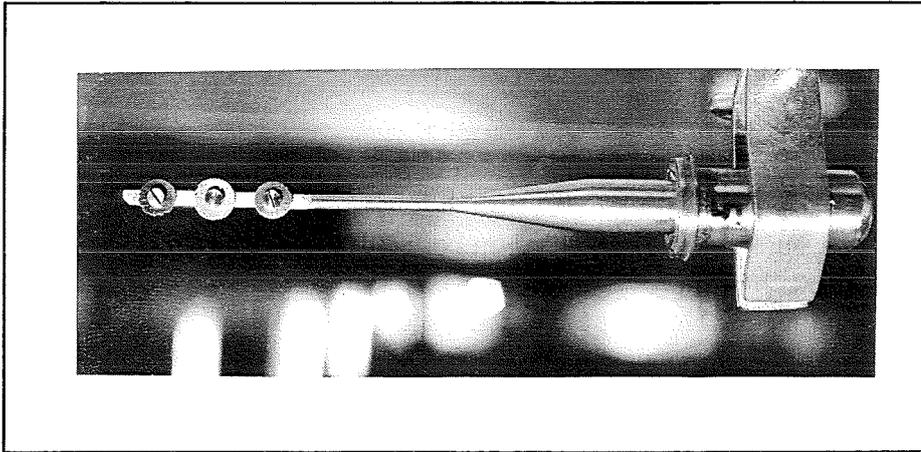


Fig.18 : Holder with corrosion samples

2.4 Measurements and Control

2.4.1 Data registration, temperature and gas pressure

TRITEX was originally controlled by an ELZET controller with EPROM's and standard manometers. In 1990 a Philips Data-Logger with INDAS software and 386-Compaq PC were installed, separating completely control and data registration. In 32 channels temperatures, flow rates, EMP-power and gas pressures were registered. Photos 1,2,3 and 5.

The ELZET system was not flexible enough. Temperatures could not be kept constant, causing pressure differences in covergas spaces. For example the main heater MH caused fluctuations even in TS of $\pm 15^{\circ}\text{C}$ and $\pm 4\text{mbar}$. After phase IV, 19 Philips KS-40 and one KS 4290 temperature controller were installed (1991), photo 4. A constant temperature around the loop was obtained, Fig.19. The concept of thermo-boxes certainly was helpful, prevented on the other hand different temperatures for example in SS1, ET and SS2.

Except for those in cold traps, thermo couples in TRITEX were not calibrated. The deviation from true values was $\pm 2^{\circ}\text{C}$ at operating temperature, generally a sufficient accuracy.

In 1990, two absolute and eight differential capacitive meters type ARI 210 resp. ARK 200, Mannesmann H&B company, were installed for gas pressure control, sensitivity ± 1 respectively $\pm 0,1\text{ mbar}$, photo 6. Differential pressures got measured against an isothermal recipient.

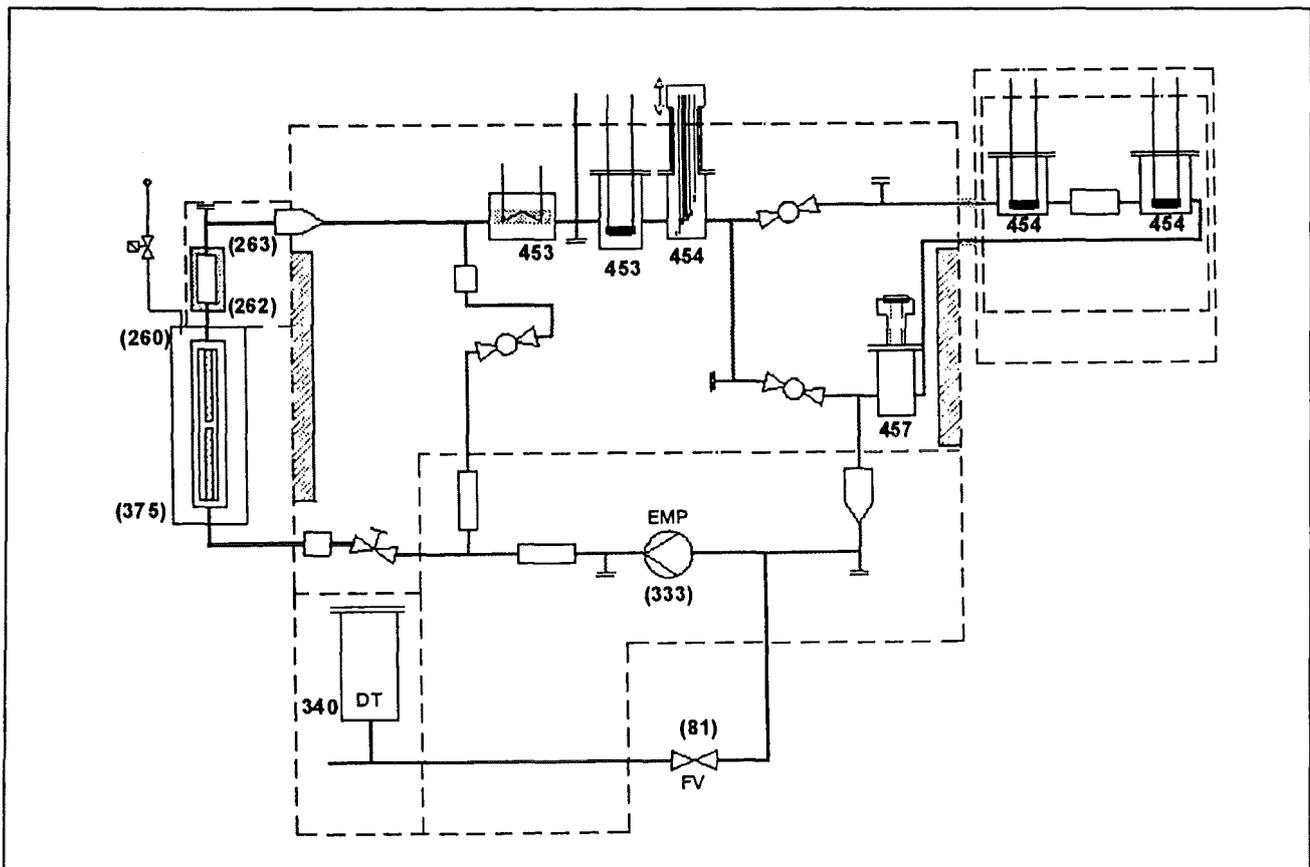


Fig.19 : LM temperatures in TRITEX in $^{\circ}\text{C}$. Phase VI, Sept. 27, 1994, 11.00.
Values in parentheses are from structure material.

2.4.2 Flow measurements

LM flow in TRITEX was always turbulent. Flow rates were changed mainly by changing EMP power. Motor-valve V3 was used to control the cold trap bypass. Larger flow rates could be seen at the observation window.

Electro magnetic flow meters

Two FM's with TZM, later V ducts, photo 20, were calibrated by INTERATOM for flowing sodium. Values for the eutectic mixture were calculated. This calculation was checked in the pre- phase by flash heating (April 8,1989):

At a length of 15mm, a power pulse of 5 kW was given to the wall for 0,1 or 0,2 s. Three in-Pb-15.8Li thermo couples, arranged over the distance of 200 mm, registered the temperature pulse. Flow rates were calculated from travel time and pipe diameter.

Found flow rates were a factor of two lower than expected. The measurements showed, that the original planned flow rates of 5 l/min were not possible because of too high liquid metal level differences in components and a too high temperature in electrical parts of EMP. Also, a better flow meter calibration was needed.

Flow resistance and gas pressure

One possibility to measure flow rates was the measurement of flow resistance's around the loop, given with closed covergas spaces by pressure differences. The pressure is highest in V3 or V4 behind EMP, and lowest in SS2 just before EMP. The flow resistance was calculated during construction of TRITEX. Fig.20 shows a good relation between gas pressure and flow rate in phase VI. In this example the cold trap loop was already partly blocked. The maximum pressure differences between V3 and SS-2 without cold traps and with open V3 and V6 at 457°C and one l/min flow rate (phase V) was 35 mbar.

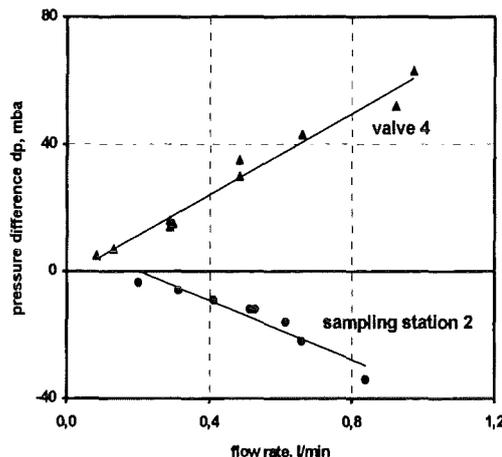


Fig.20 : Gas pressure differences V4 and SS2 against inert gas recipient. Temperature distribution of Fig.19. V3 closed, full flow over partly blocked cold traps. Flow rates measured with MFM 1.

Pressure differences in mbar are nearly equal to liquid metal level differences in mm. Higher flow rates than in the example of Fig.20 would result in not acceptable level differences in the loop. Δp values, therefore, were used for flow measurements and as an indicator for liquid metal levels. Pressure differences reflect level differences against an average level with EMP=0 and open gas volumes.

Mass flow meters

Electrical signals and gas pressure differences depend on temperatures. Two mass flow meters, MFM were installed before phase VI. Behind valves 3 and 4, the eutectic was heated by a 267 Watt heating cartridge between two thermo couples. Cartridge and thermo couples were in flowing Pb-15.8Li, the length of the device 100mm.

From temperature increase, specific heat and density of the eutectic [10], mass flow rates were calculated. Temperature differences were small, therefore heat loss to the thermo box negligible. For example, at 450 °C the temperature increased only by 9.2°C for a flow rate of one l/min. Flow rates from 0.05 to 1.5 l/min could be measured.

$$P = m' * \Delta T * Cp(T)$$

P in Watt = J/s, m' in g/s, T in K, Cp in J/g*K.

Typically for 450 °C, the flow rates v in (l/min) are given by $v = 9.23/\Delta T$.

MFM's were mainly used for calibrating other measurements. With the now installed 4 flow meters (each 2 FM and MFM) and pressure differences, flow resistance's in different parts of TRITEX could be checked. This was helpful to find blocked positions.

2.4.3 Liquid metal level indicators

Capacitive liquid metal level indicators were originally installed in DT and ET. They were of TZM and failed in the pre-phase. One five point electro-mechanical LI in SS1 was installed to check the level there, Fig.21. Until phase V, LI signals were used to control the gas pressure in DT, compensating the V2 leak.

2.5 Safety

Two kinds of safety were considered : safety of operation and safety for experiments. The second one will not be discussed in this report.

A safety report was written in 1989. It was relatively short. VDI and VDE regulations were taken into account during construction. Pb-15.8Li is not burnable and reacts only moderate with air or humidity [11]. However the heat content of more than 100 kg molten mixture at max. 550°C is very large, causing a fire hazard. To collect any leaked-out liquid metal a stainless steel pan was placed below the loop, 1800*1380*50 mm. This pan had no contact to burnable materials. Only because of the pan slow freeze valves could be installed.

Originally emergency drain was performed if several limit values were reached, including temperatures, gas pressures and liquid metal levels. With valve 7 open drain was fast through V2, causing heavy splashing in the drain tank. Because of too slow gas supply, a fraction of the eutectic remained in the lower part of the loop, e.g. in EMP. No emergency drain was provided any more after installation of freeze valves.

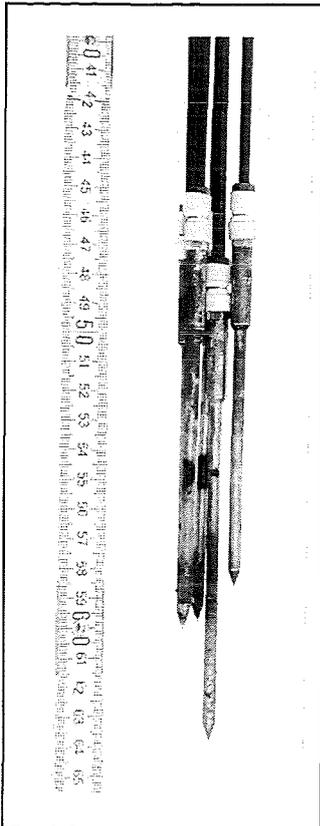


Fig.21 : Five point level indicator, 1989

In case of a leak finely distributed hot Pb-15.8Li may be formed. When using water to extinguish a fire it will react as any hot metal and form hydrogen. To avoid this and to avoid steam explosions, danger signs were set at the entrance of the room not to use water in case of a fire, fire extinguisher were water free.

Other systems like the glove box or the covergas system needed no special safety features. An exception was hydrogen (H_2 or D_2) in experiments. Usually non-burnable mixtures of less than 10 vol.% hydrogen in argon were used. Tests with pure hydrogen were performed only under permanent observation by an operator. The only used radioactive nuclide was tritium. No special precautions were needed, TRITEX was located in a radioactive controlled laboratory.

2.6. Operation

Even if only 3 persons were concerned with operation, a handbook and several procedures were written. Pressure equilibration, described in chapter 2.2, was one procedure. Filling and draining TRITEX were two others.

2.6.1 Filling the drain tank

TRITEX was filled three times with fresh eutectic mixture.

