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THERMOSIPHON LOOPS FOR CORROSION MEASUREMENTS IN HIGH TEMPERATURE TERPHENYLS

by

G.C. IMARISIO

1971

Joint Nuclear Research Centre
Ispra Establishment - Italy

Materials Department
Physical Chemistry

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The general design criteria are described as well as some peculiar features of the plant. Continuous operation of the loops during about 20,000 hrs has shown that these apparatus are well suited for the long term corrosion measurements which require a troublefree operation. The loops are provided with control of the atmosphere over the terphenyl and of its composition. The water content of the circulating liquid is controllable and constant within ± 10 % at the 100 ppm level; the temperature is held within ± 1 °C around 400 °C.

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ABSTRACT

Eight thermosiphon loops have been designed and built for corrosion measurements in high temperature terphenyls.

The general design criteria are described as well as some peculiar features of the plant. Continuous operation of the loops during about 20,000 hrs has shown that these apparatus are well suited for the long term corrosion measurements which require a troublefree operation. The loops are provided with control of the atmosphere over the terphenyl and of its composition. The water content of the circulating liquid is controllable and constant within $\pm$ 10 % at the 100 ppm level; the temperature is held within $\pm$ 1 °C around 400 °C.

KEYWORDS

COOLANT LOOPS
TECHNICAL SPECIFICATIONS
CORROSION
HIGH TEMPERATURE
TERPHENYLS
TESTING

I n d e x

List of figures

1) Introduction *)

The group of loops described here has been built in 1963 for the measurement of the corrosion behaviour of Zirconium alloys in high temperature terphenyl in the frame of the development of the Orgel reactor. When the decision to start this research was taken the equipments available were quite unreliable for this type of work. On the other hand also the technologies associated with the high temperature terphenyls were not very developed so that successful choices were difficult to do.

Little information was available also on the factors affecting the corrosion of the Zirconium alloys in terphenyl: the few experimental measurements available reported that their corrosion resistance was very poor. As a consequence the equipments had to be provided with enough flexibility to permit a broad range of experimental conditions.

Different choices were possible: autoclaves and capsules was the most straightforward extrapolation from corrosion work in water and steam. The most costly alternative was to choose loops with forced circulation. It is difficult, however, to maintain in terphenyls the required degree of control over the environment.

In fact, the commercial "terphenyl" (however "clean") is a technical mixture of the three isomers (ortho-, meta-, para-terphenyl), containing also small amounts of diphenyl, of heavier omologues, and various inorganic impurities.

These terphenyls are partially pyrolized at the higher working temperatures (380 - 450°C), and are solid at room temperature.

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*) Manuscript received on 16 December 1971

It is clear that the control of water chemistry is far easier than that of terphenyls, at least for corrosion work. The above mentioned degradation of the organic material precluded the straightforward use of simple autoclaves. The more sophisticated "refreshed" autoclaves had the problems of finding a reliable feed pump and long lived valves.

Low reliability (and high cost) of suitable pumps as well as the high cost of the associated and necessary instrumentation is also the main problem of forced circulation loops.

The whole corrosion facility had to operate with a minimum of surveillance, while the cost per operative unit had to be as small as possible (to multiply the number of units).

As a compromise between the contrasting needs under the suggestion of Mr. F. H. Krenz from AECL it was decided to develop very simple thermosiphon loops of low therphenyl hold-up, using only well-known and reliable components.

The main advantage of a thermosiphon loop is the inherent stability and reliability of the pumping action so that the other advantage of a recirculating loop can be exploited at the expense, however, of a very modest available head.

2.1 Design of the main mechanical components

In designing the loop for thermosiphon circulation some difficulties had to be coped with. In fact the high specific heat of the terphenyl and its modest heat transfer properties together with its pyrolysis at high temperature require a careful choice of the working conditions.

The useful height of the loop being constant the speed of the liquid is proportional to the temperature difference between the hot and the cold legs. The driving pressure is given by:

$$\Delta P_m = K_t \cdot g \cdot h \cdot \Delta T \qquad (1)$$

where g is the gravity acceleration; h is the height of the liquid column; ΔT is the temperature difference between the hot and cold leg; K_t is the temperature coefficient of the density; ΔP_m is the resulting pressure differential.

This driving pressure is equilibrated by the total pressure drop in the whole loop which is function of the velocity of the fluid: $\Delta P_f (v)$.

The resulting velocity v of the liquid is implicit in the relation:

$$\Delta P_m = \Delta P_f (v) \qquad (2)$$

The heating power needed for the above ΔT is:

$$W = v \cdot \pi \frac{d^2}{4} \rho C_p \Delta T \qquad (3)$$

where: W is the heating power; d the diameter of the tube; C_p the specific heat, and ρ the density of the liquid.

The diameter of the tubes is fixed by the space required by the corrosion samples. On the other hand to limit the wall temperature of the heater (to avoid catastrophic fouling) the specific thermal load has to be kept low (3). The heat transfer on the heater has been evaluated with the approximate formula (used for oils):

$$Nu = 0.023\ Re^{0.8}\ Pr^{0.4} \qquad (4)$$

where Nu is the Nusselt number
Re is the Reynolds "
Pr is the Prandt "

From which the area needed to transfer the power W is obtained; this area in turn increases the pressure drop of the circuit (due to the equivalent length of tube added to supply the said area).

At the limit each further increase of Δ T (required by an increased pressure drop) leads eventually to increase the heater area of such an amount that the added pressure drop due to the increased size of the heater, nearly counterbalances the increase in ΔT.

The hydraulic diameter of the heater had to be roughly optimized, on the basis of a temperature drop at the heater wall not higher than 10°C with a clean surface.

The final design is sketched in fig. 1: the heater is made of two concentric tubes. An elicoidal channel is formed on the outer surface of the inner tube, so that the resulting structure is equivalent to a long spiral tube of rectangular cross section.

The heater can be opened for cleaning and repair, by cutting as shown in fig. 1 the main welding (2) between the two tubes.

Two insertion places are provided for thermocouples: one in T_1 for regulating the temperature of the heated liquid, the other in T_2 to shut off the heaters if the liquid does not circulate. The two thermocouples act through separate galvanometric type regulators (with integral action) to increase the overall reliability of the system.

Special sample holders, shown in fig. 2 have been designed to minimize the pressure drop and to give an uniform flow of liquid around the test specimens. The sample holders charged with the test specimens are inserted into the test sections as a string held in place by two elastic retaining rings at the two ends of the string.

Control thermocouples are provided at the inlets and outlets of the test sections for monitoring the temperature of the fluid stream where the specimens are exposed. Auxiliary thermocouples are provided at the cooler outlet and in the main reservoir of the loop.

