

MASTER

CONF-790855--3

DESIGN AND INITIAL TESTING OF
HIGH TEMPERATURE/PRESSURE SAMPLERS

by

T. R. Bump and H. Chang

Prepared for
1979 Symposium
on
Instrumentation and Control for Fossil Energy Processes
Denver, Colorado
August 20-22, 1979

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.



U of C-AUA-USDOE

ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

Operated under Contract W-31-109-Eng-38 for the
U. S. DEPARTMENT OF ENERGY

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

The facilities of Argonne National Laboratory are owned by the United States Government. Under the terms of a contract (W-31-109-Eng-38) among the U. S. Department of Energy, Argonne Universities Association and The University of Chicago, the University employs the staff and operates the Laboratory in accordance with policies and programs formulated, approved and reviewed by the Association.

MEMBERS OF ARGONNE UNIVERSITIES ASSOCIATION

The University of Arizona
Carnegie-Mellon University
Case Western Reserve University
The University of Chicago
University of Cincinnati
Illinois Institute of Technology
University of Illinois
Indiana University
The University of Iowa
Iowa State University

The University of Kansas
Kansas State University
Loyola University of Chicago
Marquette University
The University of Michigan
Michigan State University
University of Minnesota
University of Missouri
Northwestern University
University of Notre Dame

The Ohio State University
Ohio University
The Pennsylvania State University
Purdue University
Saint Louis University
Southern Illinois University
The University of Texas at Austin
Washington University
Wayne State University
The University of Wisconsin-Madison

NOTICE

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States nor any agency thereof, nor any of their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for any third party's use or the results of such use of any information, apparatus, product or process disclosed in this report, or represents that its use by such third party would not infringe privately owned rights. Mention of commercial products, their manufacturers, or their suppliers in this publication does not imply or connote approval or disapproval of the product by Argonne National Laboratory or the United States Government.

DESIGN AND INITIAL TESTING OF HIGH TEMPERATURE/PRESSURE SAMPLERS

T. R. Bump and H. Chang*
Components Technology Division
Argonne National Laboratory
Argonne, Illinois 60439

ABSTRACT

The ANL High Temperature/Pressure Samplers Program develops, under guidance of the Morgantown Energy Technology Center (METC), samplers for installation at selected locations in coal-process PDU, pilot, and, eventually, Demonstration plants, to improve monitoring of plant performance. Two samplers have already been designed and procured, a Non-insertable, Isothermal Gas Sampler and an Insertable Pyrometer. The former component is designed to collect gas samples near the wall of a water-cooled coal process vessel, such as the METC low-Btu gasifier, and deliver the gas isothermally to an analyzer. The sampler line heaters and their control console, which controls the heaters at the gas sample stream inlet temperature, have already been tested at 1500°F. Also, the pressure letdown system, which provides for a 300 psi drop from gasifier to analyzer, has been calibrated for use as a flowmeter.

The Insertable Pyrometer is designed to penetrate into the interior of a pressurized coal-process vessel, such as the METC gasifier, and measure radial temperature distributions, to at least 2000°F. The reference probe houses a quartz rod that transmits infrared signals from the vessel interior to a signal analyzer outside the vessel. The entire component has already operated satisfactorily away-from-vessel. In addition, an Insertable, Combined, High-velocity Thermocouple and Isothermal Gas Sampler, that combines many features of the first two components, has been designed and is under procurement. Finally, the conceptual design of a Coring-type Solids Sampler has been completed.

INTRODUCTION

Current coal-process PDU and pilot plants operate essentially as "black boxes," with characteristics of the process ingredients known only at the inlets and outlets of the process streams. Basic engineering data are needed for other locations within plant process streams operating at high temperatures and pressures; therefore, it is essential to install samplers to determine the extent of reaction completion and other process parameters, at those locations. A recent example of such a sampler for a non-coal process application is described in Ref. (1). Reference (2) describes samplers for coal-process use, but these samplers are intended primarily for characterization of particulates in gasifier product streams.

Because samplers for coal-process use are generally unavailable, ANL has been funded by DOE to develop them, under the guidance of the Morgantown (WV) Energy Technology Center (METC). Two samplers have already been designed, fabricated, and partially tested: a Non-insertable, Isothermal Gas Sampler, and an Insertable Pyrometer. A third unit called

an Insertable, Combined, High-velocity Thermocouple and Isothermal Gas Sampler, has been designed and is currently under fabrication. A fourth unit, a Coring-type Solids Sampler, has been brought through the conceptual design stage. Details of each of these samplers are given in the following sections.

NON-INSERTABLE, ISOTHERMAL GAS SAMPLER

This component, with nominal design conditions of 300 psig and 1500°F, is designed to collect an up-to-100-std- ℓ /m gas sample stream near the wall of a water-cooled coal-process vessel, such as the METC low-Btu gasifier, Ref. (3), and deliver the gas isothermally to a spectrometer for on-line analysis. Isothermal delivery is required to maintain the sample stream in chemical equilibrium so that ex-vessel spectrometer data can be readily extrapolated to indicate gas compositions inside the vessel. A side benefit of isothermal delivery is that tar condensation in the sample stream is discouraged, thereby increasing analysis accuracy and decreasing the possibility of sample line plugging. Gasifier gas compositions are of interest to METC researchers because, in their efforts to develop improved product stream cleanup systems, they desire to correlate gasifier operating conditions with performance of cleanup system components.

Because heat loss conditions vary considerably along the sample line between vessel and analyzer, seven separate heaters are provided. Each of the heaters is controlled independently to maintain the local sample line temperature equal to that of the gas entering the sample line. The gas inlet temperature is measured by a "high velocity thermocouple," Ref. 4. Such a thermocouple is designed to (a) promote convective heat transfer to the thermocouple hot junction by providing high gas velocity at the junction, and (b) reduce radiative heat transfer from the hot junction, by surrounding the junction with a radiation shield(s), preferably heated to the junction temperature. In this way, the junction temperature can be very close to that of the gas passing the junction.

Three of the seven heaters are incorporated in an Alumina Heater, Fig. 1, which is comprised of three lengths of doubled, 20 ga., Incoloy 800 wire* wound at three axial locations on a 3/8 in. OD x 1/4 in. ID x 38 in. long high-purity alumina tube. The wire is bonded to the tube, and electrically insulated, with alumina cement, and covered with Refractory Products WRP-X-AQ thermal insulation. Also bonded to the tube are control and limit thermocouples, one each for each heater, consisting of 1/16 in. OD, Inconel 601 sheathed, ungrounded, Type K wire. The gas inlet temperature is measured with a similar thermocouple (the high velocity thermocouple) that travels the length of the alumina tube between the heater wire and thermal insulation and is bent to enter the inlet end of the tube as can be seen in Fig. 1. In event of failure of this thermocouple, a backup unit, centered by "spiders," would be installed along the axial centerline of the tube.

*Incoloy 800 (and Inconel 601) are used because of their superior resistance to sulfidation. Alumina is used because of its chemical inertness and high-temperature strength.