For the pre-phase a special filling device was used to load DT with about 120 kg Pb-17Li, Fig.22. Argon pressed the molten mixture from the device into DT, any oxides were floating on the surface and kept back. After the pre-phase, loop surfaces were wetted by the eutectic and came in contact to air at room temperature. To avoid small amounts of oxides was not so important any more. Before phase I and again before phase VI solid bars of Pb-17Li were placed in DT, the surfaces cleaned by machining, Fig.23.

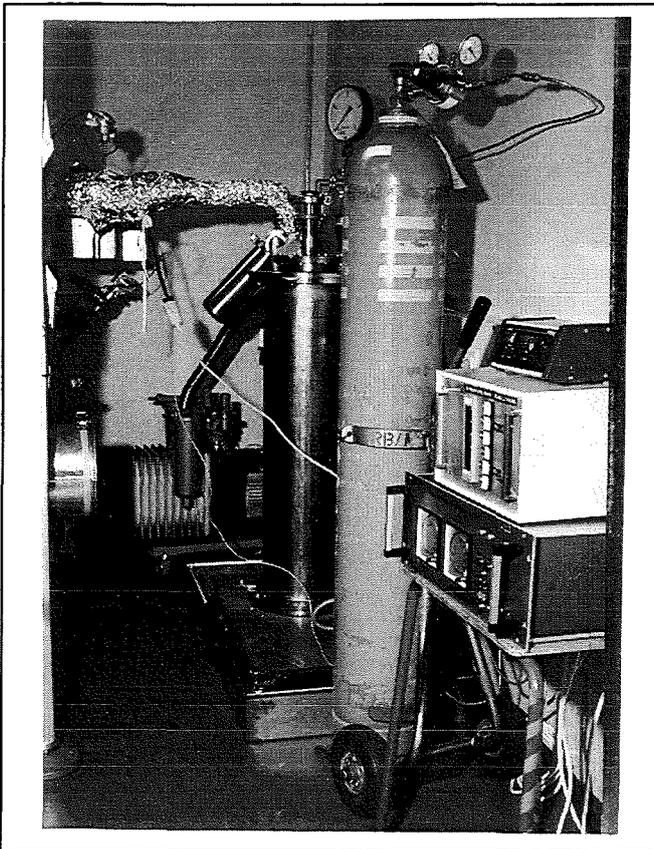


Fig.22 : Filling equipment for pre-phase, March 3, 1988

DT was closed, evacuated and checked for leaks. Leak rates of 10^{-7} mbar*1/s were acceptable. When heating DT, a kind of thermo-analysis could be seen. At 230°C, just below the melting point of 234°C, the temperature rise stopped for about 3 hours, Fig.24. During cooling, the temperature stopped for less than 2 hours, again at 230°C. The thermocouple was in the center of DT, it was controlled after dismantling and found to be correct within $\pm 1^\circ\text{C}$.

The next step was always vacuum degassing of the mixture at 350 to 400°C. Degassing was controlled by pressure rise, it needed several days. Considerable amounts of gases were entrained in form of bubbles.² Larger bubbles were formed with voids during solidification, causing splashing during melting. Small bubbles, most of micron size, were released over a long time.



Fig.23 : Pb-17Li bars in DT before closing, Nov.16, 1993

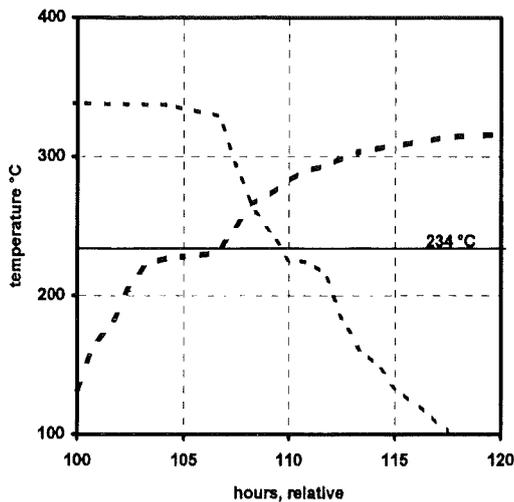


Fig.24 : Heating and cooling of 120 kg eutectic mixture in DT, 1991

² Attempts to measure Ar solubilities in Pb-15.8Li failed [8].

2.6.2 Loading the loop

At first leak tests were performed by two ways. When ever possible a He-leak detector (Leybold UL-100) was used, especially for localizing. However, deuterium from experiments caused problems. It get released from structural materials over a long time. He and D₂ have both M=4, the leak detector could not distinct. Pressure rise after evacuation, in some cases also pressure drop with the loop at more than 1.5 bar were mostly used for leak detection. While single leaks were seldom higher than 10⁻⁷ mbar*s, the whole loop was in the range from 5*10⁻⁶ to 1*10⁻⁴. Then TRITEX was heated to about 400 °C. Before any filling, leak test were done again. Before valve 2 respectively a freeze valve was opened, both loop and DT were evacuated. Rising the gas pressure in DT pressed Pb-15.8Li into the still evacuated loop. Similar to the filling device any oxides remained floating in DT. To avoid splashing filling was done slowly within 20 minutes, Fig.25. Temperature differences between the rising eutectic and components of the loop allowed to follow the filling, entry of liquid metal in SS2 could be seen through the observation window, and finally level indicators showed the filled loop. The numbers in Fig 25 show the status of filling.

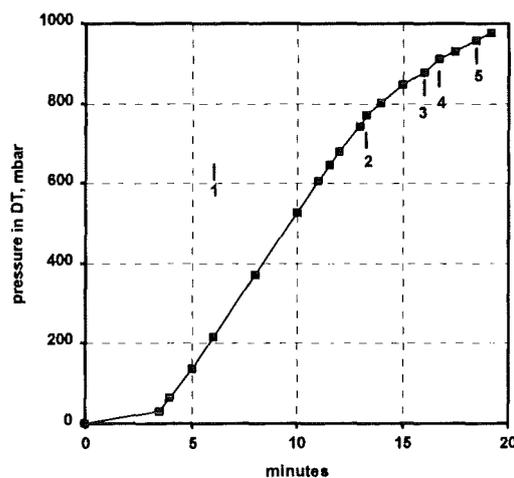


Fig.25 : Filling of TRITEX in phase VI. Filling status : 1=EMP, 2=CT1 inlet, 3=CT2 outlet, 4= molten eutectic visible in SS2, 5=loop filled.

Sometimes the liquid metal level had to be risen, for example to allow higher flow rates. This could be done as before by filling from DT. Two times however molten Pb-15.8Li was added at TS in the glove box. After a larger loss at beginning of operation more than 20 kg were filled in by this way.

2.6.2 Drain procedure

At EMP=0, all covergas spaces were opened as the first step. With DT at filling pressure, V2 respectively FV was opened. Draining the loop was done by reducing the pressure in DT. When parts of the facility were plugged, the pressure in the loop could be increased.

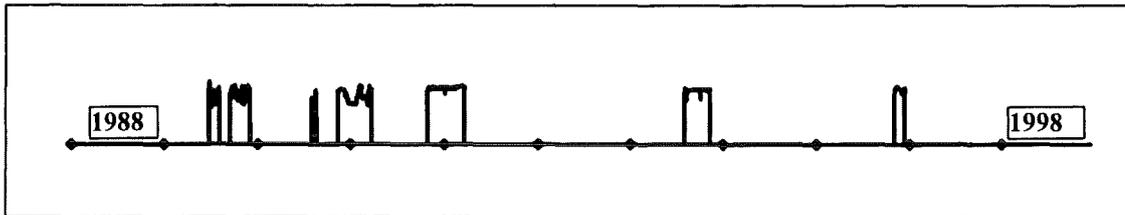
Whenever possible, drain was done at higher temperatures. But there remained still a film of Pb-15.8Li on surfaces. At 477°C drain temperature (phase V) $87 \pm 61 \text{ mg/cm}^2$ were found. The wide scattering shows that there is not a homogeneous film. This effect has to be considered when draining a system. In TRITEX nearly 1 kg Pb-15.8Li were not drained, 1% of the inventory.

(In batch type experiments, $45 \pm 12 \text{ mg/cm}^2$ were found at walls.)

3. Operation history of TRITEX

This chapter gives a short description of loop operation.

Loop TRITEX was ordered in December 1985 and delivered in summer 1987. Between 1989 and 1997, the eutectic mixture was circulating for 13003 hours in seven experimental phases.



(Y-axis relative average temperature of main circuit, X-axis 1988 to 1998)

Fig.26 : 10 years operation of TRITEX

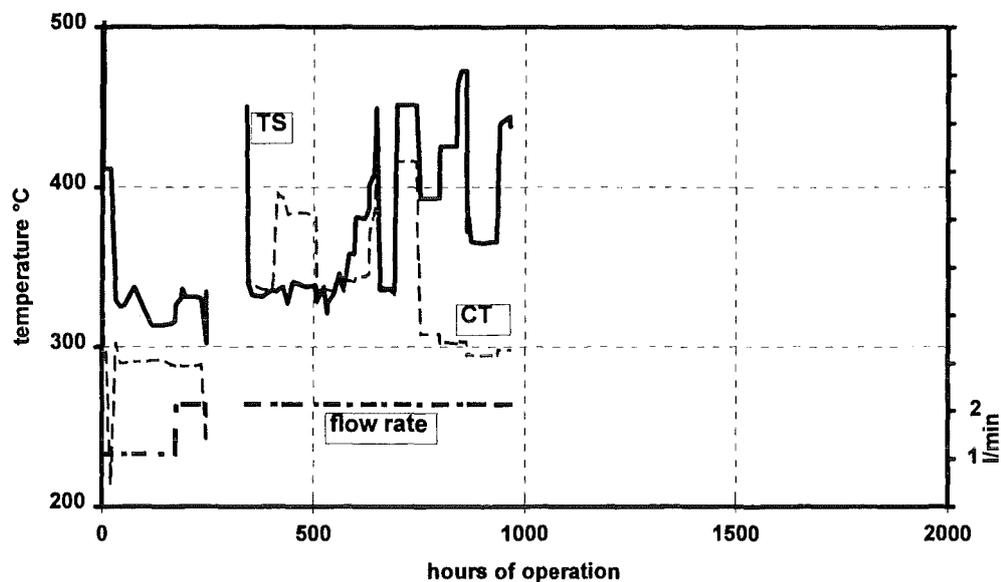
During and after an operation phase samples were taken at different positions and analyzed. Between phases the loop was modified depending on experimental requirements. The program with Pb-15.8Li in FZK ended in 1996. Therefore experiments were stopped after phase VII.

3.1 Pre-phase

Before a first filling of the loop, the permeation of deuterium D_2 through loop walls was measured. The pre-test phase began in 1988. In March, about 120 kg of the eutectic were filled into the drain tank using a special device. In April, after a first filling of the loop the TZM tube of the EMP broke, about 15 kg of the eutectic were lost. To continue start-up, an austenitic steel tube was installed. After this, because of a broken TZM level indicator at the drain tank, Pb-17Li there was heavily oxidized. About 3.5 kg oxides were formed, the remaining metal contained less than 5 at.% Li. The tank had to be cleaned. Also all liquid metal valves had to be cleaned because of Pb-15.8Li at metal bellows. Pb-17Li in form of solid bars were put in the tank before closing. A second pre-test phase was in April 1989, with several fillings and drains. Flow rates were measured by flash heating before three thermo couples in Pb-15.8Li. Because of malfunction, a pipe broke. Some Pb-15.8Li was lost, it was refilled via TS in the argon glove box. Altogether the loop was operated with molten eutectic for about 50 hours.

3.2 Phase I, 960 hours

June 26 to August 8, 1989



(temperature measurements in flowing eutectic, flow rate given for main flow)

Fig.27 : Operation phase I

New components

A new EMP tube was constructed and fabricated (PLANSEE company), consisting of an austenitic steel tube with TZM liner. It was installed in April 1989. An similar construction was used for MT1.

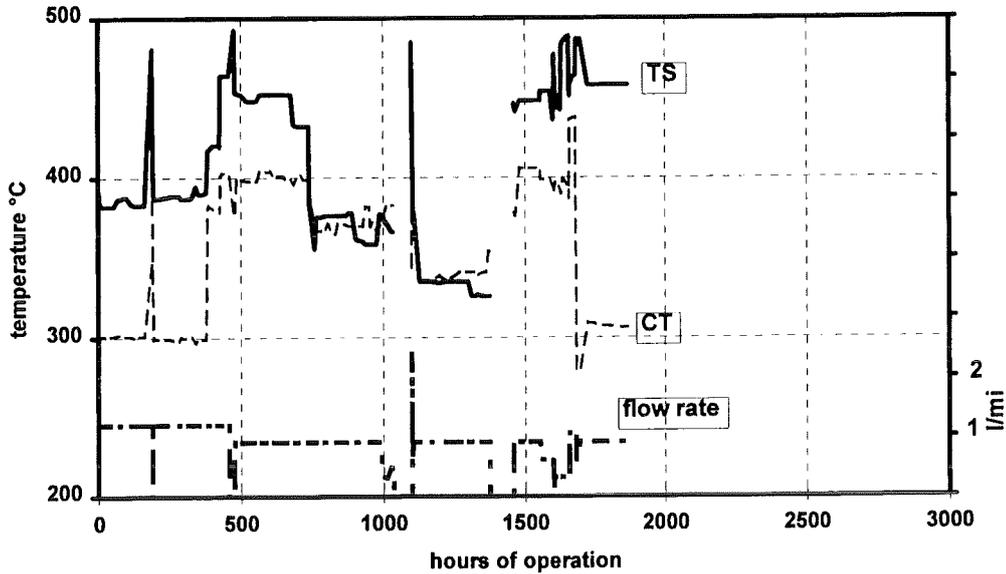
Operation

Because of problems with electronics the loop was drained for 93 hours. During this time, loop and DT were still heated, covergas flows controlled. After refill, a leak of drain valve V2 caused a loss of about 1.4 kg Pb-15.8Li per day from the loop to the drain tank, corresponding to a drop of the liquid metal level of 6 mm/d. Increasing the gas pressure in the drain tank was required to keep the level constant.

