

## FOREWORD

This report was prepared by ManLabs, Inc. under USAF Contract No. AF33(616)-6837. This contract was initiated under Project No. 7351, "Metallic Materials", Task No. 735103, "Unique Metallic Materials and Techniques". The work was administered under the direction of the Directorate of Materials and Processes, Aeronautical Systems Division, with Mr. K. L. Kojola acting as project engineer.

This report covers work conducted from 8 March 1962 to 8 December 1962.

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## ABSTRACT

Two separate high-pressure systems have been designed, constructed and tested. Their successful use to pressures of 60 kilobars and temperatures up to 1500°C has been demonstrated. It is anticipated that both of these systems will be utilized for high-pressure heat treatment of large specimens in the current high pressure program.

This technical documentary report has been reviewed and is approved.



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**I. INTRODUCTION**

By the terms of an amendment to the present contract, AF33(616)-6837, the following new Phase VI has been added to Exhibit A.

a. The contractor shall develop, modify and construct a specialized test apparatus of a design comparable to contractor's design of the MIA-II high pressure test apparatus. This apparatus shall be complete with all the necessary attachments for applying the auxiliary instrumentation for performing high pressure-high temperature experiments. The apparatus shall be equipped with tungsten carbide dies and pistons. The apparatus shall be capable of subjecting a 0.70 inch diameter, 1.50 inch long specimen to pressures of 60 kilobars and 1500°C simultaneously.

b. The contractor shall also develop and construct an apparatus based on the principle of the Supported Piston design for exerting ultra high pressures on research samples. This apparatus shall be complete with all the necessary attachments for applying the auxiliary instrumentation for performing high pressure-high temperature experiments. The apparatus shall be capable of subjecting a 1.00 inch diameter, 2.50 inch long specimen to pressures of 60 kilobars and 1500°C simultaneously.

c. The contractor shall conduct necessary research to obtain an indication of both pressure and temperature being exerted on various materials within the specimen chamber of each apparatus.

The above systems have been designed and tested. Data is presented to show the results of these tests.

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Manuscript released by the authors January 1963 for publication as a WADD Technical Report.

## II. APPARATUS

### A. MIA-II System

The MIA-II high pressure apparatus, initially developed in this laboratory, is an enlarged version of the MIA-I system and is capable of processing a specimen having a maximum diameter of 0.70" and a maximum length of 1.50". It is designed to achieve pressures of 60 kilobars at a temperature of 1500°C. These design capabilities are based on the use of tungsten carbide dies and pistons.

#### 1. Design and Construction

The original MIA-II system was equipped with steel dies and pistons and was demonstrated to have a pressure capability of approximately 45 kilobars at a temperature of 1300°C. The pressure limitation was imposed both by the use of steel dies and pistons and by a load limit of 1000 tons. The use of steel dies also precluded temperatures higher than 1300°C when processing a specimen of the dimensions given above. A specimen of smaller diameter than 0.70" could be heated to higher temperature because heavier insulation between the furnace and the die wall could be used. Shown in Fig. 1 is a dimensioned cross-sectional view of the MIA-II system. It will be noted that the cooling jacket is positioned directly against the die. When the system was first built, the cooling jacket was incorporated into the outer safety ring as it is in the MIA-I system. It was found during use that the temperature to which the specimen could be subjected was severely limited because of excessive die heating. The cooling jacket now used is in the form of a split shim which is channeled to permit the free flow of cooling water. The inlet and outlet connections are positioned at the top of the jacket, and designed so as not to interfere with the downward travel of the piston at maximum load. The pistons are also water cooled by means of cooling jackets incorporated into the outer piston safety rings.

Because of the weight of the die restraining ring assembly, three air-operated jacks, permanently positioned on the outer ring, are employed to raise the die to permit positioning of the lower piston and specimen cell, and to lower the die over the specimen cell after it has been placed on the lower piston.

Included in this system is a backing block placed beneath the lower piston, which incorporates a positioning ring for centering the lower piston directly under the die. The heating power supply terminal is located on the backing block.

The system constructed as required by the amendment to the contract, is similar in every respect to the original MIA-II apparatus except for the use of tungsten carbide instead of steel for the die and pistons.



## 2. Test Results

The system was first tested for pressure capability, using the calibration cell shown in Fig. 2. The results of the calibration runs using Bi, Tl and Ba wire are given in Fig. 3 which correlates imposed load with pressure. The pressures used for the pressure-induced transformation of Bi, Tl and Ba are 25, 37 and 59 kilobars respectively.

The load spread for each of the transformations shown in Fig. 3 results from pressure cycling. The highest load for a given pressure is obtained on the initial cycle with lower loads occurring as the same specimen is subjected to further pressure cycling.

Fig. 4 plots temperature versus input power. The thermocouple failed in the test at 1200°C and the temperature is extrapolated to the highest input powers used.