The Alumina Heater serves to carry the gas sample stream isothermally through the gasifier water jacket. The Heater thermal insulation fits snugly inside a 1-1/4 in., Sch. 40 Incoloy 800 horizontal pipe that is attached to the inner wall of the water jacket and provides a gas-water pressure boundary. That Incoloy pipe in turn is centered inside an existing 2 in. carbon steel nozzle, Fig. 2, welded to the water-jacket outer wall. The nozzle is flanged to a bellows Expansion Joint welded at its other end to the outboard end of the 1-1/4 in. Incoloy pipe. The Expansion Joint allows for differential expansion between Incoloy pipe and 2 in. nozzle, and is provided with a tap to allow cooling water to circulate from the water jacket, through the nozzle and Expansion Joint, and on to the suction of the water jacket circulating pump.

The outboard end of the Expansion Joint is flanged to a Type 316 stainless steel Spool Piece of 6 in., Sch. 40 pipe, Fig. 3, that is flanged in turn to a Tapered Connection, Figs. 4 and 5. The Tapered Connection provides a loose connection between the Alumina Heater and downstream metal sample lines. The Tapered Connection is centered about a 3/4 in., Sch. 80 Incoloy 800 pipe that has a loose socket for the Alumina Heater's alumina tube at one end and an Autoclave 9/16 in. OD tubing, Type 316 stainless steel modified coupling at the other. The Incoloy pipe is wound with a controlled heater to allow carrying the sample gas stream isothermally from the Alumina Heater to a Throttle and Shutoff subassembly, composed primarily of Autoclave fittings, that leads to the sample analyzer. Two reducers connect the 3/4 in. pipe to a 6 in. flange that is bolted to the outboard end of the Spool Piece. The two reducers provide a gradual temperature transition from the up-to-1500°F 3/4 in. pipe to the cool Spool Piece. Conservative calculations indicate that the Tapered Connection can withstand at least 500 thermal cycles between 70°F and 1500°F at 300 psi without risk of fatigue failure. The Tapered Connection also provides, in its 6 in. flange, the gas sample-air pressure-boundary penetrations for the Alumina Heater's mullite-beaded heater lead wires and its thermocouples, which are sealed at the penetrations with Conax fittings. WRP-X-AQ insulation is installed in the remaining gap between Tapered Connection reducers and Spool Piece.

The Throttle and Shutoff subassembly, between Tapered Connection and analyzer, includes two shutoff valves, a throttle valve, and a filter. Also included are tees for measuring gas sample temperatures, to ensure isothermal conditions are being achieved, and taps for backflushing with either steam or gas. A Pressure Letdown is provided to help reduce the sample gas pressure, from the up-to-300 psi gasifier level to the atmospheric analyzer level, by use of pipe friction (rather than by orificing which, with local velocity increases, would reduce local temperature). The Letdown consists of a 7/16 in. OD x 1/16 in. wall x 8 ft long stainless steel tube holding a second 1/4 in. OD plugged tube that is centered by dimples formed in the outer tube. Both tubes are wound into a 7-1/2 in. OD x 12 in. high helix to reduce overall length. The sample gas stream flowing through the annulus between tubes undergoes a significant pressure drop under desired isothermal conditions. The Throttle and Shutoff subassembly components are provided with three separate heaters of 1/8 in. OD sheathed heater wire, and insulated with WRP-X-AQ material. One of the three heaters is dedicated to the valves, which are not allowed to exceed 1200°F.

The Alumina Heater and the Heater Console, Fig. 6, have already been tested together, with the Heater at 1500°F in air, and operated satisfactorily after modest improvements were made. The Heater was originally made with the welds between heater wires and heater lead wires (the latter twice as big in diameter as the former) outside of the alumina bonding cement. This resulted in hot spots in the exposed heater wire at those locations, because the relatively high thermal-conductivity cement was not available to carry the heat away. The Heater was repaired to ensure that the heater wires were completely covered by cement. Also, the heater controllers were originally not provided with variable transformers, the idea being that the controllers' voltage-pulsing capabilities could avoid heater overheating regardless of maximum applied voltage. This did not prove to be the case, so variable transformers were added to allow reducing applied heater voltage.

Also, the Pressure Letdown has been calibrated for pressure drop vs. flowrate, so that it can be used as a gas-sample-stream flowmeter. Unfortunately, the Pressure Letdown was found to produce less pressure drop than desired, primarily because last-minute parts availability necessitated using parts that produced a slightly larger flow annulus than was originally selected. It is not planned to procure an additional higher-pressure-drop device of this type until the first has been tested under prototypic conditions; the device, with its narrow annulus, may tend to plug too readily and have to be re-designed.

INSERTABLE PYROMETER

This component, also with nominal design conditions of 300 psig and 1500°F, is designed to measure radial temperature distributions within a water-cooled coal-process vessel such as the METC low-Btu gasifier. Gasifier temperatures are of interest to METC researchers again because they wish to correlate them with performance of product stream clean-up system components.

Figure 7 illustrates an assembly of a number of subassemblies of the Insertable Pyrometer, namely the Guide Rod and Support Subassembly, the Slide Subassembly, the Probe Subassembly, and a hydraulic cylinder. The reference Probe Subassembly consists of a housing of 1 in., Sch. 160 Incoloy 800 pipe which contains internals the most important of which is a stainless-steel-encased, 1/4 in. dia. quartz rod that extends along the axial centerline of the Probe. The inboard end of the quartz rod receives infrared signals from various radial positions in the gasifier's bed, and the rod transmits the signals to a Vanzetti signal analyzer (not shown) at the outboard end of the Probe.

The actual connection between the quartz rod and the signal analyzer consists of a short length of flexible fiber optics, provided by Vanzetti. The correlation between signal analyzer output and temperature is dependent on the gasifier bed's emissivity at the inboard end of the quartz rod. (The temperature recorder, Fig. 8, has provision for emissivity input.) It is expected that the bed's emissivities are very close to unity; however, even if the emissivities were as low as 0.8 the error in recorded temperatures, assuming $\epsilon = 1$, would be less than five percent.

It is anticipated that differential thermal expansion, between the quartz and potential deposits from the gasifier bed, may keep the inboard end of the quartz "clean". If not, steam-jet cleaning is a possible backup, as is substitution of a thermocouple(s) for the quartz rod. Potential advantages of the quartz rod over thermocouples are faster time response and longer life. Even though the design temperature of the Probe is "only" 1500°F, it is anticipated that it will be possible to insert its inboard end momentarily to the center of the METC bed, where temperatures as high as 2400°F could be encountered.

A concentric ring is welded to the outboard end of the Probe, and this ring is "trapped" by the Slide Subassembly which rides, courtesy of Thomson ball bushing pillow blocks, on the guide rods of the Guide Rod and Support Subassembly (Fig. 7). The weight of the latter is supported by calibrated-spring hangers. A hydraulic cylinder bolted to the Guide Rod and Support Subassembly has its piston rod bolted to the Slide Subassembly and provides the force necessary to insert the Probe against the gasifier pressure. The Probe is sealed by a Stuffing Box Subassembly, Fig. 9, which uses Garlock graphite packing and is flanged at its inboard end to a bellows Expansion Joint flanged at its own inboard end to an existing 2 in. OD carbon steel nozzle welded to the gasifier water-jacket outer wall. Inboard of the Stuffing Box Subassembly, the Probe slides inside a 1-1/4 in., Sch. 40 Incoloy 800 horizontal pipe that is attached to the inner wall of the water jacket. The outboard end of this pipe is welded to the outboard flange of the Expansion Joint. As with the Non-insertable, Isothermal Gas Sampler, the Expansion Joint is provided with a tap to allow cooling water to circulate from the water jacket, through the nozzle and Expansion Joint, and on to the suction of the water jacket circulating pump.