First D_2 transport measurements were performed, with several changes of parameters. The loading of the eutectic with D_2 was done over the covergas interface in the expansion volume. Because of D_2 loss through walls and a hydrogen background [8], the sensitivity for D_2 transport measurements was not good.

3.3 Phase II, 1866 hours

September 14 to December 12, 1989



(temperature measurements in flowing eutectic, flow rate given for main flow)

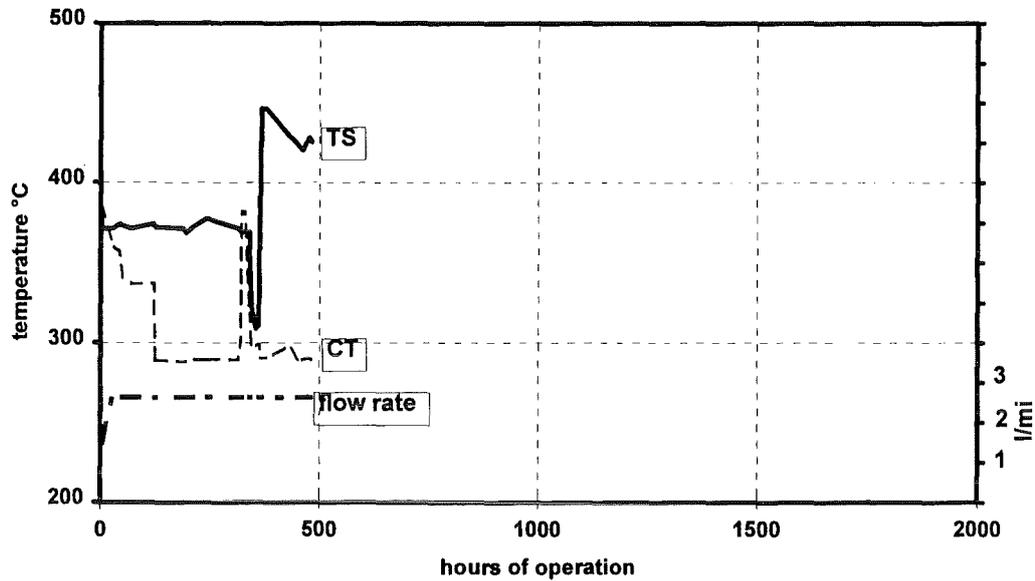
Fig.28 : Operation phase II

Operation

After about 1200 hours, an 100 cm^2 armco-iron permeation membrane was installed in the test section for a better determination of the D_2 activity in Pb-15.8Li. The loop was not drained during this installation, TS was opened in the argon glove box. For the investigation of behavior and transport of Protium (H_2) and Deuterium (D_2) in TRITEX, temperatures and flow rates were changed several times.

3.4 Phase III, 483 hours

August 1 to August 21, 1990



(temperature measurements in flowing eutectic, flow rate given for main flow)

Fig.29 : Operation phase III

New components

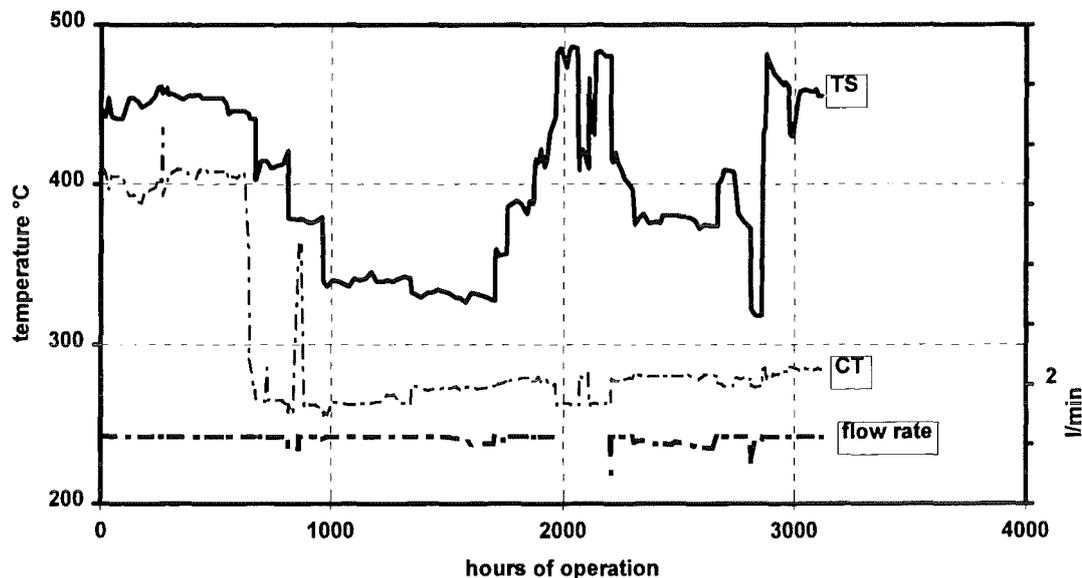
A second thermo box for parts of the main piping. The inert gas system was completely re-build : electro-pneumatic valves and capacitive differential pressure transducers between the covergas spaces and an isothermal recipient. A magnetic trap and flow meters from vanadium. Because of the small leak of V2 to the drain tank, a five point level indicator was installed in SS2. Depending on the LM-level, the gas pressure in the drain tank was controlled, compensating the leak and keeping the level in the loop within ± 2 mm. A Phillips Data-Logger with PC as new registration system, with this, loop controlling and data storage were independent. For a better loading of the eutectic with D_2 , a 1036 cm^2 armco-iron permeation membrane was installed in the expansion tank.

Operation

This was a short operation phase to check new equipment's.

3.5 Phase IV, 3143 hours

November 12, 1990 to March 22, 1991



(temperature measurements in flowing eutectic, flow rate given for main flow)

Fig.30 : Operation phase IV

New components

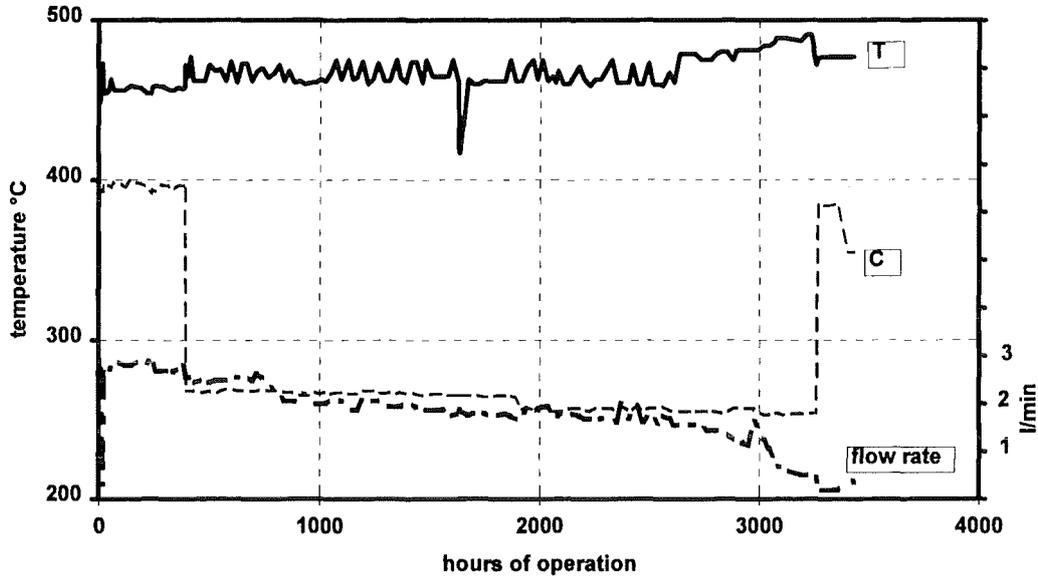
Three TS were installed, TS1 and TS3 with an 100 cm² membrane of armco iron, TS2 empty.

Operation

This was the first long time operation without any drain of liquid metal. Mainly the behavior and transport of Protium (H₂) and Deuterium (D₂) in a pumped ferritic system was investigated. Also an attempt with Tritium was made, but the total used ³H activity of 10⁶ Bq was too small. The only problem occurred after about 2000 hours at power control of the EMP. It could be solved without draining the loop. Temperatures and flow rates were changed frequently.

3.6 Phase V, 3431 hours

October 28, 1991 to March 19, 1992



(temperature measurements in flowing eutectic, flow rate given for main flow)

Fig.31 : Operation phase V

New components

Replacement of V2 by freeze valves. By this the liquid metal level in the loop remained constant. 19 Philips KS-40 temperature controller.

Operation

The behavior and transport of H_2 and D_2 was studied. During phase V a basket with 41 grams vanadium (290 cm^2 V-surface) got immersed in TS2. But operation was mainly for the production and deposition of corrosion products.

Because of problems with the main heater MH the loop temperature could not be kept constant for some time. The fluctuation was $\pm 7^\circ\text{C}$. After about 2000 hours, flow resistance in main loop and cold trap bypass increased. For some time it could be compensated by increasing the EMP power, Fig.32. But flow resistance caused finally the end of phase V. A plug of LiPb was found later at the exit of CT2, particles + LiPb mainly in flow meters. After drain, the loop was heated without Pb-15.8Li for about 600 hours to study D_2 degassing behavior of structural materials.

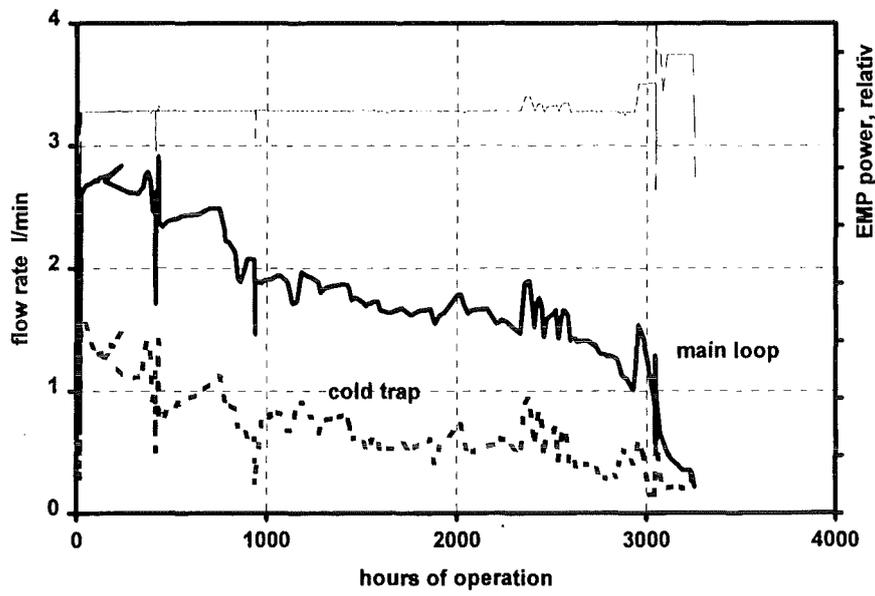
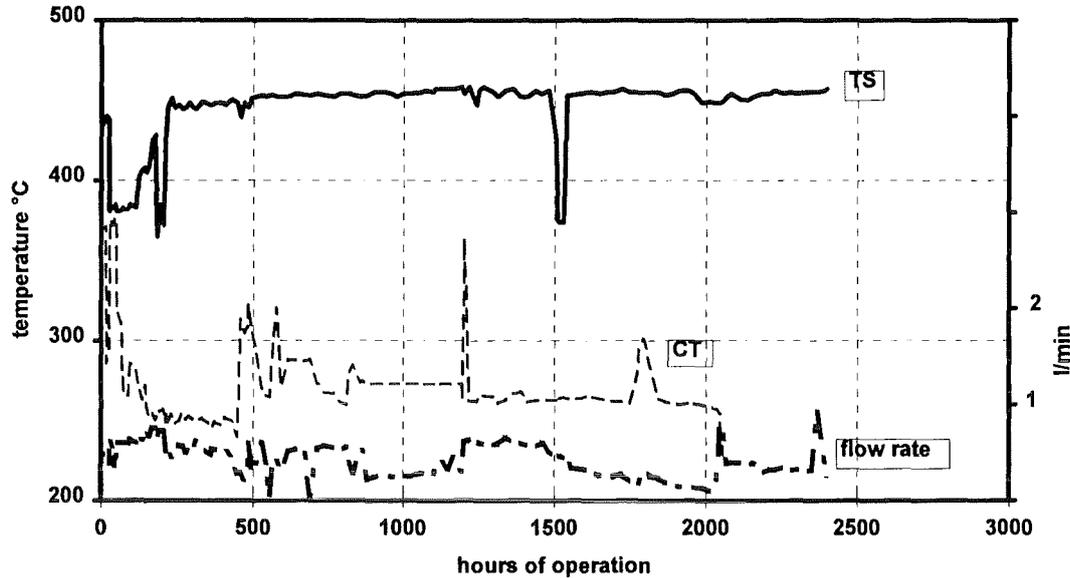


Fig.32 : Pb-15.8Li flow rates in phase V.

