Chapter III

EXPERIMENTAL INVESTIGATION

3.1 Equipment Design

3.1.1 Glass column as fluidized bed

The detailed design of the equipment is shown in Fig.
3.1. The general arrangement of the equipment is shown in
Fig. 3.2-A. The equipment consisted of two parts. The upper
part was a glass tube of 2 inches ID., 18 inches in height.
The lower part was conical with bottom opening of ¼ inch ID.
and connected with rubber tubing. The conical part was
filled with porcelain balls ¼ inch in diameter for the pur-
pose of homogeneous distribution of gas. Between the two
parts, a stainless steel perforated plate was associated with
a 120 mesh stainless steel screen and sealed by rubber ring.
This glass tube was used as the fluidization testing column.
Two glass tubes ½ inch ID., one from the upper part and
another one from the lower part of the column, were connected
by rubber tubes to a manometer. The glass column was sup-
ported by a clamp and a hook on a steel rod, two feet in
height.
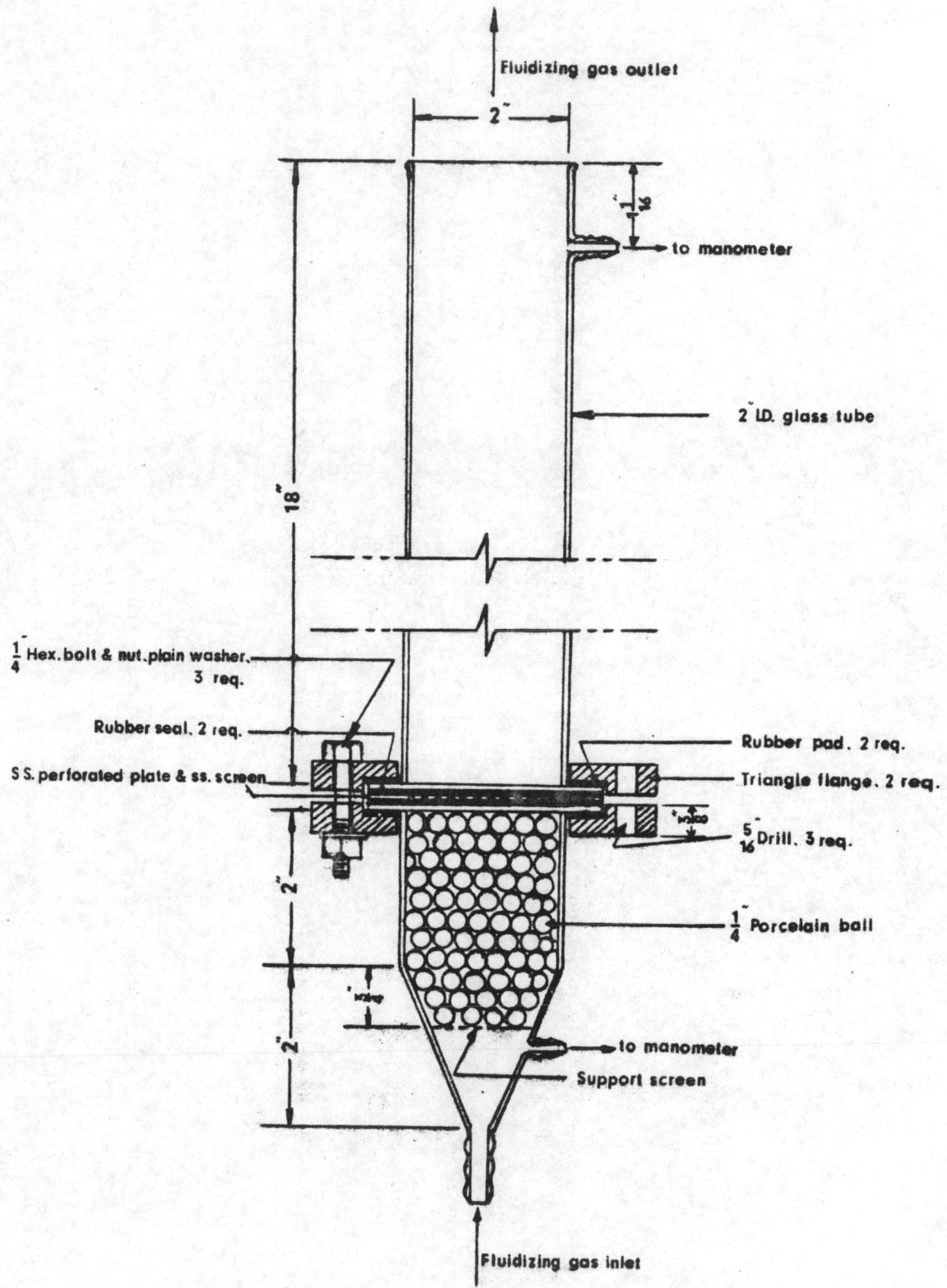
Fig. 3.2-B. shows the fluidized bed of oil shale parti-
cles.

Fluidizing gas outlet

2"

$\frac{1}{16}$"

→ to manometer

2" I.D. glass tube

18"

$\frac{1}{4}$" Hex. bolt & nut, plain washer, 3 req.

Rubber seal, 2 req.

S.S. perforated plate & ss. screen

Rubber pad, 2 req.

Triangle flange, 2 req.

$\frac{5}{16}$" Drill, 3 req.

