Mr Sa

FRI 17/1973

F.R.I. 47

VERSLAG NR.

VAN

REPORT NO. 17

OF 1973

BRANDSTOFNAVORSINGSINSTITUUT VAN S.A.
VERTROULIK
CONFIDENTIAL
FUEL RESEARCH INSTITUTE OF S.A.

U1/E/3/2

BRANDSTOFNAVORSINGSINSTITUUT

VAN SUID-AFRIKA

FUEL RESEARCH INSTITUTE

OF SOUTH AFRICA

ONDERWERP:
SUBJECT: A DESCRIPTION OF THE DELAYED COKING APPARATUS

AFDELING:
DIVISION: ENGINEERING

NAAM VAN AMPTENAAR:
NAME OF OFFICER: T.C. ERASMUS

Cosmi 9228 - B4

FUEL RESEARCH INSTITUTE OF SOUTH AFRICA

REPORT NO. 17 OF 1973

A DESCRIPTION OF THE DELAYED COKING APPARATUS

1. The essential feature characterizing the delayed-coking process is the high speed with which the temperature of the material to be processed is brought to the level at which the coking process is to be carried out. Ideally, the transient temperature period should be of such short duration that the coking process is initiated only once the predetermined processing temperature has been attained, and not before.

   In practice, the molten material is passed through a suitably designed heat exchanger, in which the temperature of the material is raised rapidly to the so-called coking temperature, and then allowed to react in a pressurized reaction vessel until the coking process is completed.

   At the instigation of Sasol, the Fuel Research Institute undertook to construct a small-scale version of the delayed-coking apparatus to study the fundamentals involved. It was proposed that the apparatus should be capable of batch-processing 1-litre quantities of various substances, including a low-ash-content coal extract.

2. In order to obtain the necessary background experience, an inexpensive experimental rig was constructed in which the maximum use was made of readily available components. A diagrammatic representation of the rig is reproduced in figure 1.

   The pumping stage consisted of a storage tank feeding a variable-speed dieselene injector pump. Both units were heated indirectly by means of an electrically heated oil bath. The heat exchanger consisted of a coiled length of tubing passing vertically through an electric furnace, the temperature of which determined the rate of heat transfer. The electrically heated tubular reaction vessel was fitted with a water-cooled reflux condenser and the system was pressurized, using compressed air. Pressure regulation was effected by means of a spring-loaded release valve.

At the end of the charging operation, the reaction vessel was isolated from the remainder of the apparatus. Flushing fluid was admitted to the storage tank and continually circulated through the heat exchanger until the furnace temperature had dropped to a safe level.

Several test runs were conducted, using Sasol pitch which was selected chiefly because of its availability. In brief, the results obtained revealed the following:

a) For a small-scale version of the apparatus, a pumping system incorporating non-return valves is unreliable. Unsteady and even interrupted flows were observed.

b) In the event of an interruption in the flow, rapid cooling of the heat exchanger is required to prevent charring taking place within the heat exchanger tube.

3. In the present version of the apparatus, pumping is effected by means of a piston pump capable of performing the entire pumping operation in a single stroke. The pump serves a dual purpose in that it also serves as the storage tank. Details of the pump are given in figure 2.

A standard Fordson tractor sleeve, having a nominal bore of 98 mm, served as the cylinder. This was fitted with a 2 kW sheath-type electric heater (not shown in the figure) capable of melting the charge in approximately

/20 .....

20 minutes. The temperature of the cylinder was measured with a thermocouple, welded to the outer wall, and temperature regulation was effected by means of an Ultrakust solid-state controller, adjustable within the range 50 - 500°C.

The piston was constructed from cast iron and a complement of five compression rings was needed to eliminate leakage entirely. A constant piston displacement velocity was effected by means of a constant speed screw-drive arrangement. The latter is also fitted with a shearing pin as a safety precaution against pressure rupture. The piston stroke is adjustable and the limits of the stroke are determined by means of micro-switches which, when activated, interrupt the power supply to the drive motor. The circuit diagram is given in figure 3. The drive motor was fitted with a direction change-over switch.

The cylinder head, which is attached after the charge has melted, is fitted with a manually controlled booster element. By means of this, its temperature, measured by a thermocouple, is raised above the melting point of the test material before the pumping operation is commenced. Thereafter, its temperature is maintained by the heat conducted from the molten charge.