The Probe insertion load provided by the hydraulic cylinder is borne by the water-jacket nozzle flange, which the Guide Rod and Support Subassembly reacts against. The hydraulic pump for the hydraulic cylinder is shown in Fig. 10. The pump discharge line is provided with a relief valve to prevent excessive load from being exerted on the water-jacket nozzle.

An electro-mechanical position indicator is mounted on the Guide Rod and Support Subassembly. The analog signal from this device, as well as the one from the infrared signal analyzer, is recorded by the unit shown in Fig. 8. Thus one is able to read how gasifier bed temperature varies with radial position, each time the Probe is inserted into the gasifier.

Testing of the Insertable Pyrometer to date has consisted of connecting the hydraulic cylinder, Fig. 7, to its hydraulic pump, Fig. 9, and checking Probe movement under substantial load. The assembly operated satisfactorily. Also, operation of the temperature-position recorder, Fig. 8, was checked, using an infrared calibration source provided by Vanzetti. Again, the system tested operated satisfactorily.

INSERTABLE, COMBINED, HIGH-VELOCITY THERMOCOUPLE AND ISOTHERMAL GAS SAMPLER

This "ICHVT & IG Sampler" combines the isothermal gas sampling and

high-velocity-thermocouple capability of the Non-insertable, Isothermal Gas Sampler with the insertion capability of the Insertable Pyrometer. In fact, development of the latter two devices deliberately preceded that of the former, so that its potential problems could be divided into roughly two parts for parallel efforts toward solving them. Thus, the Alumina Heater, Expansion Joint, Tapered Connection, Throttle and Shutoff Subassembly, Pressure Letdown, and Heater Console of the Non-insertable, Isothermal Gas Sampler, and the Probe, Guide Rod and Support Subassembly, hydraulic cylinder and pump, Slide Subassembly, and Stuffing Box Subassembly of the Insertable Pyrometer all have their direct counterparts in the ICHVT & IG Sampler.

Specific differences between the present sampler and its two predecessors follow:

1. The high-velocity thermocouple is 1/4 in. OD rather than 1/16 in., to allow a 4x thicker sheath and correspondingly increased sulfidation resistance. Also, the heater wires are 17 ga. rather than 20 ga., for the same reason. The spider-centered 1/4 in. OD thermocouple runs the entire length of the alumina sample-gas tube, which is accordingly 5/8 in. OD x 1/2 in. ID.
2. To accommodate the alumina tube, heater wires, control and limit thermocouples, and thermal insulation, the Probe is 2 in. dia. instead of 1-1/4 in., requiring that a new 3-1/2 in. nozzle be installed on the METC gasifier water jacket.
3. The Slide Subassembly is connected to the Probe simply by being bolted with the Tapered Connection flange bolts. (The Tapered Connection, and Throttle and Shutoff Subassembly are parts of the Probe.)
4. Differential thermal expansion between the pipes attached to the gasifier water-jacket inner and outer walls (the former pipe being that which the Probe slides through, and the latter that to which the nozzle flange is welded) is accommodated by an O-ring-sealed slip joint rather than by a bellows expansion joint. However, the spool piece holding the O-ring can be replaced by a standard bellows joint of equal length if the O-ring should prove unsatisfactory.
5. The current intention is that the gas analyzer remain stationary while the Probe is moving. Therefore, the Throttle and Shutoff Subassembly are connected to the analyzer with a flexible metal hose that is equipped with heaters and covered with thermal insulation.

There are two possible ways to seal the insertable heated probe of the ICHVT & IG Sampler. First, a sliding seal could be made directly against the heated probe; this seal can be called a "high-temperature seal." Second, a sliding seal (a "low-temperature seal") could be made against a cooled connection welded to the heated probe. An early tradeoff study had resulted in selection of the "low-temp. seal" approach, because uncertainties concerning performance of a "high-temp. seal" were believed to be too great to face. However, as time passed two additional significant things were learned. First, tests on the Alumina Heater for the Non-insertable Isothermal Gas Sampler had shown that a compact heated

probe can be built with enough internal insulation to keep a "high-temp. seal" from actually becoming very hot; this eliminated the main concern over that design approach. Second, detailed calculations for the "low-temp seal" design had shown that it was difficult to provide for the high insertion force (14,000 lb) required for that design, especially when considering shock loads that might result from hydraulic line failure. (The insertion force for the "high-temp. seal" design is only 1800 lb.) Accordingly, a second tradeoff study was made, and results from that led to eventual selection of the "high-temp. seal" version of the sampler design.

The ICHVT & IG Sampler design has been completed, and this component is currently being fabricated.

CORING-TYPE SOLIDS SAMPLER

This component is designed to take core samples from the interior of a water-cooled coal-process vessel, such as the low-Btu METC gasifier, and allow the samples to be transported in a "bomb" to a laboratory for analysis. The purpose of such analyses is to increase understanding of how coal lumps or particles change size and composition as they move from the feed end to the discharge end of the vessel.

A one-end-open tube with saw-tooth leading edge is inserted radially into the vessel interior, and rotated simultaneously, the insertion being accomplished by hydraulic force and the rotation manually, at least in the initial version. Material inside the vessel is thereby trapped inside the tube, in roughly the same relative position as when undisturbed. The tube is then withdrawn from the vessel, carrying the trapped material with it as aided by ratchet-type lugs at the tube open end. The tube is withdrawn through a nozzle flanged to two open ball valves which are flanged to each other. When the sample tube is past the valves, they are closed and the flanged connection between them is broken to allow removal of the sample to a laboratory.* The outboard ball valve is piped to a stuffing box which seals the outboard end of the sample tube. The sample tube is equipped with extension pieces which must be added during sample-tube insertion, and removed during sample-tube withdrawal. This provision permits substantial reduction of the overall length of the complete sampler but of course increases the complexity of taking a sample.

The conceptual design of the Coring-type Solids Sampler has been completed. At present there are no plans for proceeding further with this component.

FURTHER TESTING

Originally it was planned to perform the initial prototypic testing, or use, of the first three samplers described here with the METC gasifier itself. However, that facility is only operated several weeks each year and during that limited time it must operate very reliably in order to serve its main purpose of providing a product stream for testing of cleanup equipment. Because testing or use of the samplers at their present stage might decrease the gasifier reliability, it has been decided to

*Chemical quench to halt the reactions is possible.

"debug" the samplers with the METC "dirty gas generator." The start of such testing has not yet been scheduled.

SUMMARY

Several components for sampling contents of water-cooled coal-process vessels operating under high temperatures and pressures have been designed and built or are under fabrication. Initial testing has been completed with satisfactory results. However, much more rigorous testing is required before the samplers will have proven themselves to be useful devices for aiding development of commercial plants for enhanced coal utilization.

ACKNOWLEDGMENTS

The helpful advice and cooperation of METC are gratefully acknowledged, especially those of Messrs. J. J. Kovach (technical monitor), K. Pater, P. Bekowies, and R. V. Rahfuse.