$\frac{1}{4}$" Porcelain ball

2"

→ to manometer

Support screen

2"

Fluidizing gas inlet

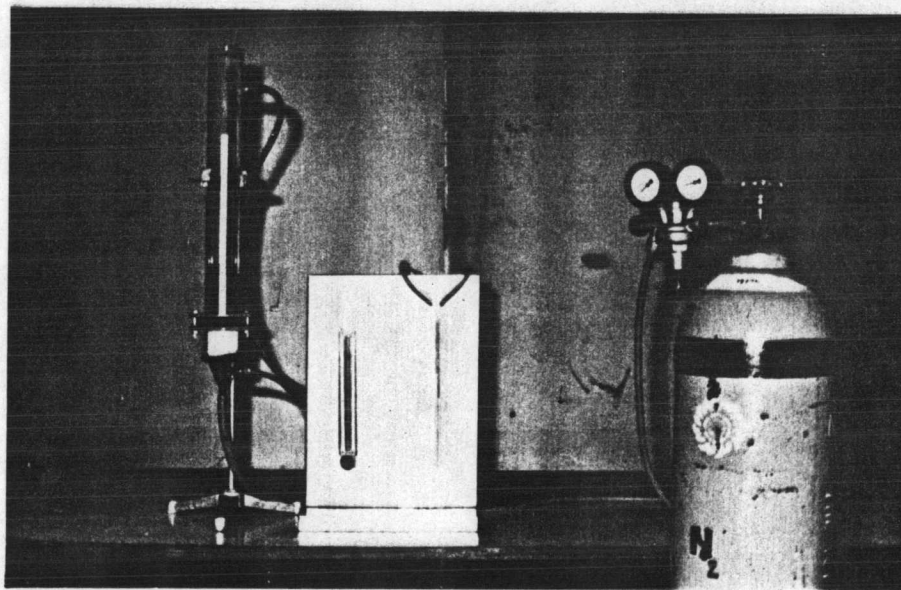**FIG. 3.1- FLUIDIZED BED FOR TESTING Umf**

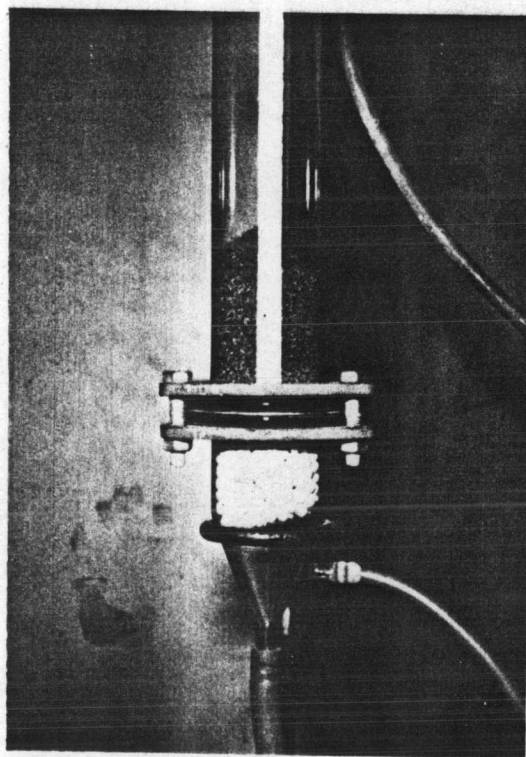FIG. 3.2 –A– FLUIDIZATION TESTING

FIG. 3.2 –B– FLUIDIZED BED OF OIL SHALE PARTICLES

3.1.2 Oil shale retorting equipment

The arrangement of the equipment for retorting oil shale, in order to study the effects of retorting temperature and particle size of oil shale on oil yield, is shown in Fig. 3.3 and Fig. 3.4.

Detailed calculation on the design of retort size and power required for the electrical heater is given in Appendix A.1 and A.2.

The detailed design of each unit of the equipment is presented separately as follows :

