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Slag Characterization: Viscosity of Synthetic Coal Slag in Steam

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National Bureau of Standards
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SLAG CHARACTERIZATION: VISCOSITY OF SYNTHETIC COAL SLAG IN STEAM

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FOREWORD

Since 1974, the National Bureau of Standards has been engaged in research designed to address materials problems and needs pertinent to coal conversion and utilization technologies. This work, sponsored in part, at first by the Office of Coal Research, then ERDA and currently the DOE, focuses on test method development, particularly accelerated procedures for materials behavior and durability; on the determination of the mechanisms of materials degradation; and on the development and operation of materials data centers to provide evaluated information on properties and performance including failures occurring in operating plants.

During the FY 77-79 period the NBS program, entitled "Materials Research for the Clean Utilization of Coal" consisted of a number of interrelated tasks including:

1. Metal Corrosion

    a. Constant Strain Rate Test
    b. Pre-cracked Fracture Test

2. Ceramic Deformation, Fracture and Erosion

3. Chemical Degradation of Ceramics

    a. Reactions and Transformations
    b. Slag Characterization (viscosity)
    c. Vaporization and Chemical Transport

4. Failure Prevention

    a. Failure Information Center
    b. Materials Properties Data Center

The results of the research have been disseminated through quarterly reports of progress (available from NTIS; report designation EA-6010; Dist. Category UC-90C) as well as numerous scientific publications in technical journals. Further, as individual tasks are completed, an overall report is prepared detailing the results and accomplishments of the project. This present publication is the final report for Task 3b. Slag Characterization: Viscosity.

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ABSTRACT

A rotating cylinder apparatus has been designed and constructed for measuring the viscosity of molten slags under steam pressure in the range 15 to ∿300 psi and up to about 1400 °C. Two synthetic coal slags, one high in alkali and the other lower in alkali but higher in calcia were studied with this apparatus. In the high alkali slag equilibrium was probably not achieved and the viscosity appeared to increase with time. In the low alkali slag although the viscosity appeared to be increased with increasing steam pressure the ambient and higher pressure curves appear to tend to coalesce with increasing temperature.

I. INTRODUCTION

In recent years NBS has been involved in research on materials and methodology employed in coal gasification reactors which are used to convert coal into synthetic gaseous fuel. In some of these gasification processes, the coal is reacted in the presence of high pressure gases including steam at 1000 psi* and temperatures up to 1500°C. The residual molten liquid (slag) is allowed to gravitate to the bottom of the reactor for extraction from the system. The integrity of the process is dependent upon the continuous flow of this liquid. The objective of this study is to obtain high pressure-temperature viscosity data on molten liquids which may be representative of those encountered in the gasification process. With this basic information the slag can be modified if necessary to enhance uniform flow and to prevent catastropic reactor failure.

Various methods have been described in the literature for measurement of viscosity of glass melts at ambient pressure [1]. However, the literature is apparently void of techniques to measure viscosity under steam pressures in the >15 to 1000 psi range. The rotating cylinder method [2] has become over the years a standard method of measuring viscosity up to 10^7 poises. The viscosity limit 10^7 poises is well within the range of interest for molten coal slag. For this investigation an apparatus has been constructed for measurements in this range employing an internally heated pressure vessel.

2.0 APPARATUS AND PROCEDURE

2.1 GENERAL FEATURES

The apparatus (Fig. 1,2) was fabricated from 304L stainless steel and is approximately 24" long and 10" in diameter. The main body of the vessel was fabricated from schedule 40 pipe. The top and bottom (Fig. 3) are class 300 flanges which are 17.5" in diameter and fastened with sixteen one inch bolts. The two side ports are 4" pipe also ASI class 300. The flanges are held in place with eight 3/4" bolts. These flanges (Fig. 4) serve to introduce two massive copper leads necessary to conduct sufficient current at low voltages to heat the platinum heater in the presence of high pressure steam while maintaining the pressure integrity of the vessel. The leads are isolated from the

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*The use of psi, bar and kbar follows the current common practice of workers in the field. Note that 1 bar = 10^5 N/m^2 (or pascal) = 10^6 dyn/cm^2 = 0.9869 atm = 14.504 psi. The accepted international standard (SI) unit of pressure is the pascal or newton per meter squared.

vessel body by mica sleeves with the pressure seal being made with the flange using an "O" ring in conjunction with a split lock collar (Fig. 5) and a retaining ring (Fig. 6).

The steam used in the apparatus is generated internally by vaporizing water using nine bayonet type heaters which together dissipate approximately 3600 watts. These heaters are isolated from the internal pressure by three finned copper heat exchangers. There are three heaters in each copper radiator. The internal and external surfaces of the radiators are pressure isolated by an O-ring seal. All O-ring seals employed in this apparatus were fabricated from ethylene propylene, this material being appropriate for use with high pressure steam. Figure 7 shows the relationship between the heaters and the heat exchangers. The bayonet heaters are supplied power from a 10 KVA S.C.R. power supply used in conjunction with a current adjusting type controller. These bayonet heaters must be controlled since they in effect control the steam pressure in the vessel. A baffle assembly is used above the copper heat exchangers to reduce bumping caused by the evolution of steam. A copper-copper constantan thermocouple is used with this device to monitor steam temperature, hence total pressure.

2.2 PLATINUM HEATER

The platinum heater (Figs. 8,9) consists of a hollow right cylinder 2" I.D. and 6" long. The cylinder was fabricated from sheet 0.040" thick. Two large platinum tabs approximately .120" thick, 3.152" long and 6" high were welded to the cylinder at 180 °. These tabs are clamped into the copper electrodes. A cylinder of high alumina material 0.5" thick was cast around the platinum cylinder. A second layer of material 0.5" thick was cast around the first to further insulate the heater from the steam enviroment. Both layers of castable were reinforced with platinum wire. The cooling of the platinum heater by the steam is the greatest single problem encountered in the operation of the viscometer. The platinum crucible used to contain the molten slag is 1.5" O.D. and 2.5" long. A tab was welded on the bottom of the crucible which was embedded into a notch at the top of the alumina spindle. The alumina spindle is in turn clamped at the opposite end into a stainless steel rotating shaft. This interlocking device helped to prevent accidentally contacting the crucible with the heater.

The stainless steel rotating shaft which supports the alumina spindle passes through the bottom flange. This shaft is sealed with two ethylene proplene O rings. Outside the vessel the shaft is cooled to prevent degradation of the O ring. This cooling is accomplished by passing water through a jacket (Fig. 10) surrounding the shaft. The shaft is attached to the motor drive transmission by a universal coupling.

2.3 VISCOSITY TORSION SUSPENSION

The viscosity torsion suspension is shown in Figures 11, 12. The platinum bob consists of a small cylinder 9/16" O.D. and 1 1/8" long with a flat disc welded to the top. A platinum tube with the end sealed containing the thermocouple was welded through the top of the bob to a depth of about 1/8 of an inch. This small sealed projection provided the necessary isolation between the thermocouple bead and the molten slag. The thermocouple wire employed was 0.015" diameter Pt-Pt10%Rh inserted into 2 mm - 2 hole thermocouple tubing. Two short lengths of smaller Pt-Pt10%Rh thermocouple wire were passed out of the suspension through ceramic isolation yokes and welded to finer .005" diameter Pt-Pt10%Rh thermocouple wire, to allow unhindered deflection of the torsion wire to which they run parallel. Above the suspension the thermocouple wire is passed through a teflon pressure seal to the outside of the vessel. This design provided a means to measure the temperature of the melt simultaneously with viscosity.

A short length of spring wire .013" diameter served as a torsion member which was attached to the thermocouple assembly at the lower end. The upper end of the torsion member is attached to a stainless steel tube which served to raise or lower the entire assembly.

A permanent magnet attached to the suspension served as a rotation indicator when used in conjunction with a small compass located on the outside of the pressure vessel.

2.4 VISCOSITY MEASUREMENT

A typical viscosity measurement was conducted in the following manner. The temperature of the slag was raised to about 800°C (below melting) and the steam pressure was generated. The system was vented of air. The temperature and pressure were allowed to rise concurrently until the slag was melted, and the steam pressure was adjusted to the desired value. The molten material was allowed to come to temperature-pressure equilibrium with the surroundings for several hours. The bob, prewarmed at the surface of the melt, was slowly introduced into the melt. The bob was manually rotated until the compass pointer was at zero degrees. This position on the scale was now defined as zero for reference. The compass is situated on an arm which was rotatable about the long axis of the pressure vessel. The arm holding the compass was rotated an appropriate amount, usually about 10 to 20 degrees. The amount depending on the viscosity of the melt and the diameter of the torsion members. These values were empirically established to produce the desired angular deflection. The melt was rotated at a speed sufficient to return the compass needle to the zero. The motor speed was determined by visually counting the revolutions over a fixed period

of time. Previously the torsion wire was calibrated with a silicone fluid with a known viscosity in the range of interest. The motor speed was recorded for various angular deflections. The viscosity was determined by the following relationship:

$$\eta \text{ unknown} = \frac{(\text{calibrant viscosity})(\text{calibrant RPM})(\text{measured deflection})}{(\text{measured RPM})(\text{calibrant deflection})}$$

at constant T and P, η = poises

where:

η = viscosity of the calibration liquid in poises; calibrant RPM = motor speed necessary to produce calibrant deflection; calibrant deflection = preselected deflection of calibrant; measured deflection - deflection of compass; measured RPM = the RPM necessary to return the compass needle to the zero position.

3.0 RESULTS AND DISCUSSION

Two slags were investigated in this study: The first, a slag high in alkali similar to a western coal ash contained SiO_2, 42%, Al_2O_3, 3%; Fe_2O_3, 25%; CaO, 5%; MgO, 5%; Na_2O, 15% and K_2O, 5% (expressed in wt percent).

The viscosity of this melt was measured at 55 psi steam pressure and ambient pressure in air at 1197°C, 1143°C and 1081°C. The viscosities in steam were $\log_{10} \eta$ = 1.90, 2.05 and 2.33, respectively. Each of these measurements was made after the temperature was held for 1/2 hour. The results are plotted on Figure 13, curve 3. When these data are compared to those values measured at ambient pressure it appears that the viscosity for a given temperature increases with pressure. The same experimental procedure was employed and the viscosities were measured at 75 psi and 1192 °C, 1133 °C, 1106 °C and 1070 °C. The values were $\log_{10} \eta$ = 2.20, 2.37, 2.77 and 2.76, respectively. These results are plotted in Figure 13, curve 4.