3.7 Phase VI, 2398 hours

August 2 to November 10, 1994



(temperature measurements in flowing eutectic, flow rate given for main flow)

Fig.33 : Operation phase VI

New components

An electro magnetic pump with a larger annular gap (smaller pressure drop). Two magnetic traps from vanadium. Two mass flow meters MFM. Cold trap 1, containing chambers with measured temperature. Cold trap 2 with 1.7 m² wire mesh packing of ferritic steel. To operate CT2 isothermally, a Philips KS 4290 temperature controller was required. Cold trap 3, a diffusion type CT with solid phase. Coiled test section 2 with vanadium. New designed freeze valve because of some problems in phase V. Furthermore, 120 kg Pb-17Li in form of solid bars from Meteaux Speciaux company were put in the drain tank before closing.

The long delay between phase V and VI was caused mainly by purification of TRITEX. After each operation phase, some components were replaced and analyzed. After phase V, however, the whole loop was dismantled, all parts investigated and chemically cleaned. This lasted for more than one year. It was required to have a clean loop for corrosion product studies. 10% HNO₃ or a mixture of 2% acetic acid + 0.5% H₂O₂ was pumped at 60 to 80°C through sections of the loop. All solutions were analyzed for Pb, Li and components of steel. After this process all surfaces were metallic clean.

Operation

After 450 hour a power supply failure (thunderstorm) caused freezing of all Pb-15.8Li in the loop. By very slow and controlled reheating it could be remolten without any break of a pipe. As before, the flow resistance in main and by-pass loop increased slowly, Fig.34. Even by increasing the EMP power, CT2 was blocked after about 2000 h. The dashed line in Fig.34 is corrected for a 'normalized' EMP-power. Flow reversal by changing EMP polarity did not help much. For the last 550 hours TRITEX was operated without CT. Mainly one position was found blocked : A plug of solid LiPb at the outlet of CT2.

TRITEX was operated in operation phase VI mainly for the production and deposition of corrosion products. But also H_2 and D_2 transported was studies again, together with Tritium. $5 \cdot 10^9$ Bq 3H - tracer were used, released by heating a Ti - 3H target. The H_2 (and also remaining D_2) background however was too high, the behavior of 3H followed the behavior of H_2 .

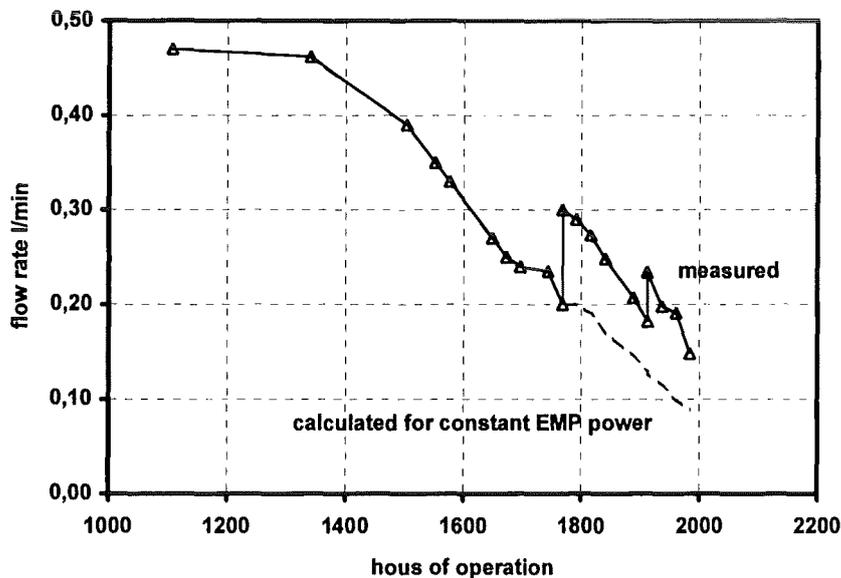
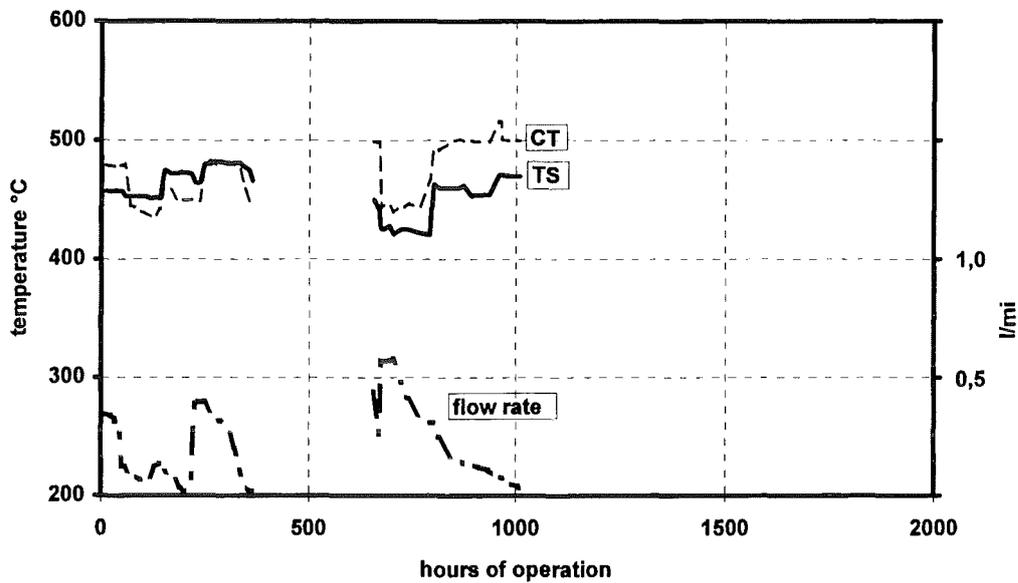


Fig.34 : Operation phase VI, flow rate of CT bypass at 260°C

3.8 Phase VII, 722 hours

October 31 to December 12, 1996



(temperature measurements in flowing eutectic, flow rate given for main flow)

Fig.35 : Operation phase VII

New components

Magnetic trap MF1 and cold traps 1 and 3 were replaced. A new cold trap 2 for trapping of particles at covergas interfaces was constructed and installed.³

Operation

TRITEX was blocked within a short time. The positions could be localized and some parts replaced. During the last time of operation, cold traps were operated with a higher temperature than the main loop. But finally phase VII ended. Mainly the blocked magnetic trap MF1 was responsible for the end of phase VII.

³ A second one was already fabricated for phase VIII, but the program ended.

4. Photographs of TRITEX

Loop TRITEX was ordered in December 1985 and delivered in summer 1987. Experiments were performed between 1989 and 1997. Often parts of TRITEX were modified depending on experimental requirements. Over the years many photographs of loop and components were taken. This chapter shows a selection to give an impression of the facility.

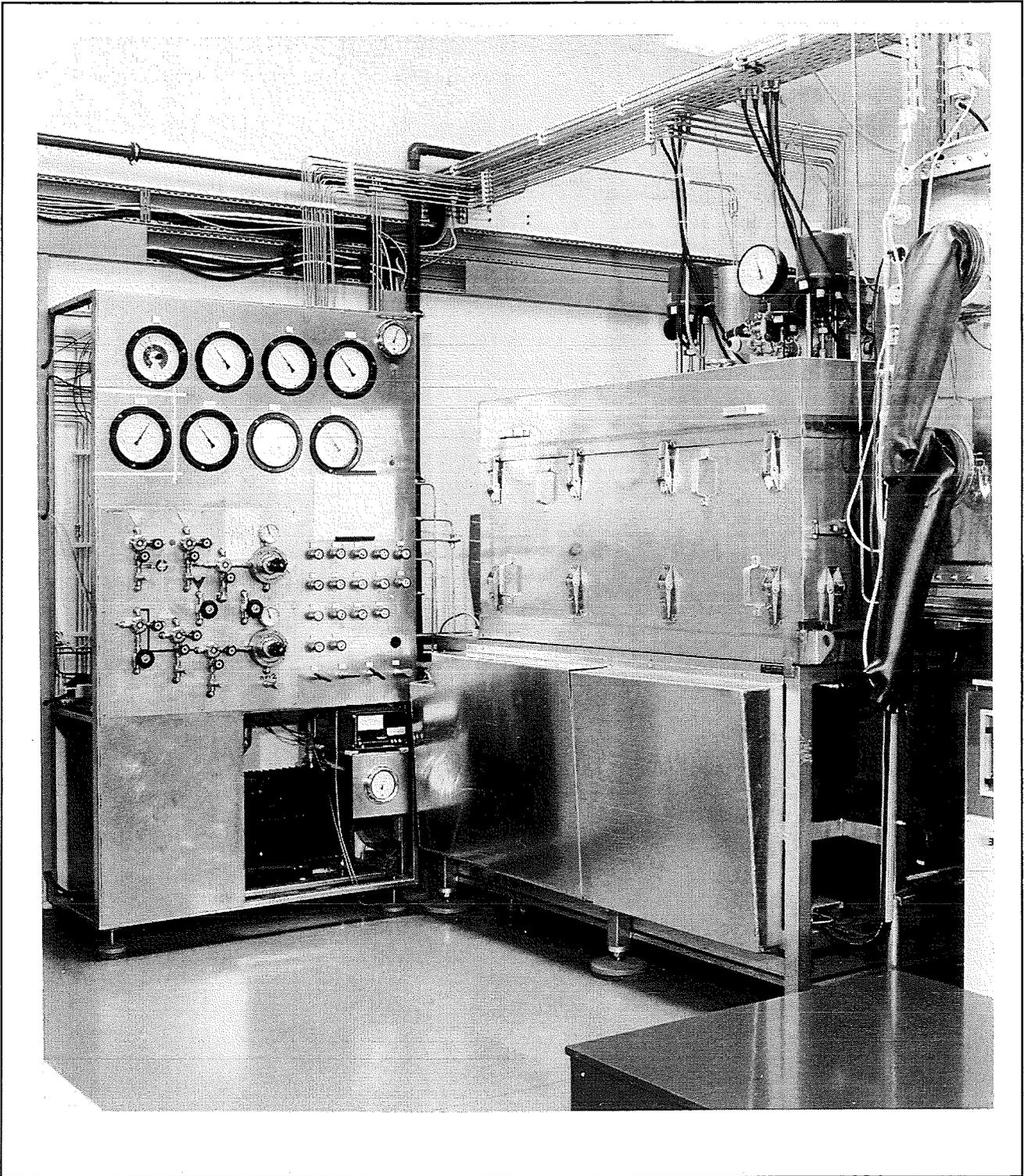


Photo 1 : 1989. TRITEX facility with thermal isolation. From left : first covergas control panel, thermo box system, glove box with test section.

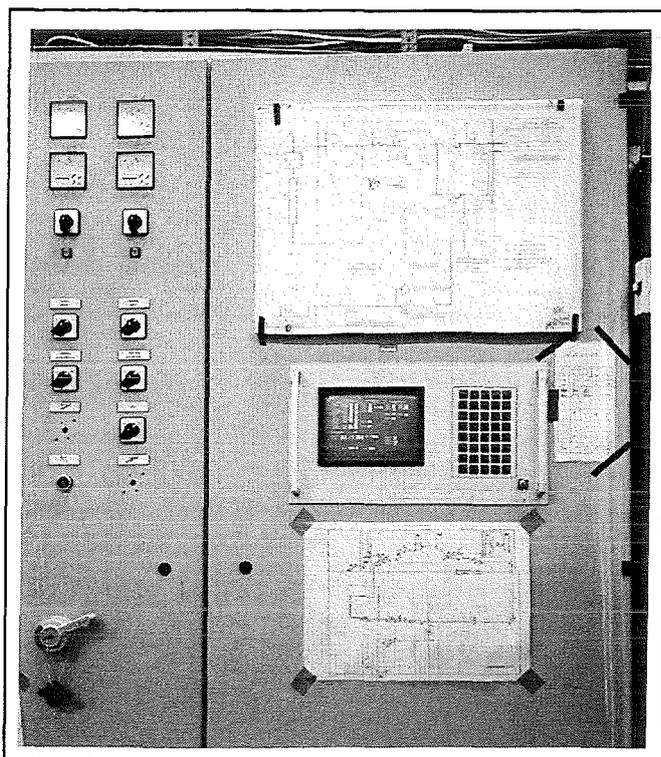


Photo 2 : 1988. First control board. From top : flow sheet with indicator lamps, ELZET controller with monitor, LM level diagram (needed for filling)