3.1.2.1 Retort. A 100-gram capacity (weight of raw oil shale) retort was built and operated as a batch unit consisting of a cylindrical stainless steel 2 inches ID. by 18 inches high. The retort comprised of a distributor as shown in Fig. 3.5 and the packings which were of fire resistance characteristics. A copper tubing ¼ inch ID., 2 meters long was coiled about 3 inches in diameter and welded to the bottom of the lower part. The coil was used for preheating the fluidizing gas. Three thermocouples were jointed on a conical cap which covered the top of the retort and was partially opened for filling of raw shale or removing of spent shale. A stainless steel tube ½ inch in diameter by 10 inches long was welded to the side of the column, one inch below the upper flange making minus 45° in angle to the column. This
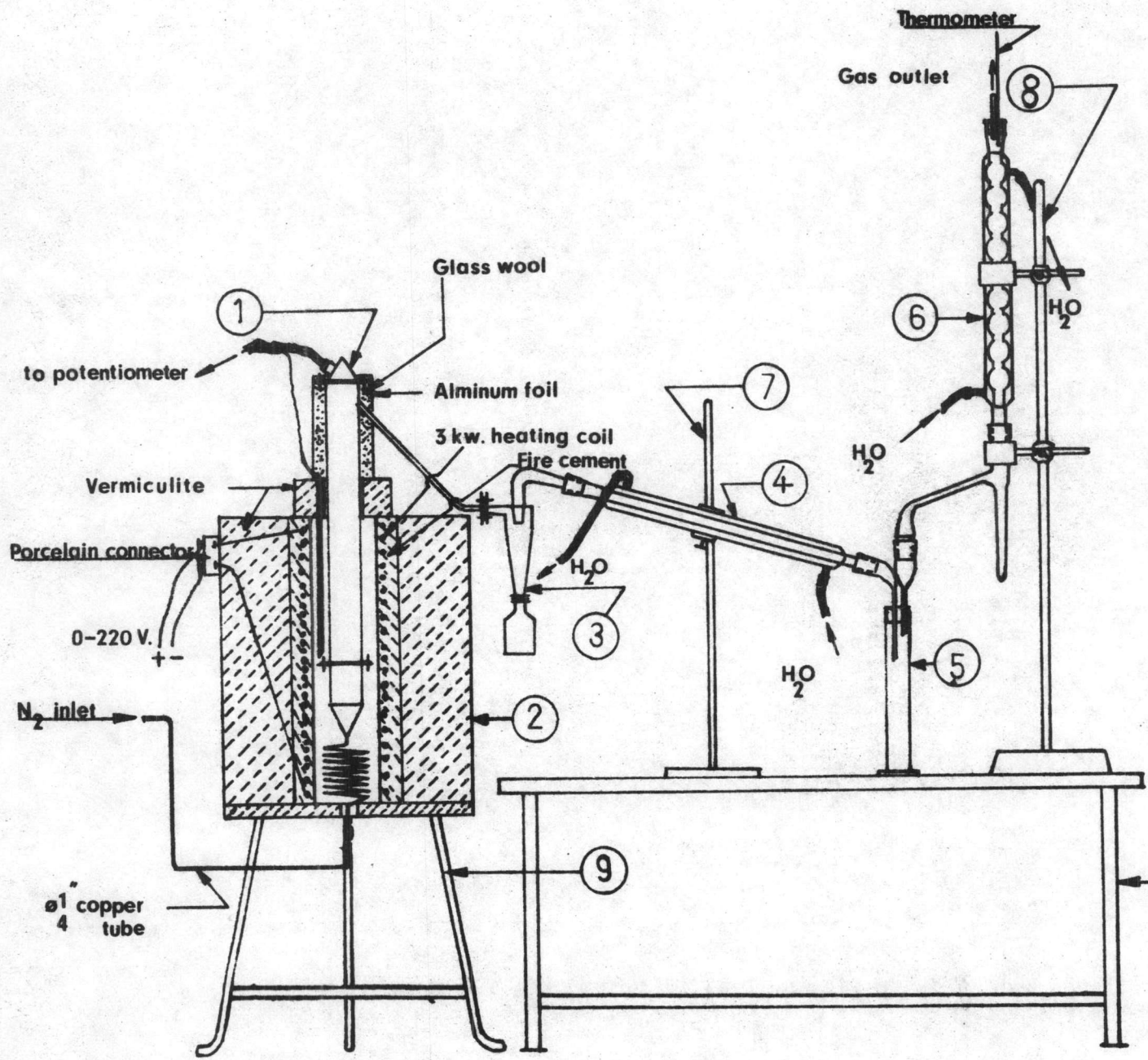
List of equipment

1. RETORT
2. HEATER
3. CYCLONE
4. CONDENSER NO.1
5. MEASURING CYLINDER
6. CONDENSER NO.2
7. STAND NO.1
8. STAND NO.2
9. TRI-POD
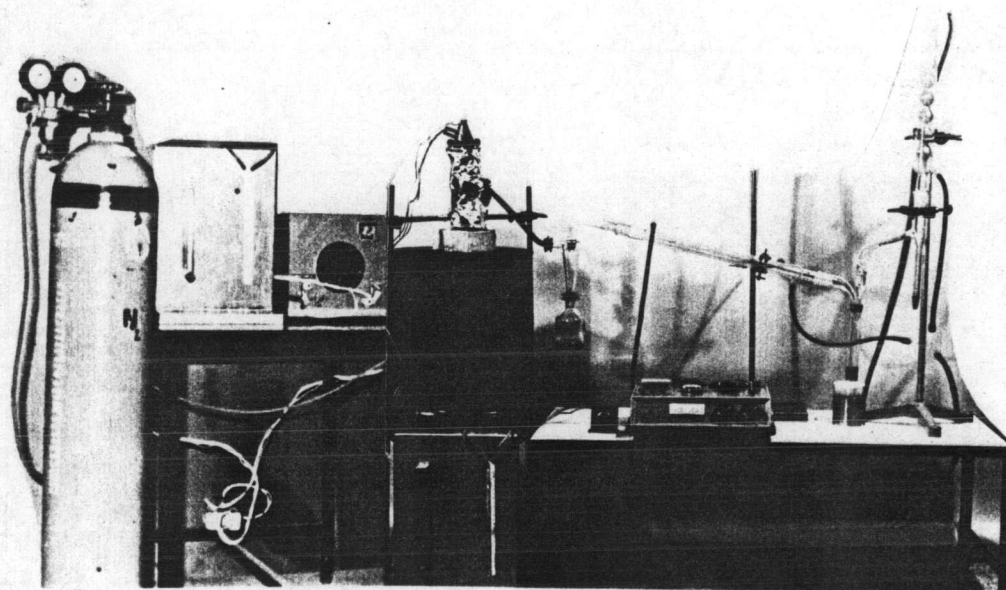10. TABLE

FIG. 3.3- ARRANGEMENT OF EXPERIMENTAL EQUIPMENT

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FIG. 3.4 – A PHOTOGRAPH SHOWING THE ARRANGEMENT
OF EXPERIMENTAL EQUIPMENT

$\frac{5}{16}$ Drill. 4 req.

120 mesh ss. screen

Fire resistance packing. 2 req.