At this point it was thought that either equilibrium was not achieved or that crystallization had taken place in the melt. Several attempts were made to achieve equilibrium by heating the melt for longer periods of time. The first experiment was conducted at 1150°C, 77 psi for 185 minutes. During the duration of the run, the values varied from $\log_{10} \eta$ = 2.23 to 2.69.

A similar experiment was conducted at higher temperatures (1170 °C) and 80 psi. While the temperature remained constant ±5 °C, the viscosity of the melt increased from $\log_{10} \eta = 2.09$ to 2.34 in 135 minutes. Some of this material heated in 80 psi steam pressure was remelted in air and the viscosity was compared to that of the starting material. The results shown in Fig. 13, curves 1 and 2, show that the viscosities compare favorably. From these data, it would appear that volatility of a component was not a factor in increasing the viscosity during the pressure experiments. In the last of this series of experiments, the viscosity of the same material was rechecked at 77 psi and 1100 °C ± 5 °C. In 165 minutes the viscosity changed from $\log_{10} \eta = 2.48$ to 2.68 (Fig. 14).

Since the viscosity of this high alkali slag appeared to be time related, it was decided to "soak" the molten slag in steam at a constant temperature with continuous monitoring of the viscosity until the value appeared constant. The melt was maintained at 60 psi and 1155 °C ± 5 °C for 420 minutes (8.67 hours). The results are given in Fig. 15. From this figure it can be seen that some small short range inflections exist which might appear to enter steady state. However, with progression of time it can be seen that equilibrium is not achieved and that the viscosity of this particular melt increases with time.

A second slag lower in alkali content was investigated. This slag has the following composition: SiO_2, 48.30%; Al_2O_3, 12.10%; Fe_2O_3, 12.00%; CaO, 14.90%; MgO, 8.00% and Na_2O, 4.7% (expressed in wt percent). At the initiation of this investigation, the viscosity of the above mentioned slag was compared independently in another rotating crucible apparatus. The viscosity of the material was found to be about $\log_{10} \eta$ poises = 1.86 at 1277 °C at ambient pressure. The viscosity of this melt was measured in the high pressure steam apparatus operating at ambient pressure. The viscosity of the material was found to be $\log_{10} \eta = 1.90$ poises at 1277 °C. These values are in excellent agreement. This same material was next subjected to an environment of N_2 to reduce the partial pressure of oxygen in the steam atmosphere. The apparatus was purged four times with nitrogen; the total N_2 pressure over the slag was set at 25 psi. The temperature was maintained at approximately 1279°C, the viscosity was reduced from $\log_{10} \eta$ poises = 1.91 (value in air) to $\log_{10} \eta$ poises = 1.71 at which time the experiment was terminated. This lowering of viscosity was probably due to the reduction of Fe^{+3} to Fe^{+2} in the melt.

An attempt was made to reoxidize the melt previously heated in nitrogen while monitoring the viscosity. The viscosity was then remeasured at 1277°C in air. This temperature was maintained for 2 hours 15 min and the viscosity increased steadily from $\log_{10} \eta = 1.82$ to $\log_{10} \eta$ poises = 2.22 which greatly exceeded the original value in air. A large mass of well formed crystals were found radiating from

the bob. This material was examined by x-ray powder diffraction and found to contain a phase which is apparently isostructural with a naturally occurring mineral in the pyroxene family, fassaite (an Al-augite).

The effect of varying the partial pressure of oxygen in steam on viscosity of the melt was investigated with a different gaseous media. The viscosity was measured in 50% CO_2 and 50% steam at a total pressure of 97 psi. At 1321 °C, 1240 °C, 1232 °C and 1161 °C, the viscosity was found to be $\log_{10} \eta$ = 1.46, 1.72, 1.73, 1.98, respectively. From Figure 16 it can be seen that the effect of reduced partial pressure of oxygen significantly lowered the viscosity in the melt when compared to the values obtained at ambient pressure. With this effect noted, it was decided not to continue the viscosity measurements in mixed gaseous environments but to attempt to increase the temperature of measurement in effort to determine if the viscosity curves in Fig. 16 would converge. To accomplish this the pure platinum heater was replaced with a 60% platinum 40% rhodium heater.

The new heater was installed and several attempts were made to remeasure the viscosity of the low alkali slag at higher temperatures. Since heat loss due to steam cooling is significant, a flat disc or lid with a small hole to allow passage of the bob was placed over the heater. This lid essentially obscured the view of the melt. In subsequent experiments when this heat shield was removed, the molten liquid was found to foam and the bob serve to "guide" or extract the liquid from the crucible. For example in one experiment all of the liquid was extracted from the platinum crucible and redeposited on the insulation of the heater. The foaming problem in this material is probably due to evolution of oxygen gas as Fe^{+3} is converted to Fe^{+2} on heating. From additional experimentation it was found that the foaming problem could be eliminated by annealing the starting melt at 1500 °C for 4 hours before placing it in the viscometer. Several experiments were conducted with this composition. The first two were performed prior to high temperature annealing in air at 1500 °C for 4 hours. The last experiment received this annealing.

In the first experiment of the series at 50 psi of steam the following values (Table 1) of viscosity were obtained:

Table 1. Viscosity of a Silicate Slag in 50 psi Steam.

Steam Pressure psi	Melt. Temp. °C	Time min	Viscosity $\log_{10} \eta$ in poises	Viscosity at ambient pressure $\log_{10} \eta$ (in poises)
50	1364	25	1.48	1.49
53	1359	45	1.50	1.51
43	1355	55	1.50	1.53
54	1357	150	1.52	1.52

From the above it can be seen that the addition of 50 psi of steam did not significantly effect the viscosity.

The results of the second experiment with foaming are given below (Table 2) at about 80 psi compared to those values obtained at ambient pressure.

Table 2. Viscosity of a Silicate Slag in 80 psi Steam (foaming detected).

ΔT min	Melt Temp. °C	Viscosity $\log_{10} \eta$	Viscosity (most reliable value) $\log_{10} \eta$ in poises	Viscosity in air $\log_{10} \eta$ poises
0	1276	1.73		
13	1275	1.74		
23	1275	1.62	1.71	1.88
27	1275	1.67		
33	1273	1.76		
46	1275	1.69		
63	1272	1.75		
0	1292	1.56		
2	1294	1.59	1.65	1.76
5	1295	1.69		
10	1296	1.69		
25	1298	1.72		
0	1351	1.32		
10	1351	1.44	1.50	1.56
14	1348	1.50		
0	1378	1.44		
3	1381	1.44	1.44	1.44

In Figure 16 the 80 psi data are compared to the ambient pressure data. From these data it appears that the viscosity values for this material are lowered with increasing steam pressure. The values of $\log_{10} \eta = 1.44$ at 1378°C at both ambient pressure and 80 psi would indicate that the viscosity curves, determined under steam pressure, tend to coalesce with increasing temperature.

In the third experiment, the melt was annealed at 1500°C for four hours in air. This material was placed in the viscometer and melted. The temperature was maintained at about 1380°C while the bob was introduced into the melt. The surface of the melt could be observed with the lid off. There was no foaming and the bob was centered in the melt. At this point steam was generated to 75 psi and the melt was allowed to saturate. The entire run took about 15 hours to complete. The results of the last experiment are given in Table 3 and Figure 17.

Table 3. Values of Viscosity at 75 psi Steam for the High Temperature Annealed Low Alkali Slag. No foam detected.

Δt min	Melt Temp. °C	Viscosity $\log_{10} \eta$	Viscosity (most reliable value) $\log_{10} \eta$	Viscosity in air $\log_{10} \eta$
0	1298	2.00		
17	1295	2.08	2.10	1.78
32	1295	2.23		
0	1343	1.91[1/]		
3	1342	1.98		
7	1341	1.96		
0	1395	1.72		
20	1410	1.67	1.70	1.36
33	1399	1.69		
53	1400	1.70		
0	1328	1.84	1.88	1.70
8	1322	1.92		
0	1222	2.52	2.52	2.20
15	1221	2.52		
0	1244	2.58	2.28	2.00
5	1272	2.28		

[1/] Pressure increased from 75 to 85 psi.

In the second experiment, foaming was probably a significant factor in producing apparently erroneous data resulting in a reduction of viscosity with pressure.

There are many possible factors which may act singly or in combination to contribute to an apparent increase in viscosity of high alkali slags under steam pressures. A few are listed below.

(1) Crystal formation in the melt is a possiblity (see Fig. 13, curve 4). However, x-ray powder diffraction shows no crystalline phases. From the x-ray data, crystals would be present to an extent of less than 5%, the detection limit of x-ray powder techniques. Examination with a polarizing microscope indicated that the primary phase present was isotropic and that a few fragments were birefringent. This birefringence does not necessarily indicate the presence of crystalline phases since glass that has been strained exhibits birefringence.

(2) The interaction of H_2O with a melt high in alkali (15% Na_2O and 5% K_2O, first slag) may contribute to a change in viscosity. It has been shown that for a given temperature the viscosity of alkali silicate glass is increased as the concentration of K_2O is increased [3] with a maximum occurring about 15 wt %. These types of glasses may be viewed as a three dimensional random network with each silicon tetrahedrally surrounded by four oxygens with each oxygen bonded by two silicons. In an alkali glass each silicon is tetrahedrally surrounded by four oxygens but some of the oxygens are bonded to only one silicon. The alkali ions are believed to be held weakly in the silicon-oxygen network. If some of the alkali were lost and the bonds were compensated in some manner by HOH then the viscosity may change markedly with changing HOH concentration. However, chemical analysis has shown that the total loss of alkali is less than one tenth percent.

(3) A change in chemical species by oxidation/reduction may change the viscosity. The Fe^{+3} ions have a tendency to reduce partially to Fe^{+2} in air. The ratio of Fe^{+2} to Fe^{+3} may be significantly different at melt condition in steam then it is in air. If the correct ratio of Fe^{+2}/Fe^{+3} can be quenched-in then Mossbauer studies of these glasses may be instructive.

(4) The increased frictional drag produced by temperature gradients may be of sufficient magnitude to affect viscosity. In this study it was determined that vertical gradients were enhanced in the melt by increasing steam pressure. At ambient pressure the difference between the surface melt temperature and the temperature at the level of the bob is about 36 °C. The increased drag produced by the cooler melt at the surface may account for some if not all of the apparent increase in viscosity with increasing steam pressure.

