AD NUMBER

AD833480

NEW LIMITATION CHANGE

TO

Approved for public release, distribution unlimited

FROM

Distribution: Further dissemination only as directed by Air Force Materials Laboratory, Attn: MANP, Wright-Patterson AFB, OH 45433, Apr 1968, or higher DoD authority.

AUTHORITY

AFML ltr, 7 Dec 1972

AFML-TR-67-426

ADO 833480

NEW THERMOSTAT FOR HIGH TEMPERATURE LIGHT SCATTERING MEASUREMENTS

MATATIAHU GEHATIA

WILLIAM E. GIBBS

TECHNICAL REPORT AFML-TR-67-426

APRIL 1968

AIR FORCE MATERIALS LABORATORY
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

NOTICE

100 - May 1968 - CO455 - 35-746

AFML-TR-67-426

NEW THERMOSTAT FOR HIGH TEMPERATURE LIGHT SCATTERING MEASUREMENTS

MATATIAHU GEHATIA

WILLIAM E. GIBBS

FOREWORD

This report was prepared by the Polymer Branch of the Nonmetallic Materials Division. The work was initiated under Project No. 7342, "Fundamental Research on Macromolecular Materials and Lubrication Phenomena," Task No. 734203, "Fundamental Principles Determining the Behavior of Macromolecules," and the research was conducted with Dr. M. T. Gehatia acting as project scientist. The work was administered under the direction of the Air Force Materials Laboratory, Directorate of Laboratories, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio.

This report covers research conducted from January 1964 to October 1967. The manuscript was released by the author in December 1967 for publication as a technical report.

This technical report has been reviewed and is approved.

William E. Gibbs

WILLIAM E. GIBBS
Chief, Polymer Branch
Nonmetallic Materials Division
Air Force Materials Laboratory

ABSTRACT

A new thermostat for high temperature light scattering measurements has been constructed. It is composed of the following major parts: (1.) metal housing; (2.) glass container with fused optical windows to contain the bath liquid; and (3.) cell adapter which also serves as a heat exchanger.

Previously designed thermostats consisted only of a metal cylinder containing a constant temperature liquid and a cell with the sample under investigation. The optical windows were glued or cemented to the wall. Such a thermostat cannot be used for experiments at high temperatures because no known chemical adhesive can resist hot corrosive liquids used as constant temperature liquids. Therefore, in the new thermostat the liquid container was constructed as a separate part, fabricated entirely from glass without any glue or cement, while other parts were fabricated from metal. This new assembly can be used for light scattering measurements up to 350°C.

This abstract may be further distributed by any holder only with specific prior approval of the Air Force Materials Laboratory (MANP), Wright-Patterson Air Force Base, Ohio 45433.

TABLE OF CONTENTS

ILLUSTRATIONS

INTRODUCTION

A light scattering apparatus is primarily used to measure the intensity distribution of light scattered from dilute polymer solutions, the ultimate aim being to determine the conformations and sizes of the molecules in solution.

A special precision light scattering apparatus has been constructed in this laboratory. The optical design is similar in principle to that described by McIntyre and Doderer (Reference 1). The mechanical arrangement, described by Katz (Reference 2) was initially constructed by the Mellon Institute (Reference 3) and later modified by this laboratory (Figures 1 and 2). The apparatus is comprised of:

1. Two light sources located on separate benches (Figure 3): (a) A mercury vapor lamp mounted on the main optical bench, and (b) A laser mounted on an additional, upper optical bench.

2. An optical system which highly collimates the monochromatic beam coming from the mercury lamp and focuses it in the light scattering cell. Another collimating lens which can be used to deflect the laser beam coming from the upper bench toward the sample cell (Figure 4) is placed on an adjustable turret.

3. Optics which deflect the laser beam toward the prism placed on the turret.

4. A chopper for periodically interrupting the light beam.

5. A beam splitter, which deflects a fraction of the primary beam to a monitoring photomultiplier. This beam serves as a reference which can be compared to the scattered light.

6. A thermostat containing solution cell and constant temperature liquid, which is the main subject of this report.

7. A detector revolving precisely around the vertical axis of the solution cell and focused on its center.

8. Electrical components which compare signals generated by the monitoring and the detecting photomultipliers.

The thermostat was originally a cylindrical metal housing. The sample cell was placed in the center of the housing and immersed in a constant temperature liquid. The optical windows were cemented to holes cut in the housing wall. Such a thermostat could not be used for measurements at high temperatures because all chemical adhesives tried were deleteriously affected by the hot bath fluids. At elevated temperatures, the adhesives became soft and caused optical disalignment of the windows. With further increase of temperature, the thermostat developed leaks.

Therefore, another high temperature thermostat has been constructed. It is composed of the following components: Glass container, metal housing, cell adapter, and lid (Figures 5-8). This thermostat does not require any chemical adhesives, and it can be used to temperatures of 350°C.

A detailed description of the new thermostat will be given in the following discussion.

THE GLASS CONTAINER

The body of the glass container (Figures 9 and 10) was built from a 5-inch-diameter Pyrex jar. Two 1-inch-diameter glass tubes were symmetrically fused horizontally on opposite sides of this jar. A circular window was carefully fused to each side tube. The following conditions were necessary:

1. The windows had to retain their optical homogeneity.

2. The windows had to be placed such that their faces are normal to a horizontal line which would represent the light beam.

Unfortunately, a glassblower cannot always fulfill the second condition with adequate precision. Therefore, several such jars were fabricated, and proper ones selected. The most precisely aligned window was placed to face the incident beam. Finally, a glass drain was also fused near the bottom of the jar to facilitate draining the bath fluid. It was equipped with a Teflon Swagelock fitting.

THE HOUSING

The housing (Figures 7 and 8) was fabricated from brass and was anodized black. Since this unit serves as a jacket for the glass container, it is similarly constructed with a vertical cylinder and two horizontal symmetrical side tubes. A horizontal slot covering about one-half the thermostat circumference has been milled in the wall between the side tubes. The housing can be dismantled into two parts along a horizontal plane intersecting the side tubes along their diameters (one version), or along a vertical plane perpendicularly intersecting the common axis of these side tubes (another version) to facilitate insertion of the glass container. Flanges with threaded holes along the matching edges of both parts are provided which can be separated or joined by screws (Figure 11). An additional hole near the bottom of the housing accommodates the glass drain tube. A layer of thermal insulation is placed between the housing and the glass container. Two more holes are drilled near the upper edge of the housing for fittings which allow circulating liquid to flow to and from the adapter (see details in "The Adapter").

The upper edge of the housing is provided with pins which support the cell adapter (see details below).

There are more accessories for optical alignment (see details in "The Optical Alignment"), such as replaceable vertical slits in the housing side tubes.

THE CELL ADAPTER

The cell adapter (Figures 12 and 13) is constructed from two brass cylinders. The outer dimensions of the smaller cylinder match the inner dimensions of the larger one. A system of connecting grooves was milled out of the inner surface of the larger (outer) cylinder (see pattern in Figure 14). The smaller cylinder was then inserted in the larger one and silver soldered. The cell adapter was also anodized black. The thermostatted liquid circulates under pressure through this system of channels, thereby, also making the cell adapter an efficient heat exchanger. A horizontal slot was milled out of the wall to match the previously described horizontal slot of the housing.

The cell adapter is provided with a flange around the outside upper edge. This flange rests rigidly on the housing's upper edge and its position is fixed by pins protruding upward from the housing. The main body of the adapter is suspended, being supported by the glass container. This system gives good thermal control of the sample cell inserted into the adapter. To increase the efficiency of thermal equilibrium around the sample cell, holes were drilled in the bottom of the adapter (Figure 15), to permit free movement of the liquid to and from the glass container.

A sample cell carrying base is located on the bottom of the adapter. It can be turned around the axis of symmetry of the cylindrical adapter, and leveled by adjusting three screws on its periphery, which press upon the bottom of the adapter. By this means the sample cell can be optically aligned.