### B. Supported-Piston-II System

During an early phase of the high pressure research which has been conducted by this laboratory, a supported-piston type of high-pressure apparatus was designed, constructed and developed. This type of apparatus is essentially a two-stage simple-piston cylinder system, in which the high-pressure piston is provided with peripheral support along that portion of its length which protrudes above the die. This support is provided by a collar of suitable material which is contained in the low pressure chamber. The collar is compressed by the advance of the low-pressure piston, which is also acting to advance the high-pressure piston against the specimen cell.

The support collar materials which have been used in the earlier, smaller supported-piston systems, have been KCl, RbCl, a stoichiometric mixture of KCl and RbCl and bismuth. The characteristic which all of these materials have in common, is that they undergo negative volume changes at the approximate pressures indicated below:

- |             |             |
|-------------|-------------|
| 1. KCl      | 20 kilobars |
| 2. RbCl     | 8 kilobars  |
| 3. KCl/RbCl | 14 kilobars |
| 4. Bi       | 25 kilobars |

The advantage to be gained from using support collars of materials which undergo this volume change is readily apparent. As the load is imposed on the low-pressure piston it is transmitted over the areas of both the collar and the high-pressure piston. In view of the fact that the latter area is relatively small, it can be seen that only a small fraction of the imposed load is actually transmitted to the specimen cell. However, when the collar undergoes a negative volume change as a function of pressure, a much larger fraction of the imposed load is placed on the high-pressure piston, resulting in considerably greater pressure

on the specimen, with a given load, than would be obtained without the transformation. Specimen pressures as high as 85 kilobars have been reached in the small system using a bismuth support collar.

## 1. Design and Construction of the Supported-Piston-II System

A dimensional cross-section view of the supported-piston system-II is shown in Fig. 5. It is similar in design to the earlier smaller system except that the cooling jacket is placed in immediate contact with the die as in the MIA-II systems in order that maximum specimen temperature can be obtained without overheating the die.

## 2. Test Results

The specimen cell used for the pressure calibration runs is shown in Fig. 6. The results of these calibrations using Bi, Tl and Ba are shown in Fig. 7 which is a plot of imposed load versus pressure. The standard pressures of transformation of these materials was used. Fig. 8 shows a plot of input heating power versus temperature.

### III. APPARATUS SPECIMEN CAPABILITY

The purpose of constructing the two systems described in this report was to develop a capability for subjecting relatively large specimens to high pressure. With the exception of the cell designed for a simple piston-cylinder device having a pressure limitation of 20 kilobars, the largest specimen cell used in this laboratory prior to the development of the two systems described herein, measured 0.50 inch diameter and 1.00 inch length. The largest possible specimen that could be used in this cell was 0.250 inch diameter and 0.300 inch length. If it was necessary to subject the specimen to only purely hydrostatic pressure, the specimen size was further reduced to 0.125 inch diameter and 0.125 inch length.

As earlier experimental work had indicated that the use of pressure during the heat treatment of certain materials appeared to produce beneficial effects, the logical next step was to perform direct mechanical property measurements on pressure-treated materials. The development of the MIA-II and the Supported-Piston-II systems arose from the need for specimens on which these measurements could be made.

#### A. MIA-II System

This system is capable of processing a solid bar or a powder compact specimen measuring 0.70 inch diameter and 1.50 inch length at pressures up to 60 kilobars at a maximum temperature of 1500°C. Room temperature processing in this pressure range permits the use of a specimen of the same length, but with a diameter of 0.875 inch. This increased diameter is gained by the elimination of the furnace and the inner insulating sleeve. If it is necessary to subject the specimen to only purely hydrostatic pressure, the specimen size is reduced to 0.25 inch diameter and 1.00 inch length.

#### B. Supported Piston-II System

For processing solid bar or powder compact specimens up to pressures of 60 kilobars at temperatures to 1500°C, the maximum specimen size of this system is 1.00 inch diameter and 3.00 inch length. Room temperature pressure operation permits the use of a specimen 1.25 inch diameter and 3.75 inch length. When it is necessary to operate at conditions of purely hydrostatic pressure, the specimen size is reduced to 0.50 inch diameter and 2.00 inch length at elevated temperatures, and 0.75 inch diameter and 2.50 inch length at room temperature.

#### C. Factors Limiting Specimen Size

Although it is theoretically possible to fill the entire high pressure chambers of these systems with a specimen, experimental considerations strongly mitigate against so doing. For example, the MIA-II system employs conical gaskets which, when under pressure, act both to retain the pressure developed in the high pressure chamber, and also to distribute the imposed pressure load over the surface of the piston in order to prevent piston failure at high pressures. If the entire pressure chamber of this system is filled with a solid

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and relatively incompressible specimen, the load on the pistons would become excessive, resulting in piston failure before the gaskets could be compressed to support the pistons. For this reason, the specimen is always encased in pyrophyllite or similar material which can be sufficiently compressed in the initial stages of load imposition to permit adequate gasket forming.