4.0 SUMMARY

A viscosity measuring apparatus was designed and constructed which has a capability of measuring viscosity of melts up to ∿1400 °C with steam pressures up to ∿90 psi. The results of the viscosity determinations were not without some ambiguity. Additional experiments would be necessary, using a series of different slags having systematic compositional variations to determine if the effects of H_2O on the viscosity of the limited compositions is in fact real and not due to other causes such as equipment errors. The data obtained here, however, indicate an increase in viscosity with increasing steam pressures.

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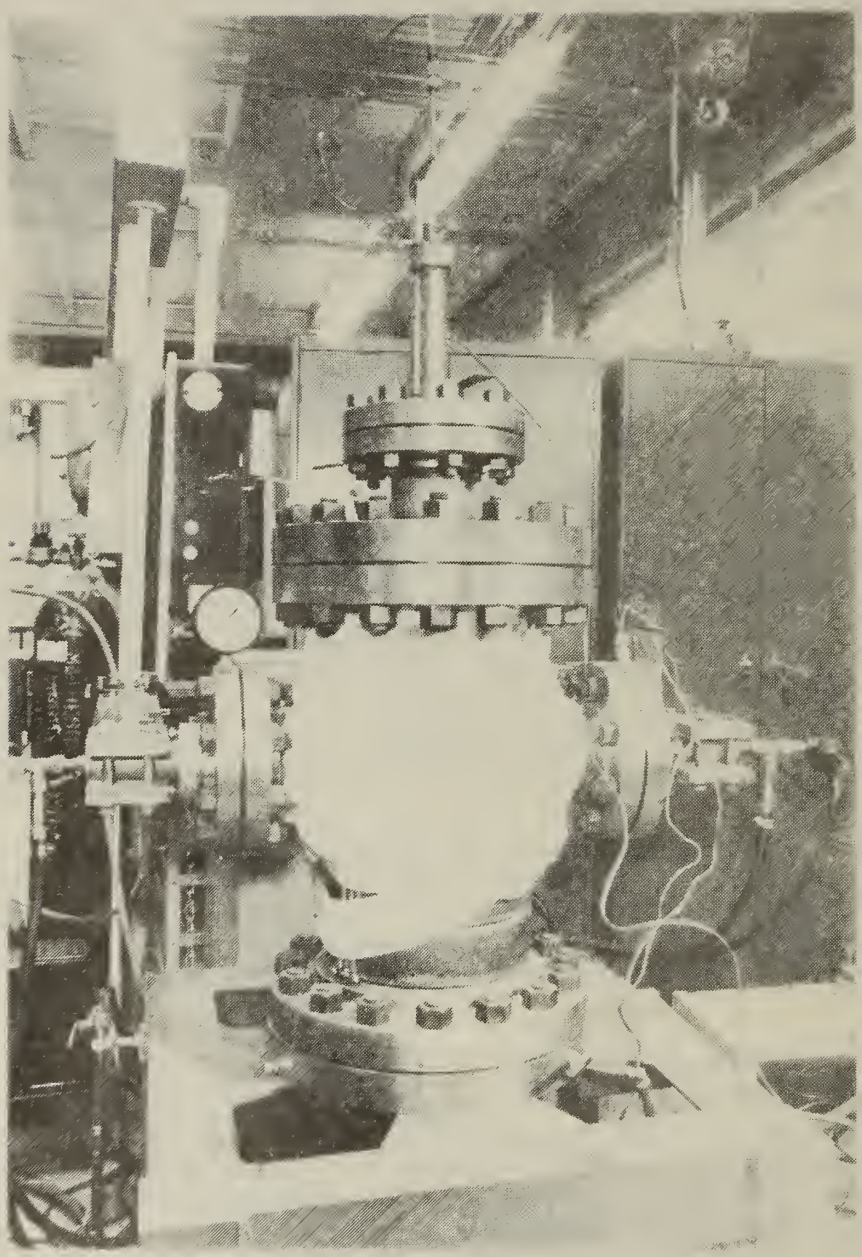


Figure 1. Assembled pressure vessel.

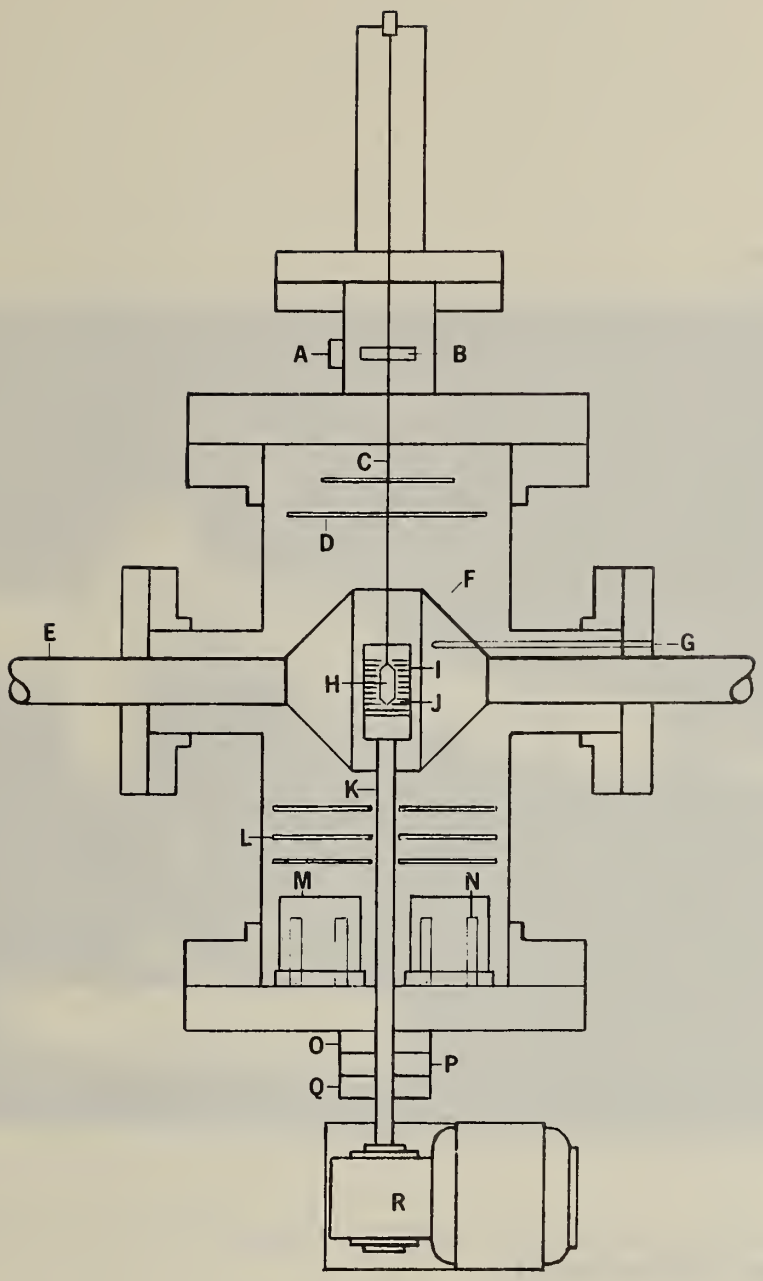


Figure 2. Full assembly schematic drawing of high pressure-temperature viscometer.

A - position detector
B - magnet
C - torsion wire
D - water condensation shields
E - electrodes
F - heating elements
G - temperature control thermocouple
H - bob
I - crucible
J - molten slag
K - spindle
L - anti-convection baffle
M - heat exchangers
N - bayonet heaters
O - shaft seal
P - shaft cooling jacket
Q - shaft bearing
R - shaft drive mechanism

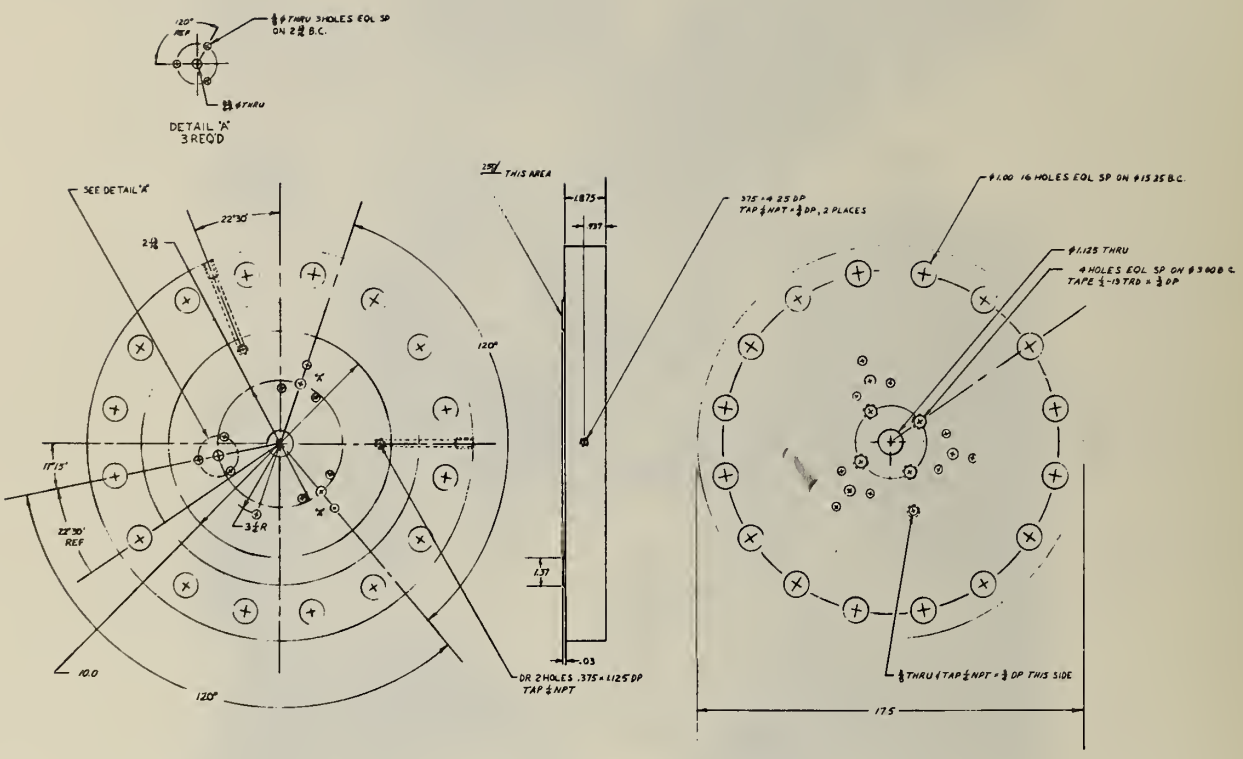


Figure 3. Bottom flange layout.