THE LID

The lid (Figures 16 and 17) was fabricated from brass and covered with a layer of thermal insulation. It was anodized or painted black. It rests rigidly on the upper edge of the cell adapter and is fixed by screws. The lid carries two vertical tubes to prevent the thermometer and stirrer from reflecting light within the thermostat. An opening in the middle of the lid is provided for inserting or removing the cell during the experiment without having to dismantle any other part of the thermostat. This opening is covered by an additional smaller lid.

OPTICAL ALIGNMENT

The optical arrangement of the thermostat assembly must provide for entrance of the incident beam, emergence of the transmitted beam, and evaluation of the light scattered from esentially 0 to 180 degrees of the incident beam in a plane horizontal to the beam path. Hence, provisions were made for the entrance and exit windows and for a horizontal slot covering about one-half the thermostat circumference, through which a photomultiplier can scan the scattered light in the plane of the incident beam. All parts of the assembly must be optically aligned.

The optical alignment of the housing can be accomplished in the following way: At first, the entire base carrying this housing is shifted across the bench to make the beam (a ray of light) pass through centers of vertical slits inserted in the side tubes of the housing. Then the base; being composed of two parts, with a threaded screw between them, is turned until the housing reaches approximately the proper height and angle. The fine adjustment of height and leveling is accomplished by adjusting a set of three screws fixed to the base which press against corresponding grooves milled out in the bottom of the housing.

The glass container rests upon a disc supported by another set of three screws fixed to the bottom of the housing (Figure 18). They control the height and leveling of the container. The container is now carefully moved on its disc to make its vertical axis match the corresponding axis of the housing. This position is then fixed by screws with rubber ends inserted in the jacket wall. Finally, the angular position of the container is controlled by adjusting screws in the housing's side tubes.

Two small horizontal tubes are screwed from inside into threads milled out in the circular openings of the adapter. The front tube contains a slit to shield the cell from stray light. In the rear tube an absorbing glass is placed at an angle which attenuates reflection of the primary beam and deflects it upwards. The cell can be also aligned by adjusting screws inserted into the base.

This thermostat is capable of operation up to 350°C. Above this limit, the silver soldered joints may melt.

REFERENCES

1. D. McIntyre and M. C. Doderer, J. Res. Nat. Bur. Std., 62, 159 (1959).
2. S. Katz, private communication.
3. G. C. Berry, et al., ASD Technical Report 61-22, April 1961.