This specimen encasing technique is required, not only because of pressure requirements, but also to provide thermal and electrical insulation when needed. For this reason it can be seen that room temperature pressure processing permits the use of a larger specimen than can be employed for elevated temperature operation.

A further limitation is imposed on specimen size when it is necessary to subject the specimen to only purely hydrostatic pressure. The solid pressure media normally used (pyrophyllite, talc, boron nitride, etc.) do not transmit the imposed load to the specimen uniformly. For this reason, a medium which remains liquid at the conditions of pressure and temperature employed must be used. This is accomplished by inserting the specimen in a stainless steel capsule filled with isopentane or similar fluid. The capsule can only be as large as the maximum dimensions indicated above for specimen sizes under various conditions. The specimen must be considerably smaller than the capsule for two reasons; 1) the compressibility of the liquid is much greater than that of a comparable solid medium and, 2) the capsule, surrounded by a solid medium, is normally badly deformed when subjected to high pressure. If the specimen is too large, the collapse of the capsule imposes a non-uniform load on the specimen resulting in deformation or breakage. The reduced sizes of specimens used for purely hydrostatic loading naturally follow from the limitation imposed by the above described conditions.

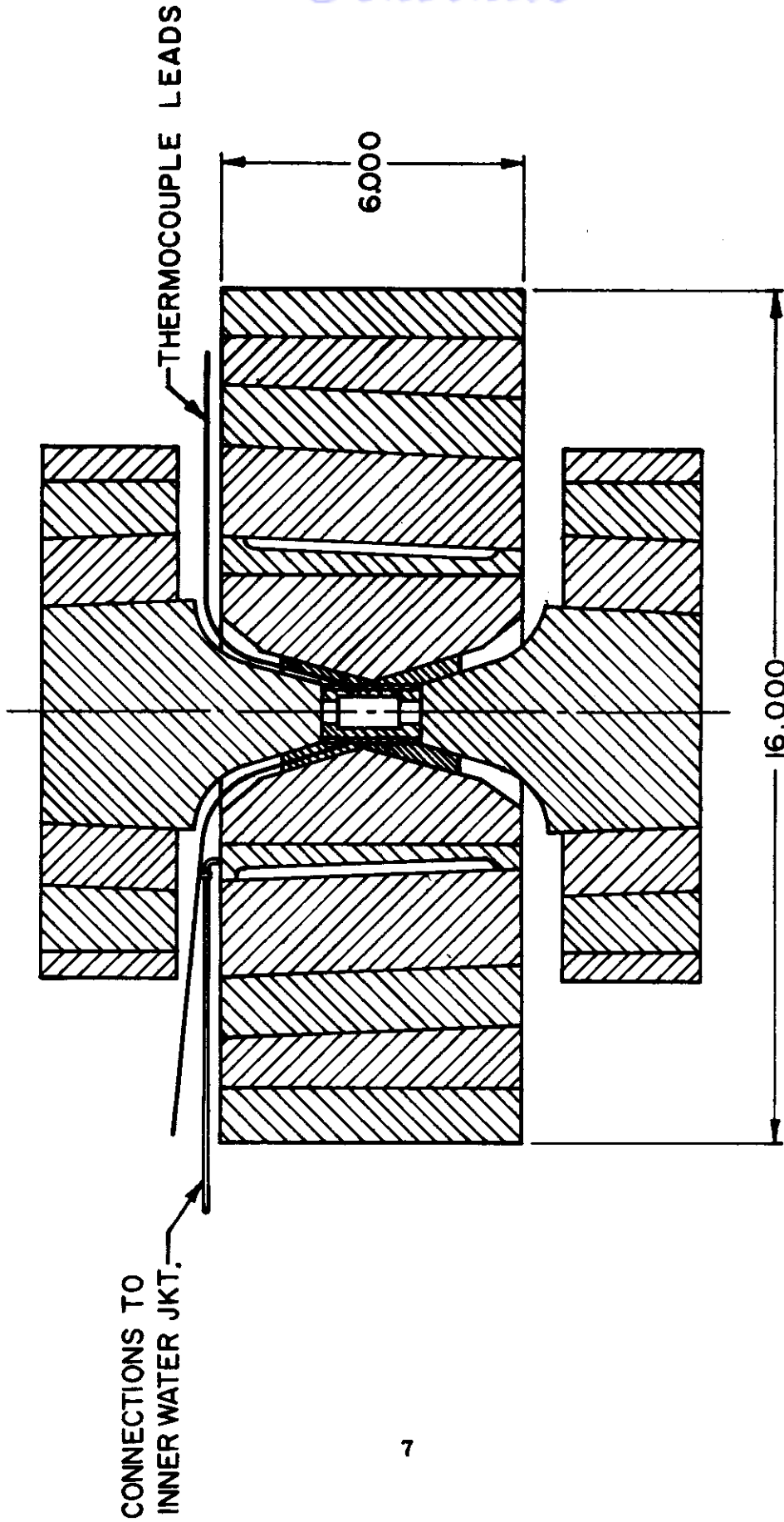
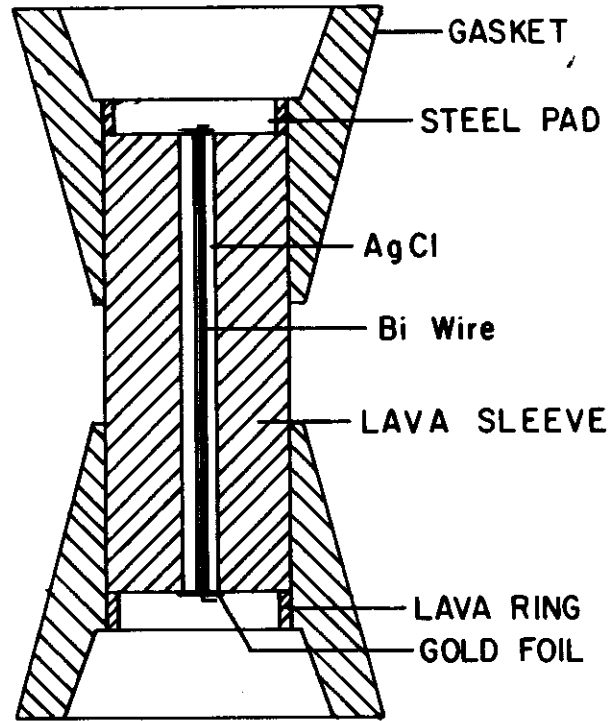