SS. perforated plate. $\frac{1}{8}$ drill by $\frac{1}{8}$

Porcelain ball. ø $\frac{1}{4}$

SS. support screen

$\frac{1}{4}$ Copper tube

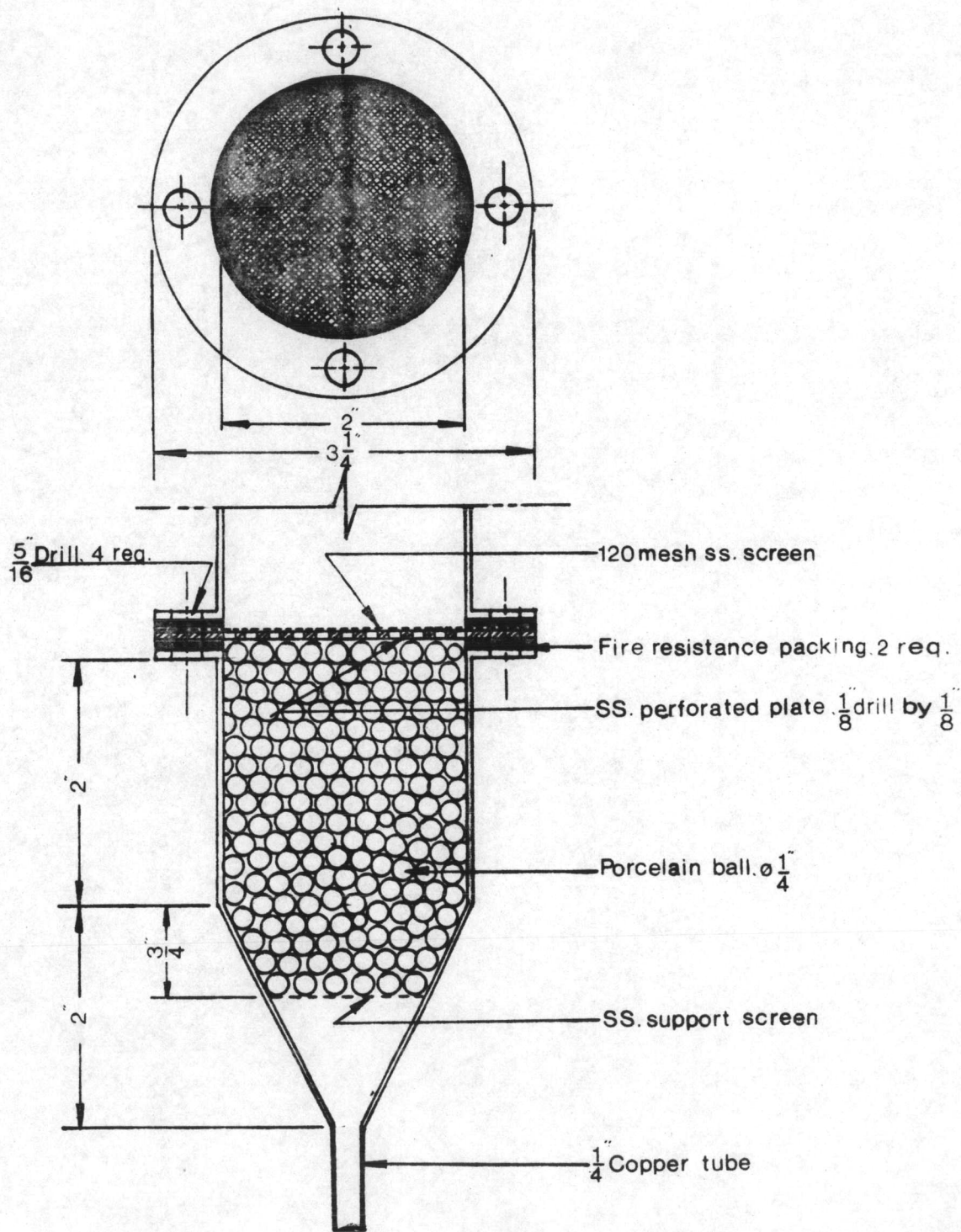2″

$\frac{3}{4}$″

2″

2″

3 $\frac{1}{4}$″

FIG. 3.5 – DISTRIBUTOR

tubing was connected to a cyclone. Fig. 3.6 and Fig. 3.7 shows the drawing and the photograph of the retort, respectively.

3.1.2.2 Cyclone. To prevent dust or small particles of oil shale from the fluidized bed (retort) to contaminate the product yields, a cyclone was used to collect the dust. A ½ inch ID. pyrex glass cyclone (Fig. 3.8) of standard dimensions (21) was connected to the retort via the stainless steel tube at the side.

3.1.2.3 Condensers. Two Jena glass condensers with standard ground joints were used to recover the liquid products such as shale oil and water. One was a sloping condenser with standard joints 24/29, length of jacket about 400 mm. The other was a condenser with standard joints 29/32 having the same length. An adapter was connected to the first condenser and its open tube to a 100 ml measuring cylinder. Another adapter was connected to a water determination apparatus which was then connected to the second condenser.

Both condensers were supported by clamps and stands, are shown in Fig. 3.3.