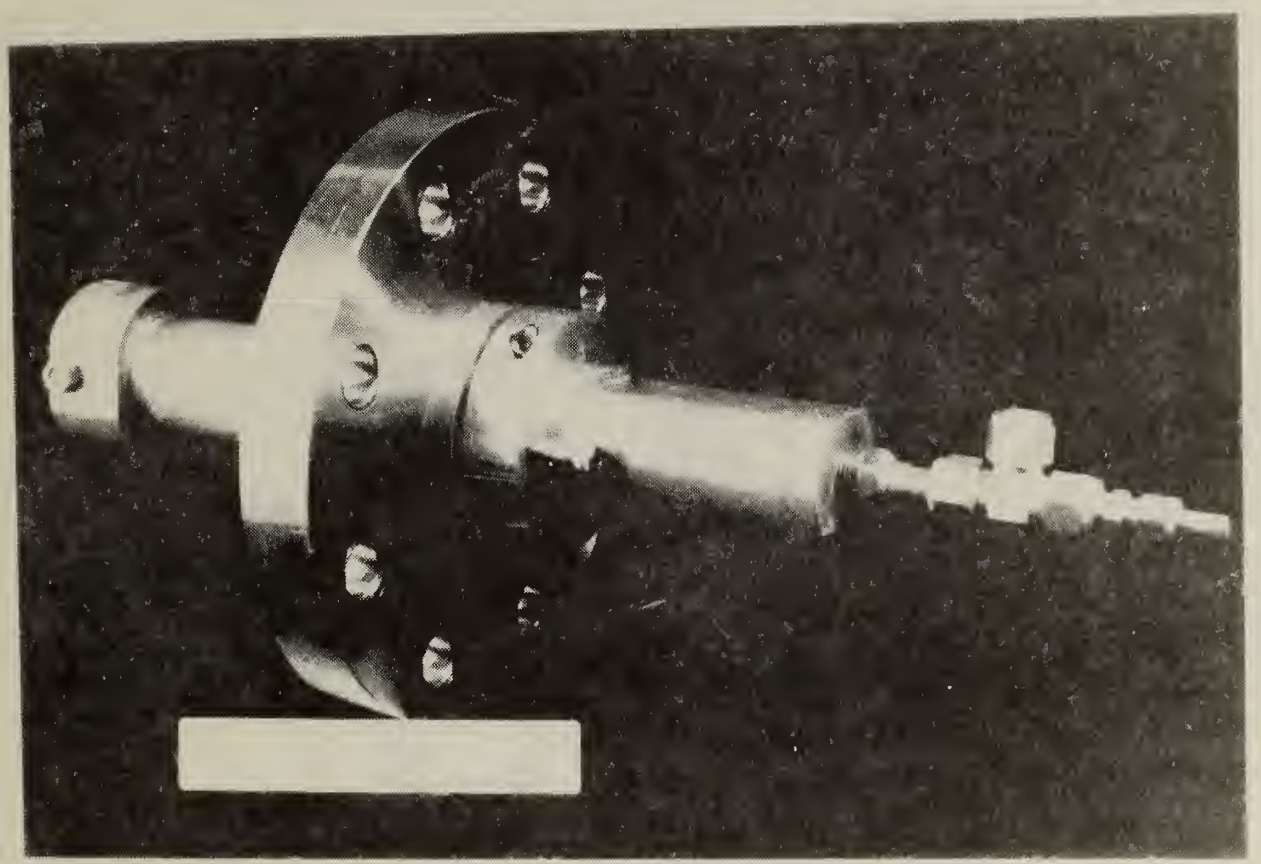


Figure 4. Side flange with copper electrode in place.

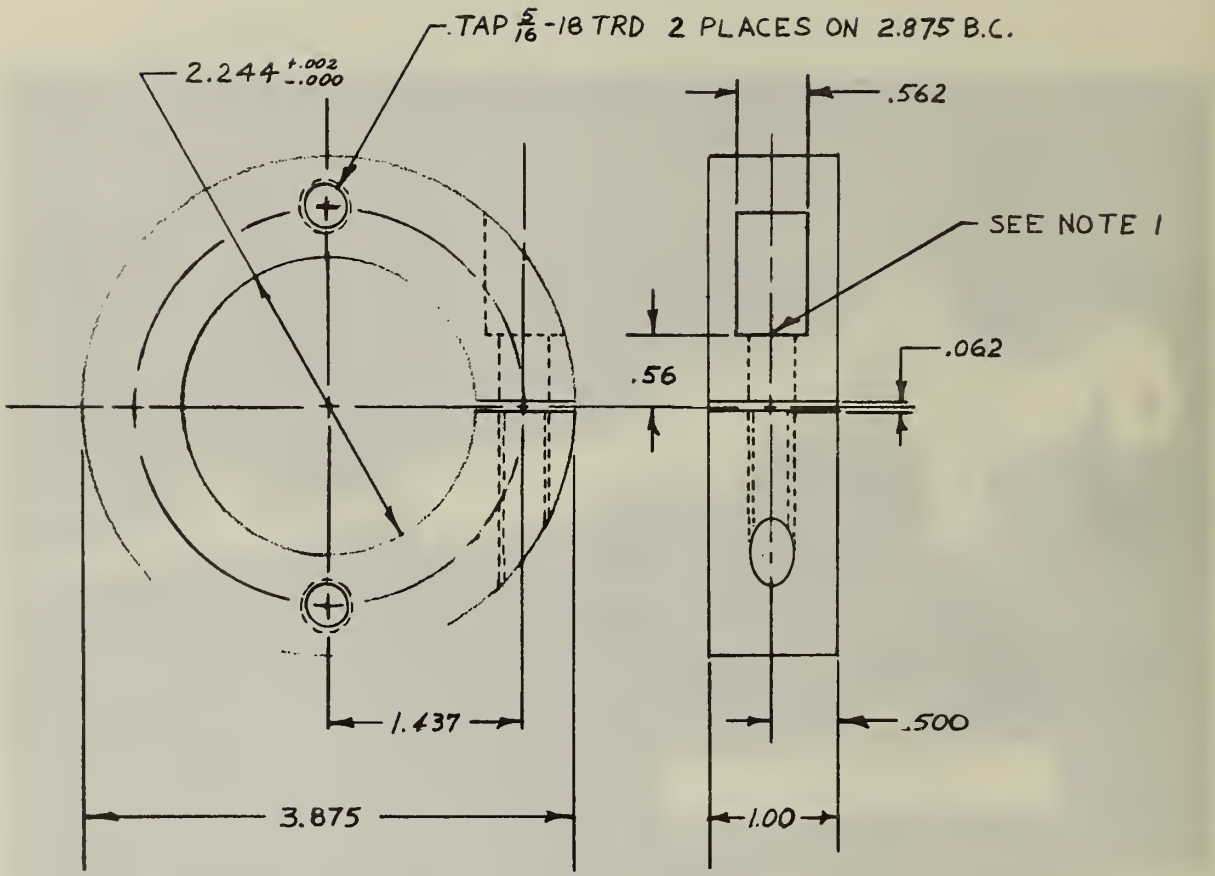


Figure 5. Split lock collar.

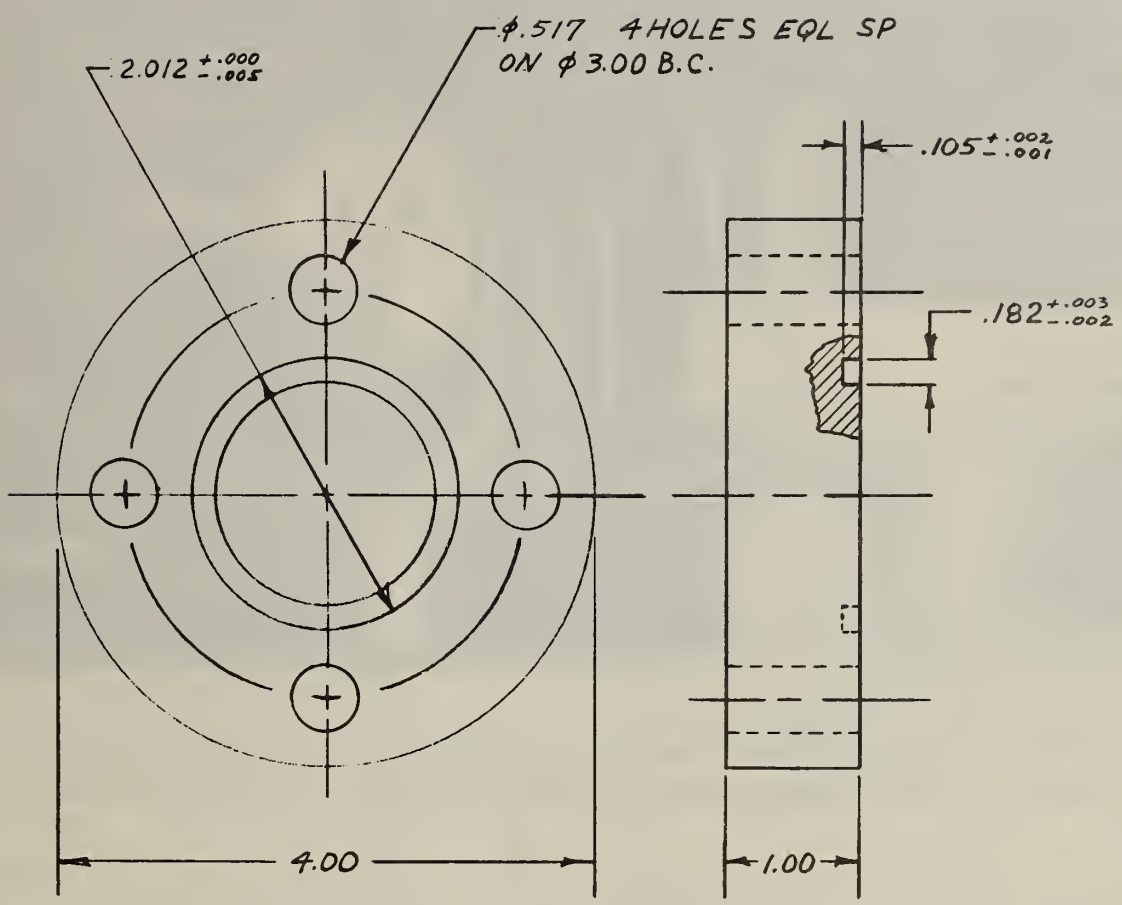


Figure 6. Retaining ring for copper electrode.