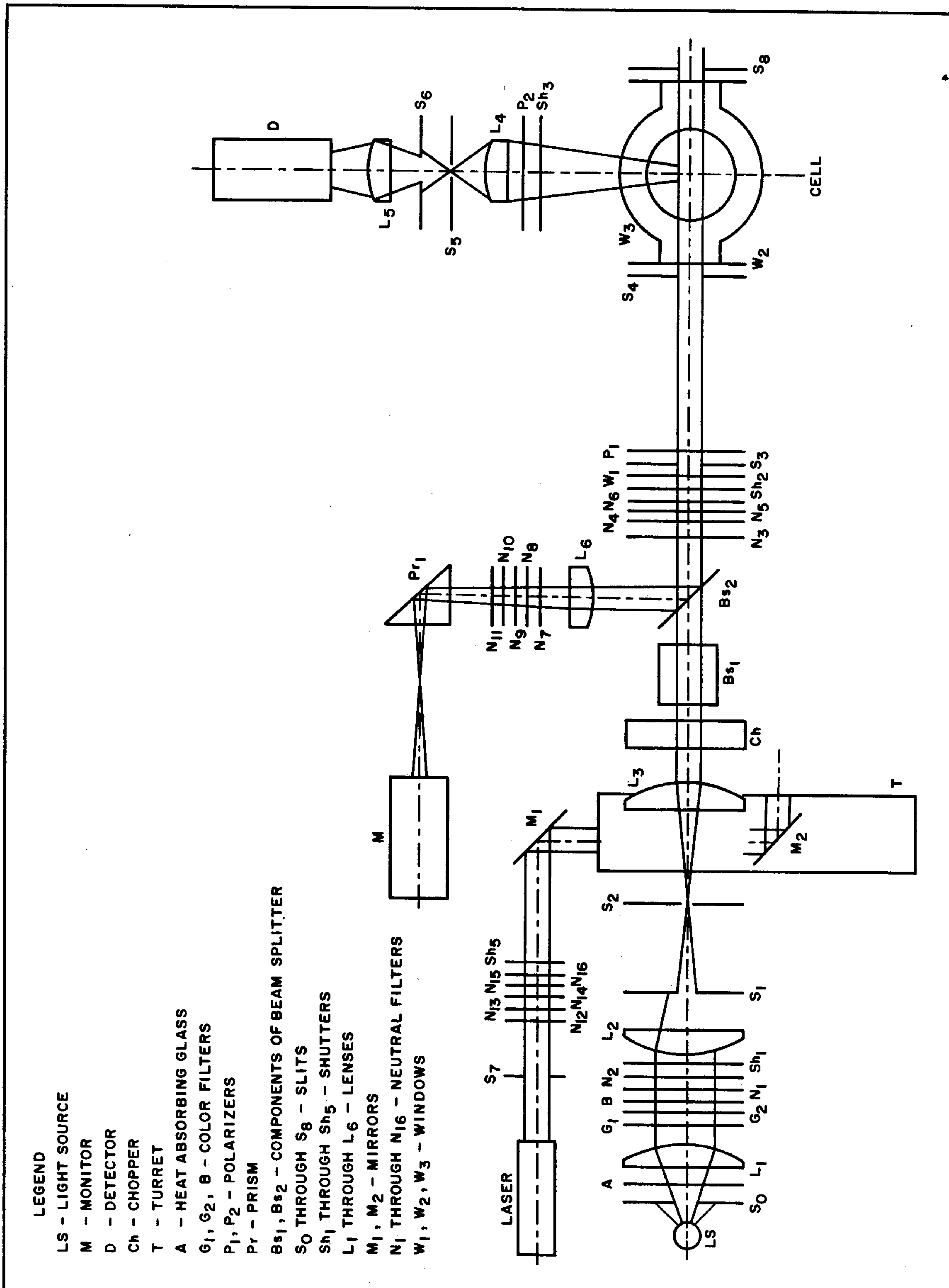

Figure 1. Optical System of Light Scattering Apparatus

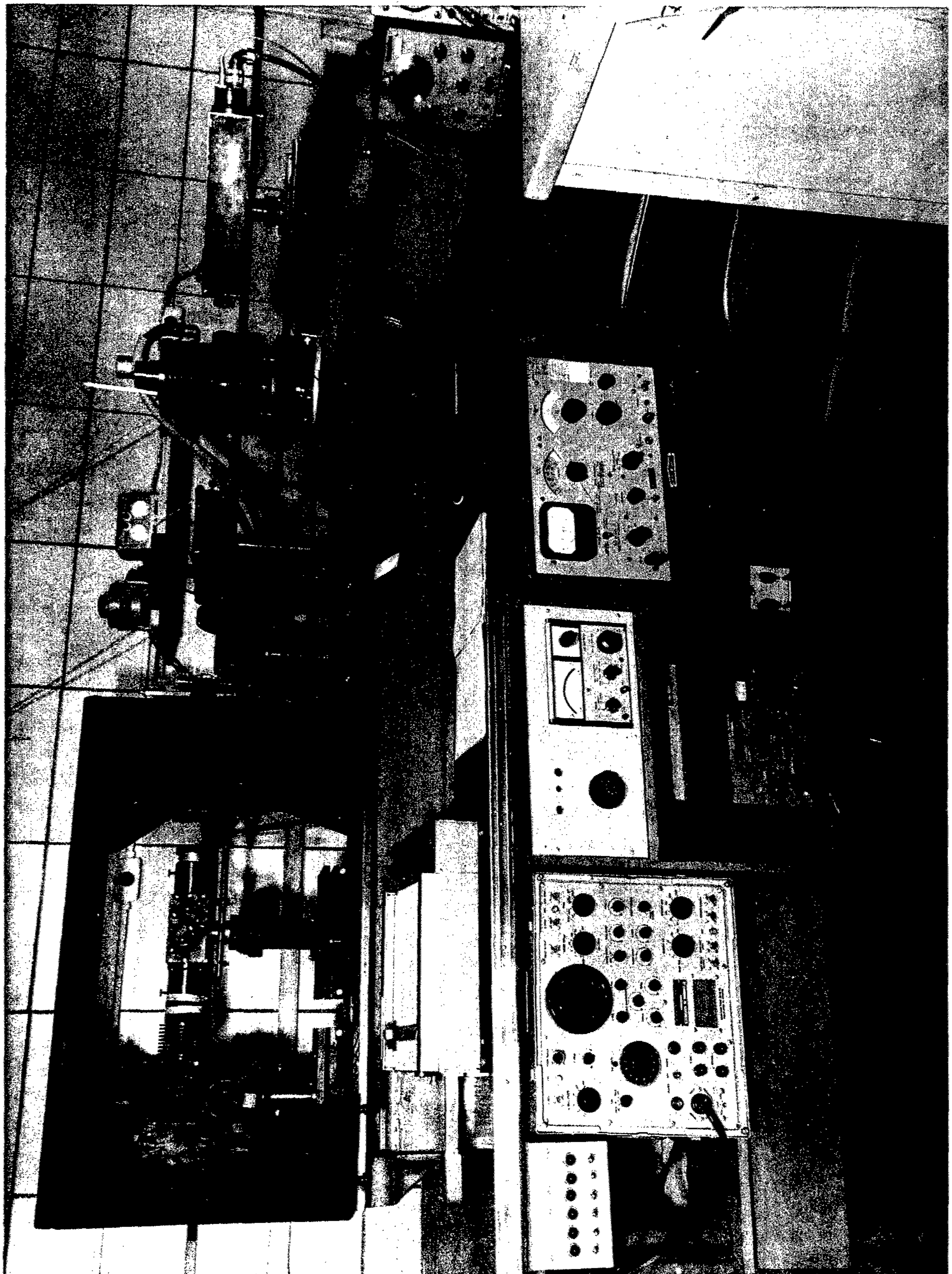

Figure 2. General View of Light Scattering Apparatus (Without Laser)