3.1.2.4 Heater. A 3000-watt heater was constructed to provide heat to the oil shale particles in the retort. The heater consisted of a steel cylinder 4 inches in diameter and 18 inches high and coiled around with heating
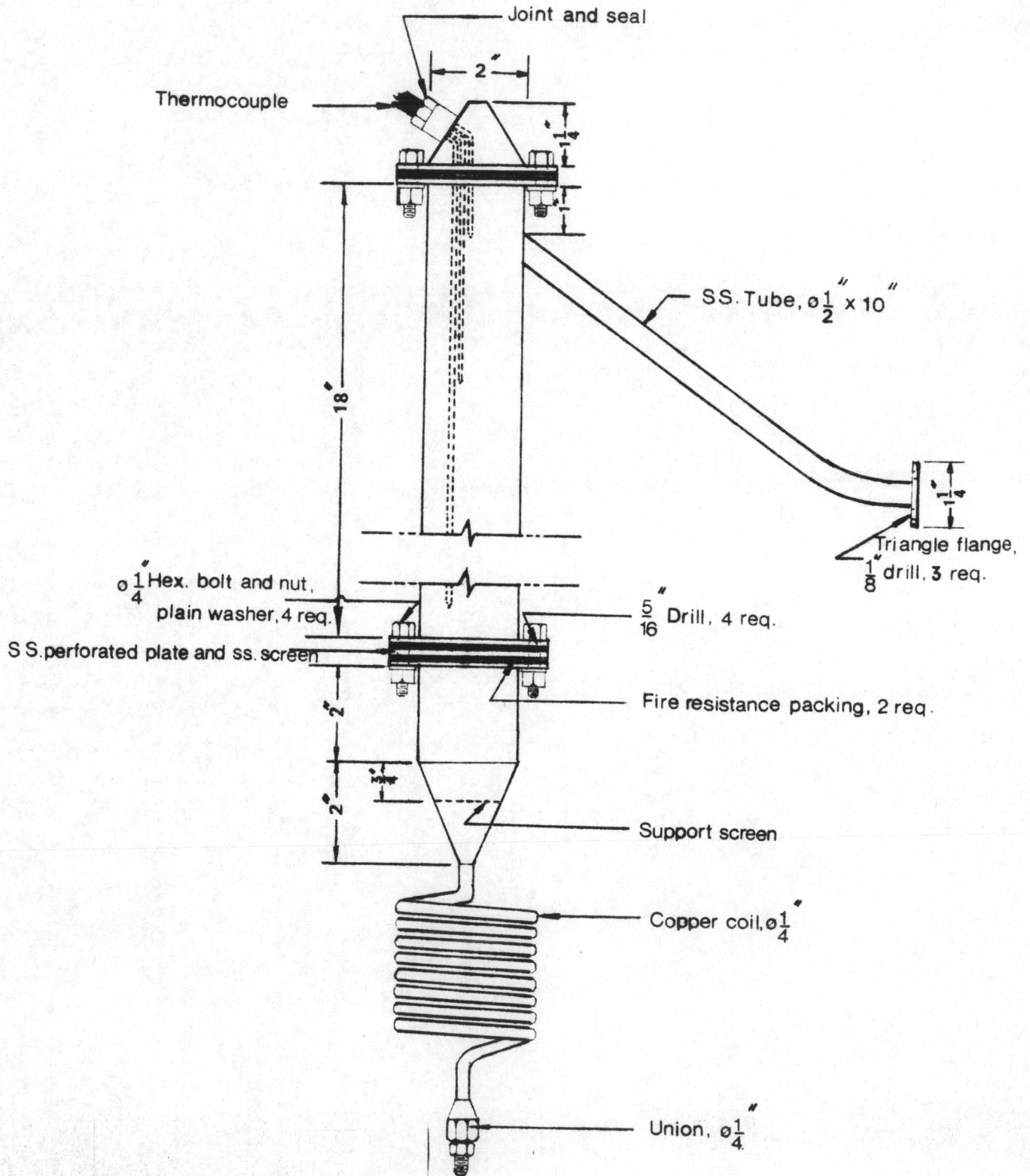
Joint and seal

Thermocouple

2"

$1\frac{1}{4}$

$1\frac{1}{4}$

18"

S.S. Tube, $\phi\frac{1}{2}"$ × 10"

Triangle flange, $\frac{1}{8}"$ drill, 3 req.

$\phi\frac{1}{4}"$ Hex. bolt and nut, plain washer, 4 req.

S.S. perforated plate and ss. screen

$\frac{5}{16}"$ Drill, 4 req.

Fire resistance packing, 2 req.

2"

2"