The sphere-on-cone joints shown in fig. 3 have been designed to avoid any trouble with gaskets.

The conical part is made of AISI 316 stainless steel, the spherical part of AISI 31o stainless steel. Special surface finish is not required.

Tightness of the seal is maintained by means of the constant elastic strain of the floating flanges and the bolts, all made of a low creep steel (17 - 22 - A, ASTM).

Very reliable leak tightness has been always obtained from the beginning without replacement or reworking of the mating surfaces, even after about 20.000 hours of operation at $\sim$ 400°C, in spite of the deformations caused by creep.

The detailed drawing of this type of joint is shown in figure (3a).

2.2 General Description of the Facility

The complicated handling and managing of the terphenyl (m.p. $\sim$ 85 - 90°C) suggested to centralize some general services in groups of loops.

A group of four loops shares: a) the pre-treatment tank, where the raw terphenyl is degassed under vacuum, b) the feed tank, where the degassed terphenyl is stored; c) the main hot water thermostat feeding water at 95 - 110°C to the terphenyl condensers at the outlet of the loops, to the pretreatment tank etc.

All the reservoirs, the feed lines and all the members in contact with the terphenyl are kept at 150 - 250°C by means of electrical heating wires (Thermocoax or Pyrothenax) fed from low voltage AC. The temperature of these components is that resulting from equilibrium between the heating power supplied and the power losses to the ambient.

All loops are alike; the schematic flowsheet of one of them is shown in fig.4. Fresh terphenyl is fed from the storage tank into the main loop reservoir R. It is pressurized there with nitrogen which is fed from a bomb N 2 through a saturator WS, where it is charged of water vapour at a very constant partial pressure. The wet nitrogen is then bubbled in the terphenyl of the reservoir R: in this way the circulating terphenyl is always kept at a well controlled concentration of water and swept free of low boiling compounds.

The gas stream is discharged to the atmosphere after leaving in a reflux condenser Cd (which is cooled by water at 95 - 110°C) all the terphenyl vapours. Terphenyl circulates by thermosiphon action in the direction shown by the arrow. A heater H and an air cooler C provide the necessary temperature difference between the "hot" and the "cold" legs of the loop. Two test sections, T1 and T2, are provided, respectively in the hot and cold legs of the circuit for the corrosion test coupons.

A connection is provided to a fore-vacuum pump for the initial evacuation of the loop. Valves for terphenyl sampling, S, and for drain, D, are provided.

In any case none of the valves is mounted in the main flow path of the loop, none is working at temperatures higher than 250°C. In spite of these precautions many troubles affected the operation with the stainless steel valves formerly chosen (completely welded, bellow-type). High pressure-high temperature cone-type stainless steel valves (*) (currently in use in the oil industry) have proven later to be the most reliable.

Preliminary experiments haven shown that the special composition of the valve gaskets (**) (asbestos-basis) is very stable and does not release objectionable impurities into the terphenyl. The average life of the gaskets is well in excess of one year of continuous operation at 150-250°C.

Side-view level indicators using the same type of gaskets with windows (***) (used in the oil industry) are mounted on the loop reservoirs R. The loop assembly is done with the minimum number of joints; these are all gasketless metal on metal already described.

Each loop, as shown in fig. 5, in enclosed for safety in a protective box, provided with fire estinguishing facilities. All the controls and regulations are centralized on remote control desks: care has been taken to reduce to a minimum the operations to be done inside the protective box while the loop is operating at high temperature.

Fig. 6 shows three of the four loops grouped around the common services.

(*) Type ABMI8 - supplied by Klinger (Germany)

(**) Type Klingeroilit AB 18/2 by Klinger

(***) Type T-D mod. IX supplied by Klinger

2.3 Electrical wiring and safety devices

The schematic of the wiring of one loop and of the general services, grouping four loops, are reported in fig. 7 and 8 respectively.

A short description of the circuit operation will be given here. The electrical power is given to the loop circuit (see fig. 7) by the main switch IG, through TG. A delayed switch CR, fed by auxiliary A.C. from a DC to AC converter (connected to a battery) switches off the loop in the case of mains interruptions longer than the delay of CR. This provision avoids unassisted automatic re-start of the loop.

A pressure gauge on the expansion tank operated PB if the pressurization is insufficient, switching off then part of the main heating, RP 1. The gauge operates PA if the pressure is higher than permitted, opening through R 2 an electromagnetic valve EV which vents the tank to the atmosphere.

In the worst case a rupture disc bursts and the escaping gas operates DR which switches off the heater RP 1.

A manual reset, IP is provided to resume the operation after a disc rupture.

The main heating power is divided in two parts: one is continuously fed, R P1, the other RP, is switched on and off by the thermoregulators TR 2 and TR 1. The first of these is the regulating one, the other acts as a safety device against overheating.

When the heating RP1 is disconnected, the cooler of the loop is kept hot by PR to avoid plugging by solid terphenyl.

The preheating power of the loop components IRA, is always on during the work, feeding a multi-voltage network for the thermocoax heating cables, which keep all the loop components at a temperature higher than the melting point of the terphenyl.

The general services, see fig. 8 provide recording the temperatures of the loop through Reg 1 and Reg 2, hot water recirculation by means of PT, RT and TR 1, automatic battery recharging, CA, and preheating of the feeding tubes, reservoirs and of all the components through IRA.

The problems of checking the efficiency of the thermocoax heating cables and of locating faults has been solved by connecting a small amperometric resistor in series with each heater. The voltage drops across each resistor are then checked by means of an AC voltmeter and a suitable (and not shown) group of switches at the console.

All the electrical switching and controls are centralized in a control room as shown in fig. 9, tc reduce fire hazard.

This system has been operating without troubles since the first start-up. The built-in interlocks and the careful design of the safety system permitted the continuous operation of the loops without any surveillance except during start-up or shut-down.

3. Starting procedures and routine controls

Referring to fig. 4 the starting procedure followed after fitting the test sections is:

a) preheat the loop, while purging it with nitrogen;

b) fill to the prescribed level with fresh terphenyl from the feed tank by pressurizing this with nitrogen;

c) pressurize the loop with nitrogen to half the final working pressure while heating at full power. During the heating period the pressure of the cover gas is gradually increased to avoid backflow of the terphenyl in the gas line;

d) When the working temperature is reached the gas pressure is brought to the working value (15 kg/cm^2) and the water saturator (previously by-passed) is connected to the gas line. Its temperature is regulated to obtain the prescribed water concentration in terphenyl.