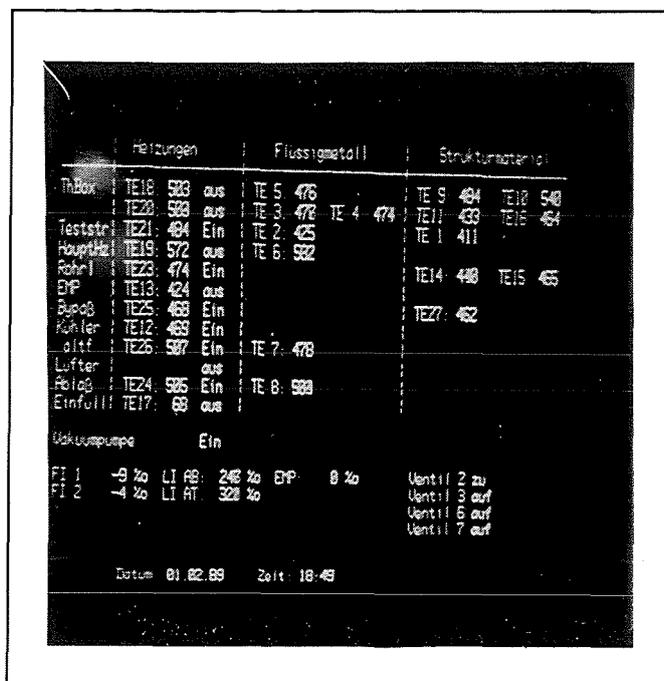


Photo 3 : 1988. Display at ELZET monitor

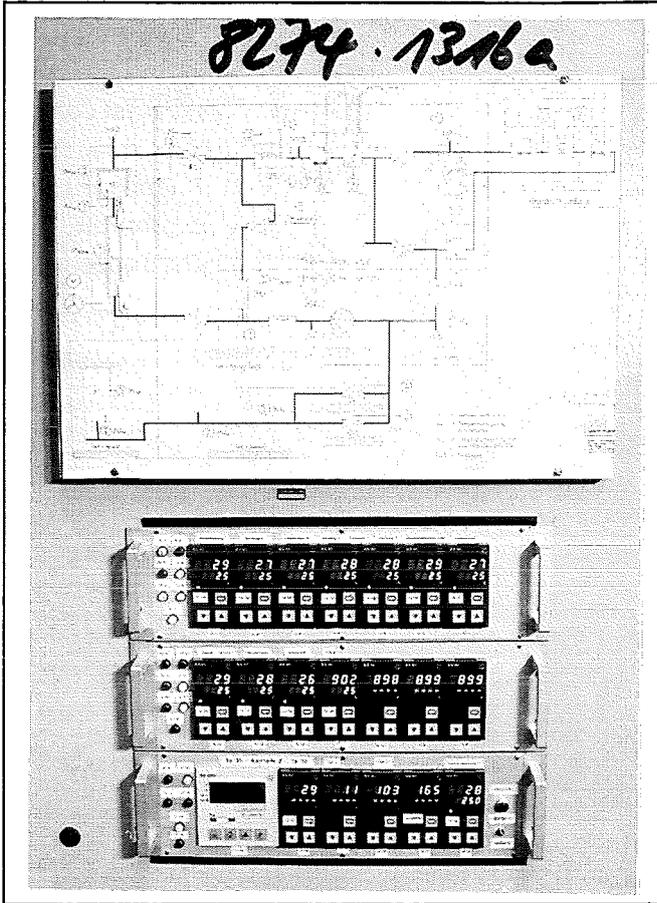


Photo 4 : 1994. New control board, flow sheet with indicator lamps and T-controller .

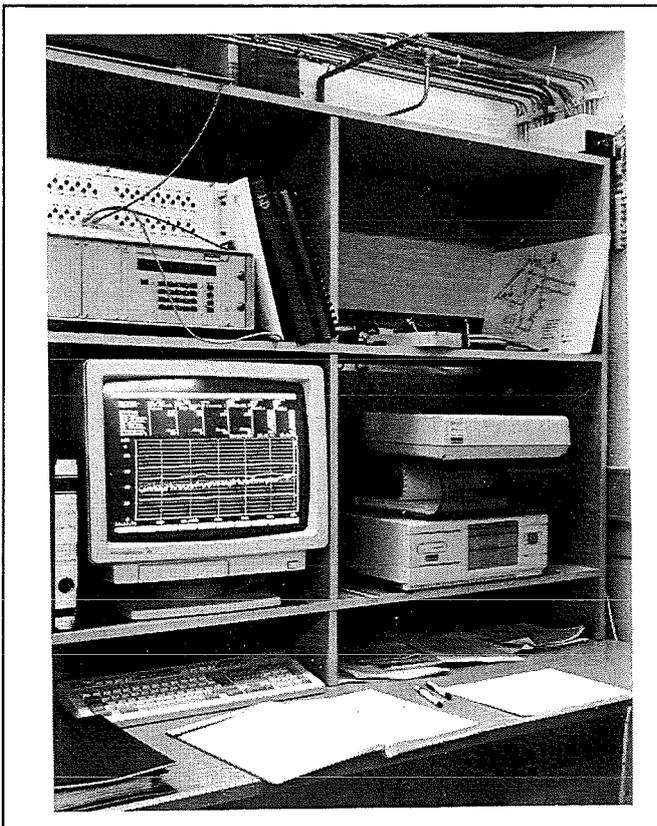


Photo 5 : 1991. Philips data-logger and PC

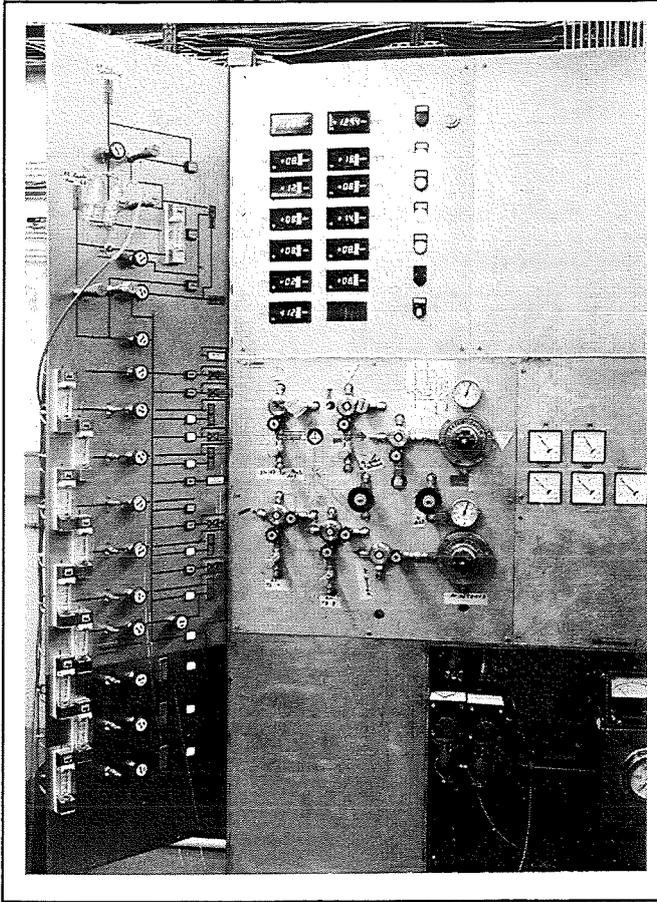


Photo 6 : 1992. New gas control panel, at right ampere meters for EMP (three phases) and box heater

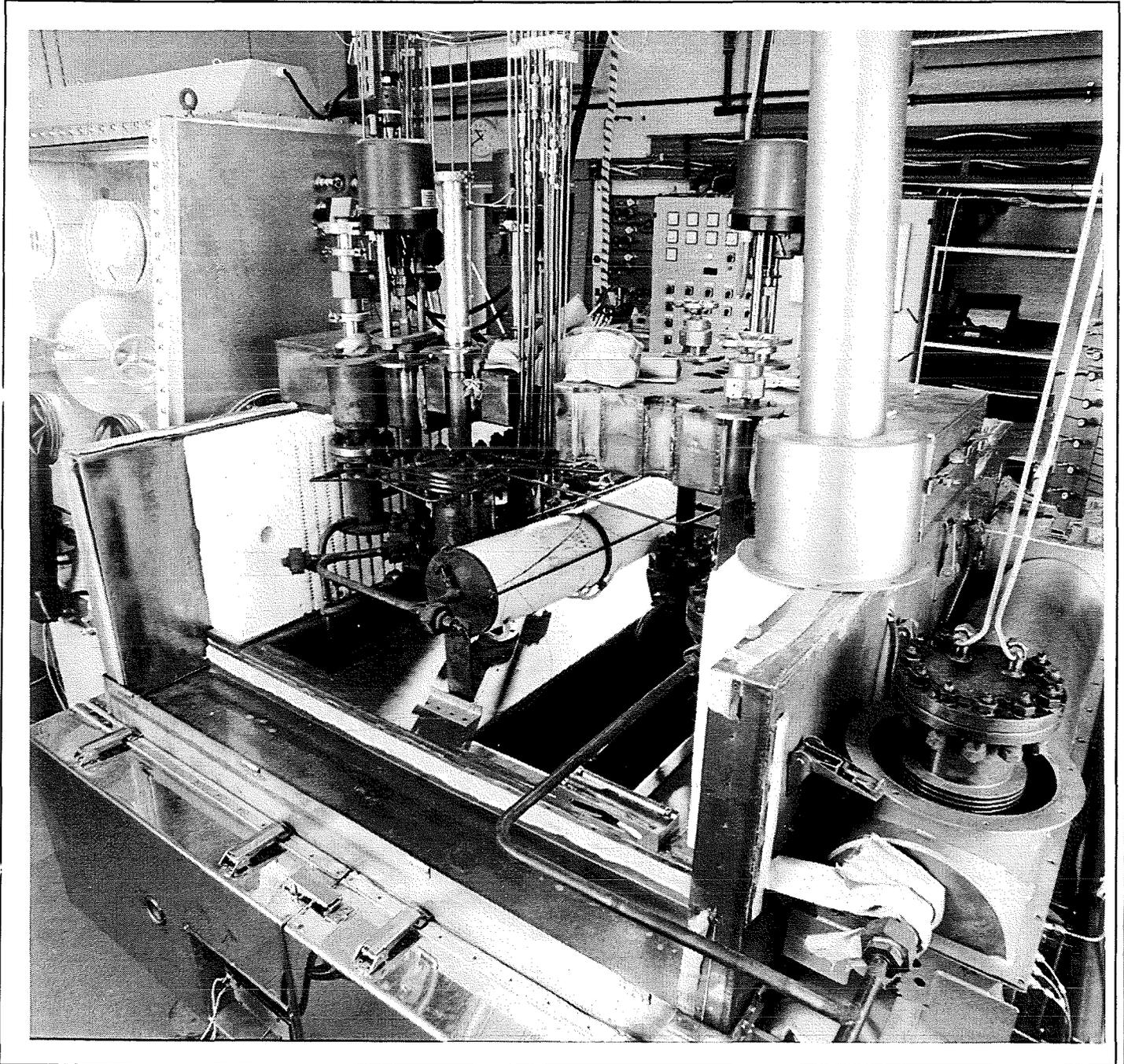


Photo 7 : 1989. Upper box, rear side from left : glove box, between box heaters' valves and main heater, cold trap without exhaust chimney (shown above cold trap)

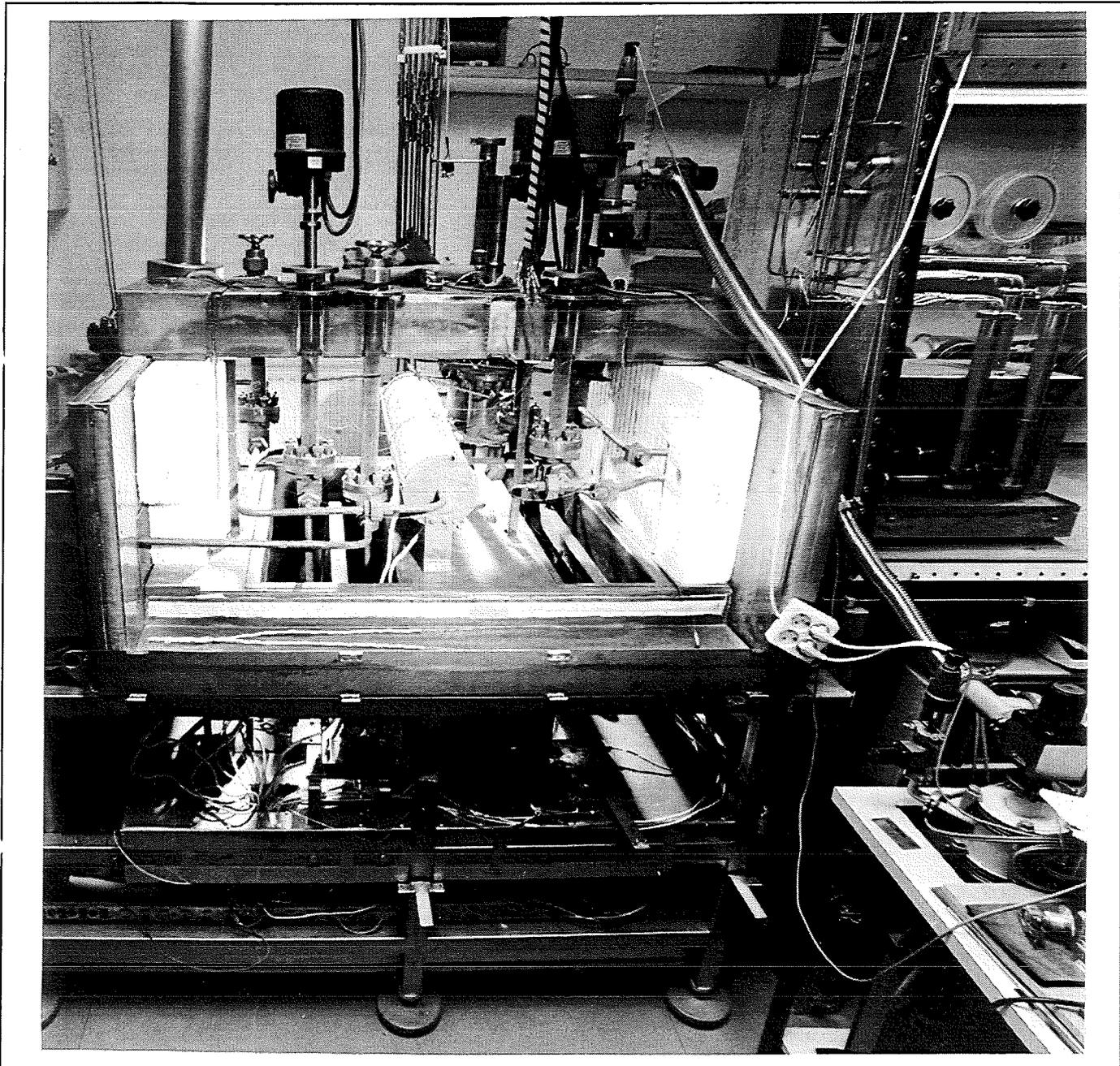


Photo 8 : 1991. Upper box, front side from left : between box heaters' valves and main heater, glove box with test section