Support screen

Copper coil, $\phi\frac{1}{4}"$

Union, $\phi\frac{1}{4}"$

FIG. 3.6 - RETORT
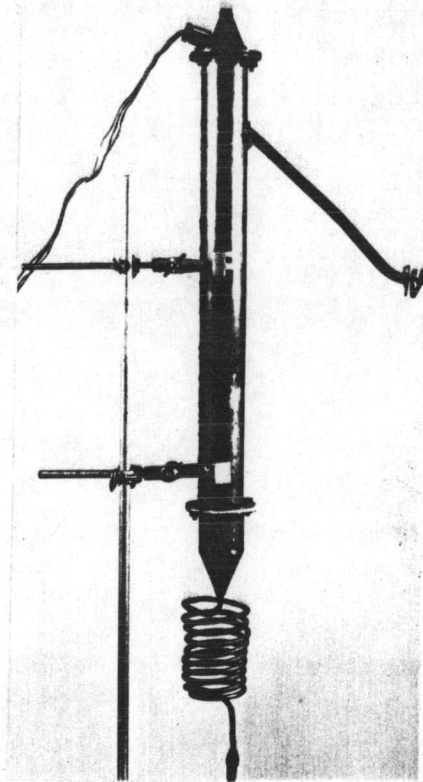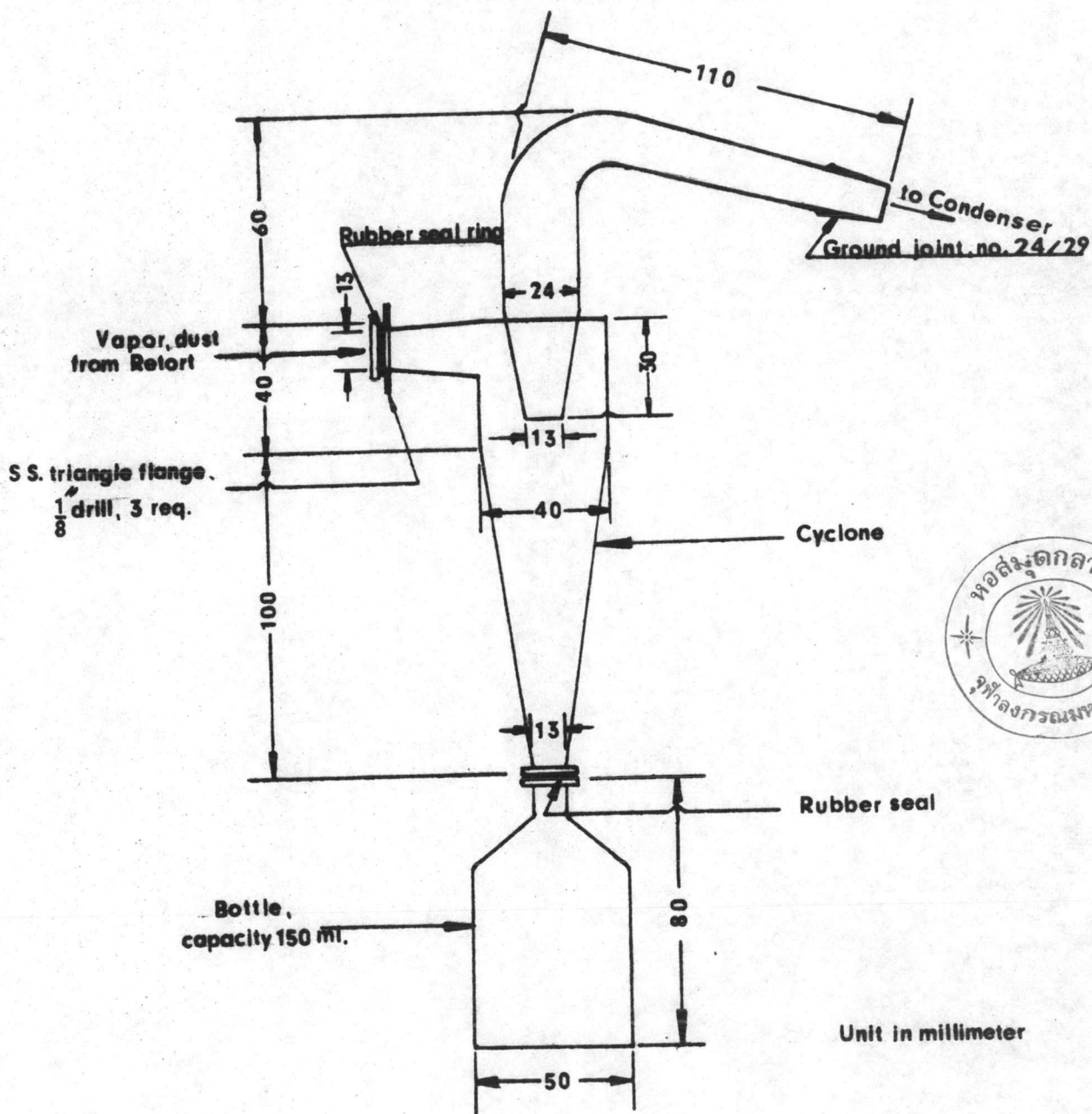
FIG. 3.7- A PHOTOGRAPH SHOWING THE

RETORT

FIG. 3.8 – CYCLONE

wire. Fire cement was used to support the coil and to pre-vent contaction of coil and steel cylinder. The heater was insulated by vermiculite, 6 inches in thickness. The out-side wall was covered by a steel plate. This electrical heater was supported by a tri-pod 15 inches in height. The drawing of the heater is shown in Fig. 3.3.

Its temperature was adjusted by a variable trans-former having the following characteristics : 50 cycles in frequency, input voltage 220 volts, output voltage 0-220 volts, current 15 amps, and power 4500 volt-amps.

3.2 Experimental Design

3.2.1 Sampling of oil shale

This method was performed to determine the particle size of oil shale by sieve analysis (22).

3.2.1.1 Apparatus

Crusher. - Roll crusher was used for reducing the size of oil shale particle.

Sieves. - Square-hole, woven wire cloth sieves of the following sizes conforming to the specifications for Sieves for Testing Proposes was used for these samples :

	Mesh number	Sieve opening (mm)
	16	1.19
	18	1.00
Sample No. 1	20	0.841
	25	0.707
	25	0.707
	30	0.595
Sample No. 2	35	0.500
	40	0.420
	45	0.354
	50	0.297
Sample No. 3	60	0.250
	70	0.210
	80	0.177
	20	0.841
	30	0.595
Sample No. 4	45	0.354
(overall average)	50	0.297
	70	0.210

The sieve frames were 8 inches in diameter, and the height of the sieve from the top of the frame to the cloth was about 2 inches.

Containers for Gross Sample. - The containers for gross samples were metal cans, provided with tightly fitting covers, such as two-gallon milk cans.

Containers for Experimental Sample. - The containers for experimental samples were cans or bottles, provided with tightly fitting covers.

3.2.1.2 Preparation of sample. The sifted samples were placed on a sheet of rubber, and thoroughly mixed by raising first one corner of the sheet and then the other so as to roll the oil shale over and over at least 100 times. After mixing, the sample was coned and quartered. The operations of mixing, coning, and quartering were continued, if necessary, until the sample was reduced sufficiently so that all of one of the quarters weighed about 100 gm, all of which would be the experimental sample.