REFERENCES

1. S. M. Csicsery, et al., "Catalyst and Gas Samplers for Fluid Catalytic Cracker Regenerator," Ind. Eng. Chem., Process. Des. Develop., 14:1, 93-96 (1975).
2. W. Szwab, W. H. Fischer, and B. N. Murthy, "Technical Support for Coal Conversion and Utilization: Techniques for Particulate Sampling and Measurement from Gasifiers at High Temperature and High Pressure," FE-2200-5 (Dec 1976).
3. A. J. Liberatore and D. W. Gillmore, "Behavior of Caking Coals in Fixed-bed Gasifiers," CONF-750868-1 (Aug 1975).
4. H. F. Mullikin, "Gas-temperature Measurement and the High-Velocity Thermocouple," in "Temperature - Its Measurement and Control in Science and Industry," Reinhold, New York, 1941, p. 775 ff.

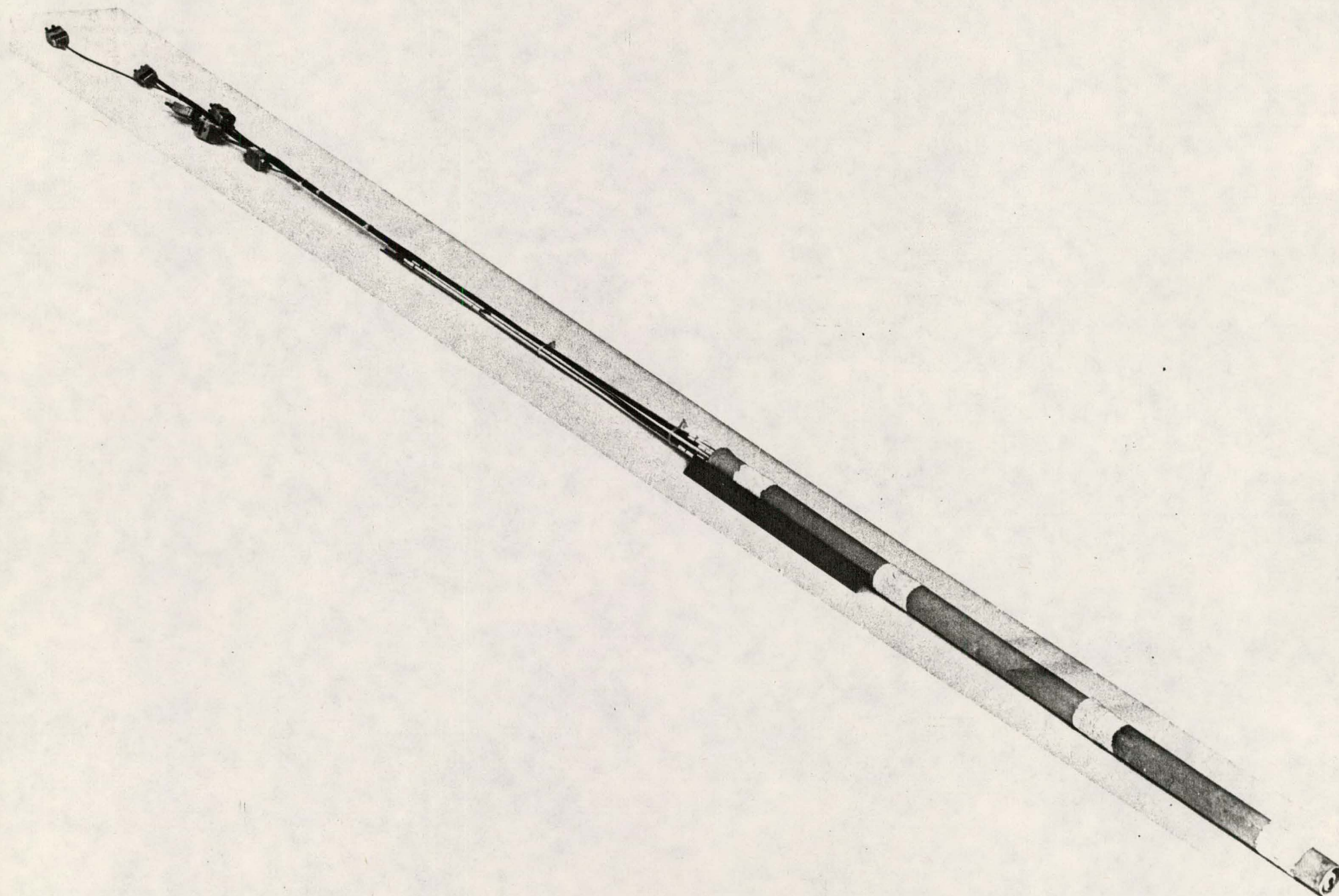


FIG. 1 ALUMINA HEATER

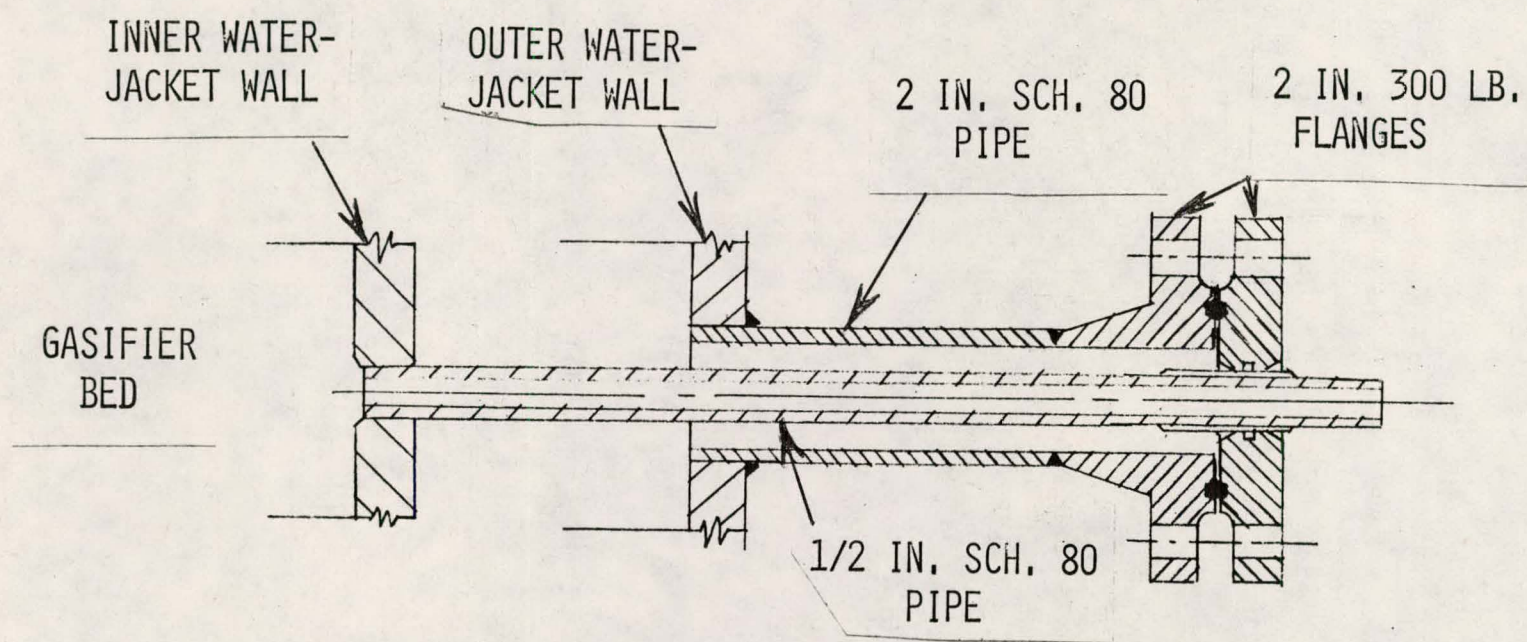


FIG. 2 EXISTING METC GASIFIER NOZZLE (TYPICAL)