Fig. 1 - MIA-II High-Pressure System



**Fig. 2 - MIA-II calibration cell**

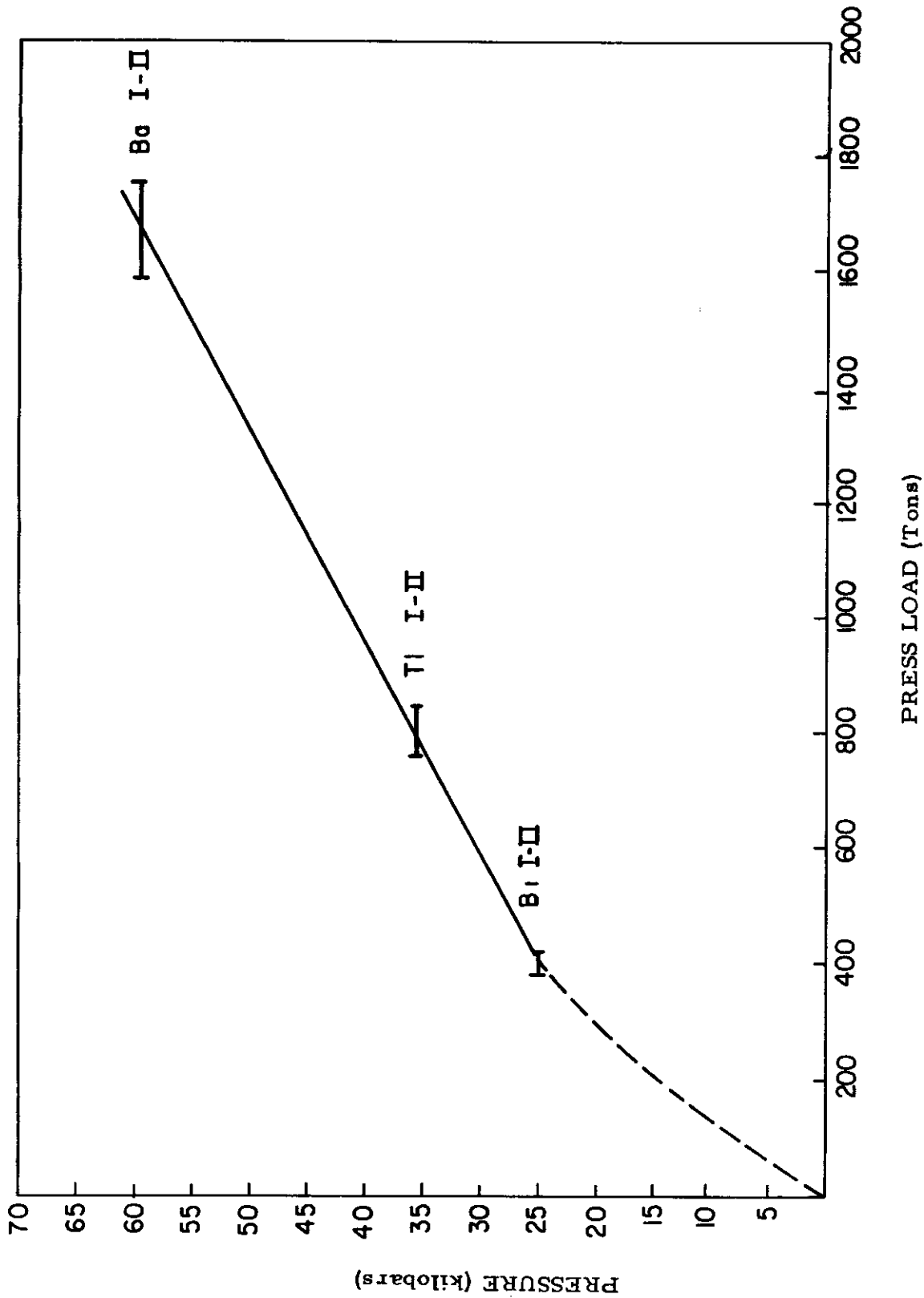