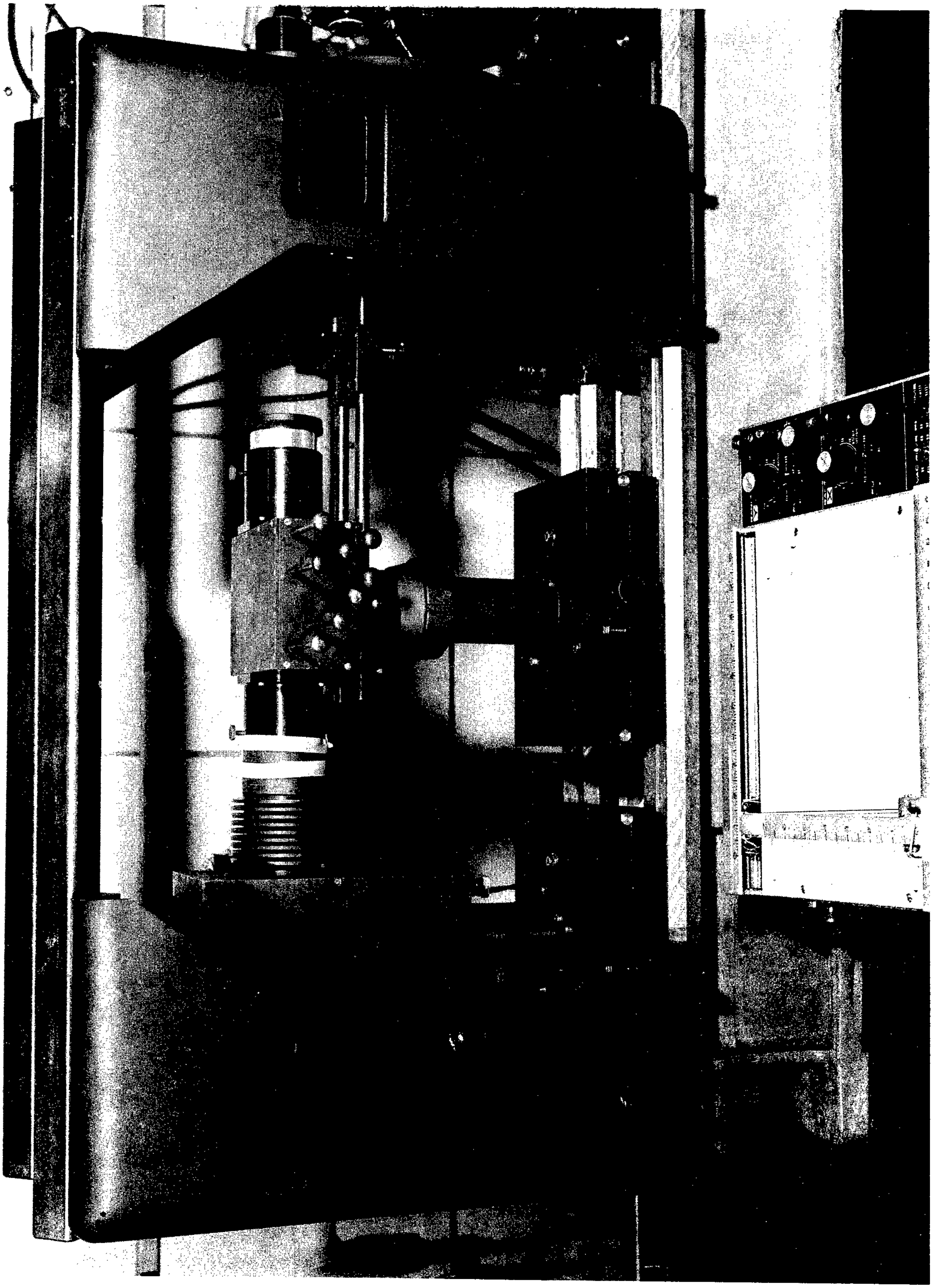

Figure 3. Conventional Light Source and Collimator

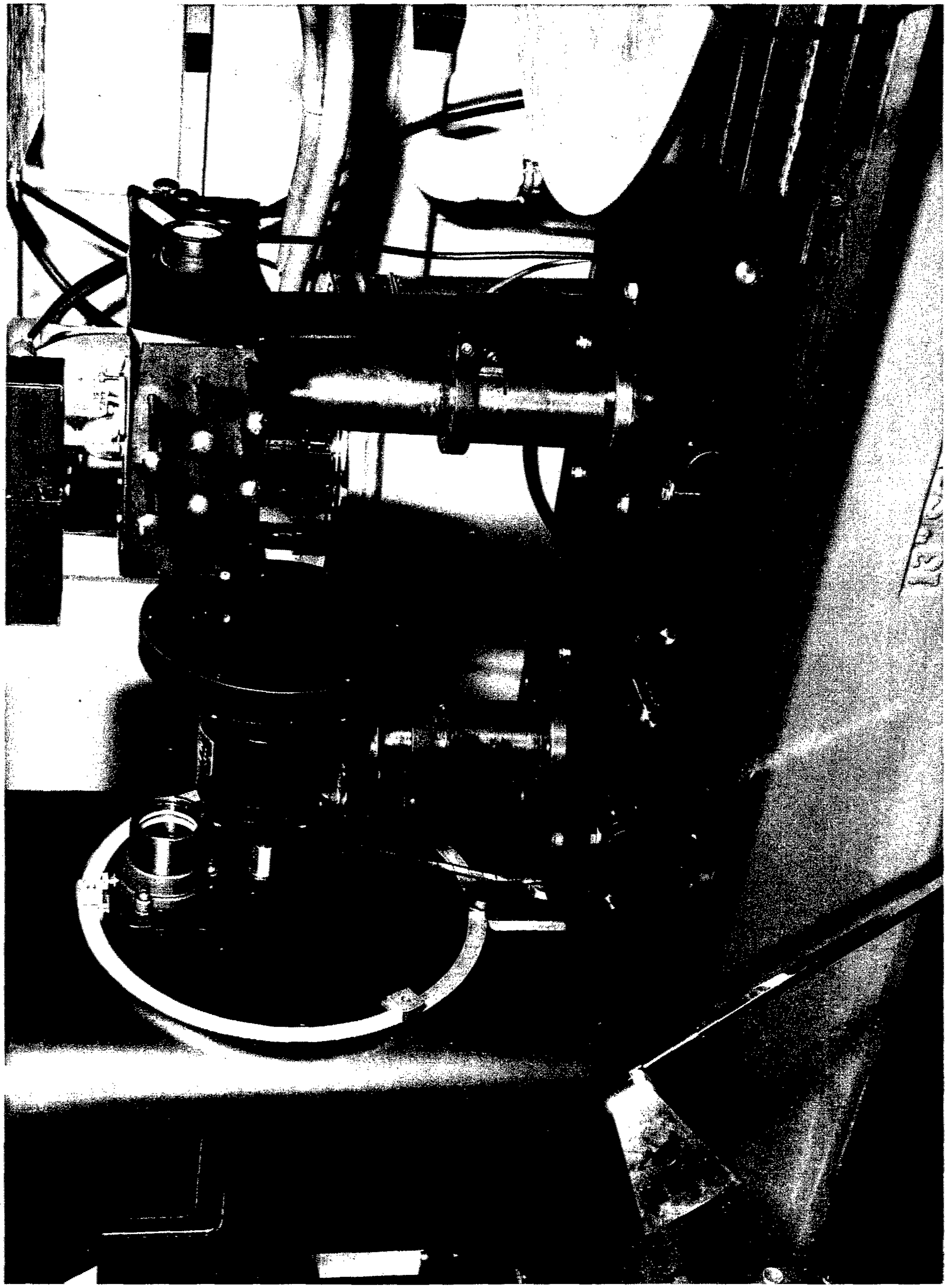

Figure 4. Turret, Chopper, and Beam Splitter

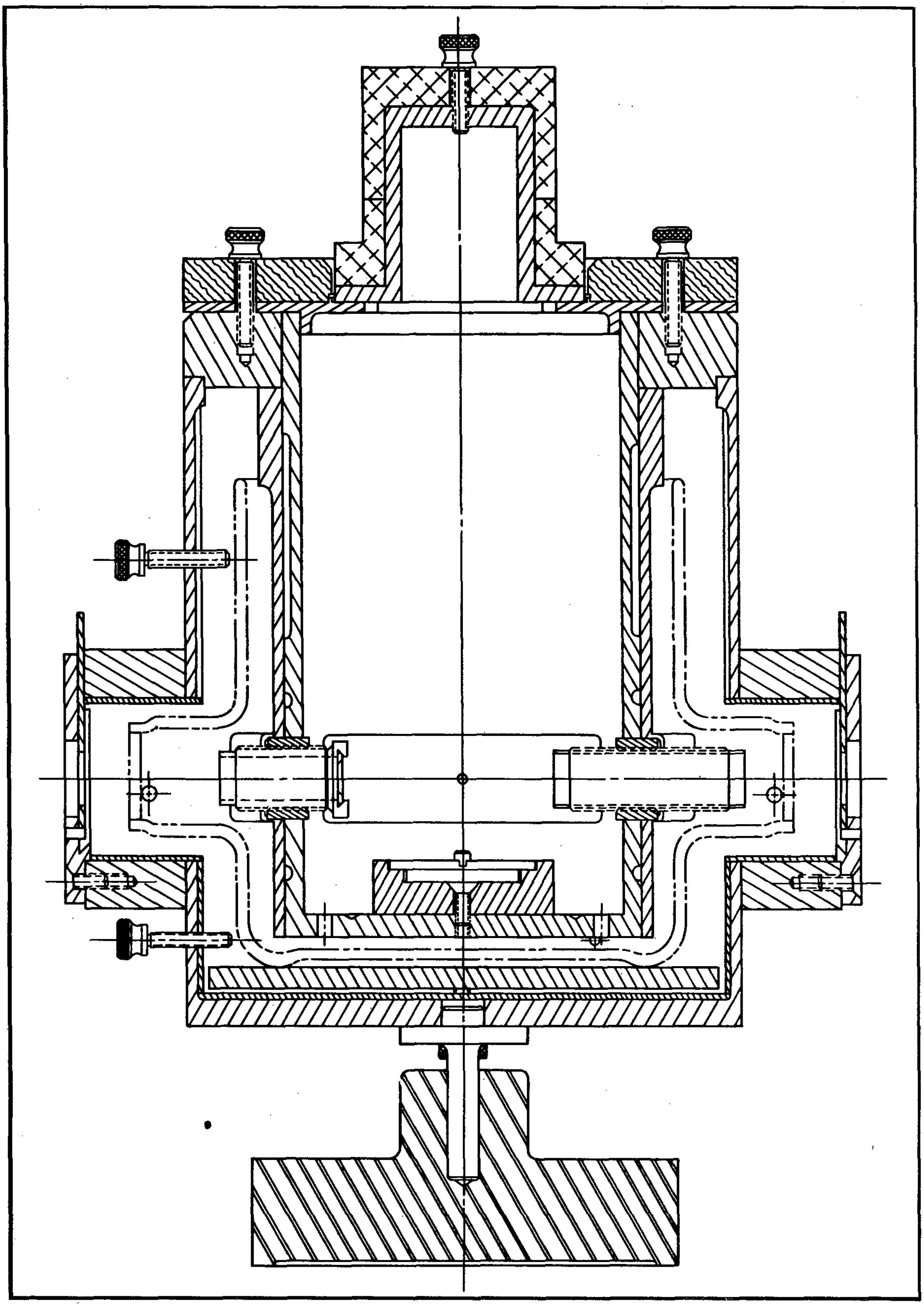

Figure 5. Longitudinal Cross Section of the New Thermostat