3.2.1.3 Calculation of oil shale particle size. The size of oil shale particles was calculated from the weights of the residue remained on the sieves, and was expressed as weight fraction of the weight of original sample, as in Eq. 3.1.

$$\bar{d}_p \;=\; \frac{1}{\sum\limits_{alli} \left(\frac{x}{d_p}\right)_i} \qquad \ldots\ldots\ldots\ldots\ldots (3.1)$$

3.2.2 Fluidizing gas selection

The fluidizing gas, which is inert or does not react with oil shale or the product yields, such as nitrogen gas or carbondioxide is suitable. Nitrogen gas was selected throughout the experiment for its convenience and availability.

3.2.3 Calculation of shape factor and density of oil shale particle.

As a fixed bed Kozeny and Carman's equation was applied

$$\frac{\Delta P}{L_m} = \frac{180 \ U_o \mu}{(\phi \ \overline{d}_p)^2} \cdot \frac{(1 - \varepsilon_m)^2}{\varepsilon_m^3} \cdot \frac{1}{980} \quad \dots\dots\dots\dots(3.2)$$

$$\frac{980}{180} \cdot \frac{\Delta P}{L_m} \cdot \frac{\overline{d}_p^2}{\mu} = \frac{(1 - \varepsilon_m)^2}{\phi^2 \ \varepsilon_m^3} \cdot U_o \quad \dots\dots\dots\dots(3.3\text{-A})$$

let $\dfrac{(1 - \varepsilon_m)^2}{\phi^2 \ \varepsilon_m^3} = K_i$

$$\therefore \quad \frac{980}{180} \cdot \frac{\Delta P}{L_m} \cdot \frac{\overline{d}_p^2}{\mu} = K_i \ U_o \quad \dots\dots\dots\dots(3.3\text{-B})$$

or $\log \left[\dfrac{980}{180} \cdot \dfrac{\Delta P}{L_m} \cdot \dfrac{\overline{d}_p^2}{\mu} \right] = \log K_i + \log U_o \quad \dots\dots\dots(3.4)$

K_i was estimated by plotting $\left[\dfrac{980}{180} \cdot \dfrac{\Delta P}{L_m} \cdot \dfrac{\overline{d}_p^2}{\mu} \right]$ versus U_o in log-log paper.

From $\varepsilon_m = \dfrac{\text{Volume of bed-Vol. of solid}}{\text{Vol. of bed}}$

$$= 1 - \frac{W}{\rho_s}\Big/ S.L_m$$

$\therefore\; 1 - \varepsilon_m = \dfrac{W}{S.L_m\,\rho_s}$

$\therefore\; K_i = \dfrac{1}{\phi^2} \cdot \dfrac{\left(\dfrac{W}{SL_m\,\rho_s}\right)^2}{\left(1 - \dfrac{W}{SL_m\,\rho_s}\right)^3}$

or $\left(\dfrac{1}{K_i L_m^2}\right)^{\frac{1}{3}} = \left[(\rho_s\phi)\left(\dfrac{S}{W}\right)\right]^{\frac{2}{3}} - \left[\left(\dfrac{\phi^2}{\rho_s}\right)\left(\dfrac{W}{S}\right)\right]^{\frac{1}{3}} \cdot \dfrac{1}{L_m}$ ....(3.5)

ϕ and ρ_s were determined by plotting $\dfrac{1}{L_m}$ versus $\left(\dfrac{1}{K_i L_m^2}\right)^{\frac{1}{3}}$ on linear graph,

letting $b = \text{slope} = \left[\left(\dfrac{\phi^2}{\rho_s}\right)\left(\dfrac{W}{S}\right)\right]^{\frac{1}{3}}$

and $a = \text{intercept} = \left[(\rho_s\phi)\left(\dfrac{S}{W}\right)\right]^{\frac{2}{3}}$

by solving, then

$\phi = a^{\frac{1}{2}} b$ ....(3.6)

$\rho_s = \dfrac{a}{b} \cdot \dfrac{W}{S}$ ....(3.7)

3.3 General Description of Experimental Procedure

3.3.1 Determination of shape factor and density of oil shale particle

One hundred gm of oil shale was weighed. It was then poured into the glass column. Oil shale samples having an average particle size of 0.310 mm (Appendix B.1) were taken for determining the shape factor and density by fixed bed method. A nitrogen tank was connected to a rotameter for measuring nitrogen flow rate. Then nitrogen was supplied slowly via a regulating valve. The pressure drop was measured by a manometer in mm. H_2O and the heights of the bed were measured in centimeter.

The pressure drop was calibrated by minus the empty column pressure drop (Appendix B.2) in the same nitrogen flow rate.

The result from the experiment was plotted and calculated to obtain the density and shape factor of oil shale particle.

3.3.2 Determination of actual minimum fluidizing velocity

Various oil shale samples having the average particle sizes of 0.715, 0.576, and 0.249 mm, respectively, were tested

for their minumum fluidizing condition. The nitrogen gas flow
rate was increased slowly through the 100 gm of oil shale un-
til the fluidizing phenomenon of particles was occurred. The
nitrogen flow rate and pressure drop and the height of bed
were measured periodically through the entire experiment.
The minumum fluidizing velocity of nitrogen gas was obtained
from the curve of nitrogen velocity versus pressure drop at
the beginning of constant pressure drop.

Besides, samples of spent shale (Appendix B.3) were
also investigated for their minimum fluidizing condition.

3.3.3 Oil shale retorting

A 100 gm oil shale of specified particle size was
filled into the retort at the top of the column, provided
with the tightly fitting cover. The heater was switched on
until it reached the desired temperature (400, 450, 500, 600,
and 650°C respectively), with the equipment set as the ar-
rangement shown in Fig. 3.3. The retort was flushed by nitro-
gen for one minute in order to prevent combustion according
to O_2 in the retort. The nitrogen flow rate was increased
slowly through the preheating coil until it reached minimum
fluidizing velocity calibrated at the retorting temperature
(Appendix B.4). The temperature of the retort was readjusted
to the desired temperature by means of a variable transformer.