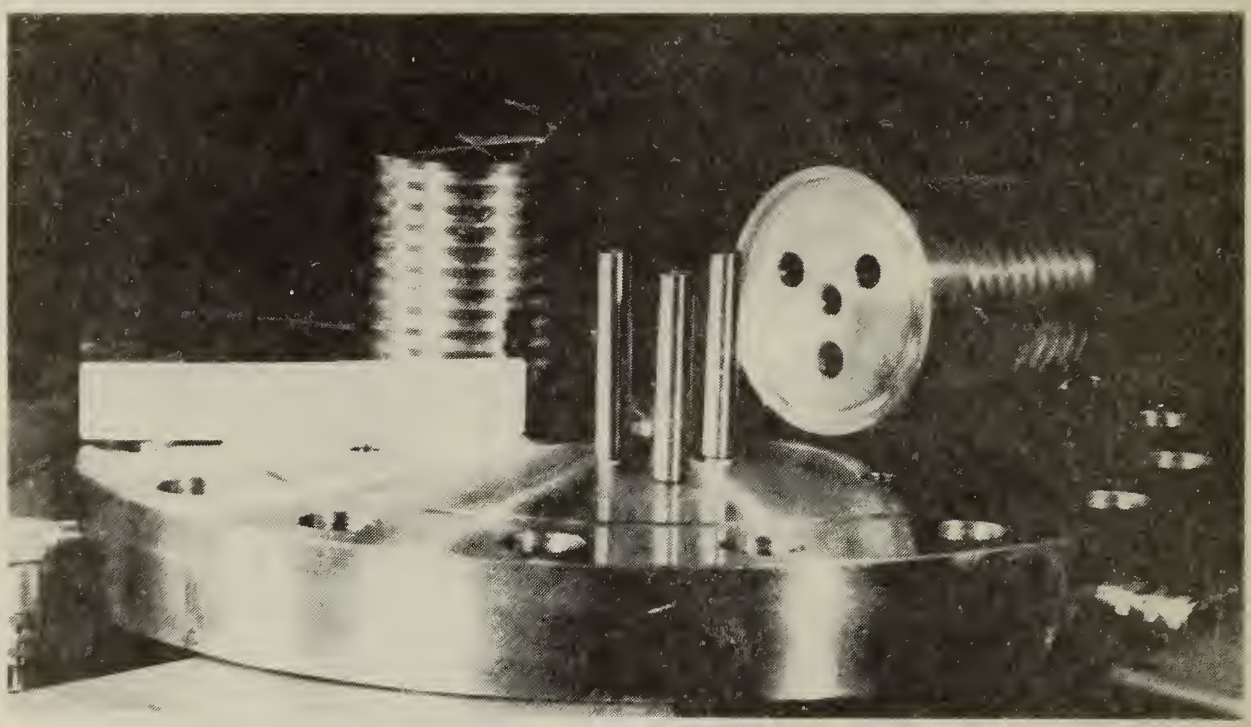


Figure 7. Relationship between bayonet heaters and heat exchangers.

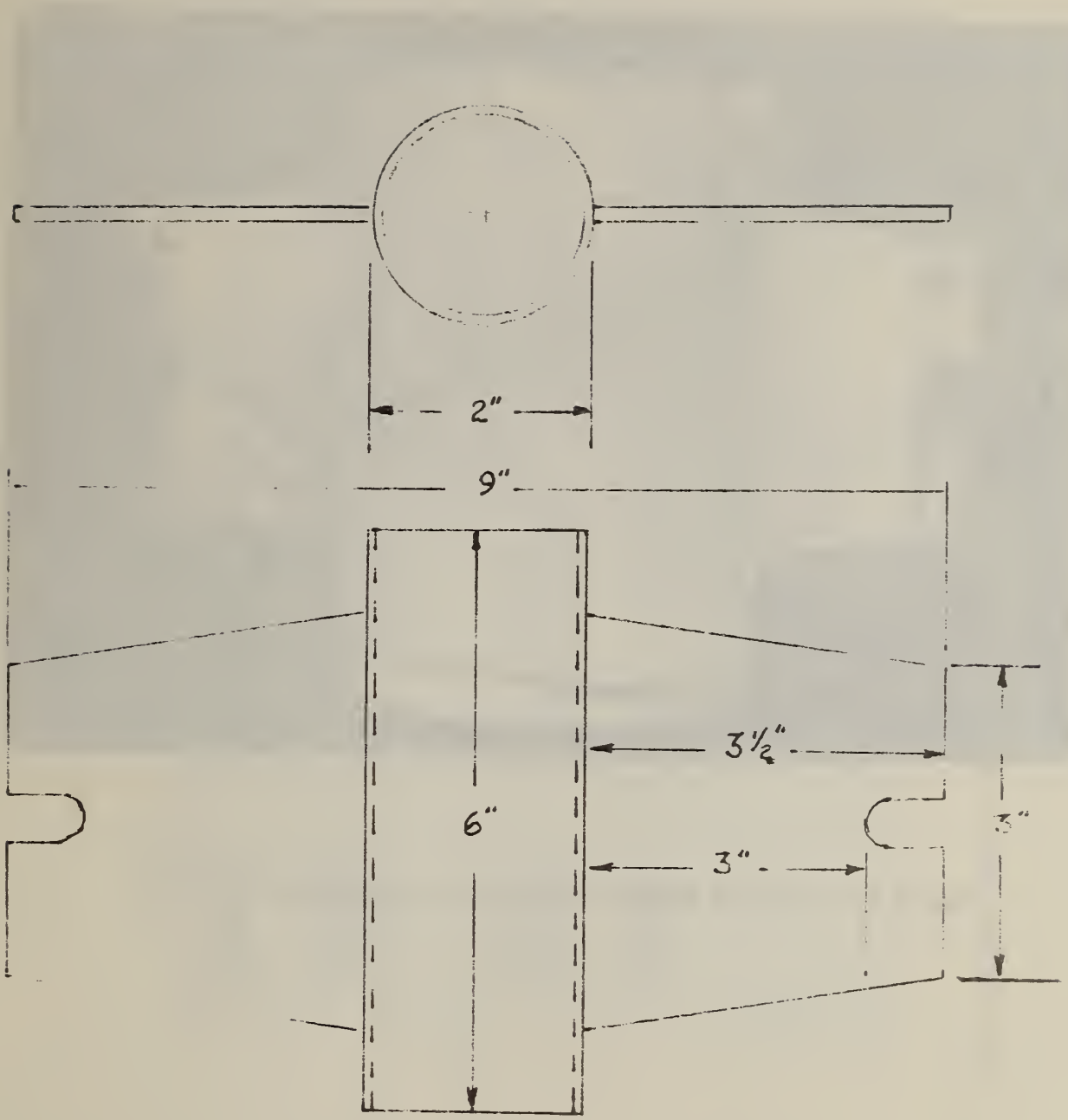


Figure 8. Platinum resistance element for heating slag container in steam.

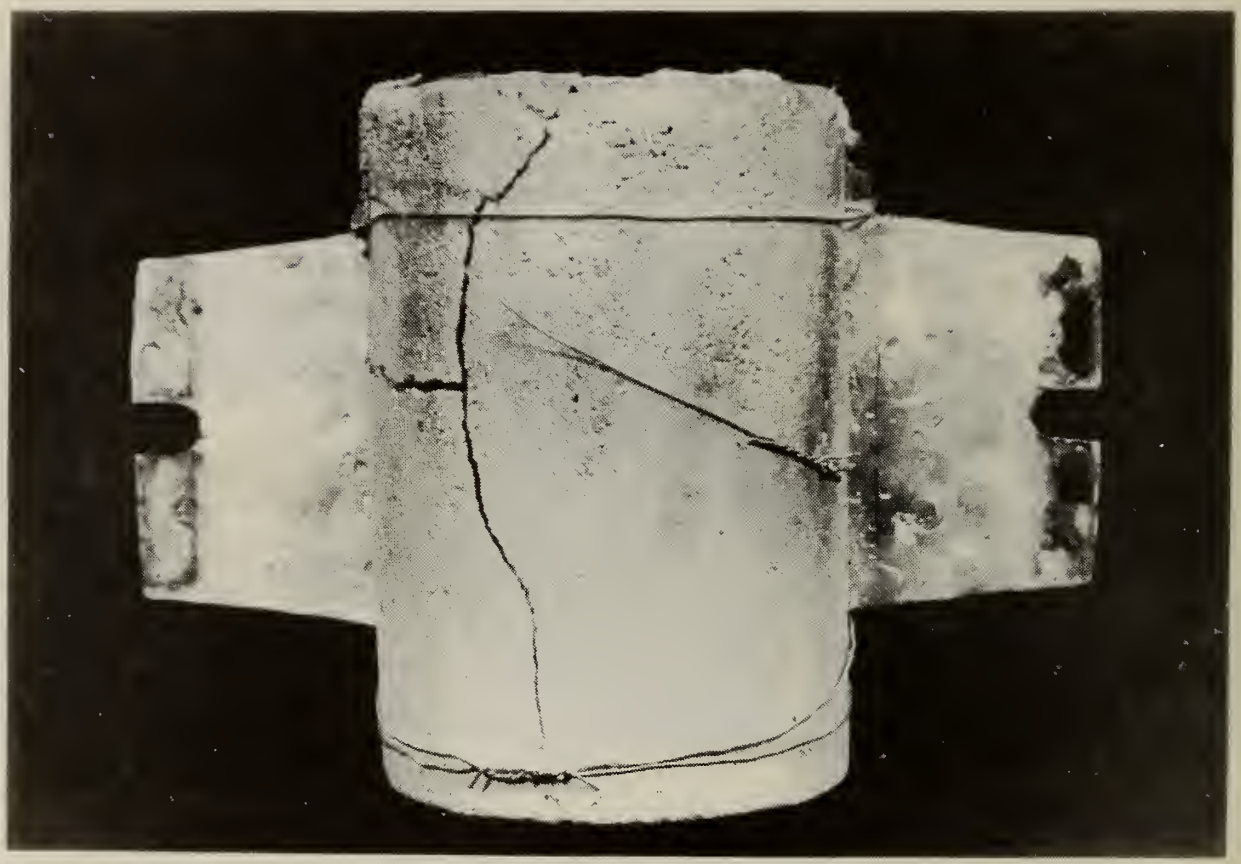


Figure 9. Platinum heater covered with refractory castable.