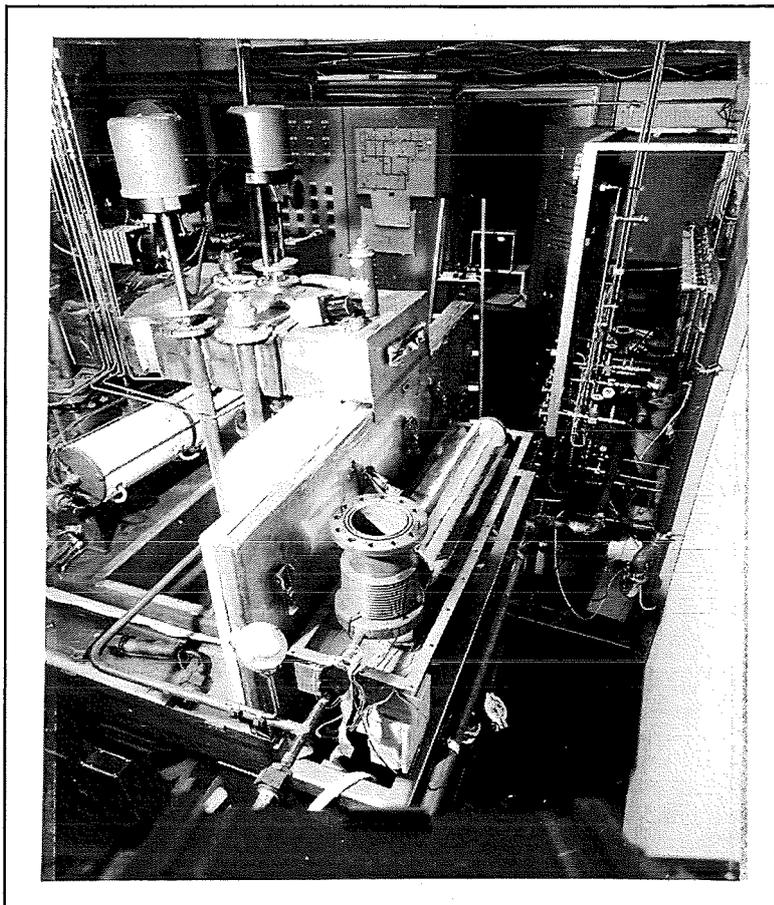


Photo 9 : 1989. Upper box rear side from left : main heater, motor-valves 2 and 3, CT1 and CT2.

Motor-valve 2 was later replaced by freeze valves, cold trap 2 shown open without basket.

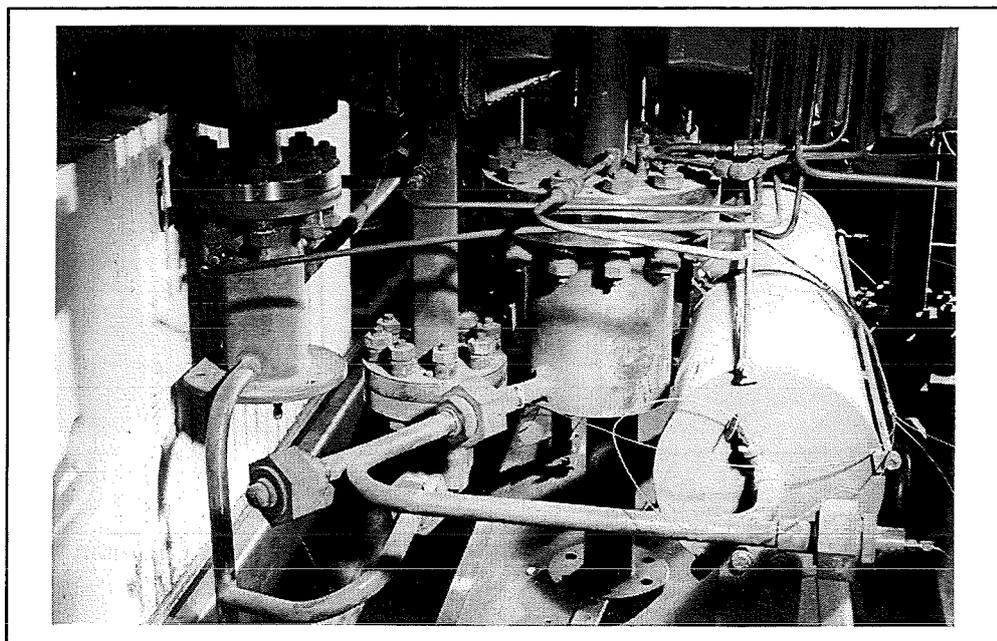


Photo 10 :1989. Upper box rear side from left : box heater, sampling station 1, valve 7, expansion tank, main heater

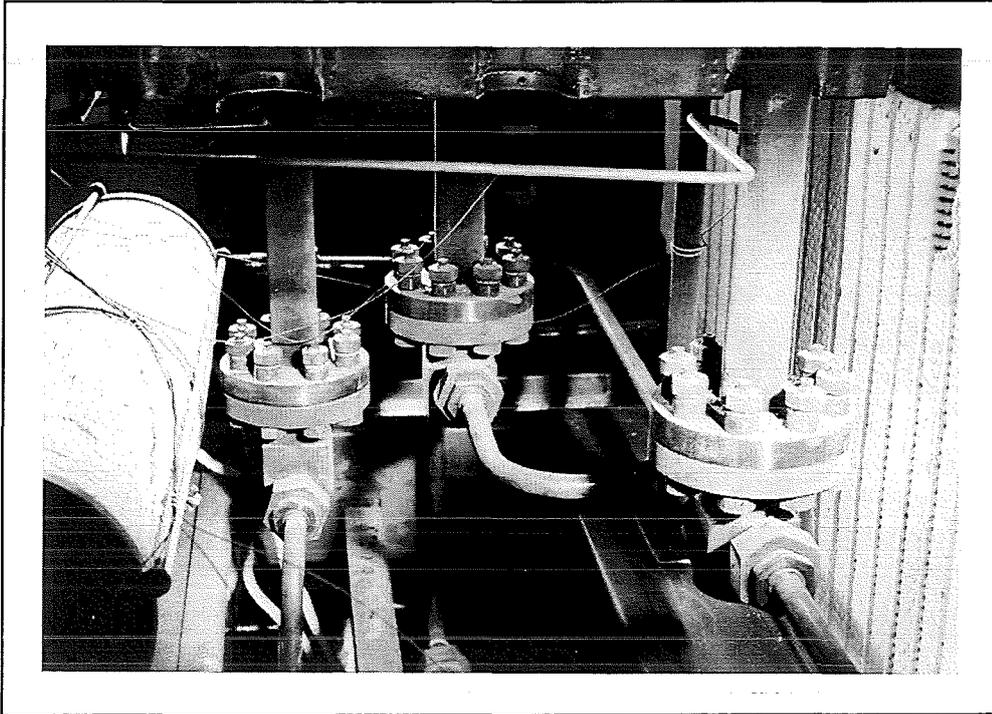


Photo 11 :1989. Upper box rear side from left : main heater, valves 4, 3 and 5, box heater

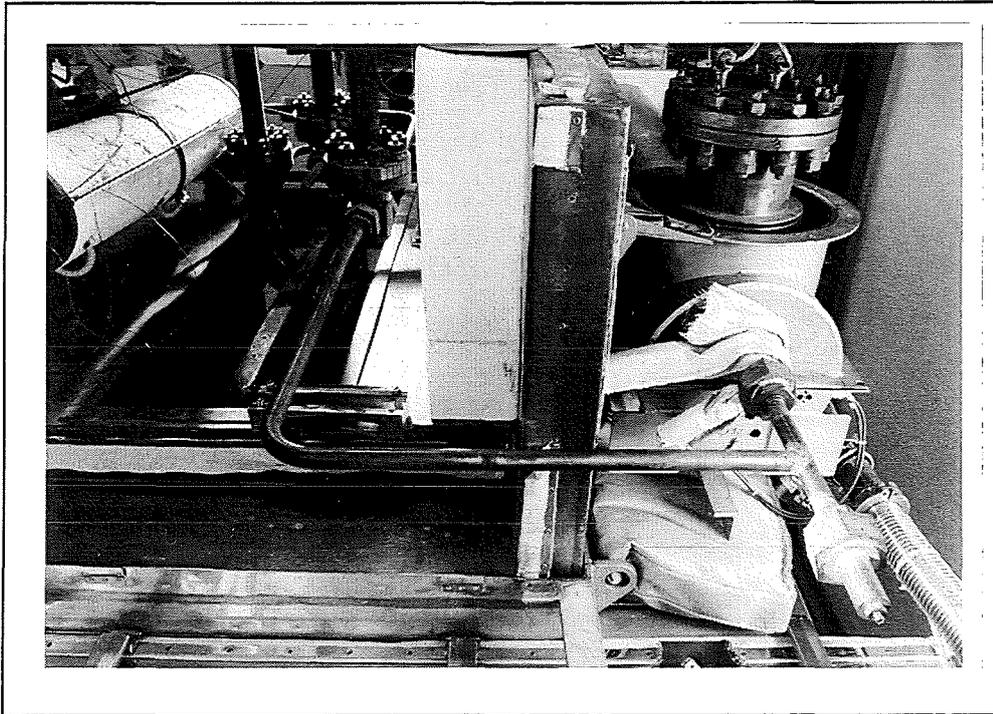


Photo 12 : 1989. Upper box rear side from left : main heater, valves (background), box heater, cold trap 2 exit

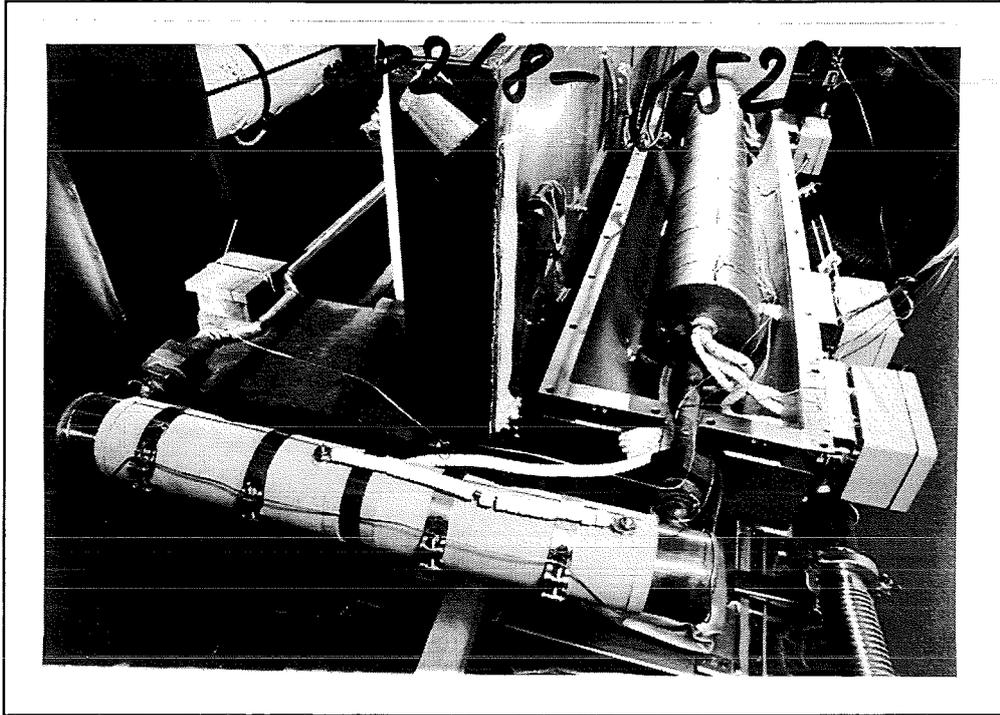


Photo 13 : 1994. Upper box rear side from left : Magnetic trap 2, cold traps 2 and 1