Each run took about 30 minutes (Appendix A.3 and A.4). Liquid products were collected in a measuring cylinder with a little of shale oil film appearing on the surface of the cyclone, condensers, and others. These parts were weighed to determined the total weight of shale oil.

3.3.4 Measurement of pressure drop

The pressure drops across the fluidized bed for determining fluidizing conditions were measured by a manometer in mm. H_2O . These pressure drops were measured both when the retort contained oil shale particles and when it was empty.

3.3.5 Measurement of superficial velocity of nitrogen

The flow rate of fluidizing gas inlet was measured by a rotameter (Sho-Rate "150", model 1355, tube R-6-15-B. foat 316 S.S.)

The correlation between rotameter reading and nitrogen flow rate in cm^3/min. is shown in Fig. 3.9.

The superficial velocity of nitrogen (cm/sec), flowing through the experimental column, was obtained by multiplying the flow rate by 8.22×10^{-4}.

3.3.6 Measurement of bed height

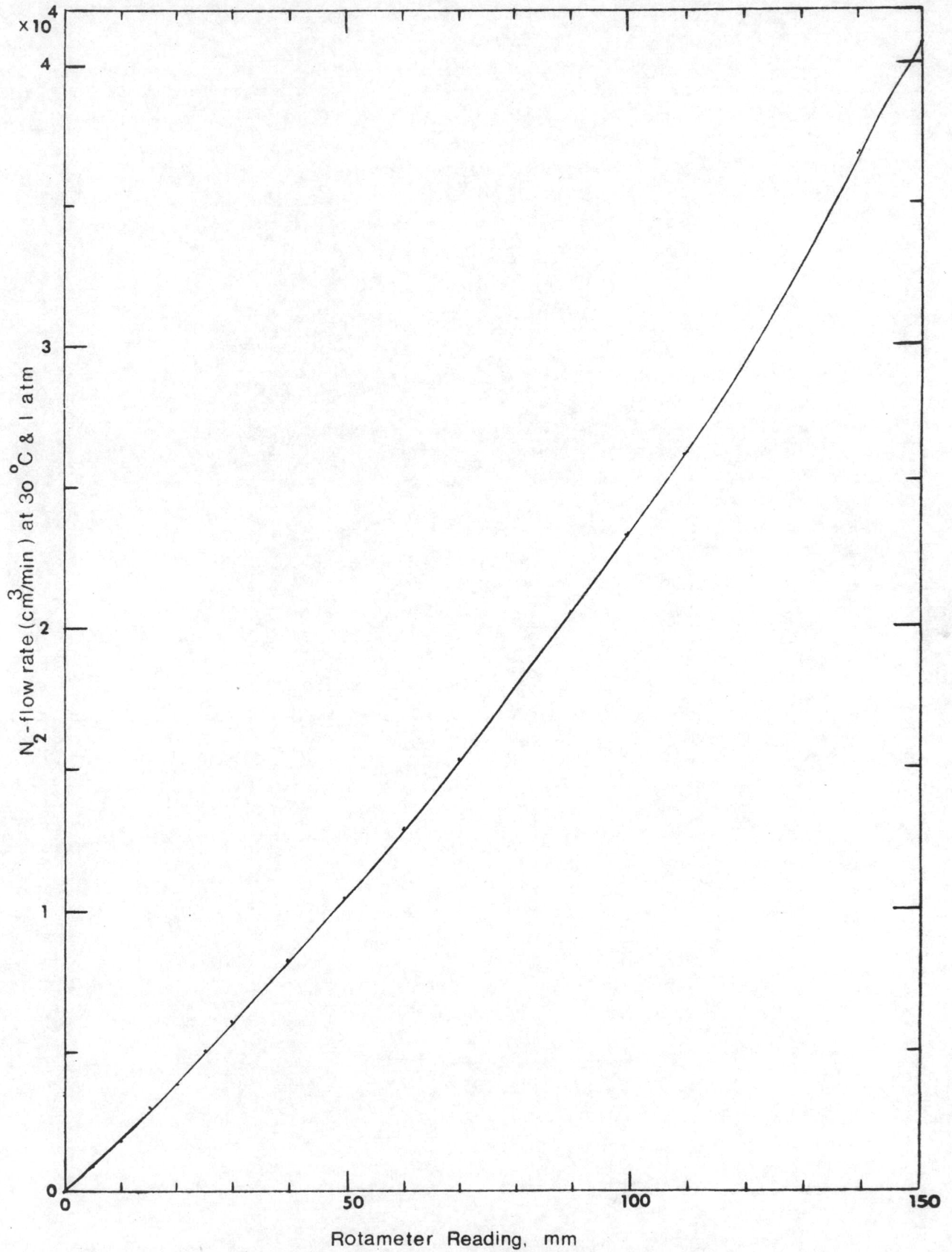The heights of bed were measured by a scale on the outside wall of the glass column in centimeter.

FIG. 3.9 – CORRELATION BETWEEN ROTAMETER
READING & N$_2$- FLOW RATE

3.3.7 Measurement of temperature

Temperatures in the retort at the fluidized bed, mid-column, top of retort, and of the heater were measured by means of a conventional high-temperature thermocouple (Nickel-Chromium/Nickel-Aluminium Thermocouple, type 6 a, 22 B & S). A potentiometer "Foster", model 3156 DPX, scale No. 2235, serial No. 69751 was used for reading the temperatures by means of a selector switch (Fig. 3.4).

Temperature of the gas outlet at the top of the second condenser was measured by a 0-100 °C mercury thermometer.