The pressure inside the pump, affording an advance warning of an impending blockage, is monitored electronically. The force exerted on the piston is transmitted, via the screw-drive, to the thrust plate. The elastic deformation of the thrust plate is detected by means of a strain gauge bridge, the amplified output of which is indicated as a deflection on a micro-ammeter.

4. The modified heat exchanger consists of a horizontally positioned, spiralled tube, sandwiched between two conventional stove hot-plates. Clamping is effected by means of a quick-release arrangement, and in the

/unclamped .....

unclamped position they may be swivelled clear of the heat exchanger tube for rapid cooling.

The temperature of the hot-plate is measured by means of a thermocouple attached to the upper hot-plate, and the temperature regulation is effected by means of an Ultrakust solid-state controller, adjustable within the range 100 - 800^{o}C.

The outlet temperature from the heat exchanger is measured by means of a thermocouple welded to the heat exchanger tube.

Several experiments, using Sasol pitch, were conducted to optimize the dimensions of the heat exchanger tube. It was found that with a 60 cm length of 0,27 cm I.D. copper tubing, outlet temperatures in excess of 500^{o}C can be obtained without difficulty. The residence time of the material within the heat exchanger is 13 seconds.

The heat-exchanger-pump complex can be isolated from the reaction vessel by means of a stainless steel gate valve, while a similar valve was employed as a pressure-relief valve.

5. The original tubular reactor had functioned satisfactorily and was thus incorporated, without modification, in the present version of the apparatus.

The reactor, 2,1metres in height, was constructed from temperature-resistant stainless steel and has an internal diameter of 82,5 mm and a wall thickness of 6 mm. The bottom of the tube is blanked-off, whereas the top flange cover gives access to the heat exchanger and the reflux condenser. Provision was also made for tilting the reactor into the horizontal position to facilitate product retrieval.

/Heating .....

Heating of the reactor is accomplished by means of two .2 kW heating elements wound at a pitch of 1 cm and covering the entire length. Each element consists of 16 gauge nichrome resistance wire, electrically isolated from the reactor body by means of ceramic beads. To ensure a uniform temperature profile, a Calrod booster element was fitted to the bottom of the reactor. Temperature regulation was effected by controlling the power supply to each element, using an on-off type Foster indicating controller.

Thermal insulation was effected by means of a 50 mm thick layer of Triton Kaowool, secured by means of sheet-metal cladding.

The reflux condenser comprises a 25 mm I.D. stainless steel tube, one metre in length, which is fitted with a water jacket. Mechanical carry-over of vapour droplets is reduced by means of segmental baffles within the condenser, as well as a low-velocity tar trap downstream to the condenser. The reactor is pressurized with nitrogen admitted at the tar trap.

The pressure within the reactor is adjustable within the range 0 - 7 bar, being controlled by means of a Foxborough pneumatic indicating controller having as final control element a stainless steel needle valve venting to atmosphere. Safety protection is afforded by means of a spring-loaded safety relief valve.

A diagrammatic representation of the present version of the delayed coking apparatus is given in figure 4.

6. The principal features of the apparatus are listed below.

/1. <u>Pump</u> .....

1. <u>Pump</u>: positive displacement, single-stroke action.
   a) Bore: 98 mm
   b) Stroke: 150 mm maximum
   c) Pumping rate: 1000 cm^3/h
   d) Temperature range: 50 - 300°C, safe.