Fig. 3 - Pressure calibration of the MIA-II high pressure system

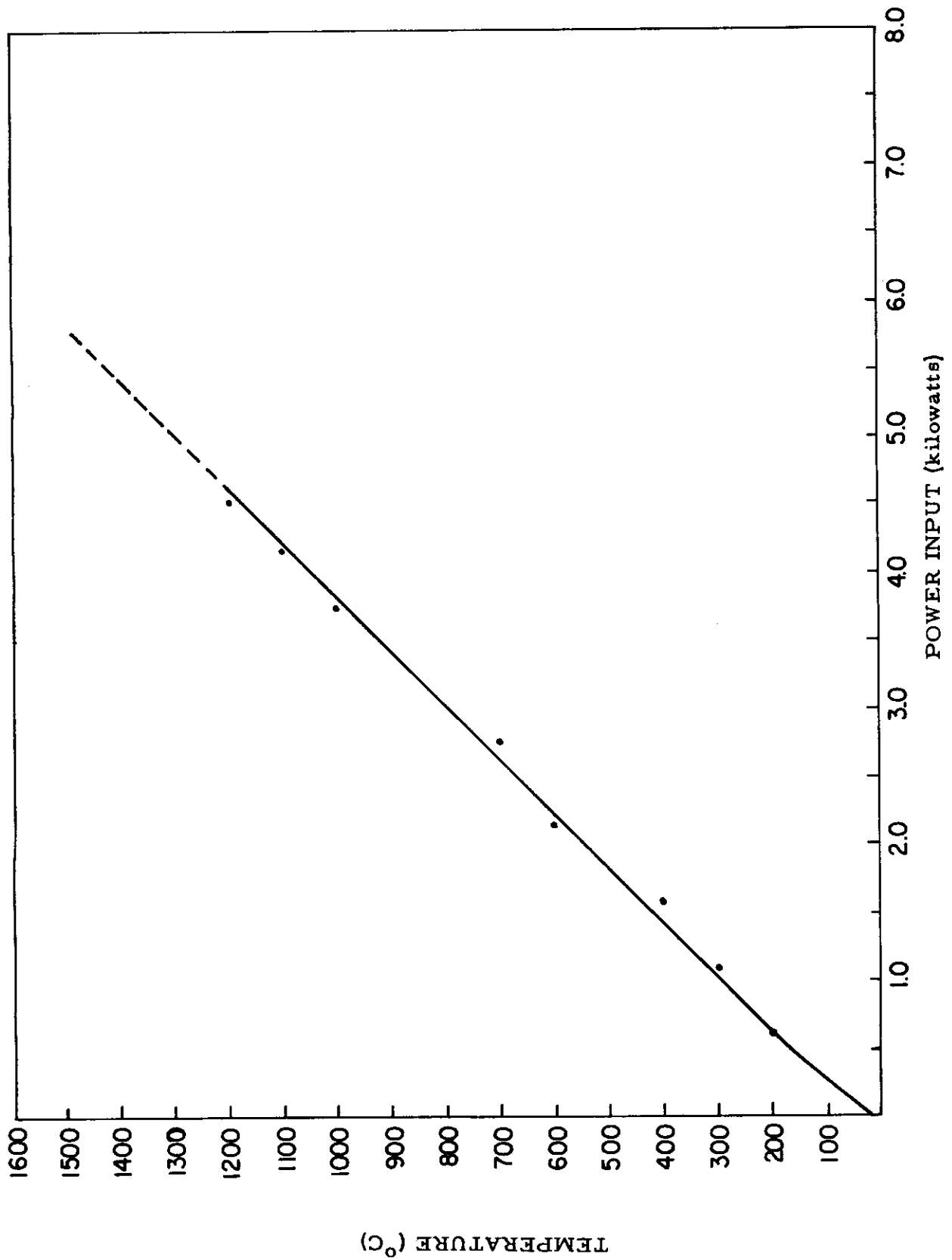


Fig. 4 - Temperature calibration MIA-II system