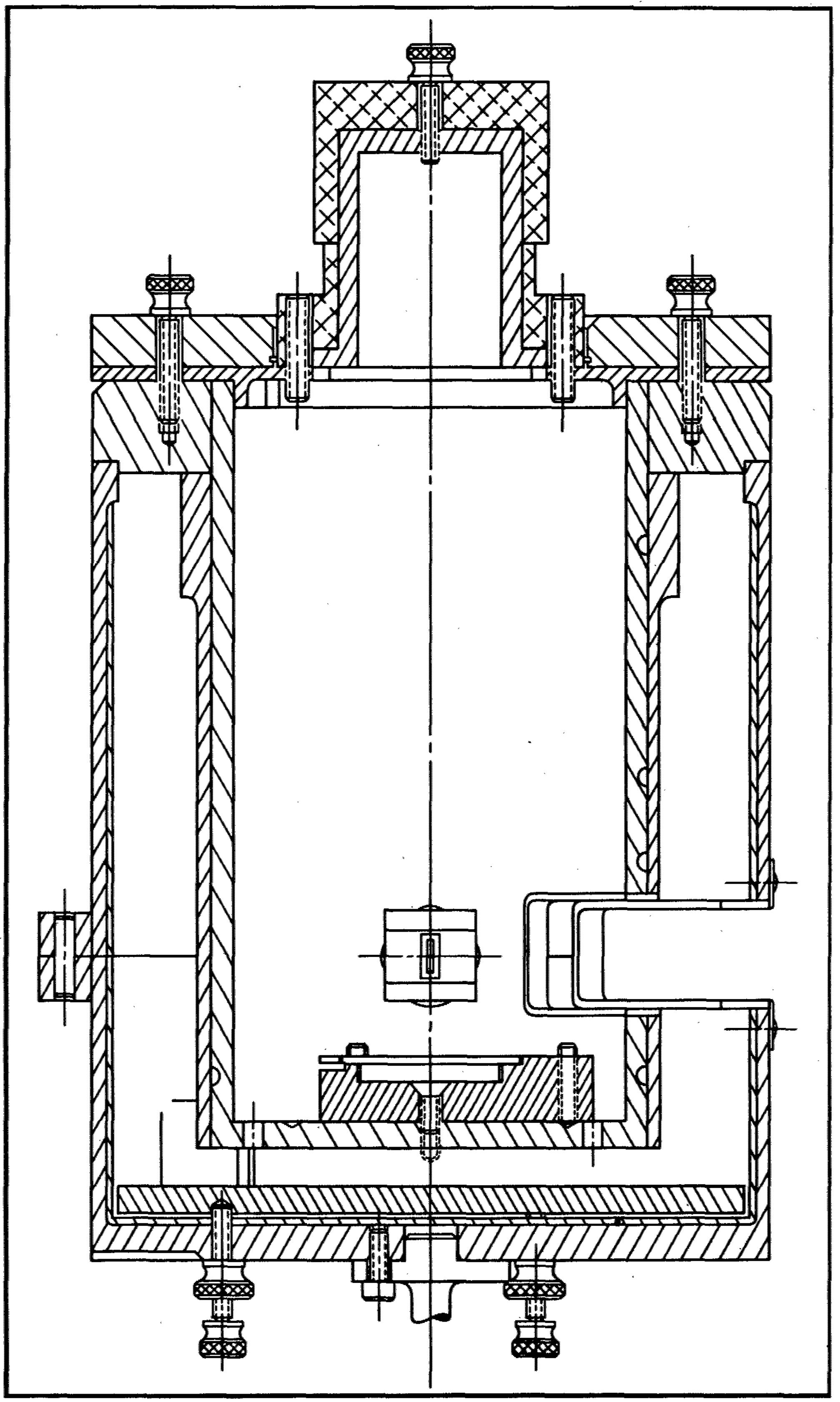

Figure 6. Cross Section of the New Thermostat by a Plane Perpendicular to the Bench

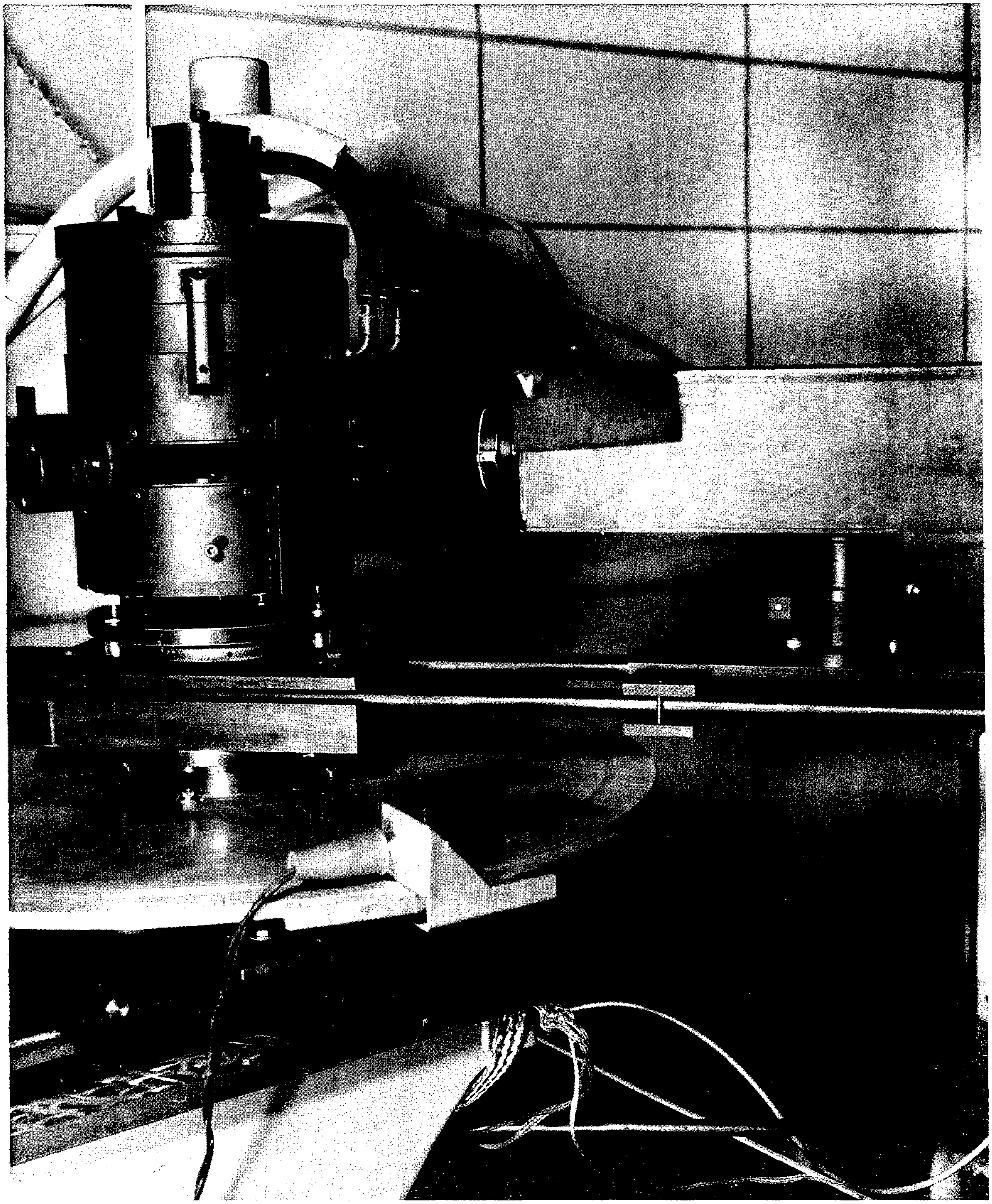

Figure 7. View of the New Thermostat on the Optical Bench (Version Two)