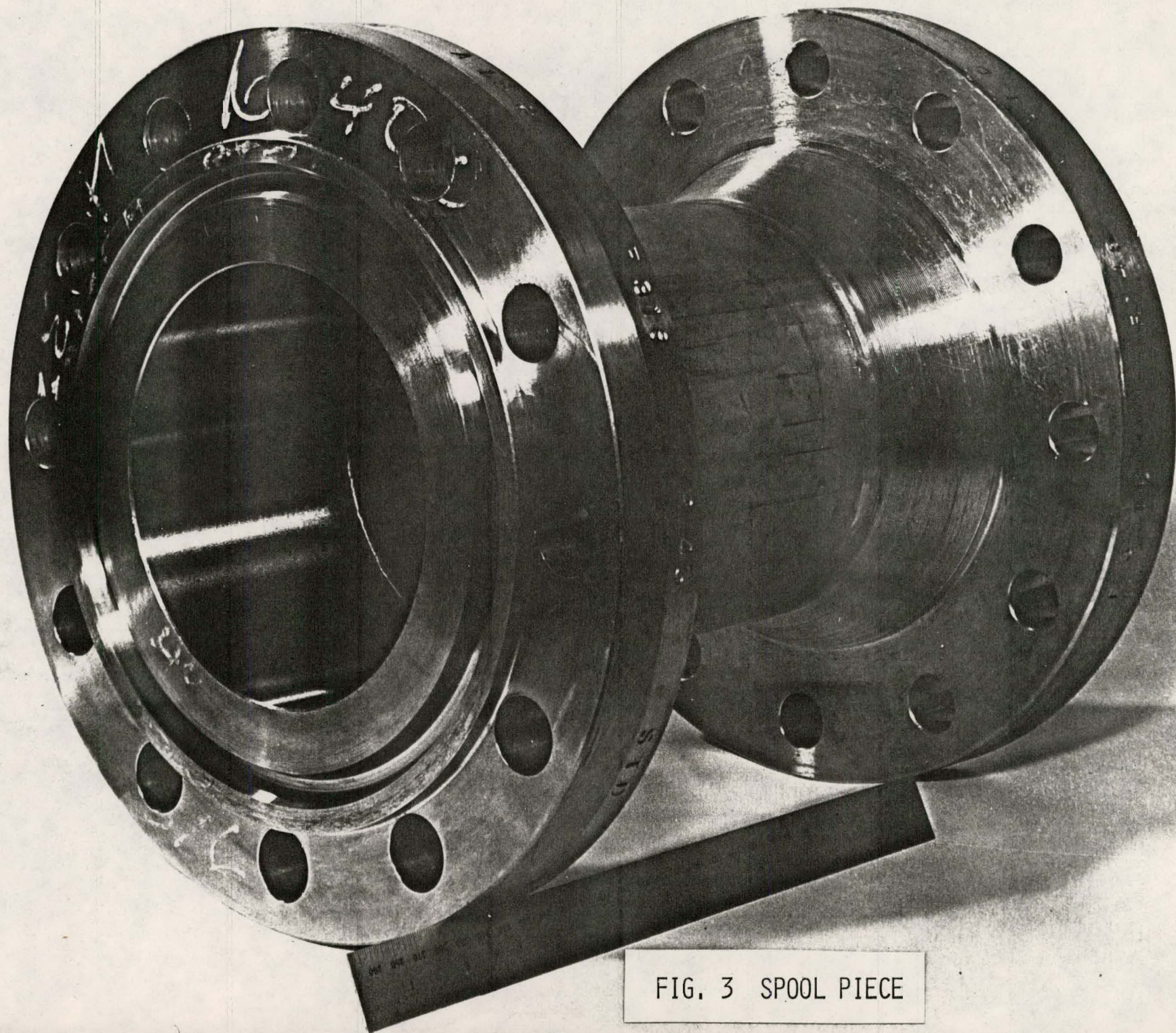


FIG. 3 SPOOL PIECE

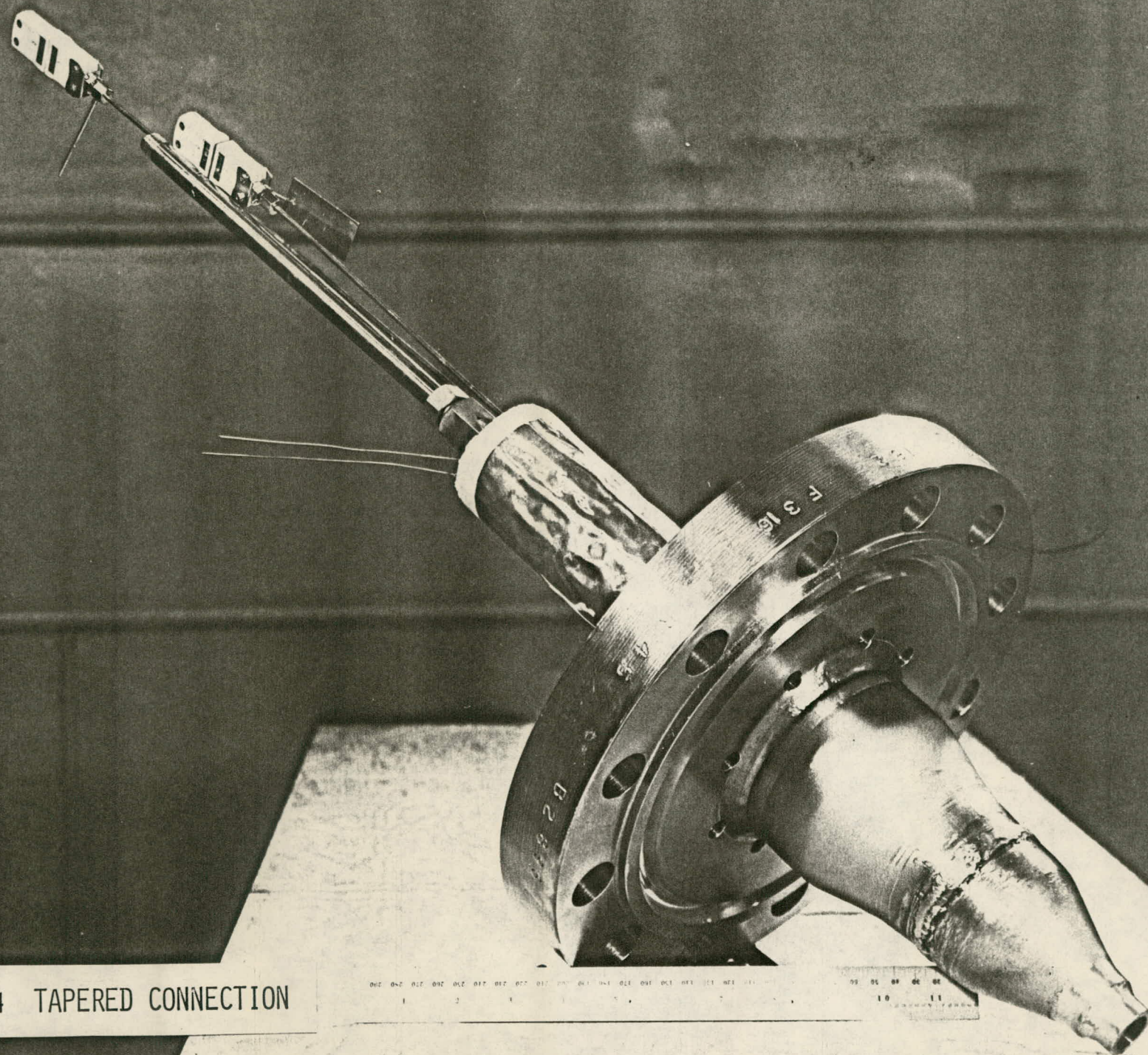


FIG. 4 TAPERED CONNECTION