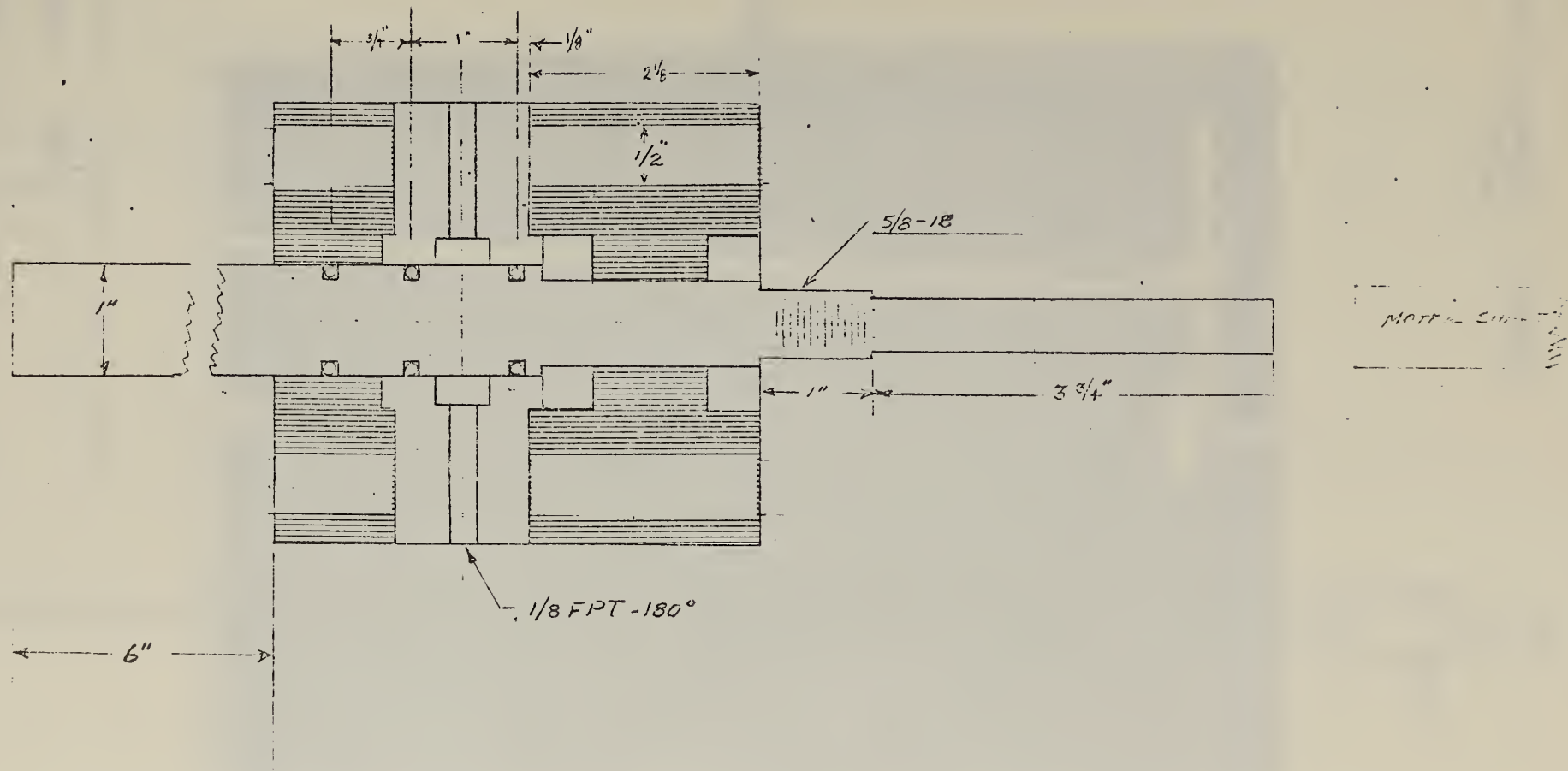
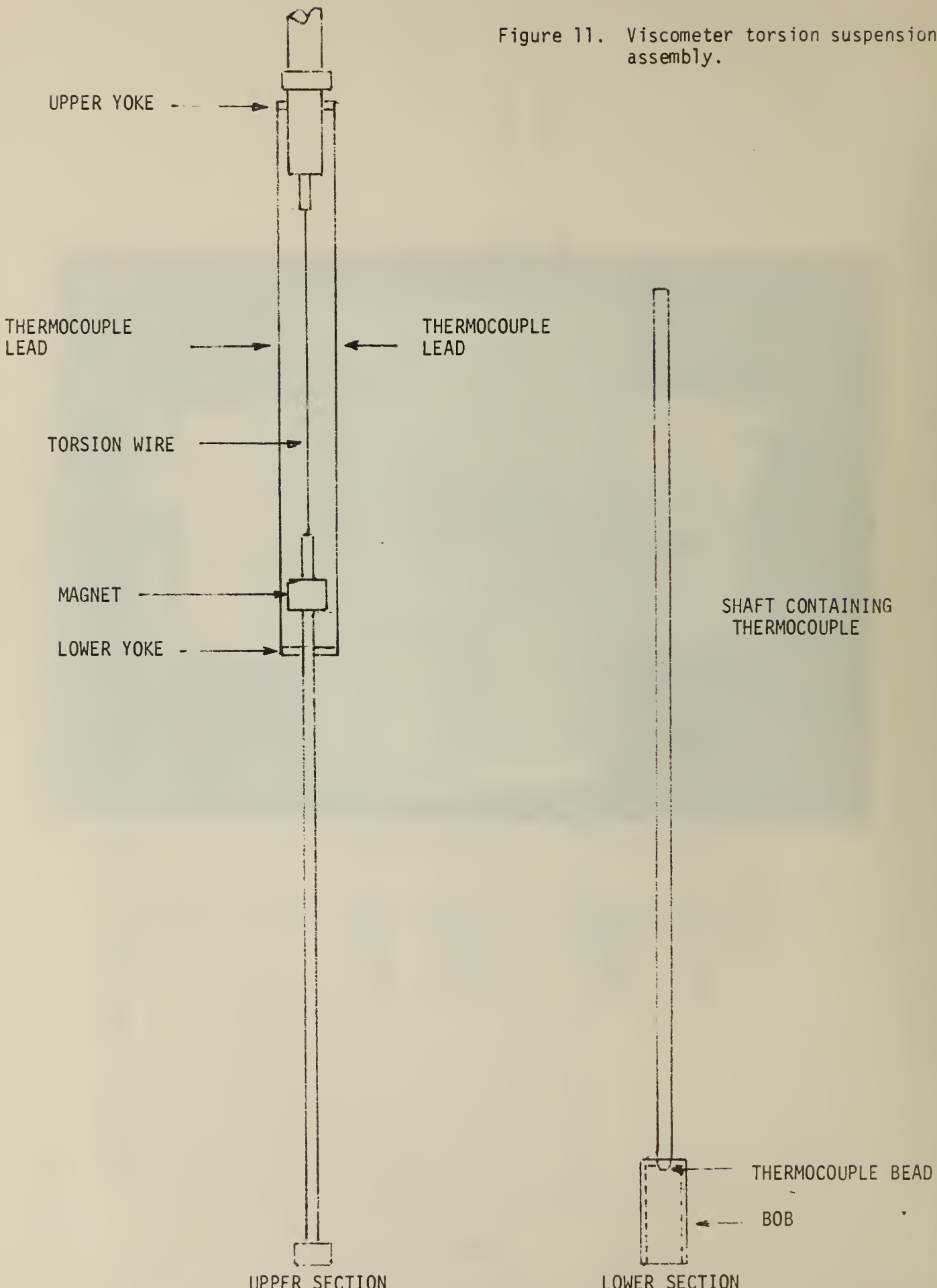


Figure 10. Water cooled seal on the rotating viscometer shaft.

Figure 11. Viscometer torsion suspension assembly.



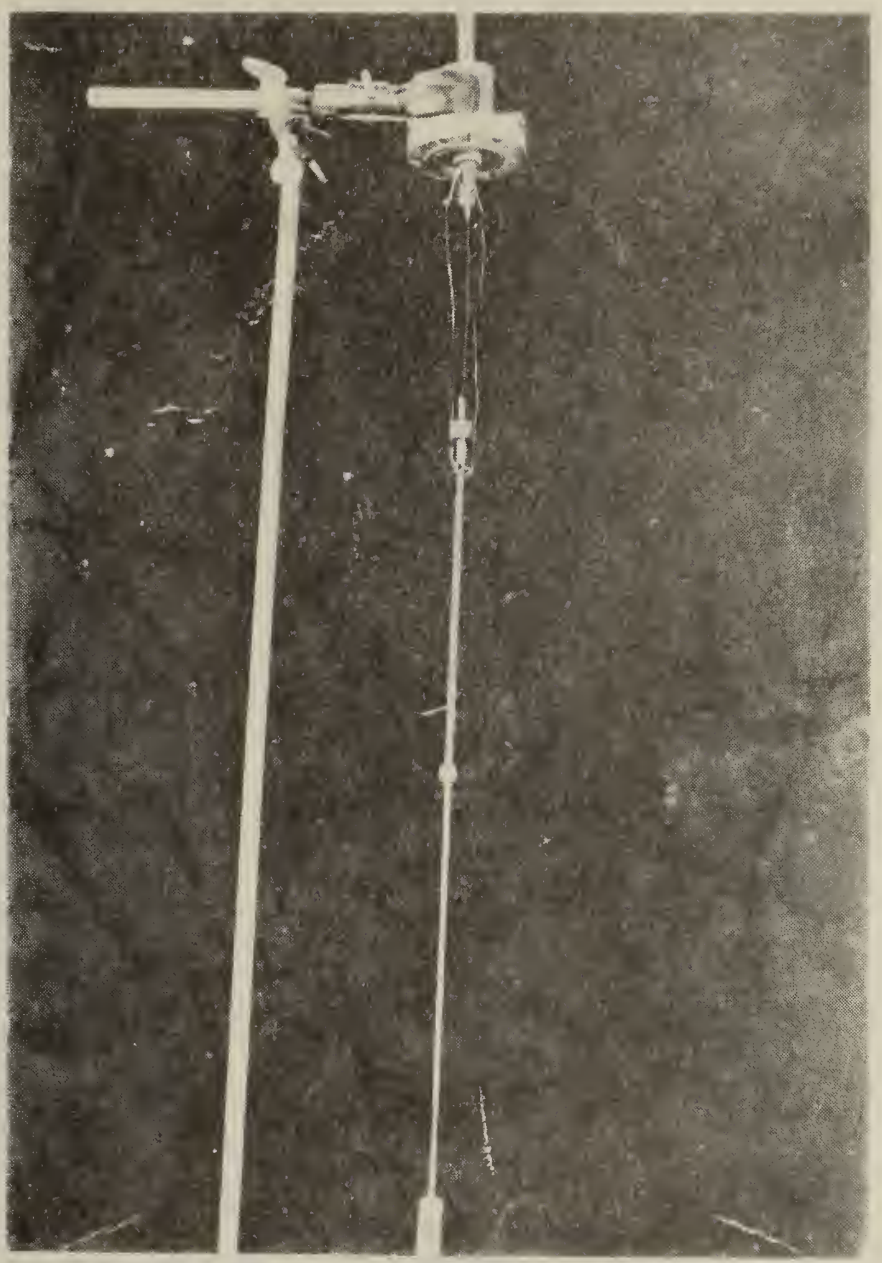


Figure 12. Viscosity torsion assembly.