The start-up of the thermosiphon circulation and the flow direction is easily checked by means of the six temperature readings taken around the flow path and recorded.

Samples of the circulating terphenyl are withdrawn and analized for water content and chlorinated impurities concentration. These two analyses are continued routinely during the run; the content of high boiling compounds is eventually measured to decide when the terphenyl has to be changed.

The frequency of the analyses is typically:

a) water content: once a day

b) chlorinated impurities

   i) low-chlorine runs: twice a month
   ii) high-chlorine runs: twice a week

c) high boilers: once a month

All temperatures and the experimental parameters are reported dayly on the operation log book.

Non-routine controls provide checks of the thermocouples and of the temperature recorders with a precisions potentiometer, as well as restandardisation of the thermocouples.

The long term stability of the controlled parameters in long-term runs is as follows:

temperature $\pm$ 1°C at about 400°C
water content $\pm$ 10 ppm. " " 100 ppm (by weight)

It is very difficult to control the concentration of the chlorinated impurities. These, in fact, are rapidly gettered by the loop and by the corrosion coupons; the analysis (by activation) is slow, so that there is actually no way of regulating this parameter.

The pressurizing gas can be changed to mixtures of different gases, if changes of the solute equilibrium in the flowing terphenyl are needed.

No detailed checks of the stability of composition are available for this case, however,

4. Measurement of the speed of the circulating terphenyl

The very low head present in the loops prevented the use of a standard method for the measurement of the speed of the liquid.

A heat impulse method has been then developed for this special case. One of the test section has been modified as shown in fig. 10: a spiral heater wire A (bare nichrome) is placed at the inlet of the test section.

Two very fine thermocouples differentially connected are placed downward the flow, one near the heater wire, the other at a known distance from the first.

The differential signal from the two thermocouples is observed on a high sensitivity DC oscilloscope and eventually recorded.

The operation of the device is very simple: a relatively long (2-3 seconds) impulse of current is fed to the heating spiral; the scanning of the oscilloscope starts at the same time; two peaks of opposite polarities are observed on the oscilloscope at the passage of the small portion of hotter fluid at the two thermocouple places. The time between the peaks (and the distance between the thermocouples) give the speed of the fluid.

Mixing and turbulence, however, deteriorate the second peak, so that only approximate measurements are possible.

Fig. 11 shows two examples of the records obtained with the above method, which, although imprecise, does not perturb the equilibrium conditions of the loop and give then the true velocity in the working conditions.

The speed measurements have been done in the following conditions:

a) various temperature differentials
b) temperature of hot leg at 380°C or 420°C
c) both the test sections empty; or one filled with test coupons (the other was measuring the speed).

The results are reported in table 1. The reproducibility of the actual speed measurement is acceptable (at least for this type of work). In fact the casual difference between the loops have a much greater influence than the errors in the speed measurements reported in table 1. The values shown are averaged over 4 - 6 time measurements whose error

Table 1

Terphenyl speed in the test section

(cm / sec) at:

Loop No.	(+) $T_{(hs)}$ = 420°C T = 20/22°C	(+) $T_{(hs)}$ = 420°C T = 15/17°C	$T_{(hs)}$ = 380°C T = 20-22°C	$T_{(hs)}$ = 380°C T = 15-17°C
1	13.1	12.6	12.8	10.7
2	-	-	15.2	13.7
3	19.9	16.1	17.2	14
4	19.8	18.7	16.3	15.4
4*	15.4	-	14.8**	-
5	15.3	11	14.9	13.9
6	15.8	12.6	15.8	14
7	16.3	15.8	14.5	12.6
8	-	-	-	14.4

All the speeds are averaged over 3 - 9 observations

*with one test section loaded **at T_{av} = 400°C

(+) $T_{(hs)}$ is the temperature of the hotter test section.

seems lower than $\pm$ 5 % in spite of the roughness of the method. Better measurements were not required so that no further data have been collected.

The speed of the liquid when the loop is in the working conditions (i.e. both the test sections loaded) has been extrapolated from the data obtained

a) with both the test sections empty

b) with one test section loaded with samples

The extrapolated speed is around 10 - 12 cm/sec. at a Δ T of 20°C for loop No. 4.

The velocity falls within the design limits and is satisfactory for corrosion work.

An independent check of this velocity (with the two test sections full) has been done by interrupting the "proportional" action of the thermoregulators. This causes sinusoidal oscillations around the average value of the temperature of the terphenyl with an amplitude of about 10°C.

The time-lag between temperature peaks (or valleys) of different thermocouples and their distance gave approximate measures of the speed of the liquid which confirmed the values given above.

5. Conclusions

The loops described here have been specifically designed for corrosion experiments in high temperature terphenyls. At present the plant has operated continuously for about 20.000 hrs without surveillance and requiring only routine maintenance with an excellent performance.

Cleaning and pickling has been done twice after the end of each major corrosion run. On this occasion leaky weldings have been spotted and repaired. These were found generally in loops working with higher (1-2 ppm) concentrations of chlorinated impurities, or in tubes working in the gas phase above the terphenyl, where hydrochloric acid from the pyrolythic decomposition of the chlorinated impurities is more likely accumulated.

These leaks have been attributed to stress corrosion at weldings, but the internal surface of a small, unwelded tube (the gas vent) has been found in one occasion reduced to a sponge by corrosion in the gas phase. For that case we don't have an explanation.

Acknowledgements

Mr. F. H. Krenz (AECL liaison scientist at the CCR of Ispra at the time of this design) is gratefully acknowledged for his very valuable suggestions and discussions.

Mr. G. Gontier is acknowledged for his continuous assistance to the loop construction.