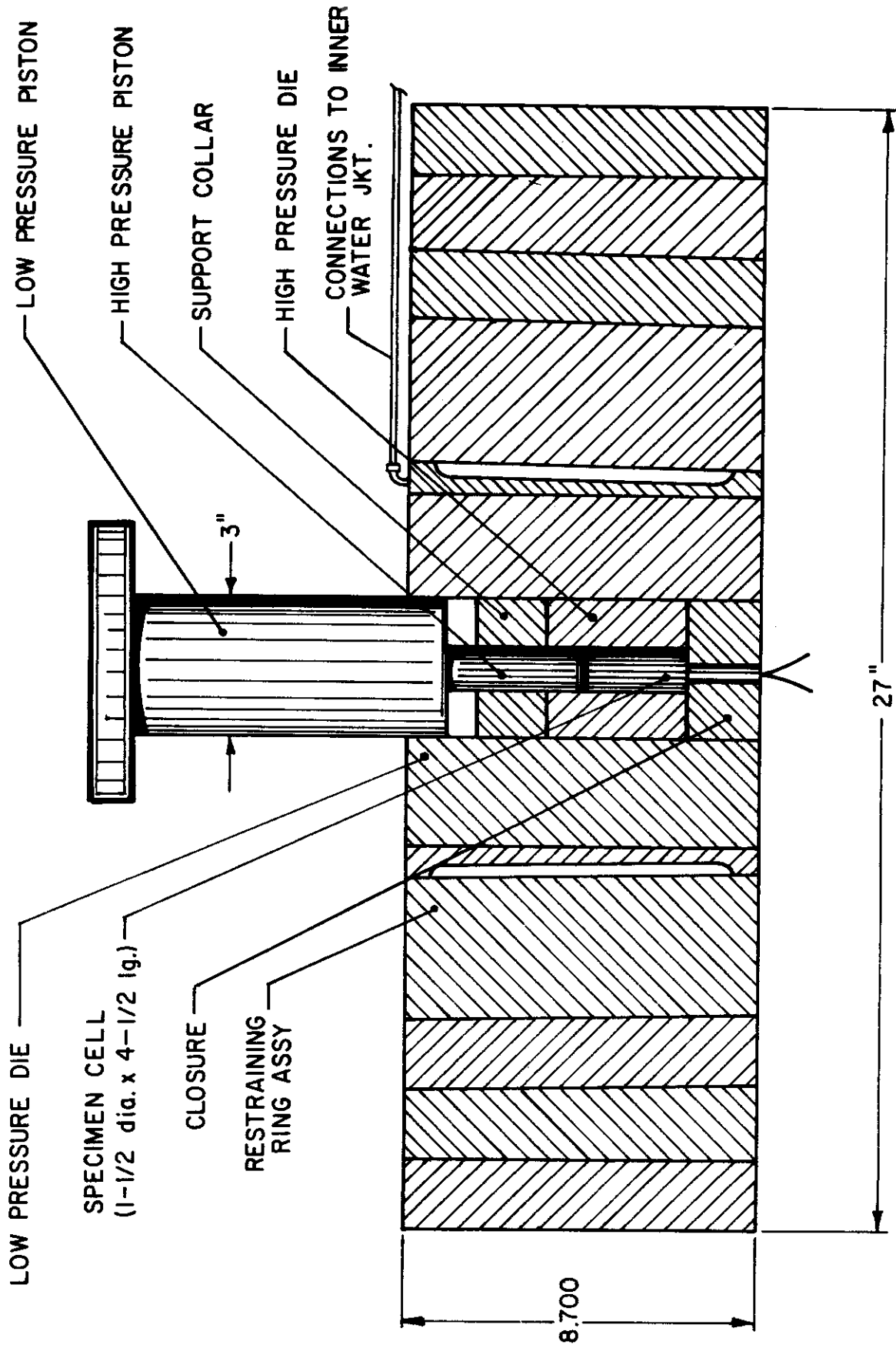


Fig. 5 - Supported-piston-II high pressure system

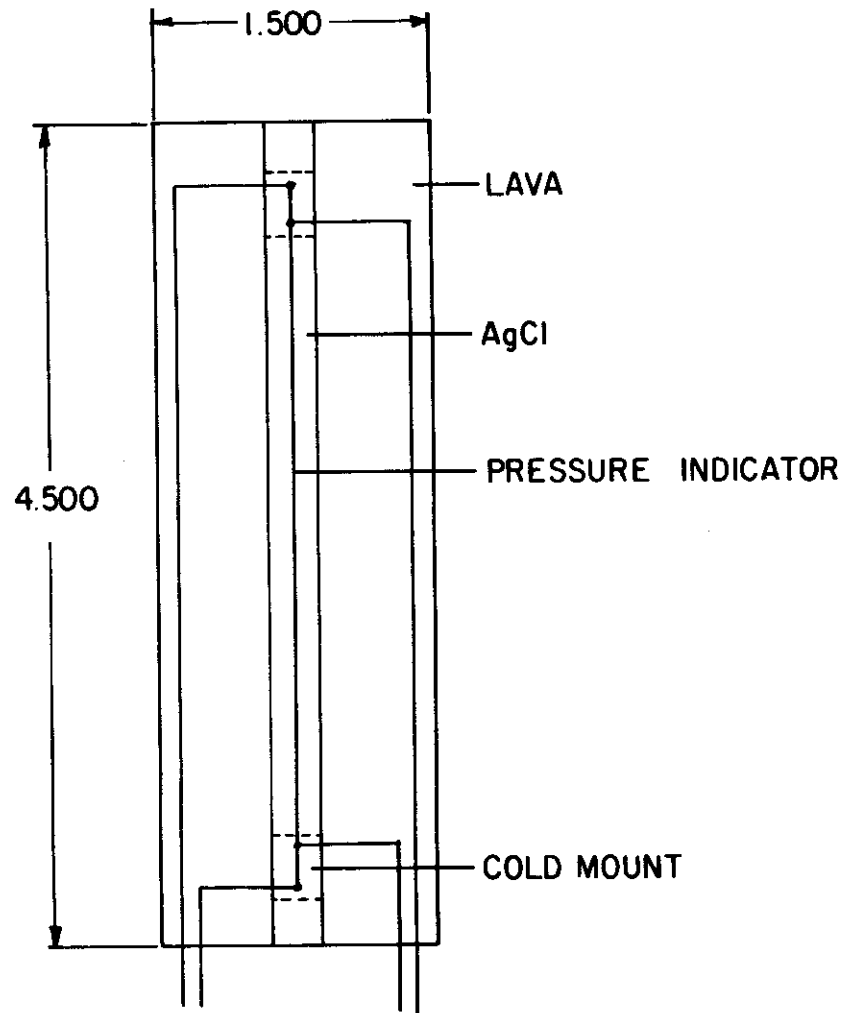