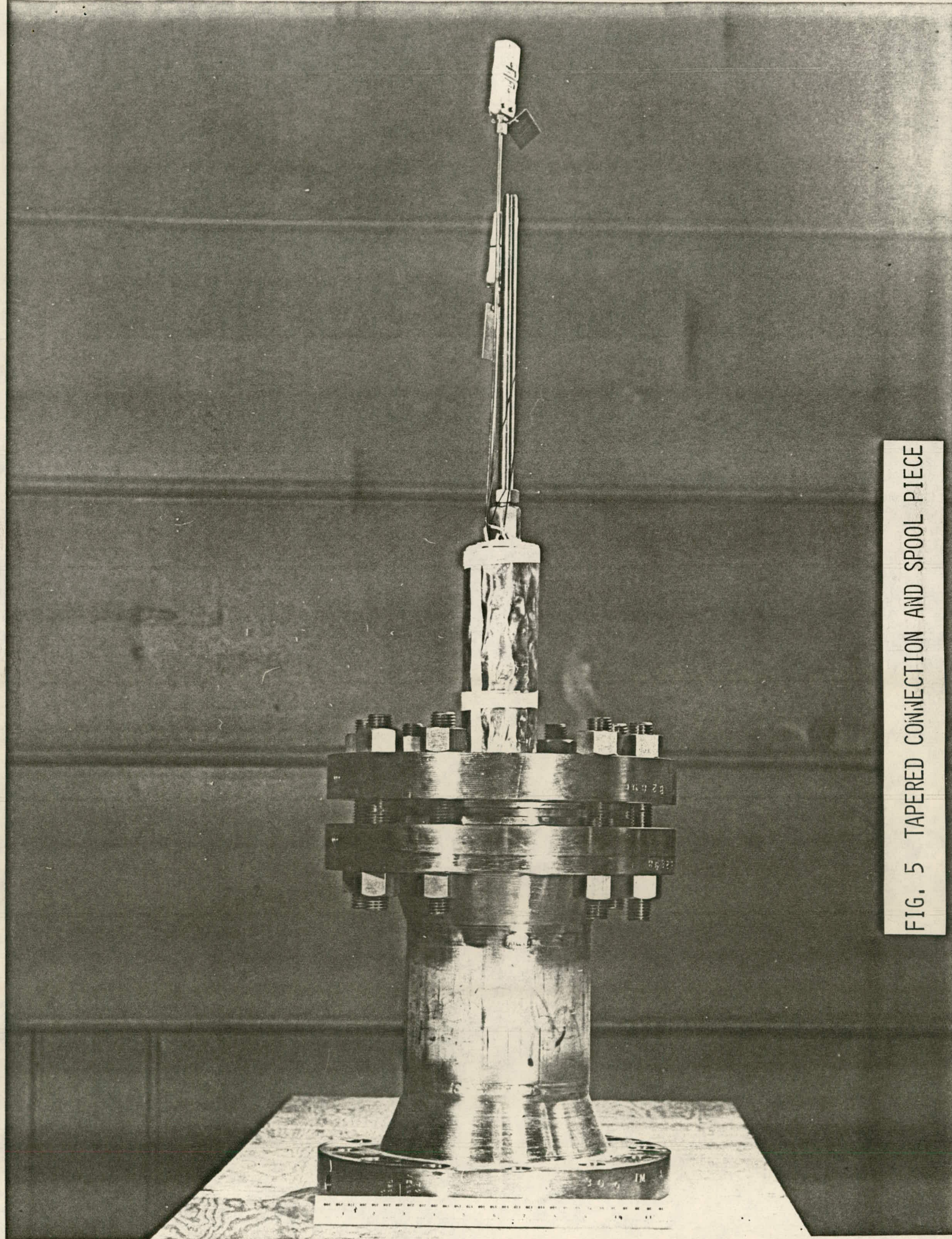
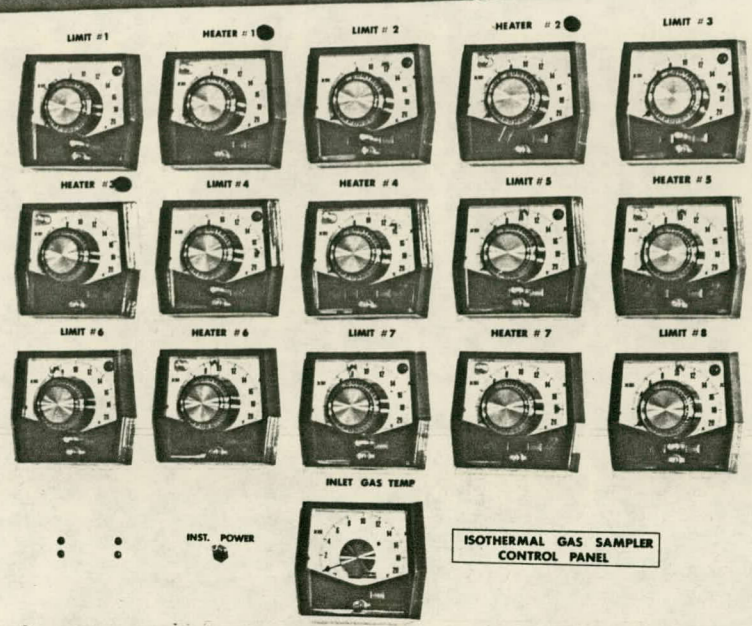
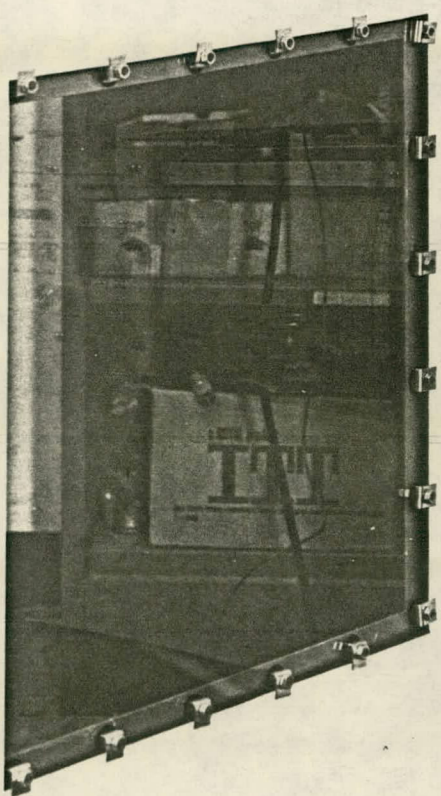
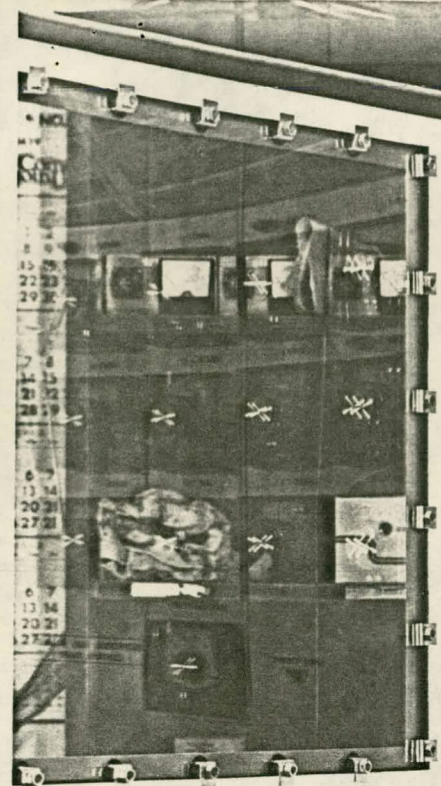