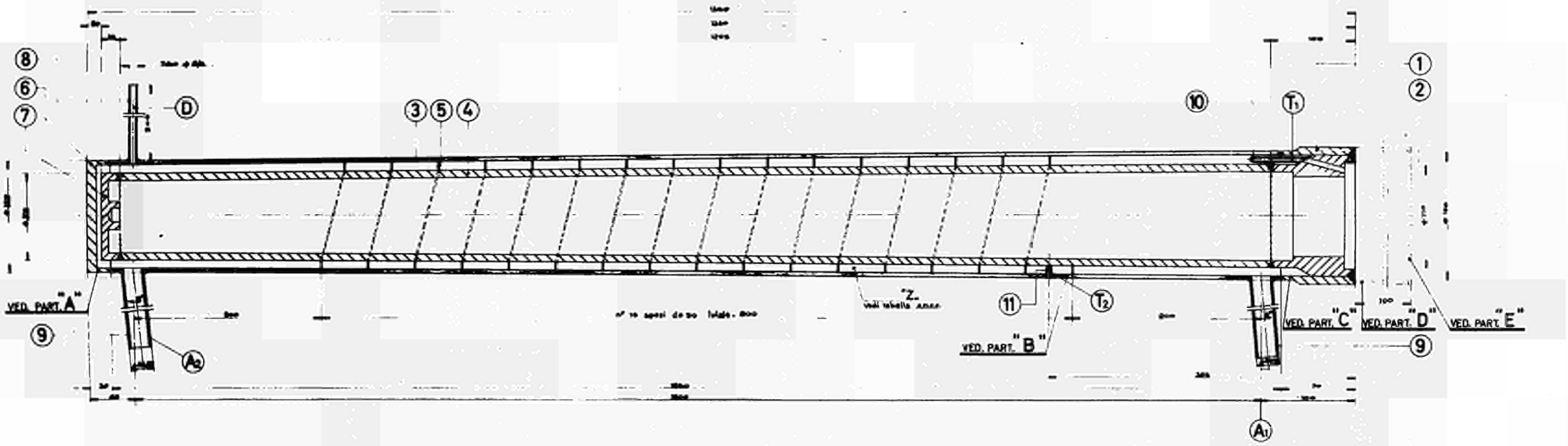


Fig. 1 : The main heater

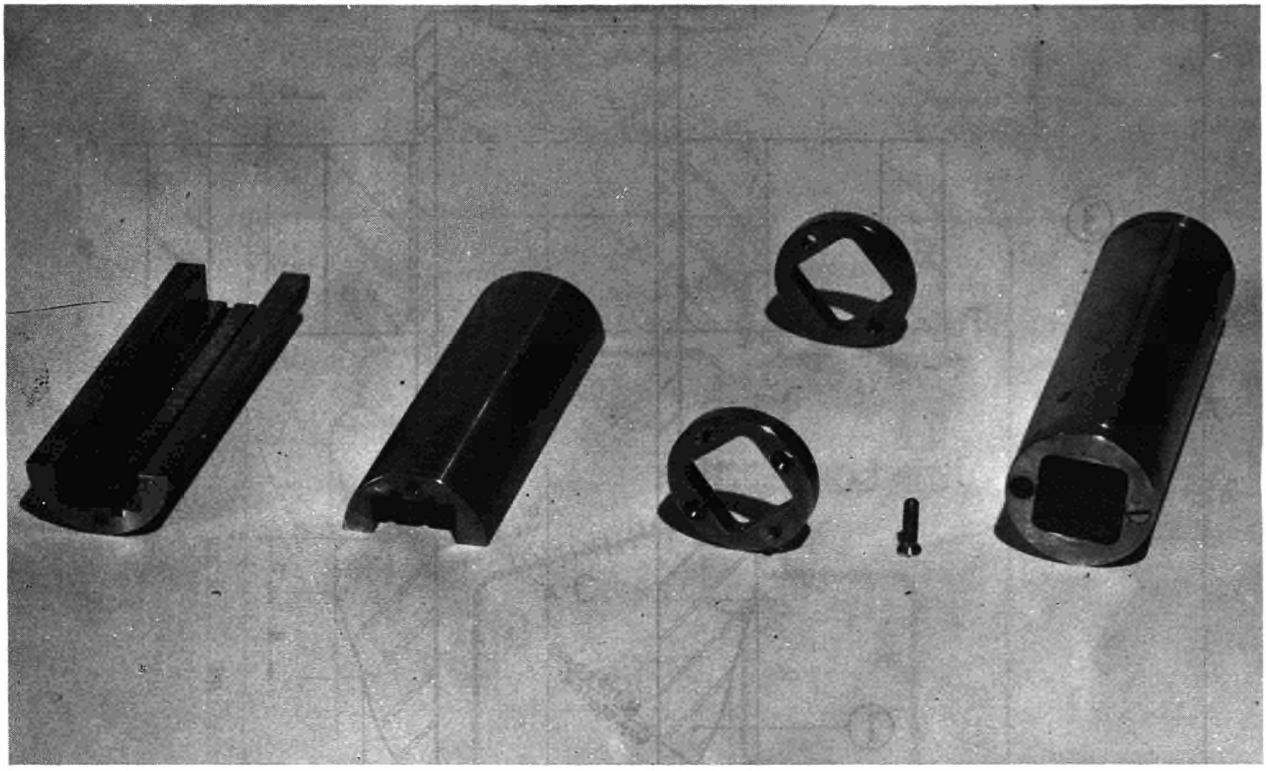


Fig. 2 : The sample holder

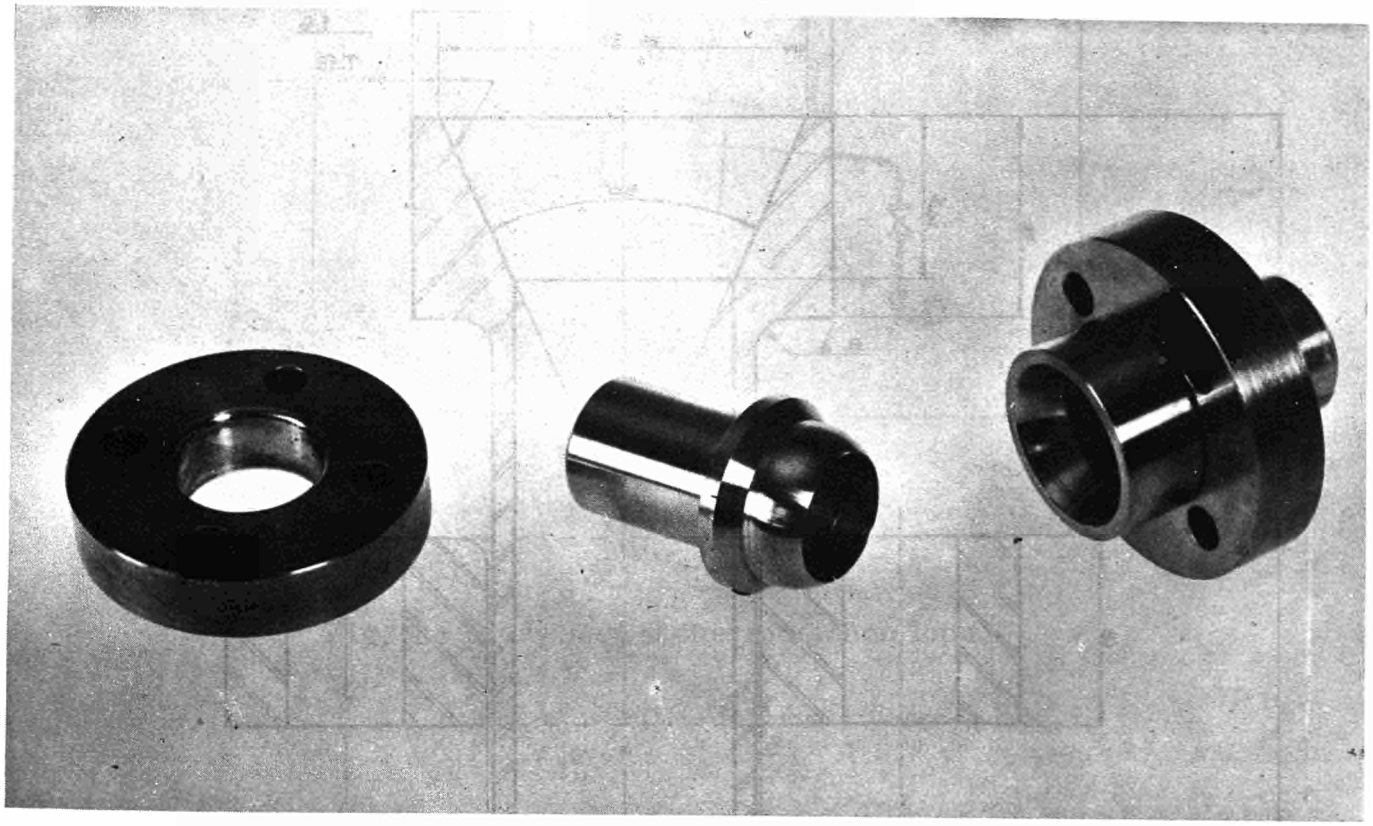


Fig. 3 : Gasketless Joints

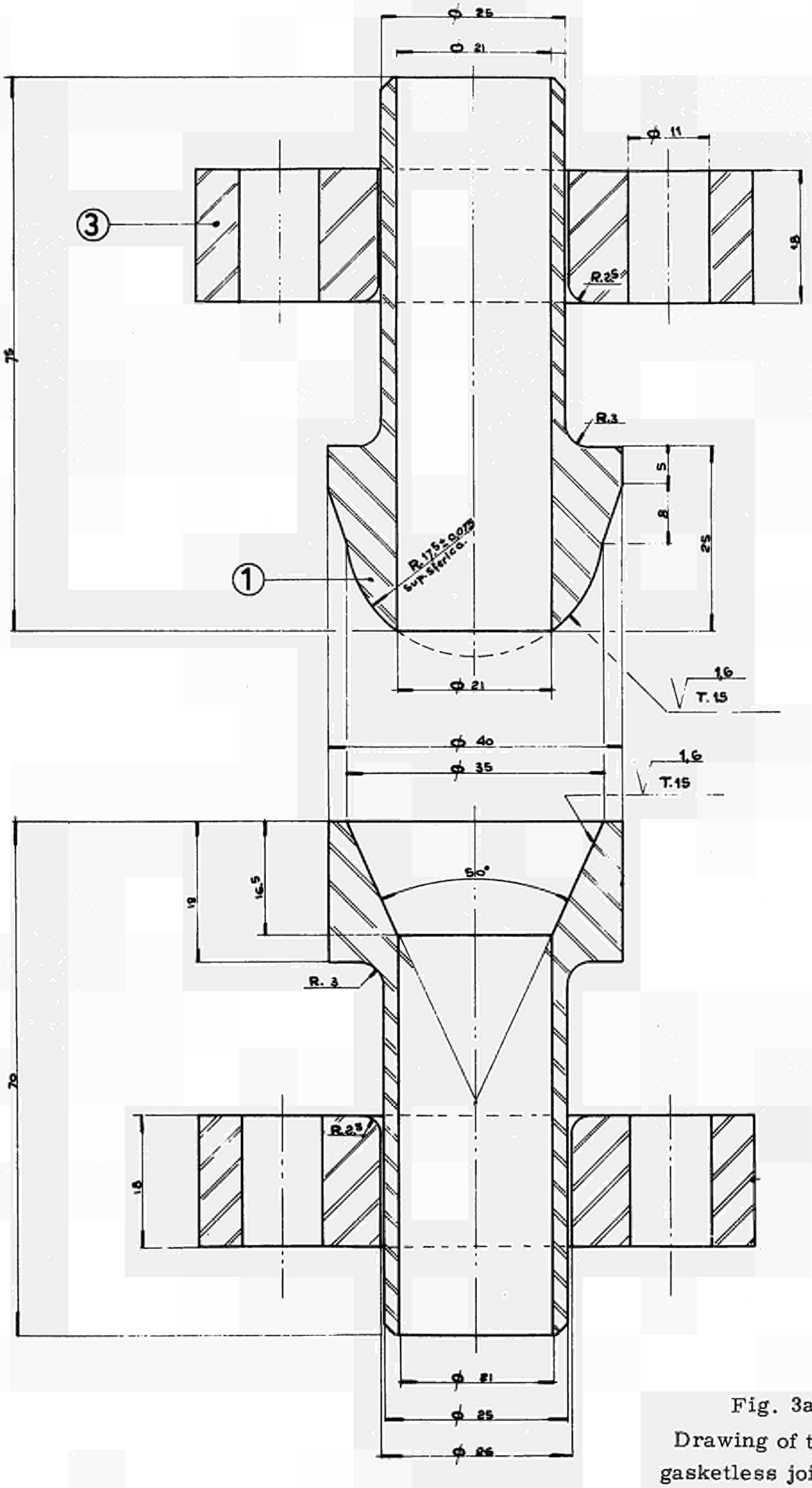


Fig. 3a :
Drawing of the
gasketless joints