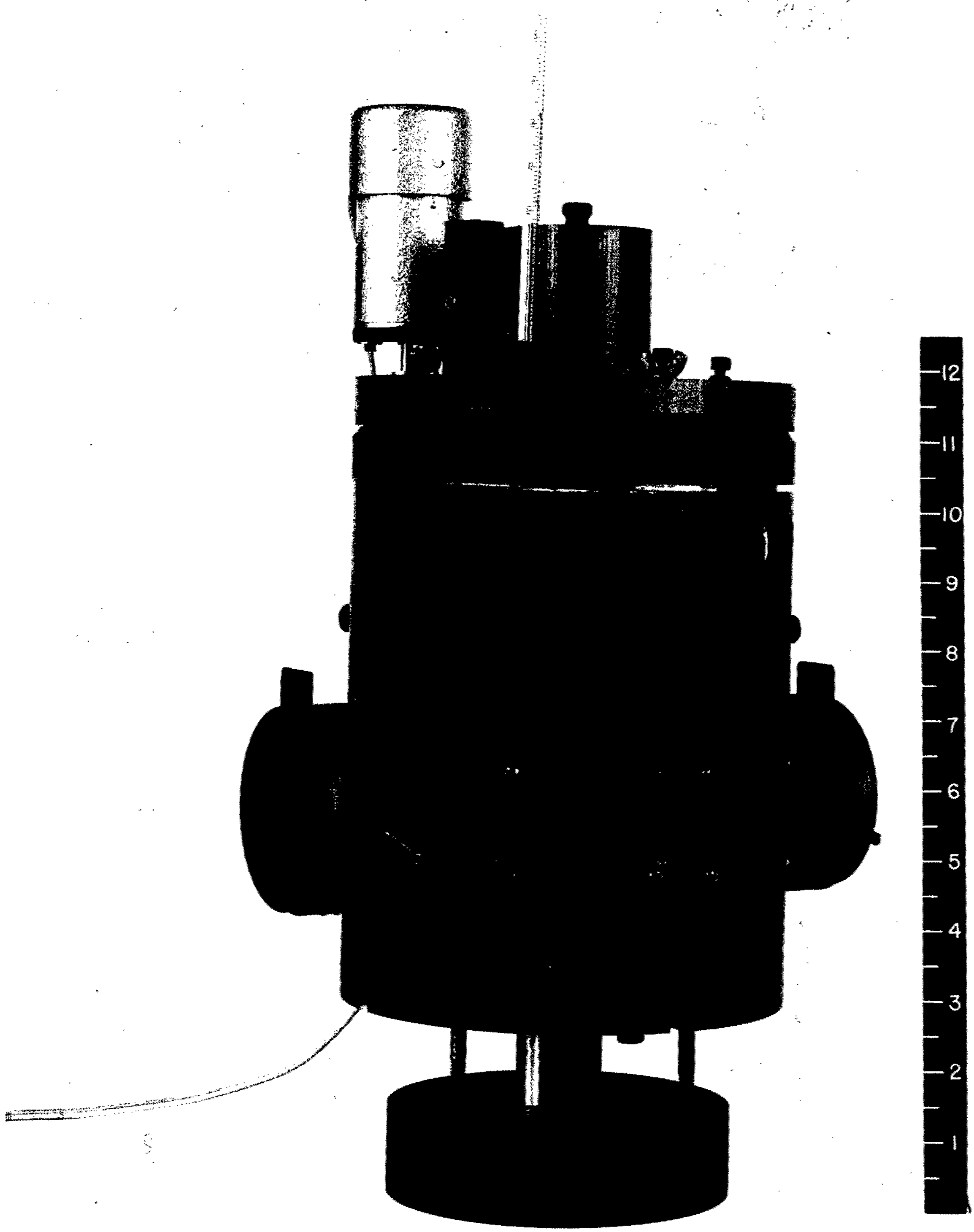

Figure 8. View of the New Thermostat Outside the Bench (Version One)