Fig. 6 - Supported piston system-II specimen cell

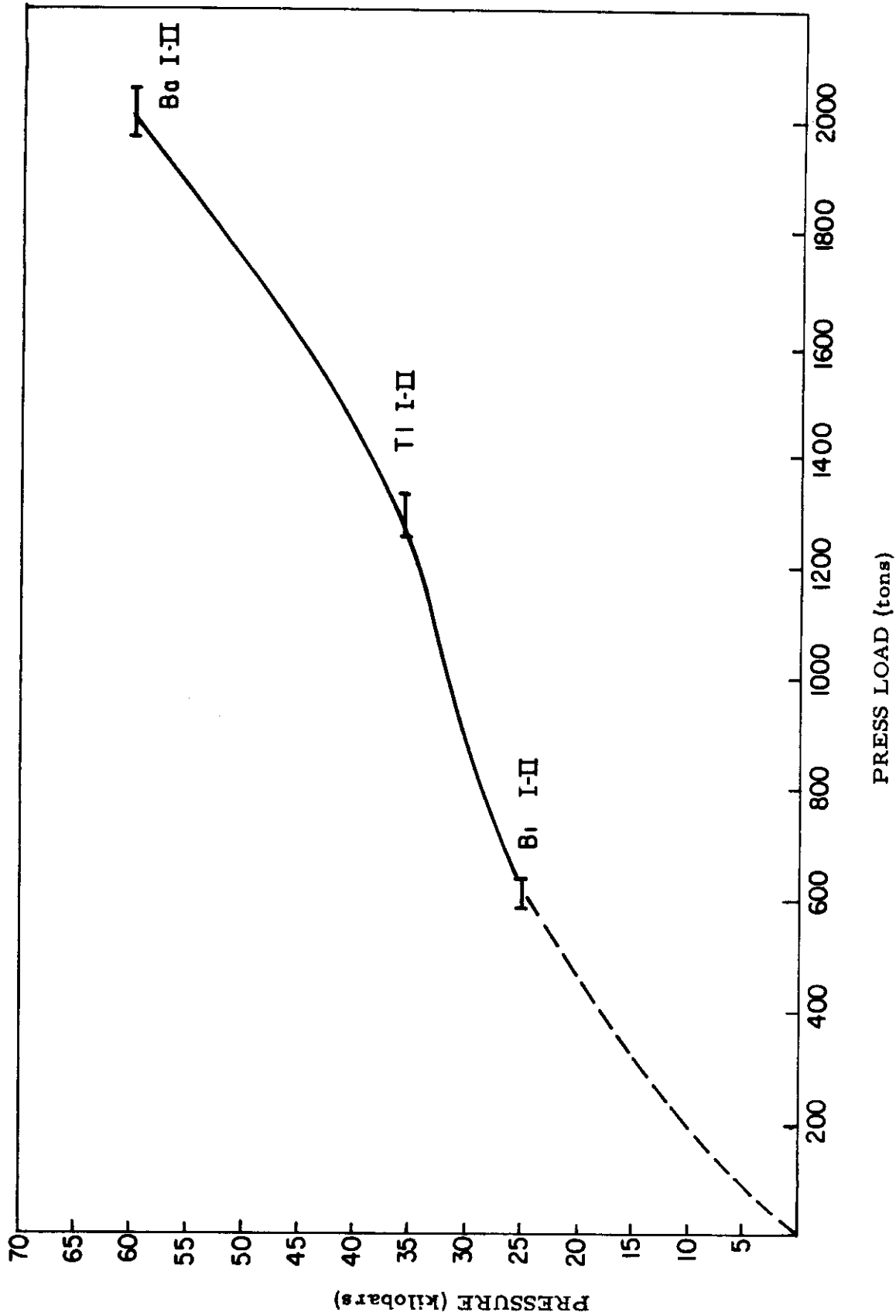


Fig. 7 - Pressure calibration