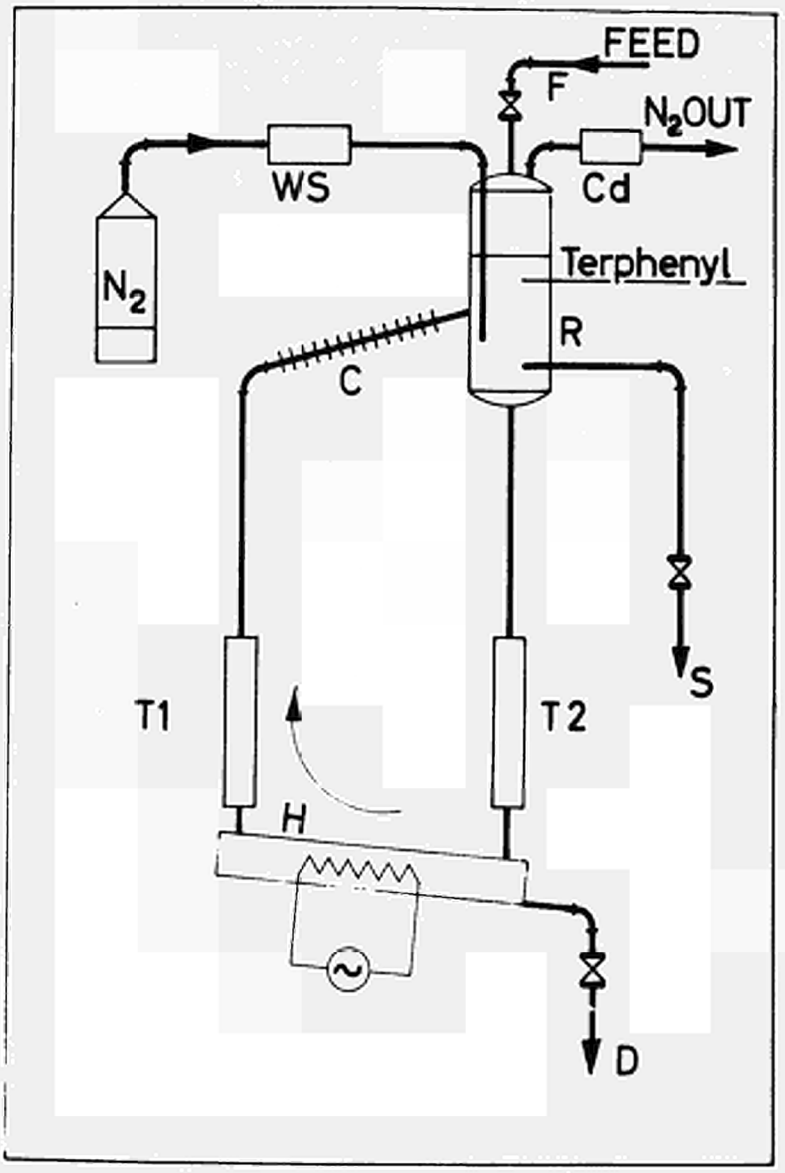


Fig. 4 : Flowsheet of thermosiphon loop

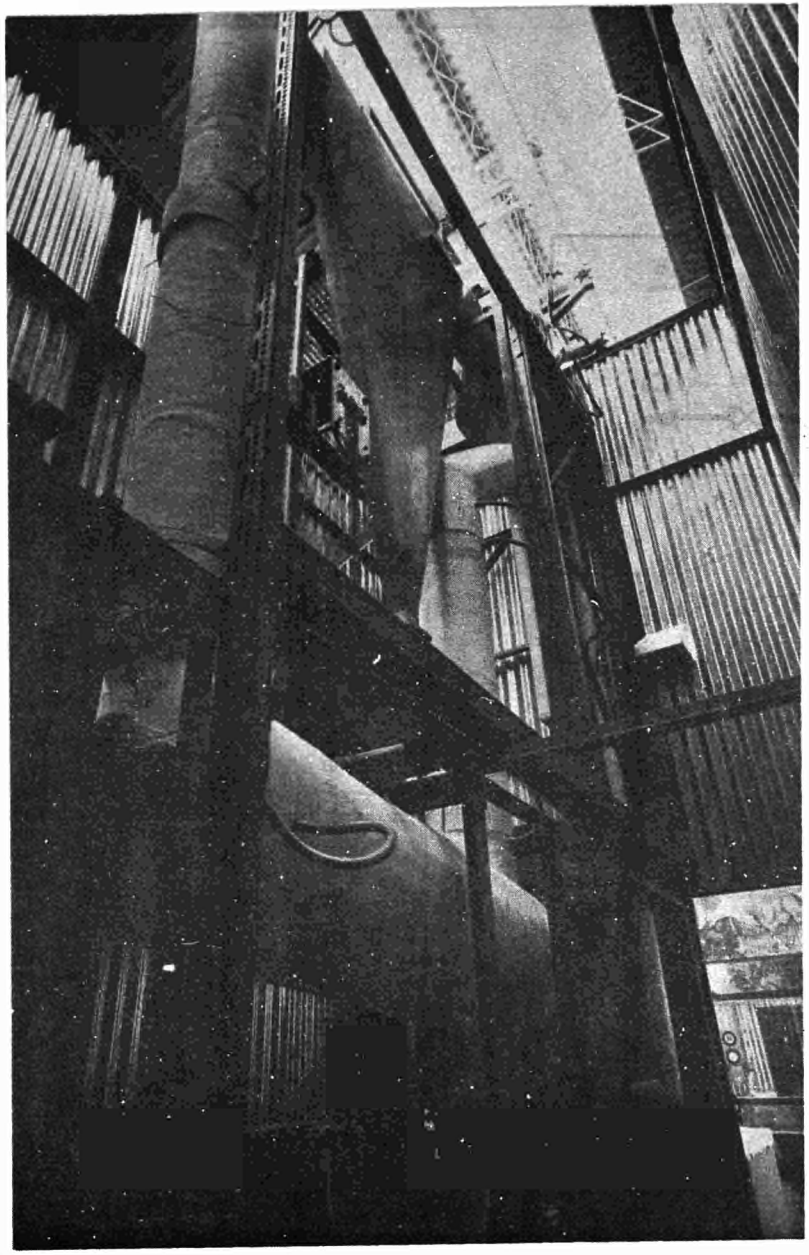


Fig. 5 : View of a loop

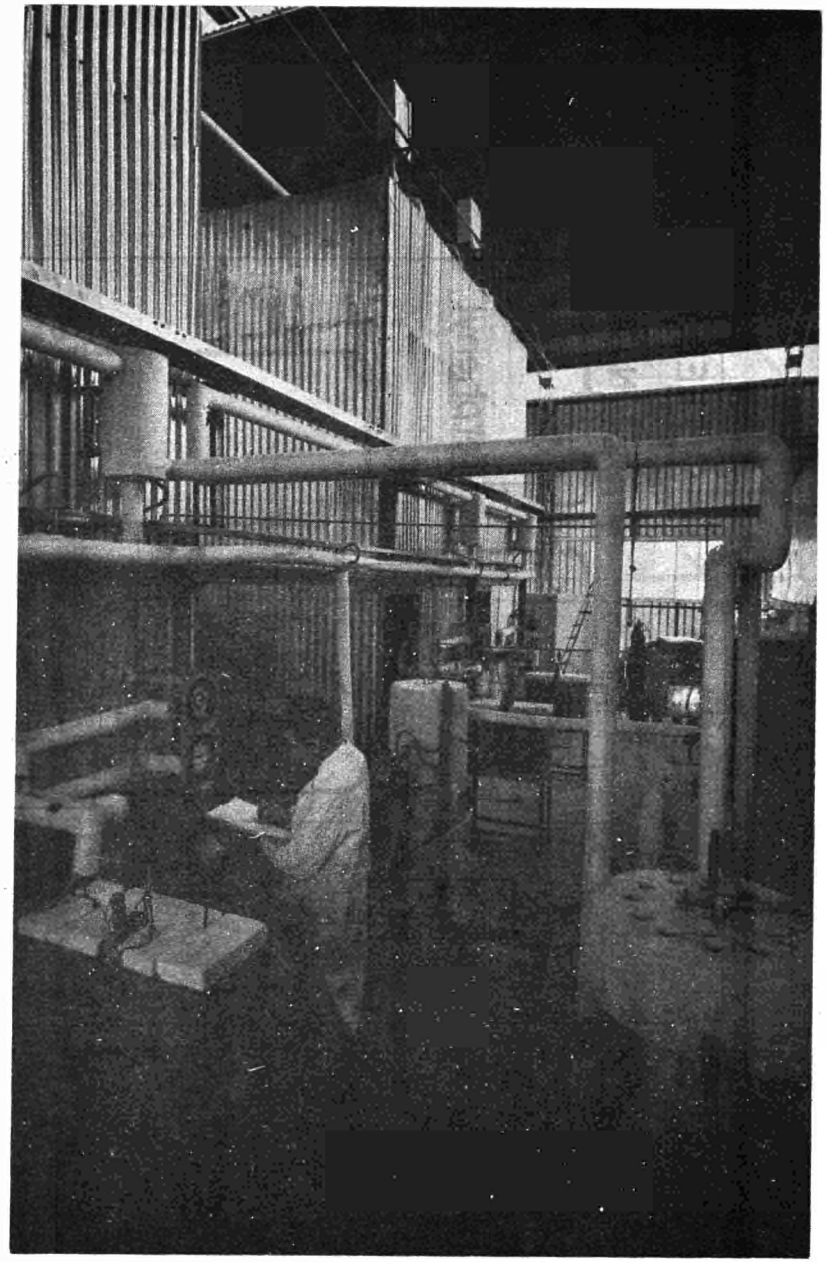


Fig. 6 : General view of a group of four loops

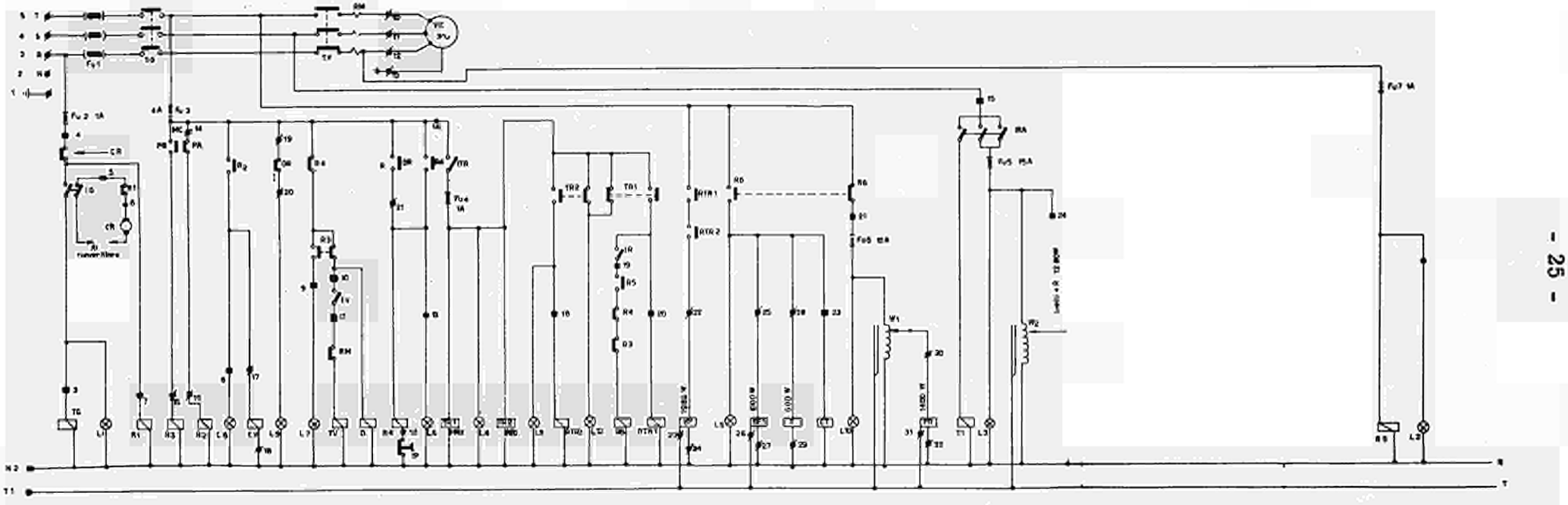