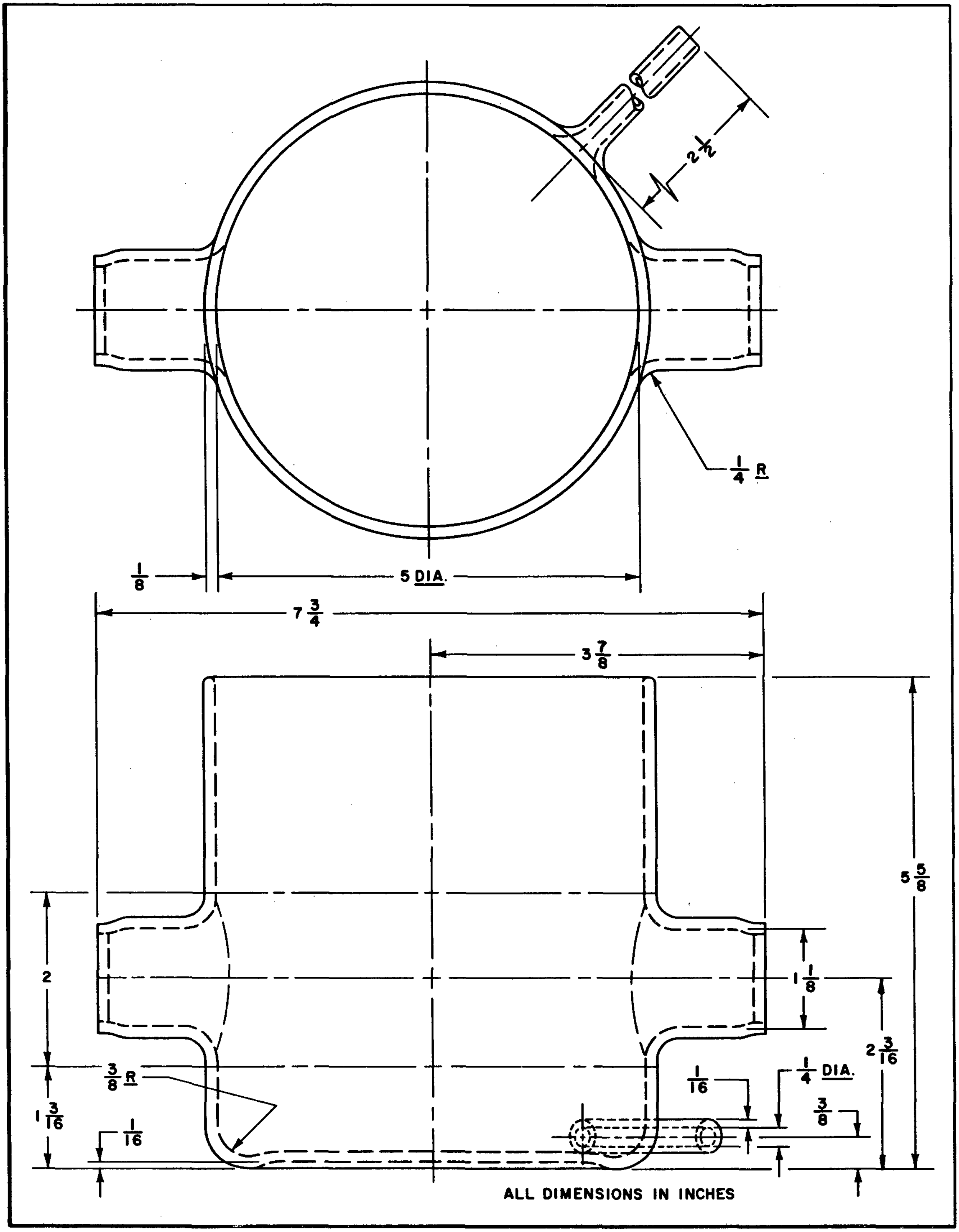

Figure 9. Cross Section of the Glass Container

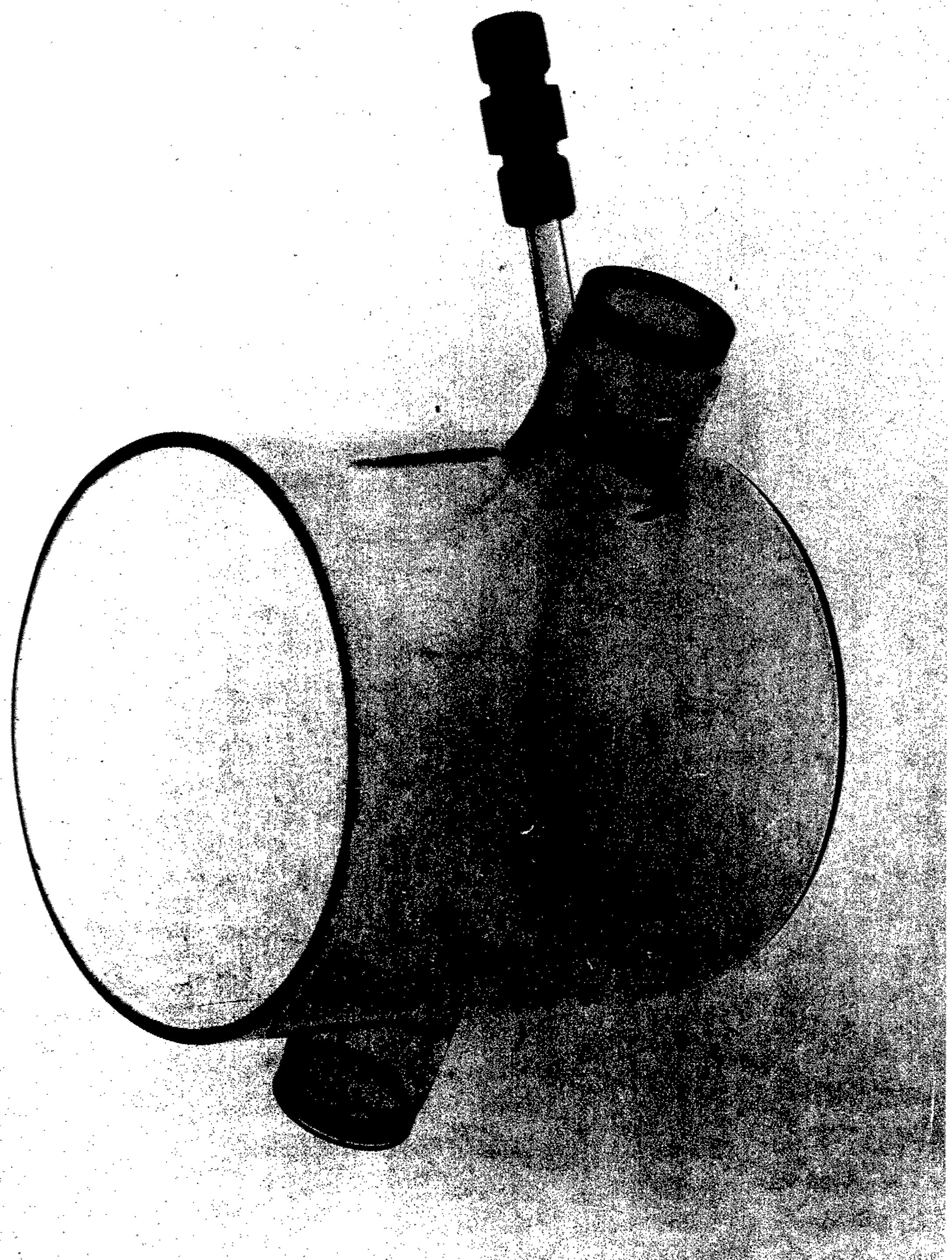

Figure 10. View of the Glass Container

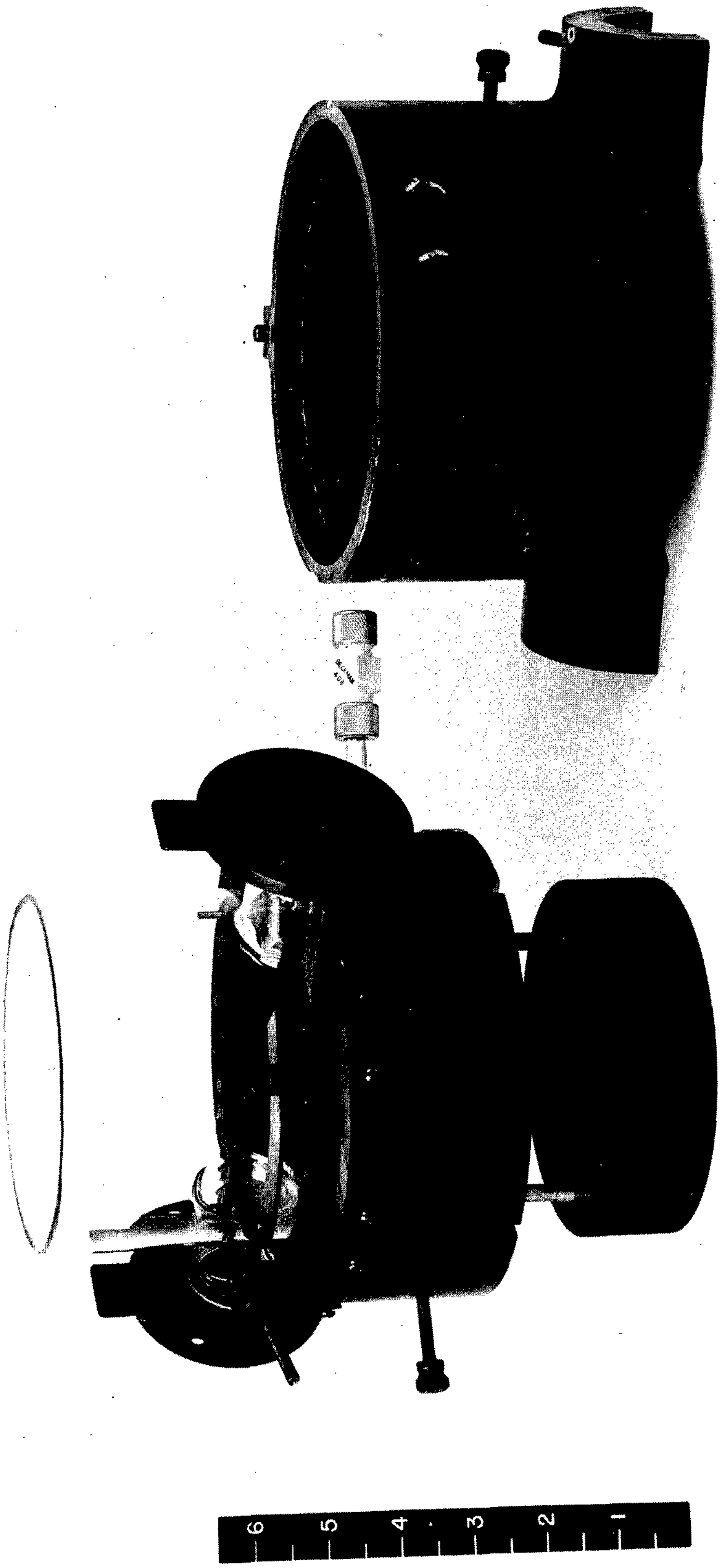

Figure 11. Dismantled Housing With the Glass Container (Version One)