FIG. 5 TAPERED CONNECTION AND SPOOL PIECE



CAUTION !
RELEASE AND CLEAR
CH. BRKR. PANEL
BEFORE OPENING

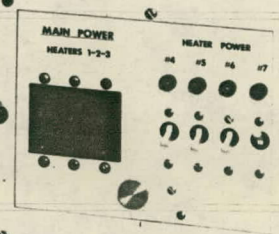
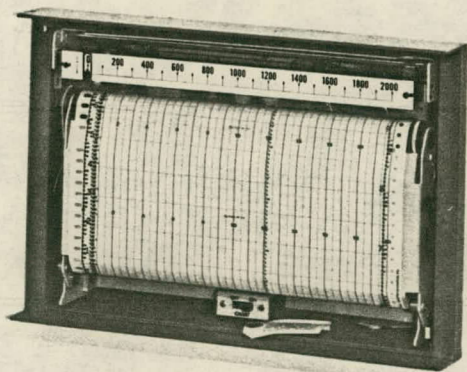


FIG. 6 HEATER CONSOLE

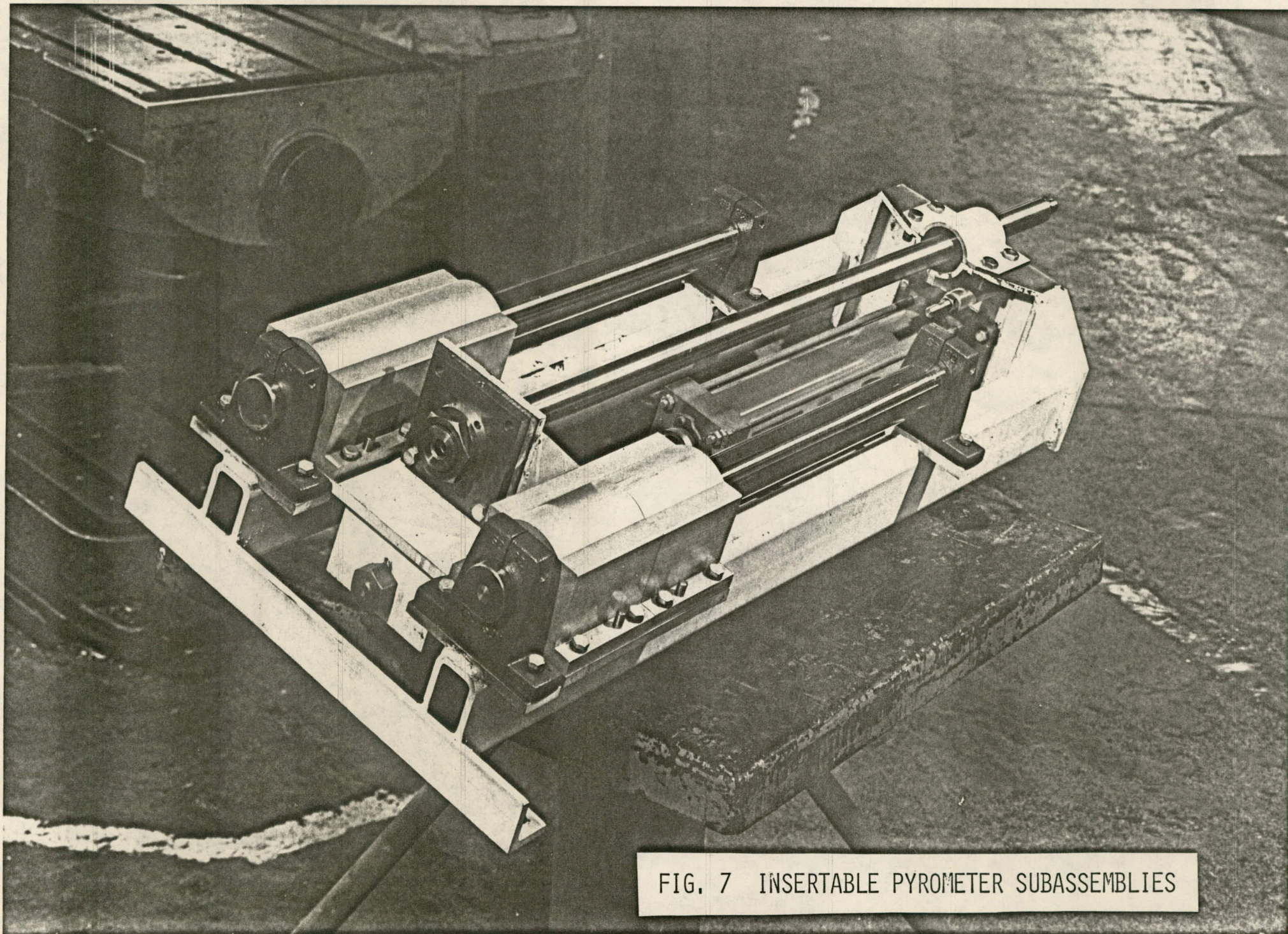