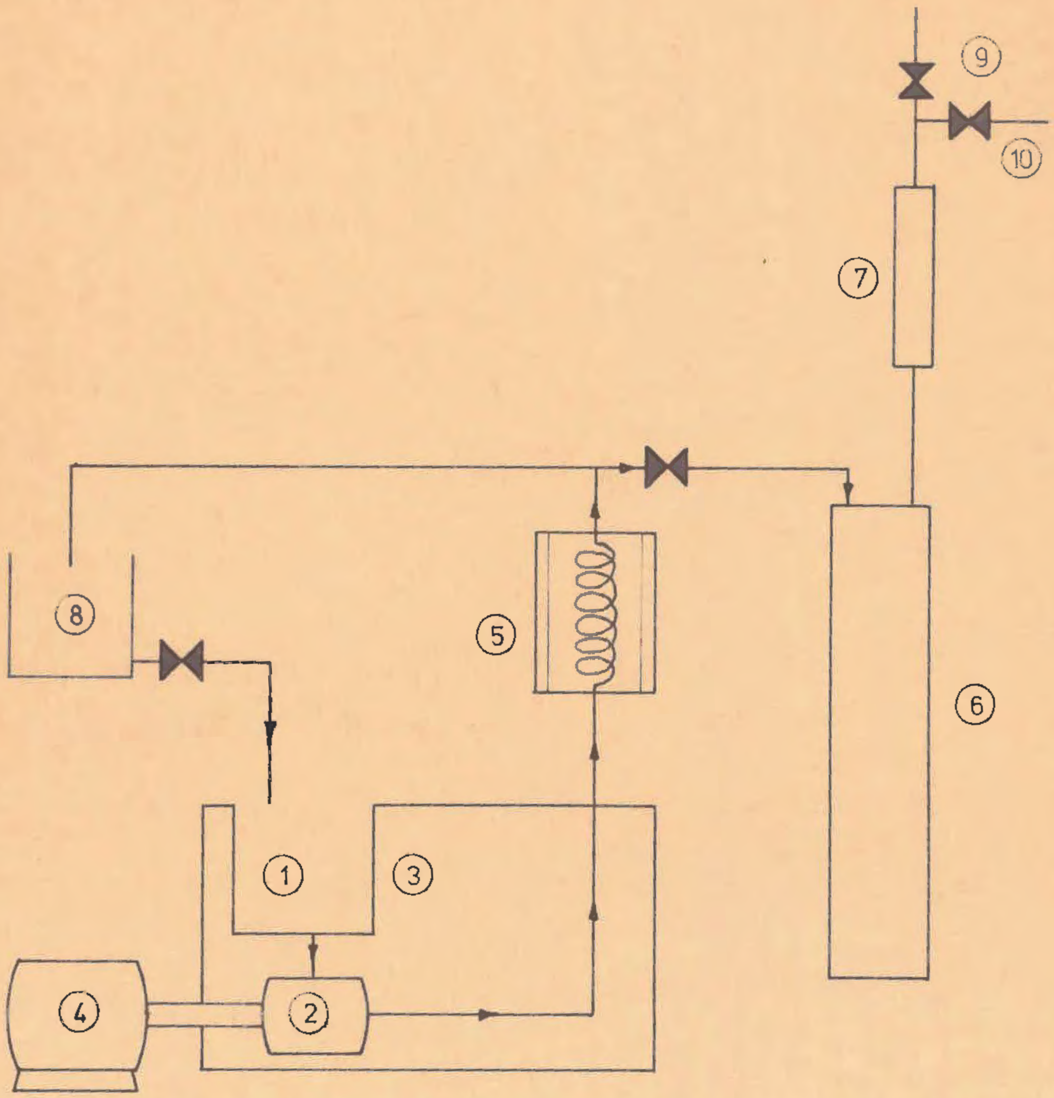
2. <u>Heat exchanger</u>: single tube sandwiched between two hot-plates.
   a) Tube length: 60 cm
   b) Tube I.D.: 2,7 mm
   c) Maximum exit temperature: ca. 600°C

3. <u>Reactor</u>: vertical tubular reactor with top feed.
   a) Height: 2,1 metre
   b) Internal diameter: 82,5 mm
   c) Wall thickness: 6 mm
   d) Maximum operating temperature: 550°C
   e) Maximum operating pressure: 7 bar

A literature survey is being compiled in report form and will be distributed in due course.