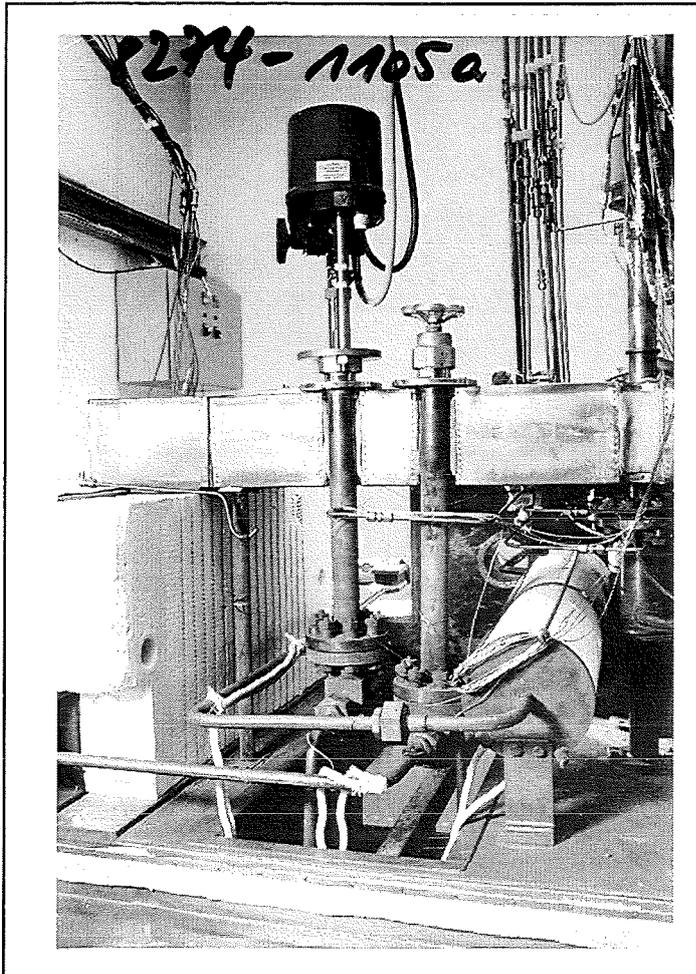


Photo 14 : 1994. Upper box front side from left : One box heater, motor-valve 3, valve 4, main heater. Front pipe going to CT1.

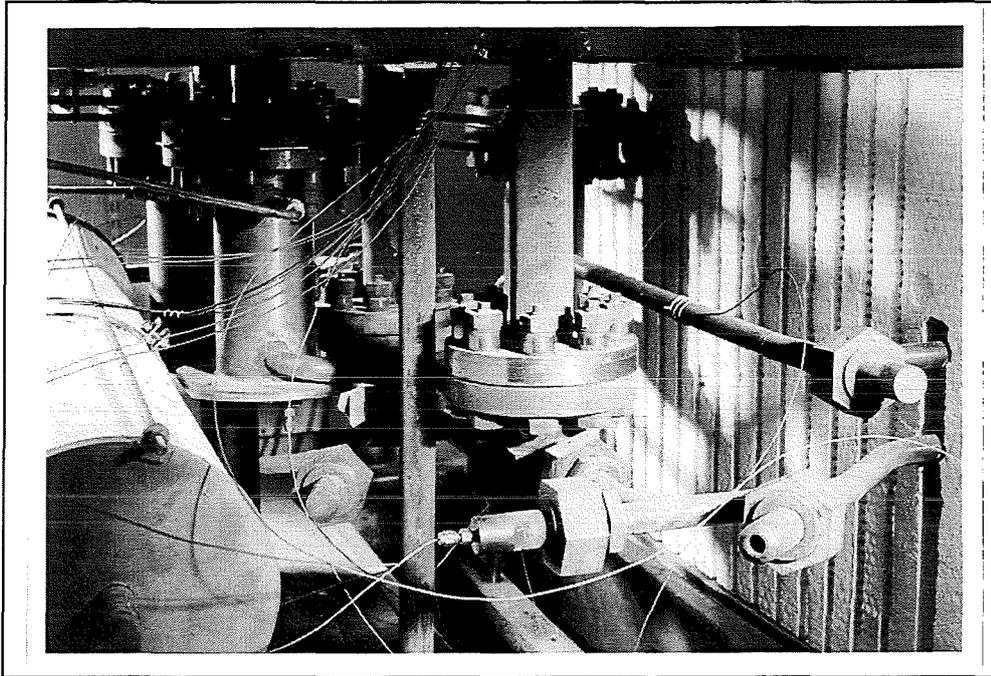


Photo 15 : 1994. Upper box front side from left : main heater, expansion tank (background), valve 4, one box heater. Front pipes through box heater going to TS.

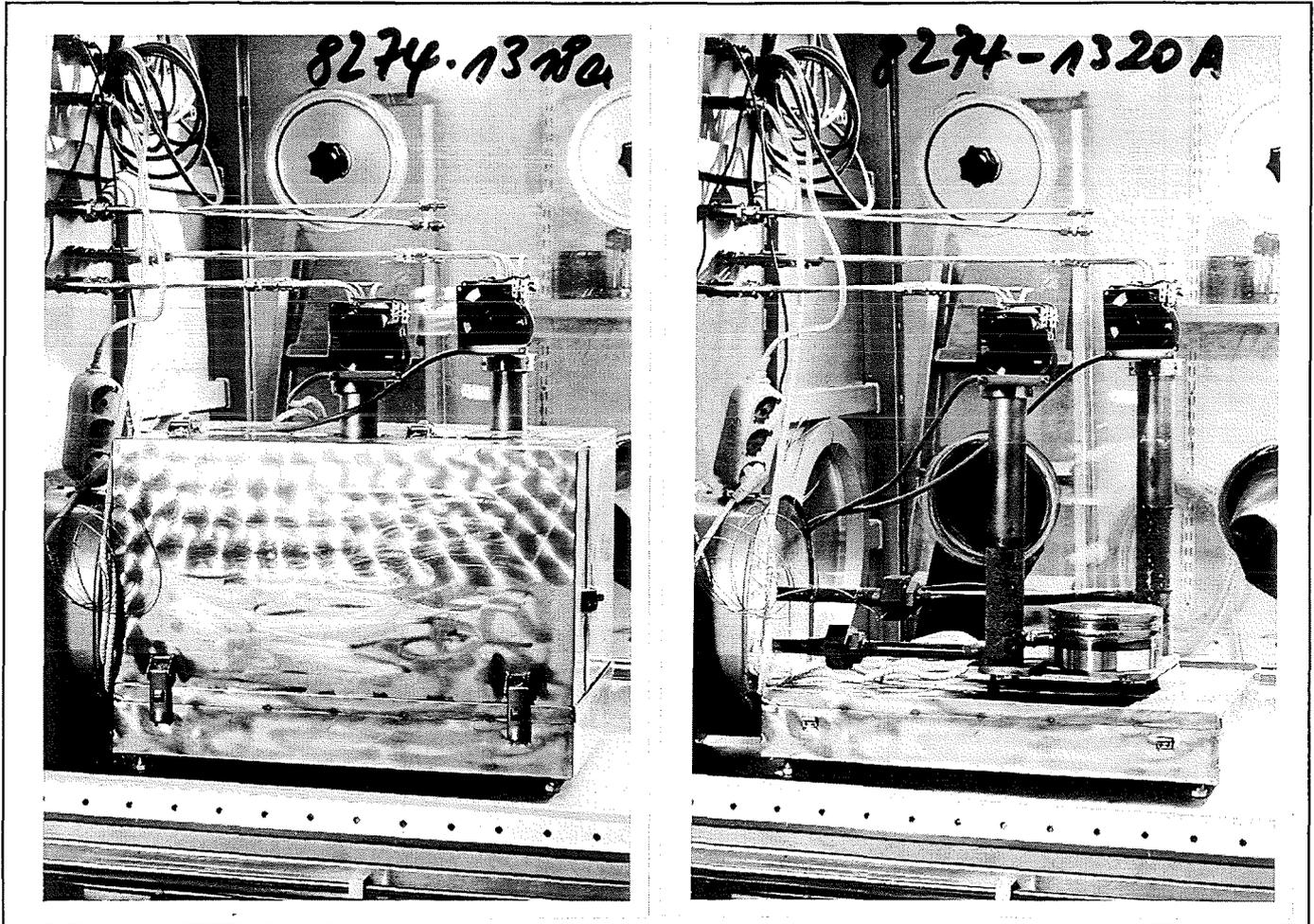


Photo 16 : 1996. Test section in an Ar-glove box, with and without thermal isolation.

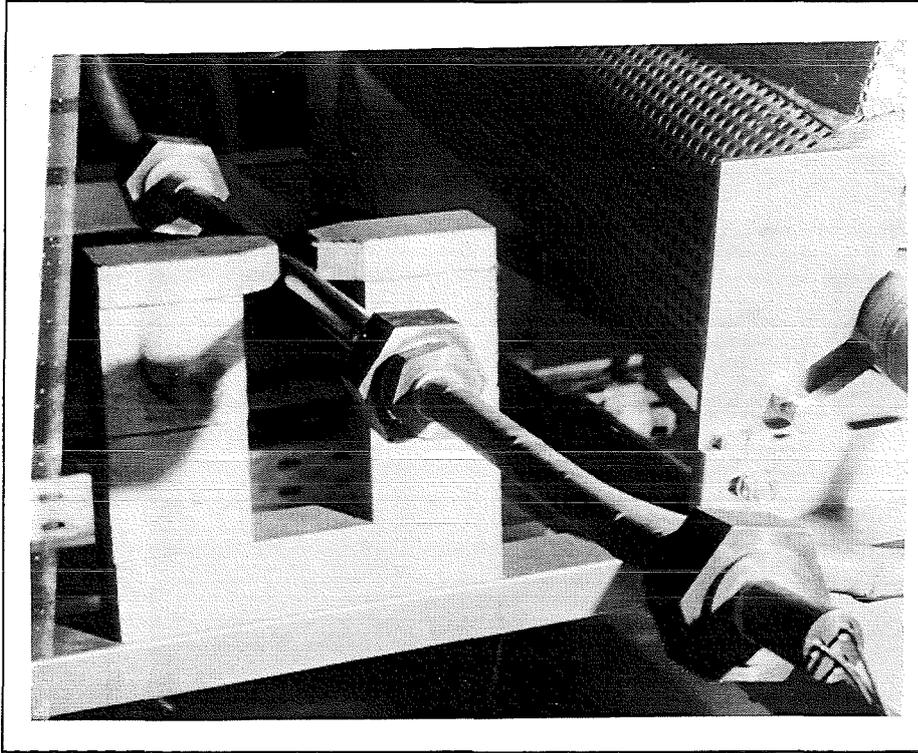


Photo 17 : 1990. Lower box rear side from left : first magnetic trap 1, front pipe to EMP inlet

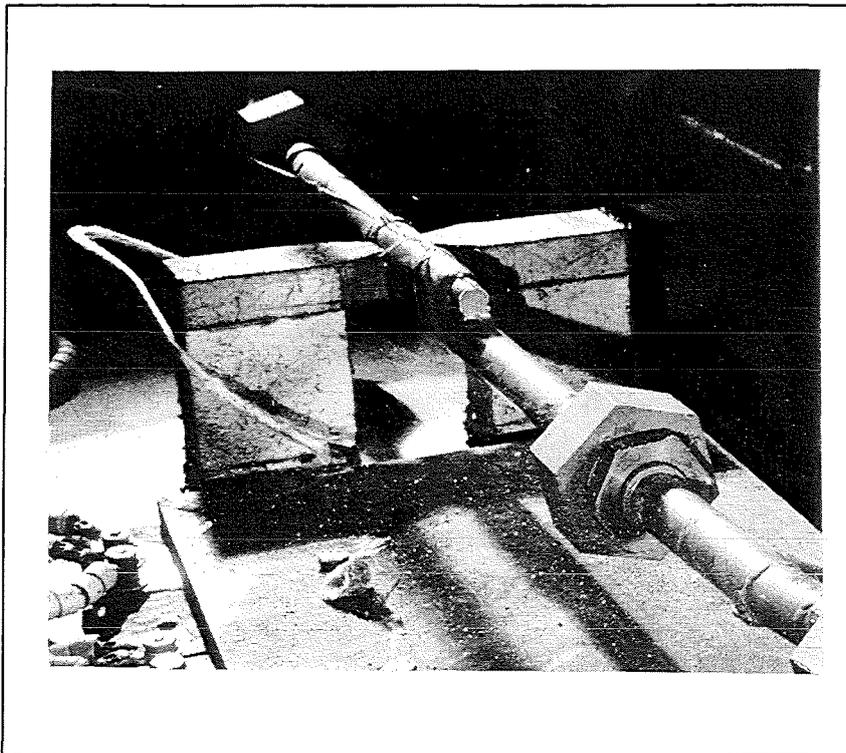


Photo 18 :1994.
Lower box rear
side from left :
last magnetic
trap 1, front pipe
to EMP inlet