of the supported-piston system II

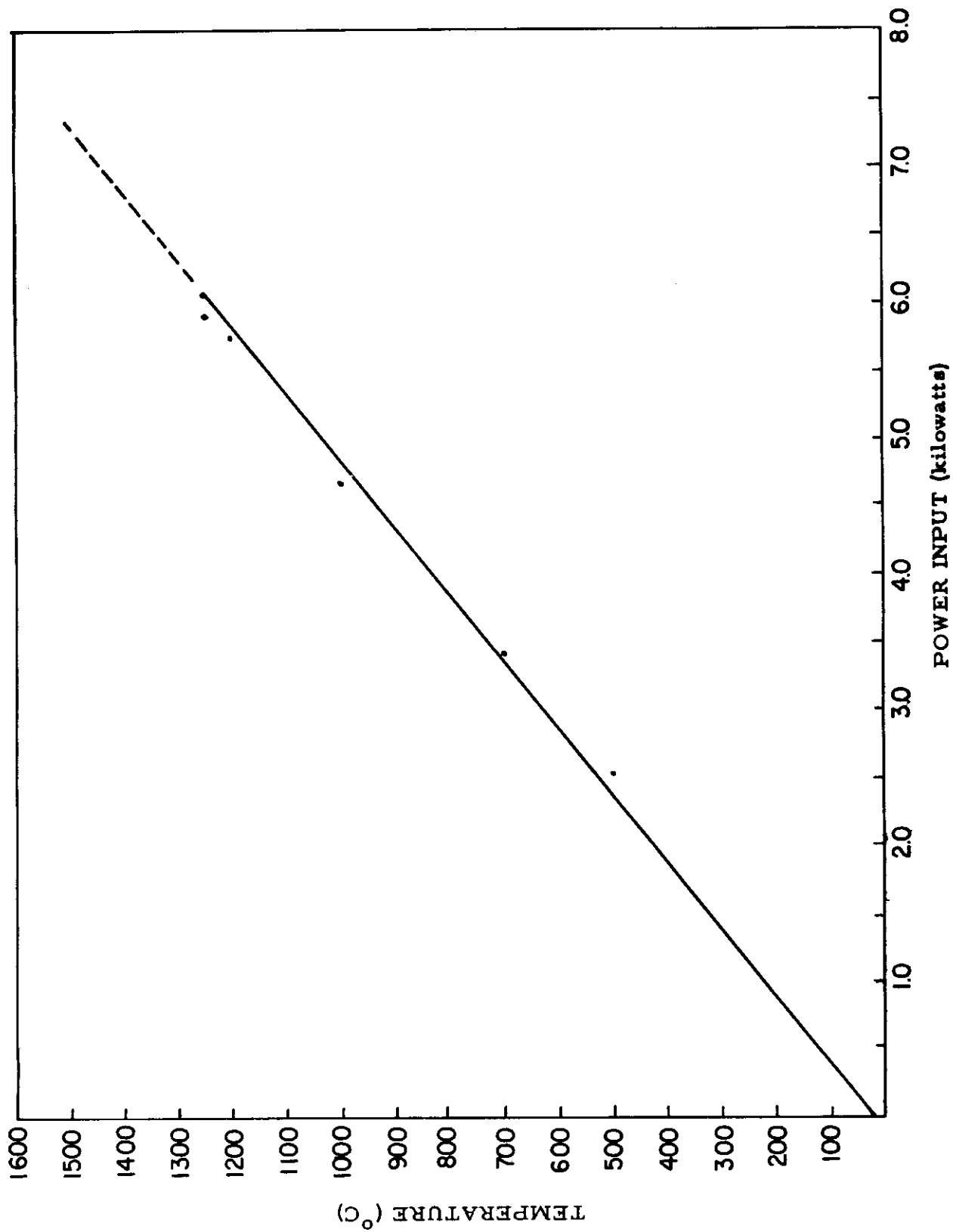


Fig. 8 - Temperature calibration supported-piston system-II