FIG. 7 INSERTABLE PYROMETER SUBASSEMBLIES

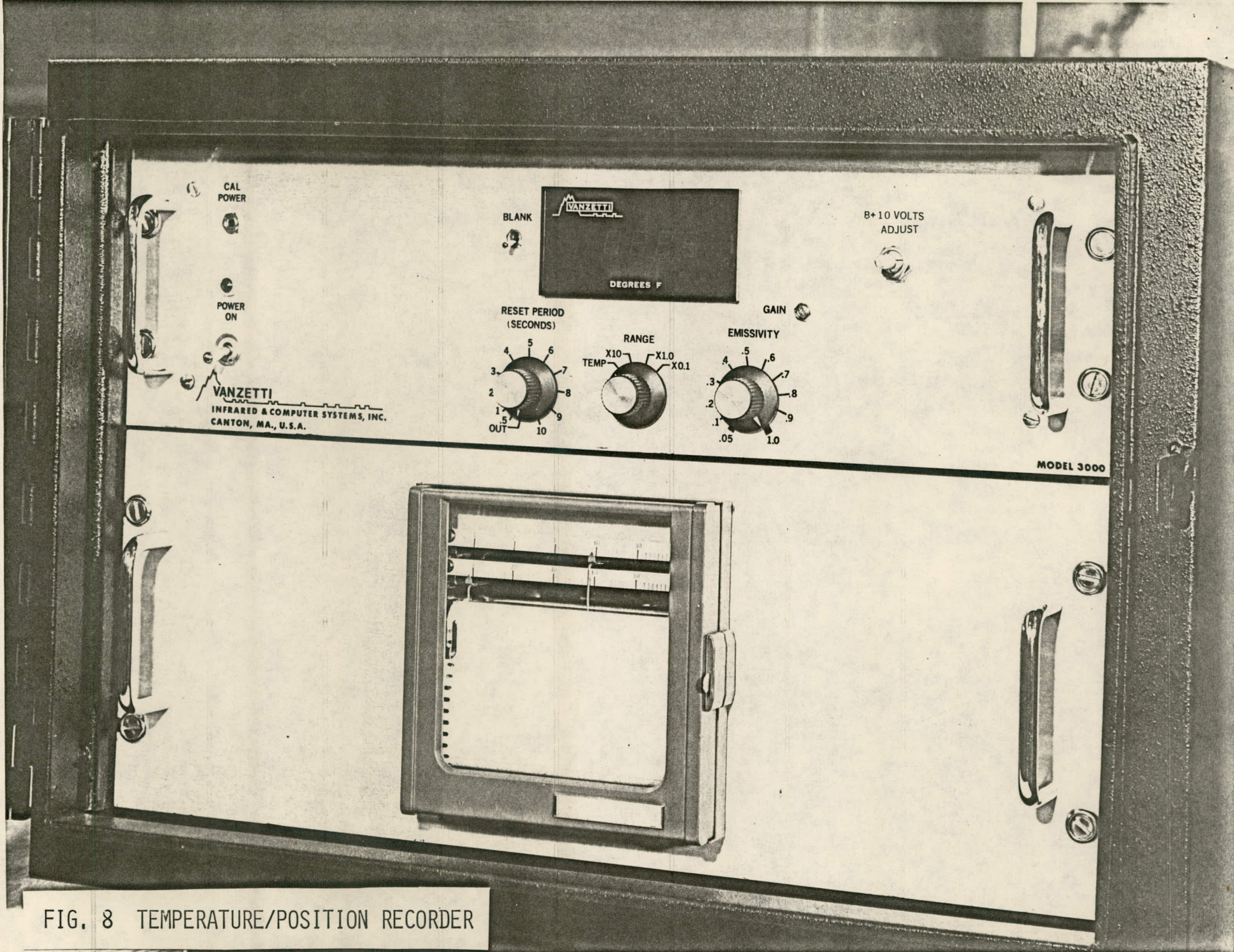


FIG. 8 TEMPERATURE/POSITION RECORDER

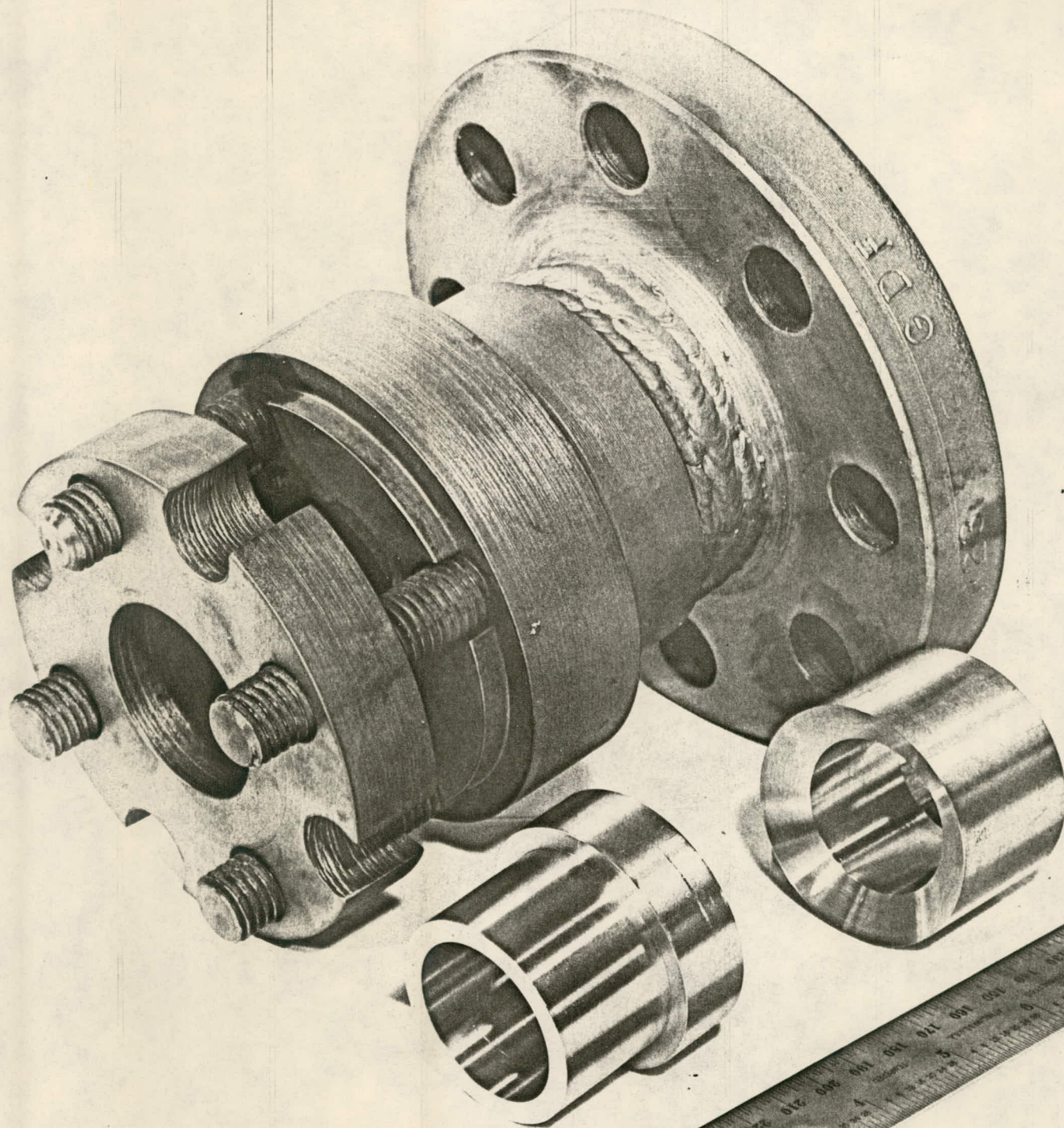


FIG. 9 STUFFING BOX SUBASSEMBLY

FIG. 10 HYDRAULIC PUMP