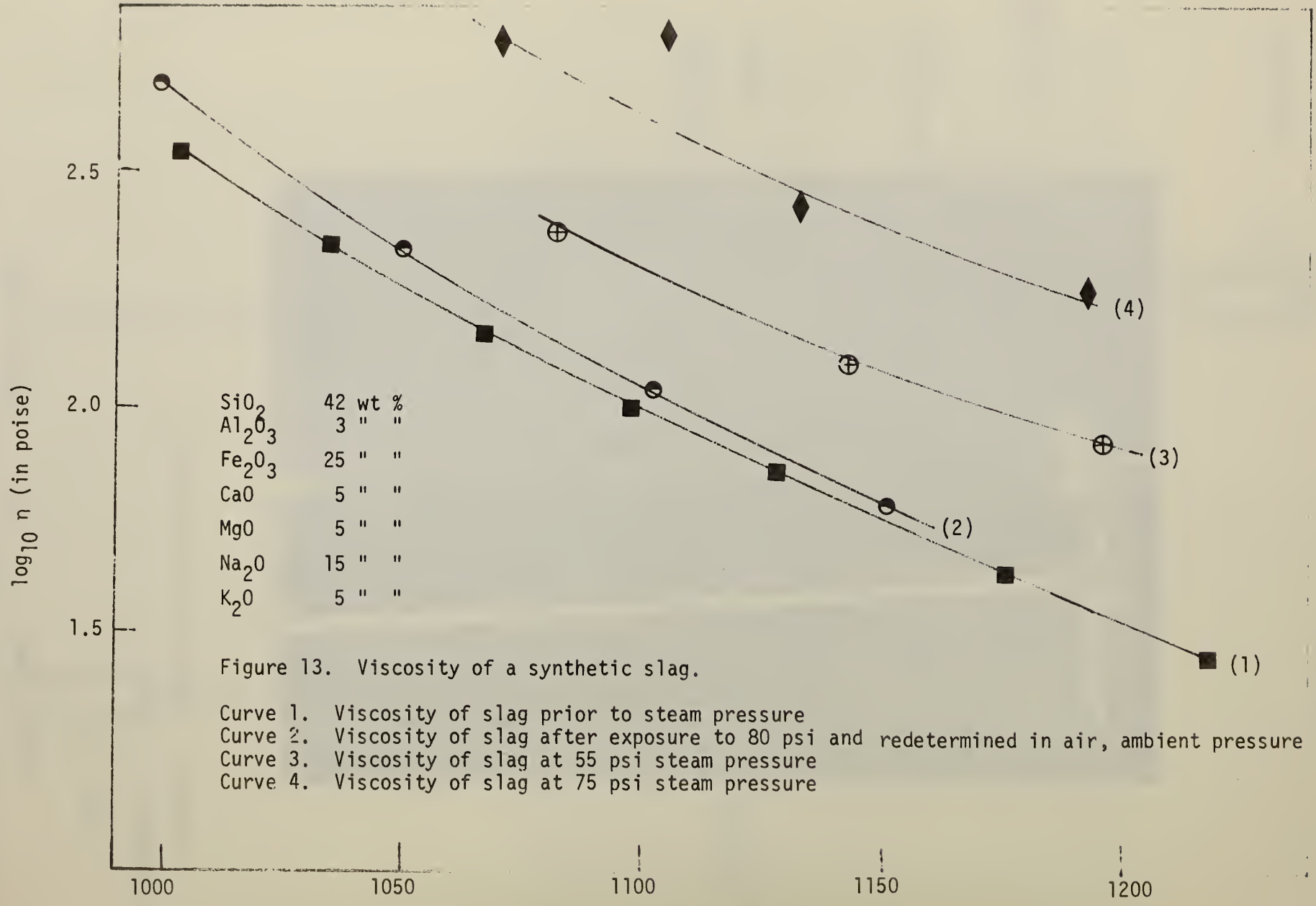


Figure 13. Viscosity of a synthetic slag.

Curve 1. Viscosity of slag prior to steam pressure
Curve 2. Viscosity of slag after exposure to 80 psi and redetermined in air, ambient pressure
Curve 3. Viscosity of slag at 55 psi steam pressure
Curve 4. Viscosity of slag at 75 psi steam pressure

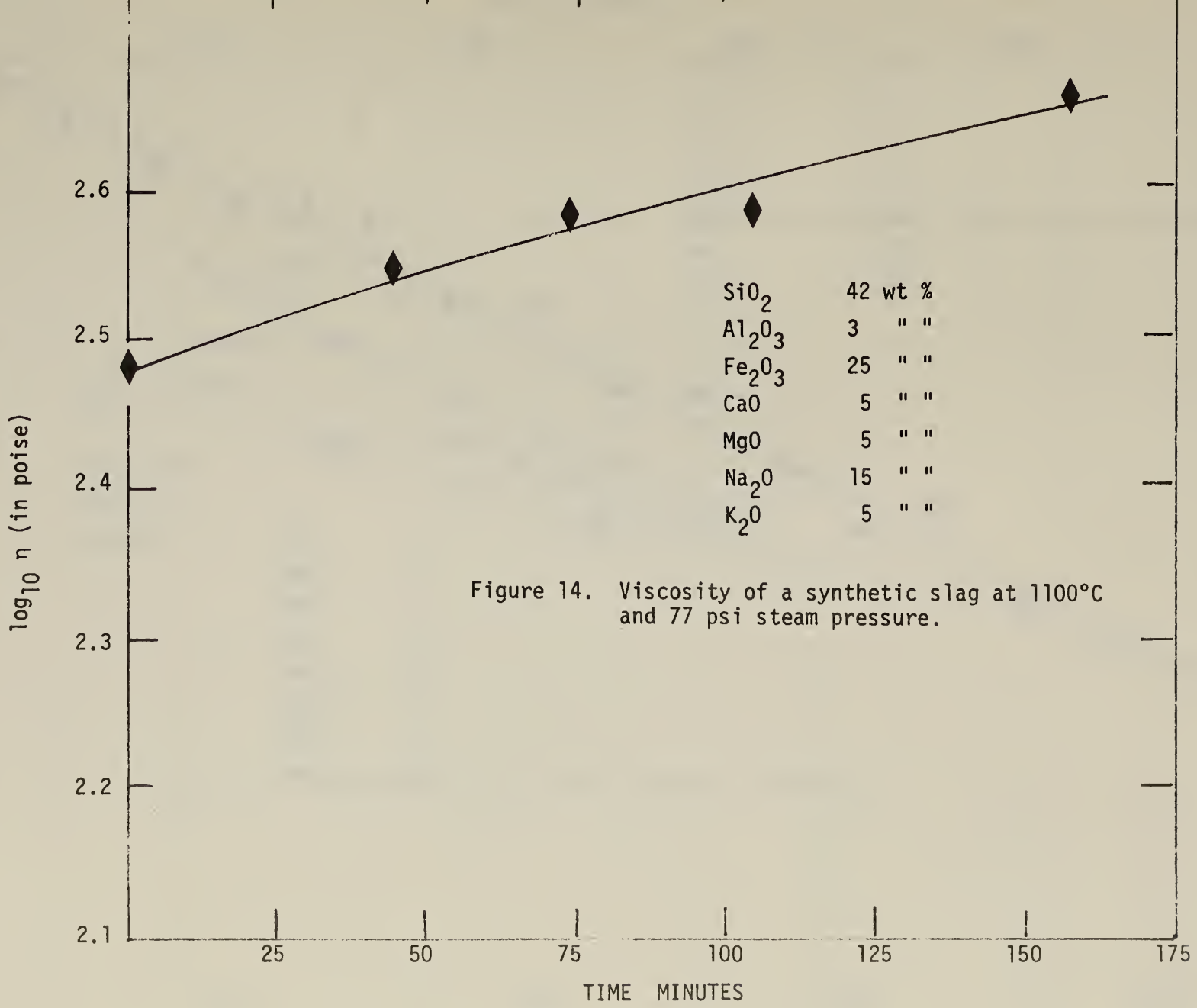


Figure 14. Viscosity of a synthetic slag at 1100°C and 77 psi steam pressure.