T.C. ERASMUS
CHIEF RESEARCH OFFICER

Pretoria.
29th November, 1973.
TCE/EMc

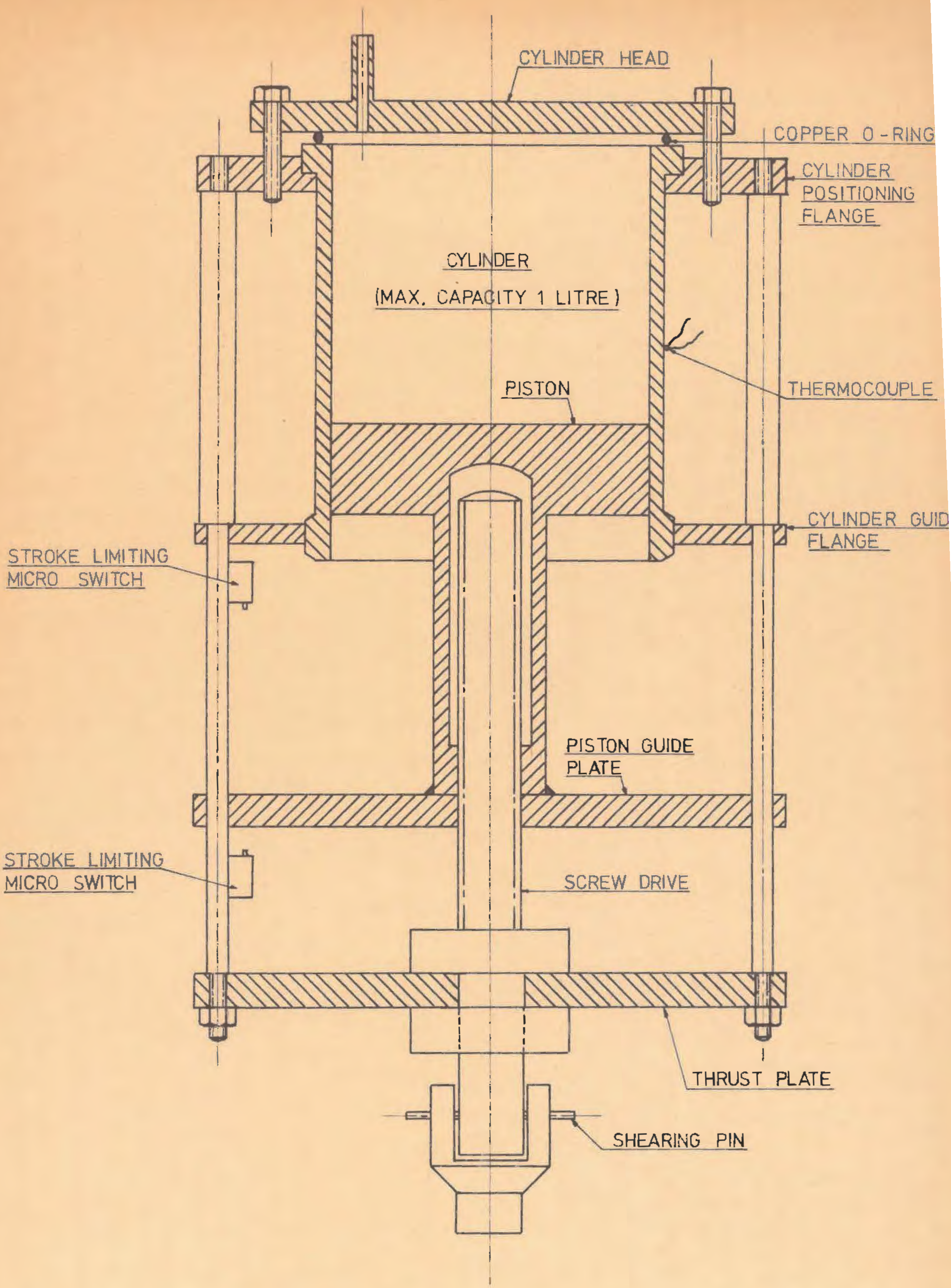


KEY:

1. STORAGE TANK.
2. RECIPROCATING PUMP.
3. OIL BATH.
4. VARIABLE SPEED MOTOR.
5. HEAT EXCHANGER.
6. REACTION VESSEL.
7. REFLUX CONDENSER.
8. FLUSHING FLUID TANK.
9. PRESSURE RELIEF VALVE.
10. COMPRESSED AIR CONNECTION.