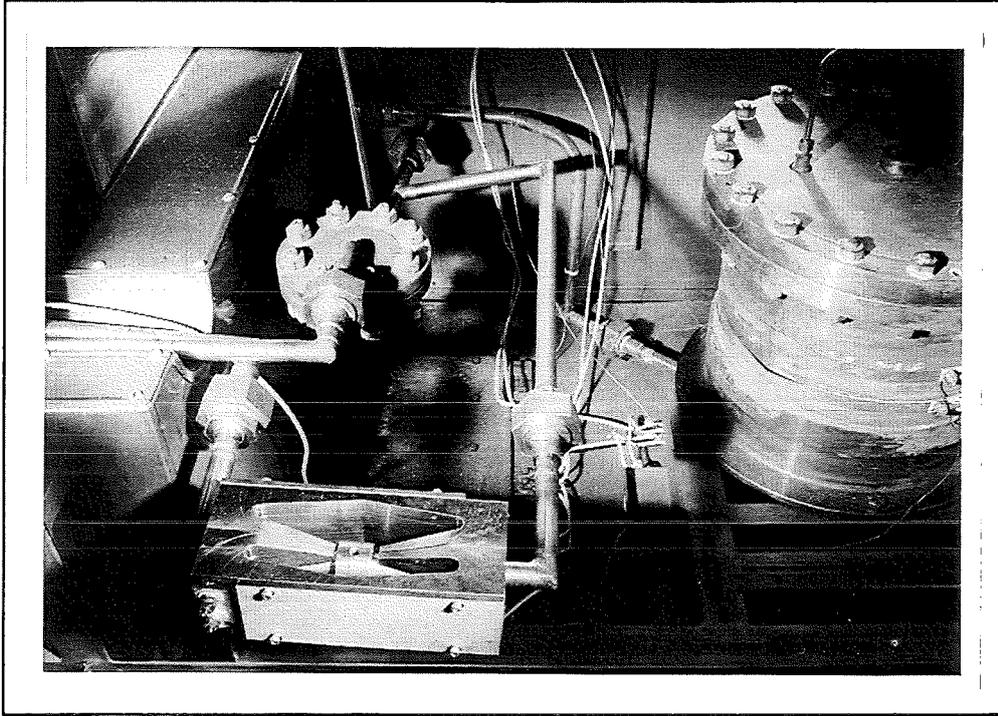


Photo 19 : 1994. Lower box rear side from left : (from inlet EMP), mechanical filter, in front freeze valve, drain tank

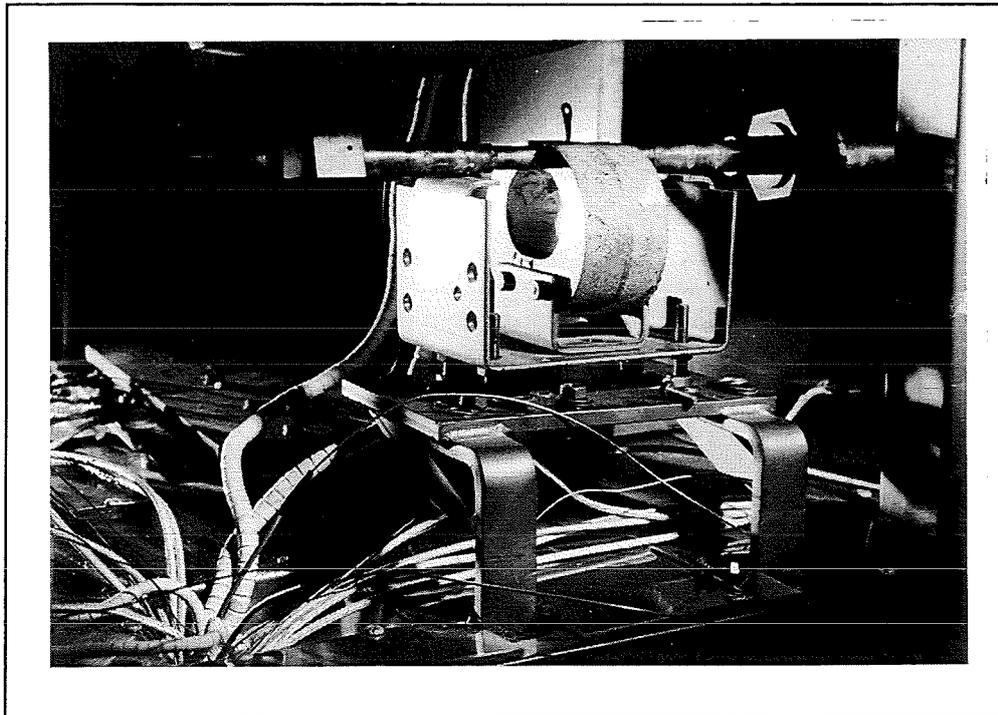


Photo 20 : 1992 : Lower box front side: flow meter 1

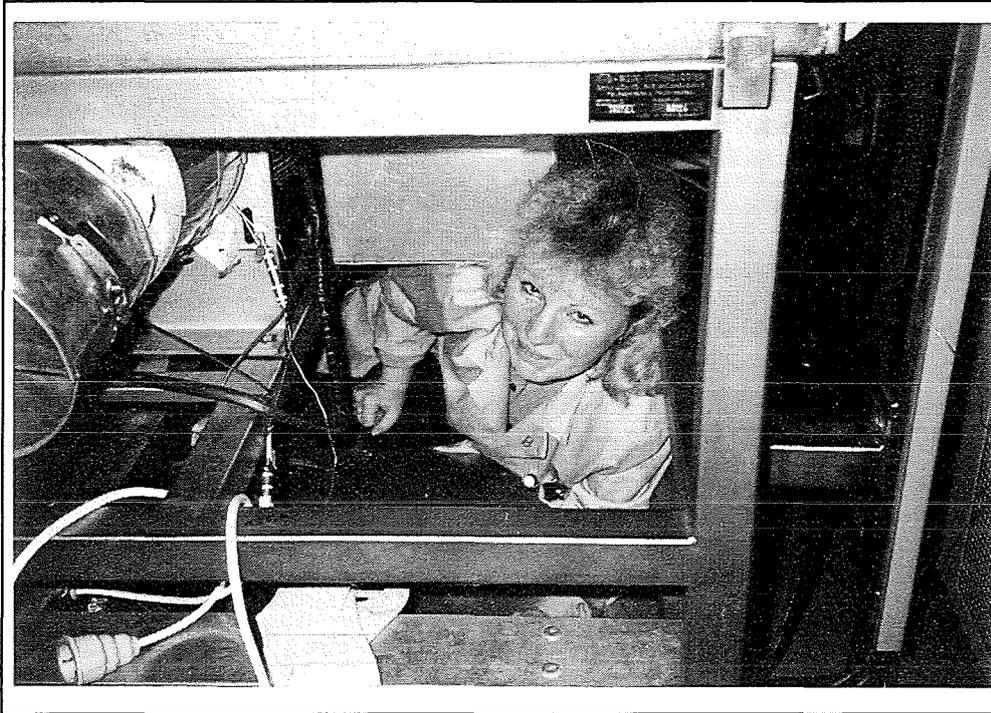


Photo 21 : 1988 : Mrs. Neumann, NTG in lower box during installation

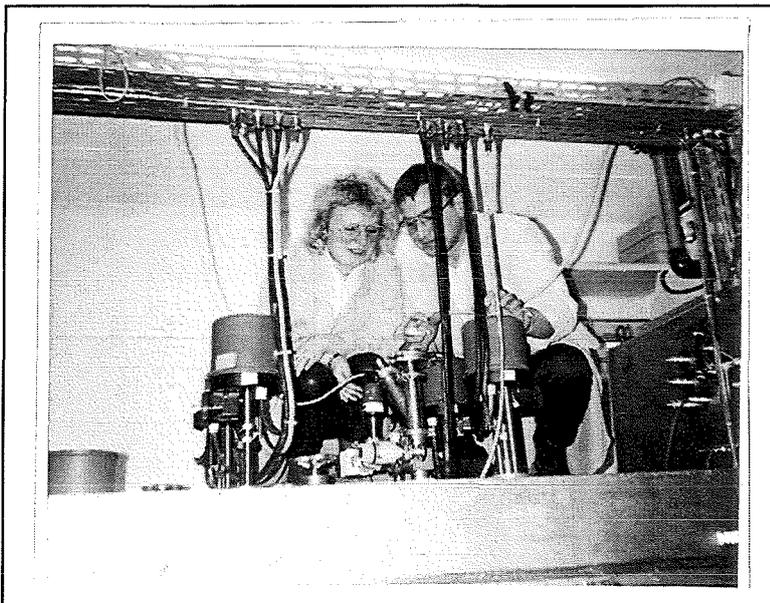


Photo 22 : 1988 :
First filling,
Mrs. Neumann and
Mr. Bott, NTG on top
of TRITEX, looking
through the observa-
tion window

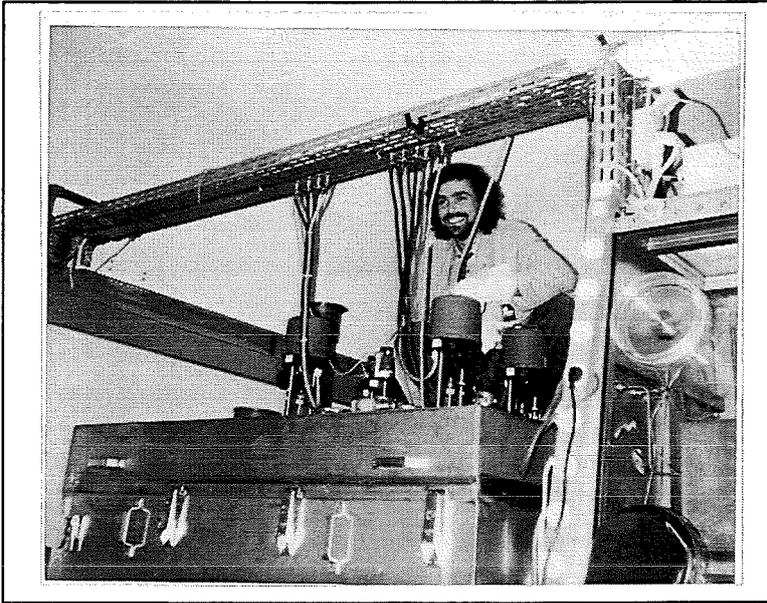


Photo 23 : 1988 :
Mr.Kieser, on top of
TRITEX looking
through the observa-
tion window; first
filling

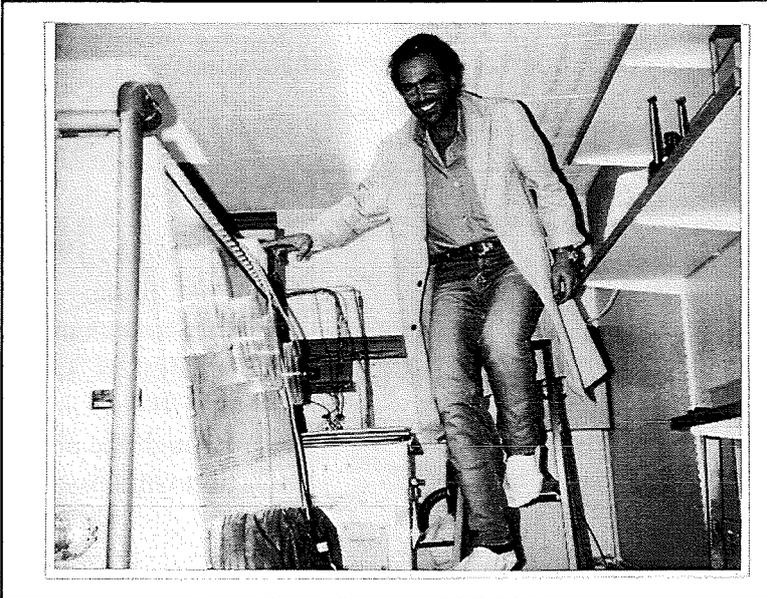


Photo 24 : 1996 :
Mr.Kieser; last filling,
'the loop is filled'

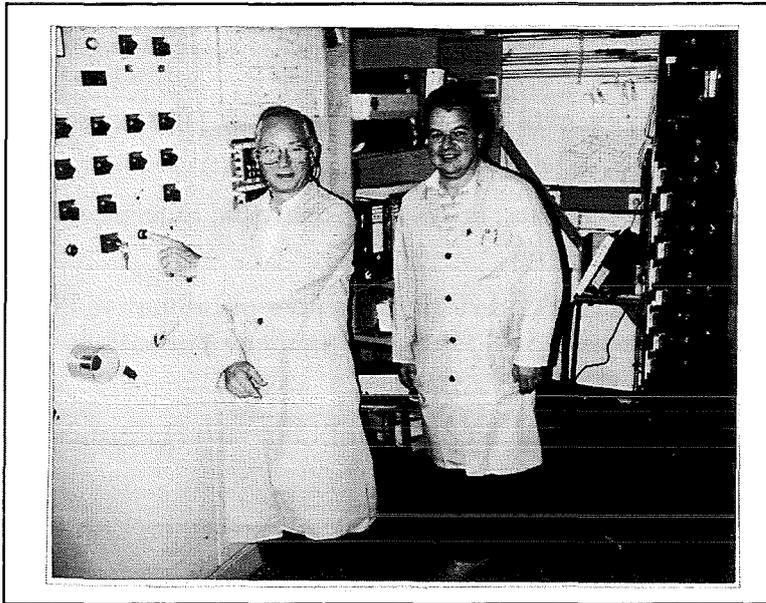


Photo 25 : 1996 : Mr. Horn, right and main author, FZK, last shut down of TRITEX. ‘ Happy ? ’

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