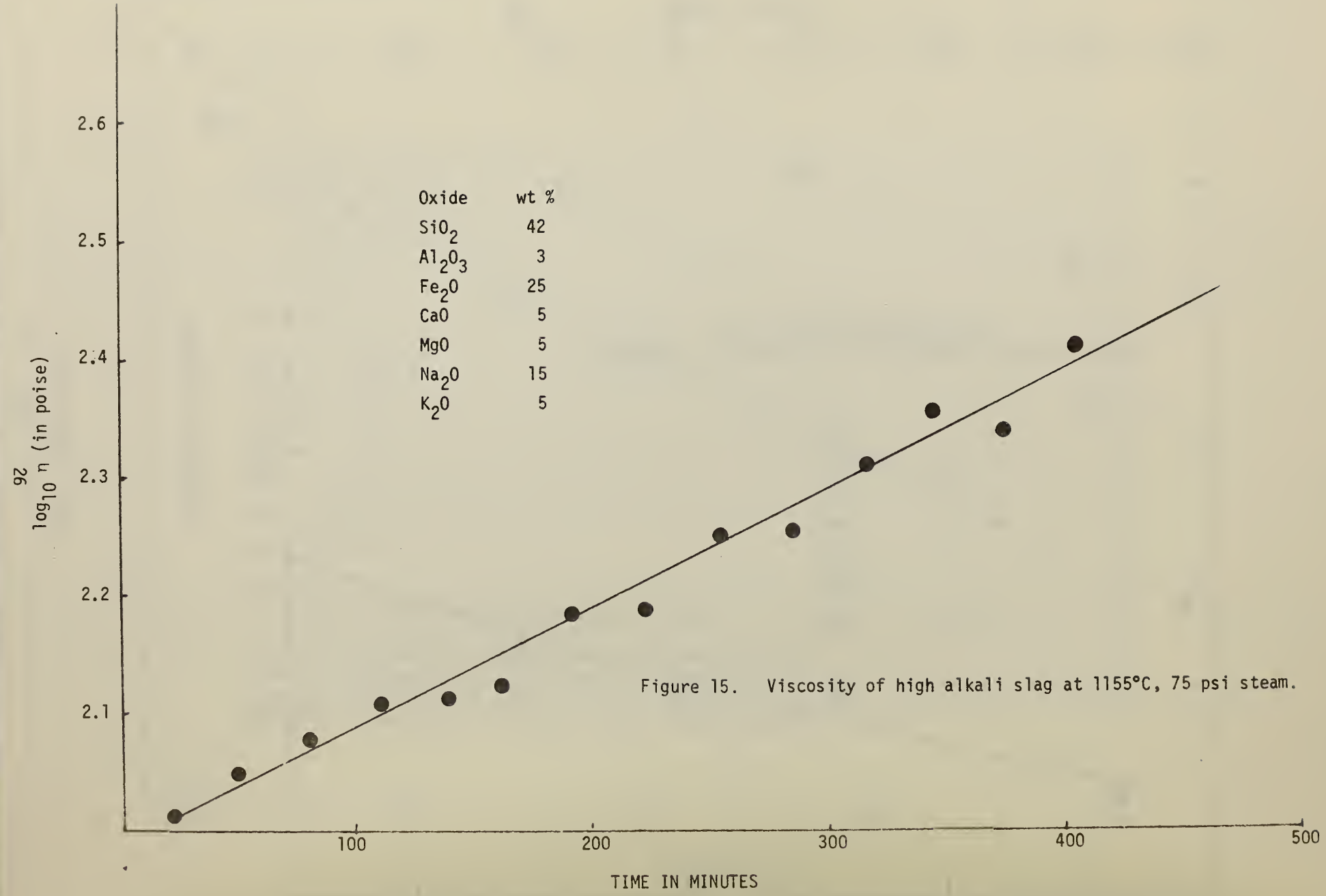


Figure 15. Viscosity of high alkali slag at 1155°C, 75 psi steam.

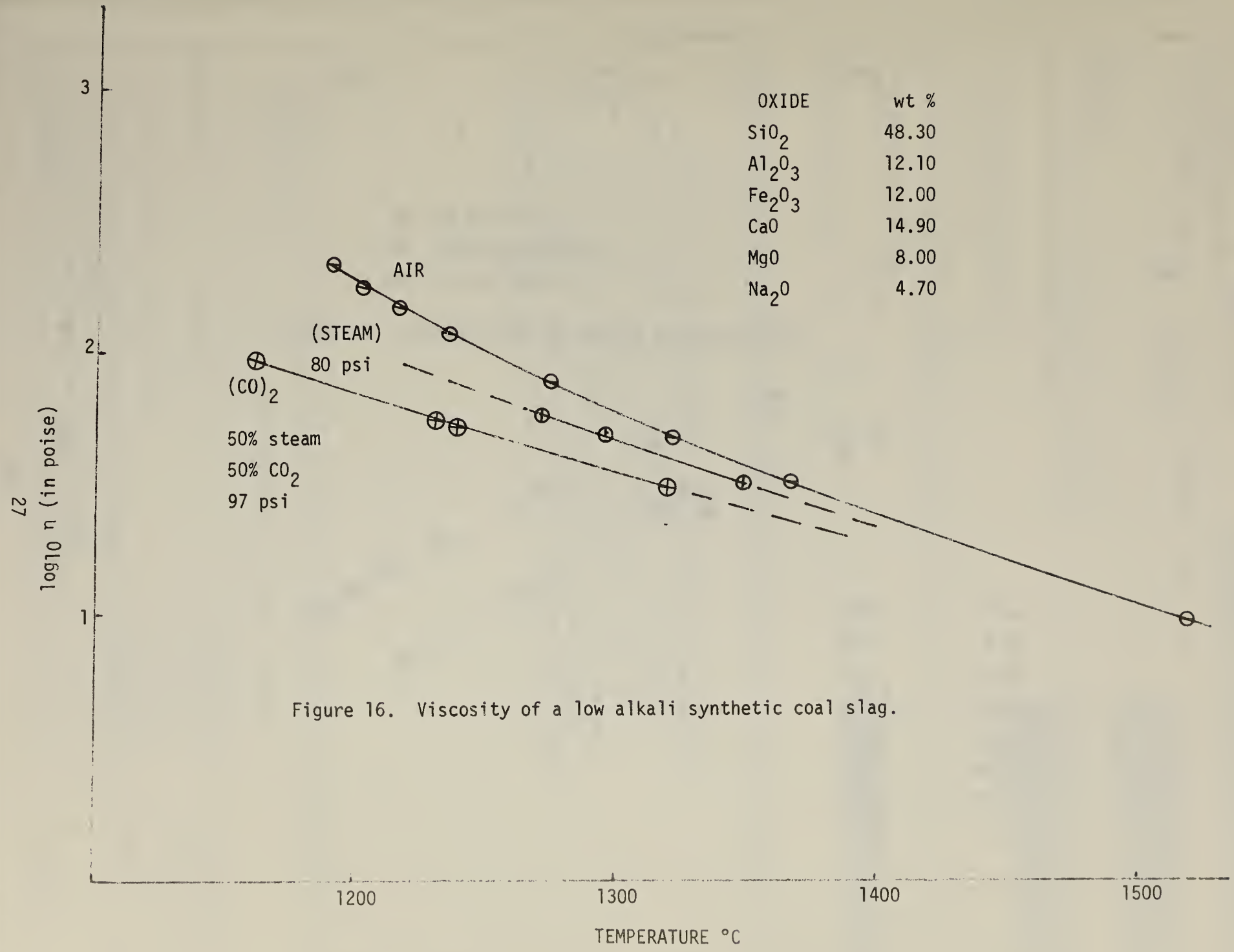


Figure 16. Viscosity of a low alkali synthetic coal slag.

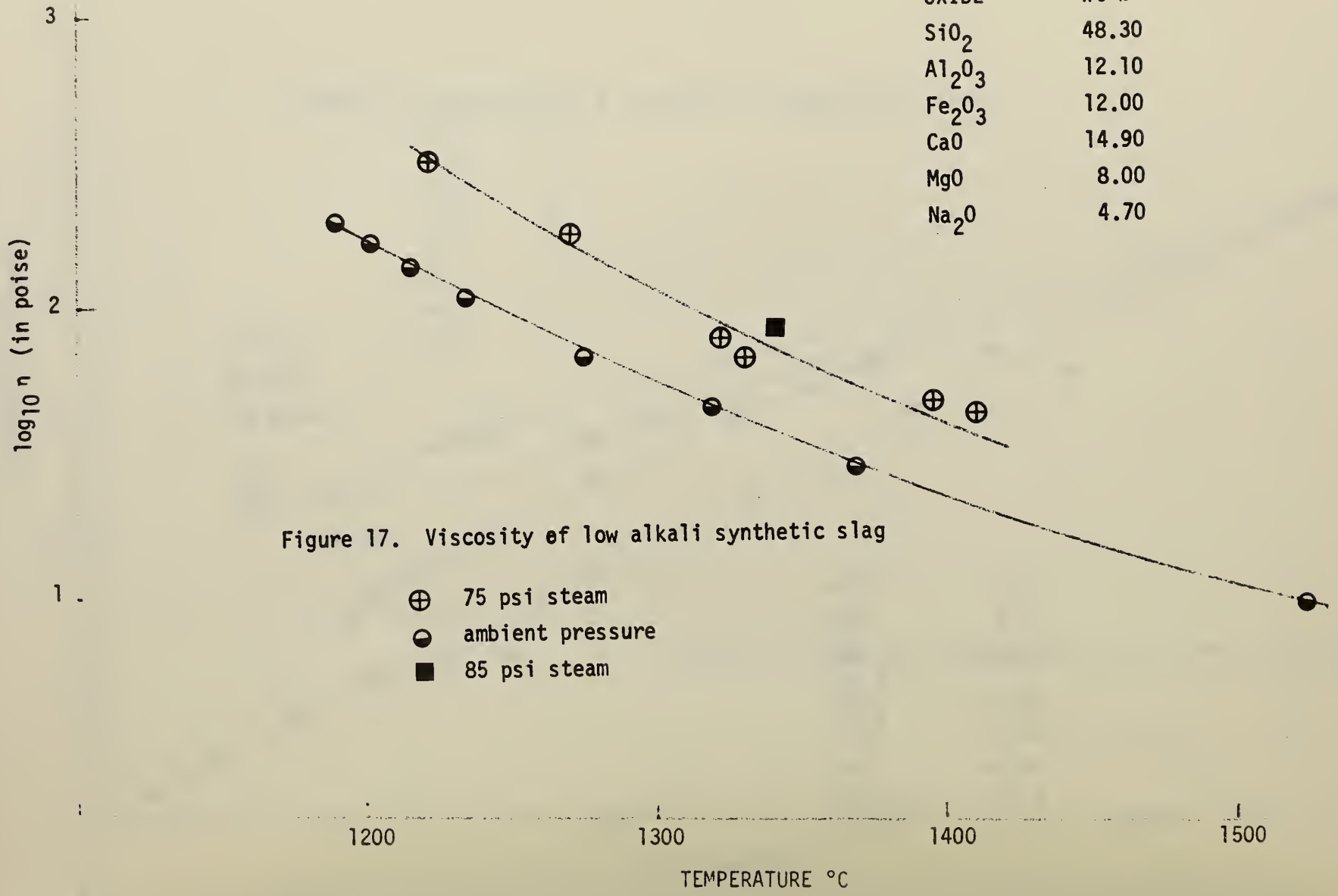


Figure 17. Viscosity of low alkali synthetic slag

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15. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

16. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)*

A rotating cylinder apparatus has been designed and constructed for measuring the viscosity of molten slags under steam pressure in the range 15 to ~300 psi and up to about 1400 °C. Two synthetic coal slags, one high in alkali, and the other lower in alkali but higher in calcia were studied with this apparatus. In the high alkali slag equilibrium was probably not achieved and the viscosity appeared to increase with time. In the low alkali slag although the viscosity appeared to be increased with increasing steam pressure the ambient and higher pressure curves appear to tend to coalesce with increasing temperature.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)*

Coal gasification; coal slag; high temperature; steam; viscosity

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