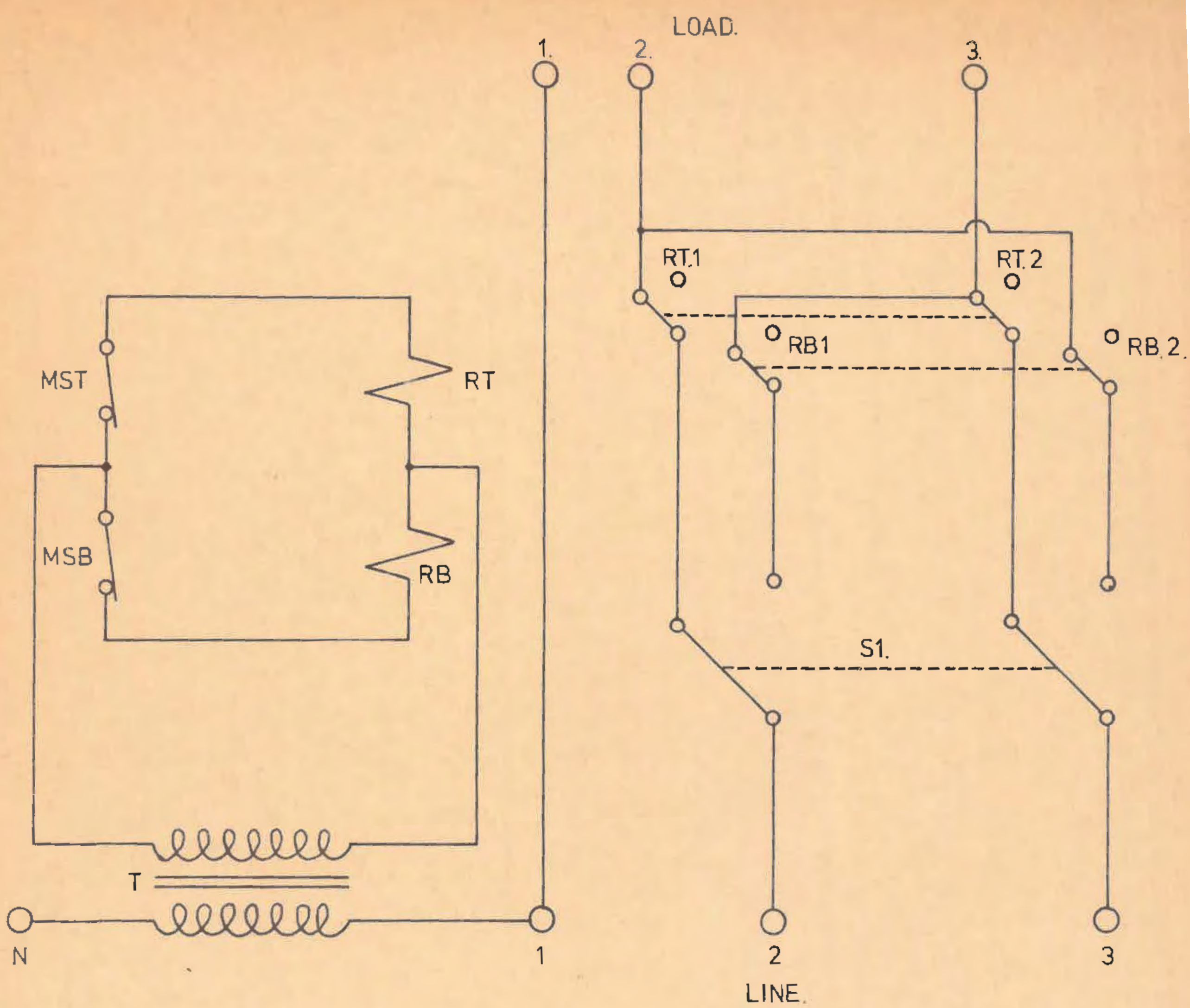
DIAGRAMMATIC REPRESENTATION OF EXPERIMENTAL RIG.