Fig. 7 : Wiring diagram for one loop

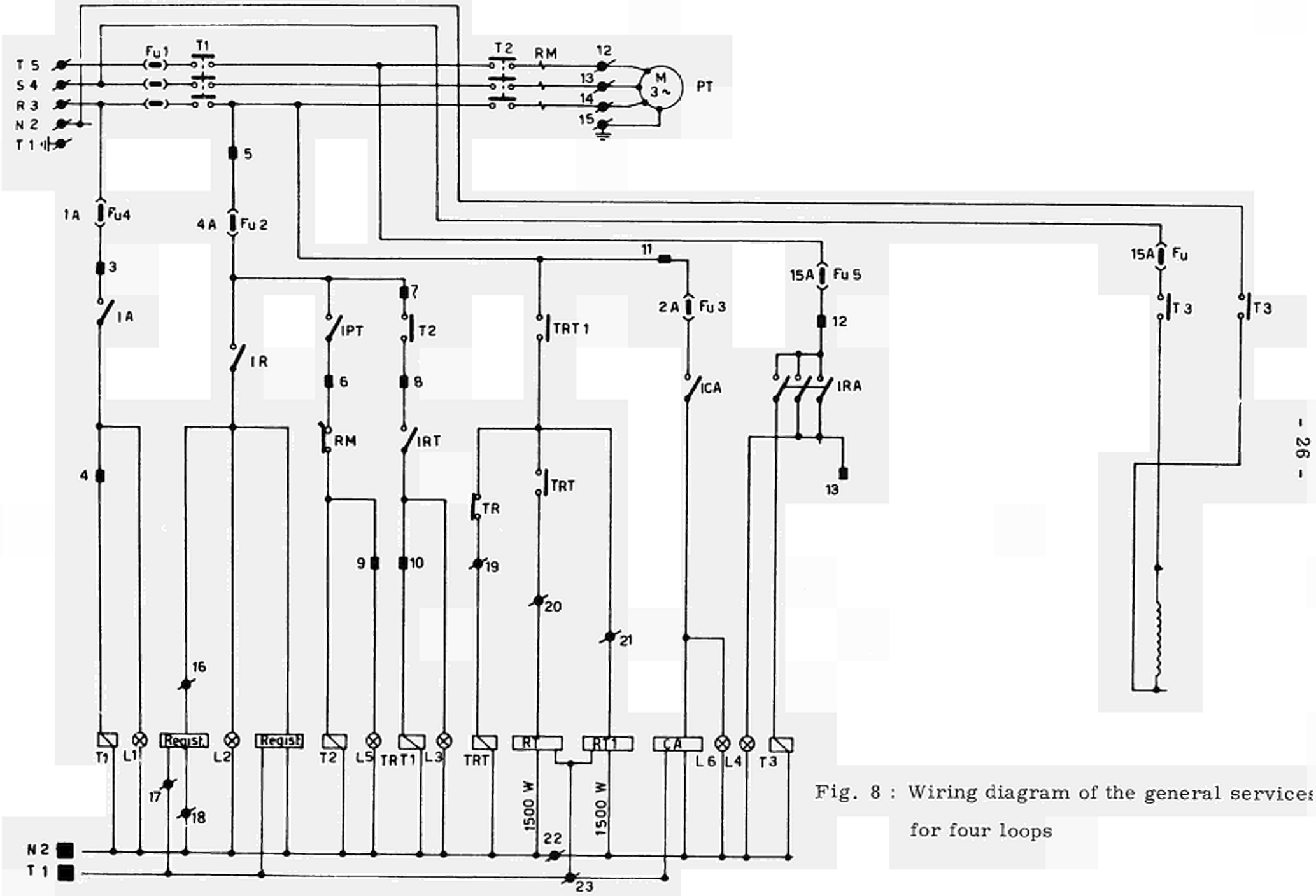


Fig. 8 : Wiring diagram of the general services for four loops

Fig. 9 : View of the control consoles

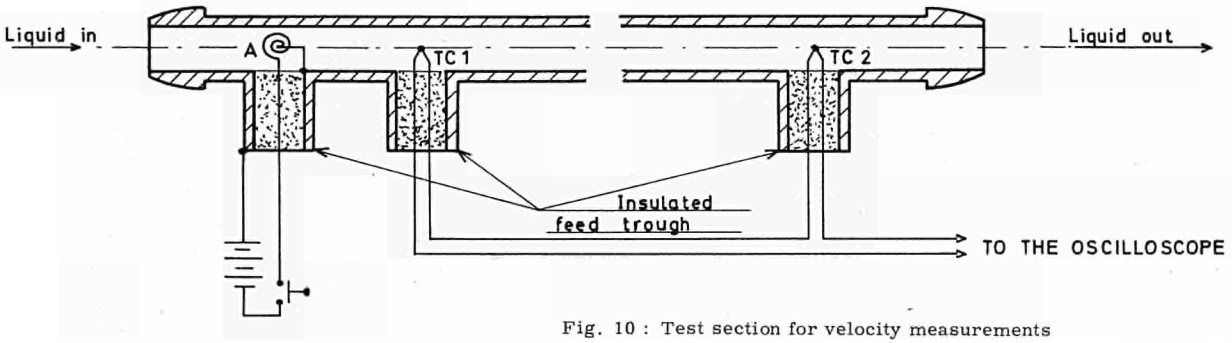
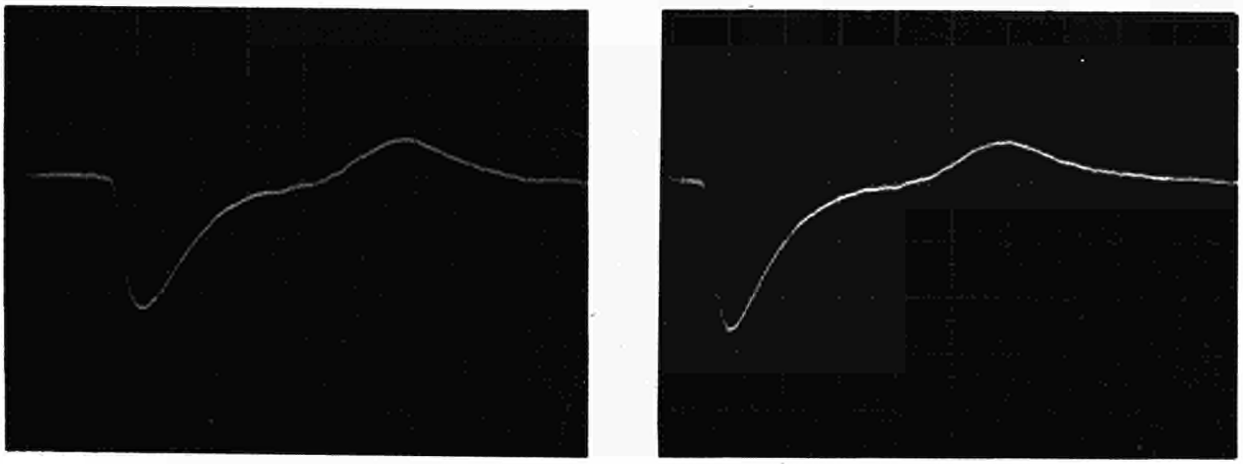


Fig. 10 : Test section for velocity measurements



a) T = 420°C

b) T = 400°C Δ T = 20°C

Fig 11 : Records of the heat pulses
taken for velocity measurements

Δ T = 20°C; horizontal deflection: 25/cm
vertical sensitivity: 200 μV/cm

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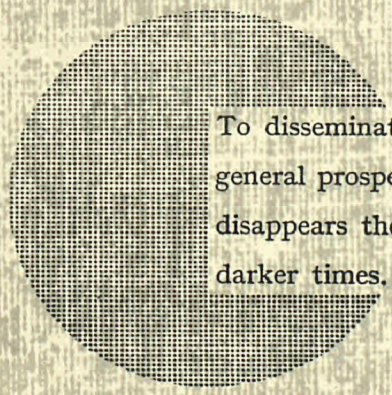

To disseminate knowledge is to disseminate prosperity — I mean general prosperity and not individual riches — and with prosperity disappears the greater part of the evil which is our heritage from darker times.

Alfred Nobel

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