FIG. 1



THE PISTON PUMP IN SECTION

FIG. 2

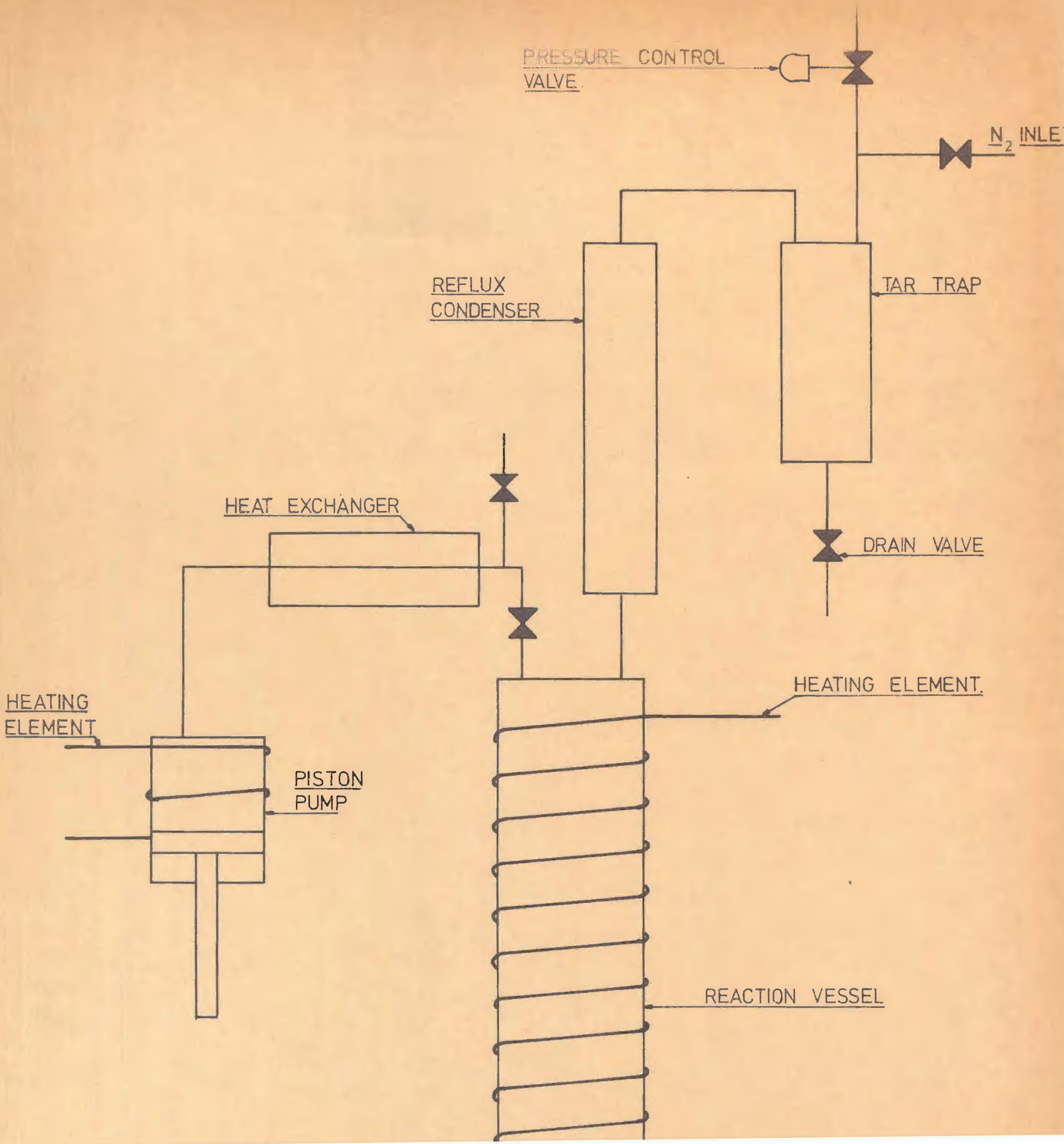


KEY:

S1.	FORWARD/REVERSE SWITCH.
MST.	MICRO SWITCH TOP (N.C.)
MSB.	MICRO SWITCH BOTTOM (N.C.)
RT.	RELAY COIL (N.O.)
RB.	RELAY COIL (N.O.)
T.	TRANSFORMER 220/6 VOLT.

3 PHASE MOTOR DIRECTION-CHANGE SWITCH WITH PISTON STROKE LIMIT PROTECTION.

FIG 3.


PRESSURE CONTROL VALVE
N_2 INLET
REFLUX CONDENSER
TAR TRAP
HEAT EXCHANGER
DRAIN VALVE
HEATING ELEMENT.
HEATING ELEMENT
PISTON PUMP
REACTION VESSEL