Figure 12. View of the Cell Adapter and Additional Parts

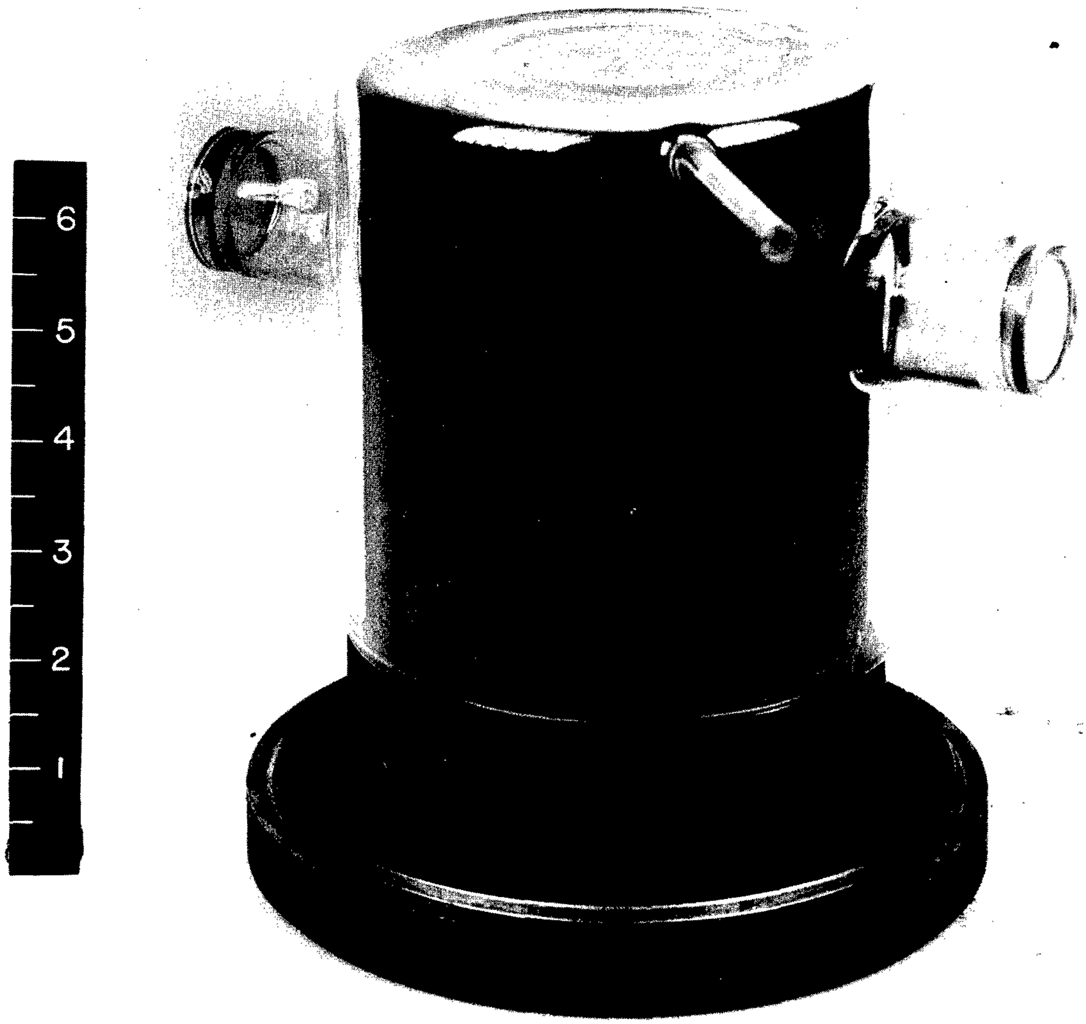

Figure 13. The Cell Adapter and the Glass Container

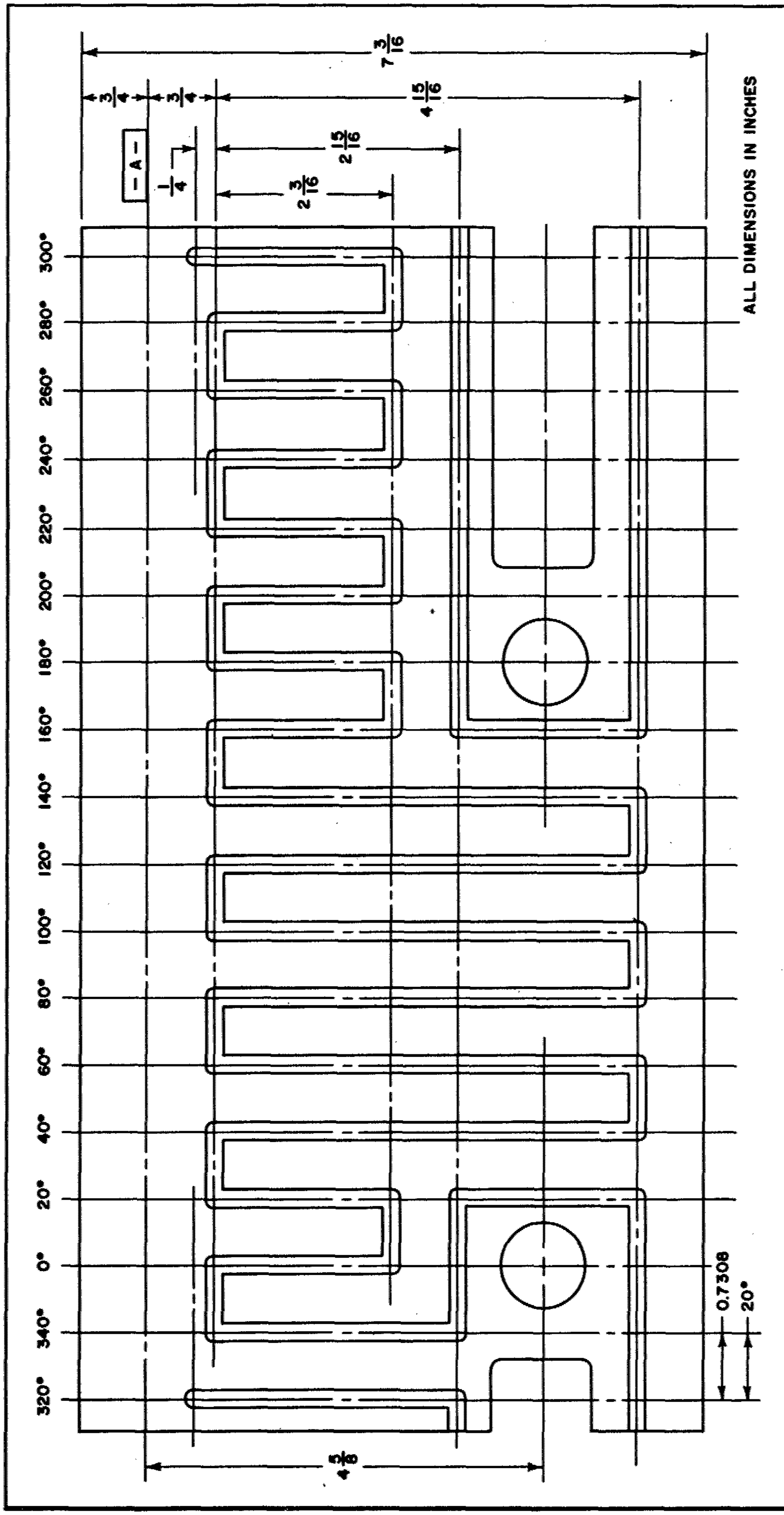

Figure 14. Pattern of Channel Grooves

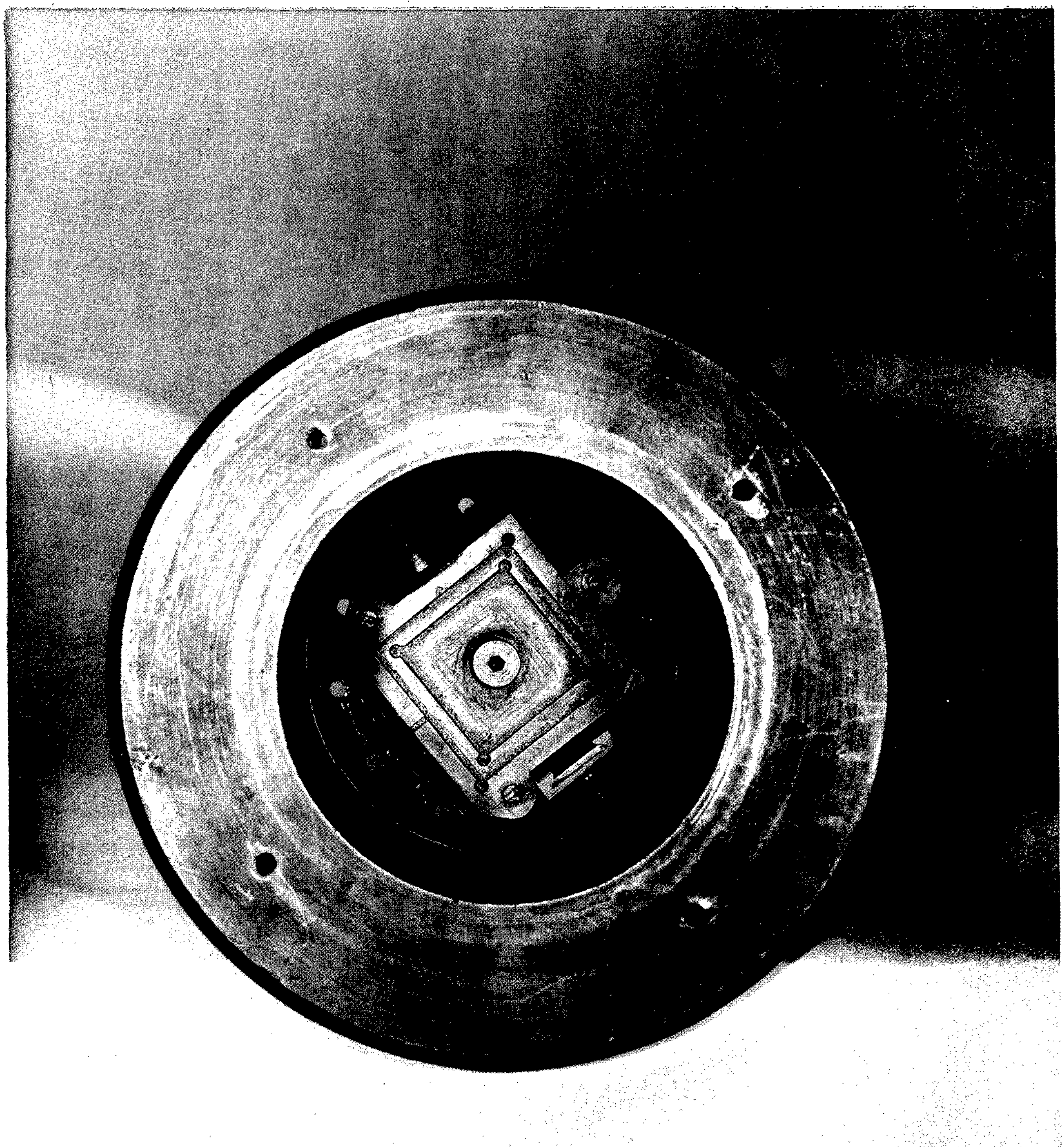

Figure 15. Bottom and the Base Surrounded by the Cell Adapter

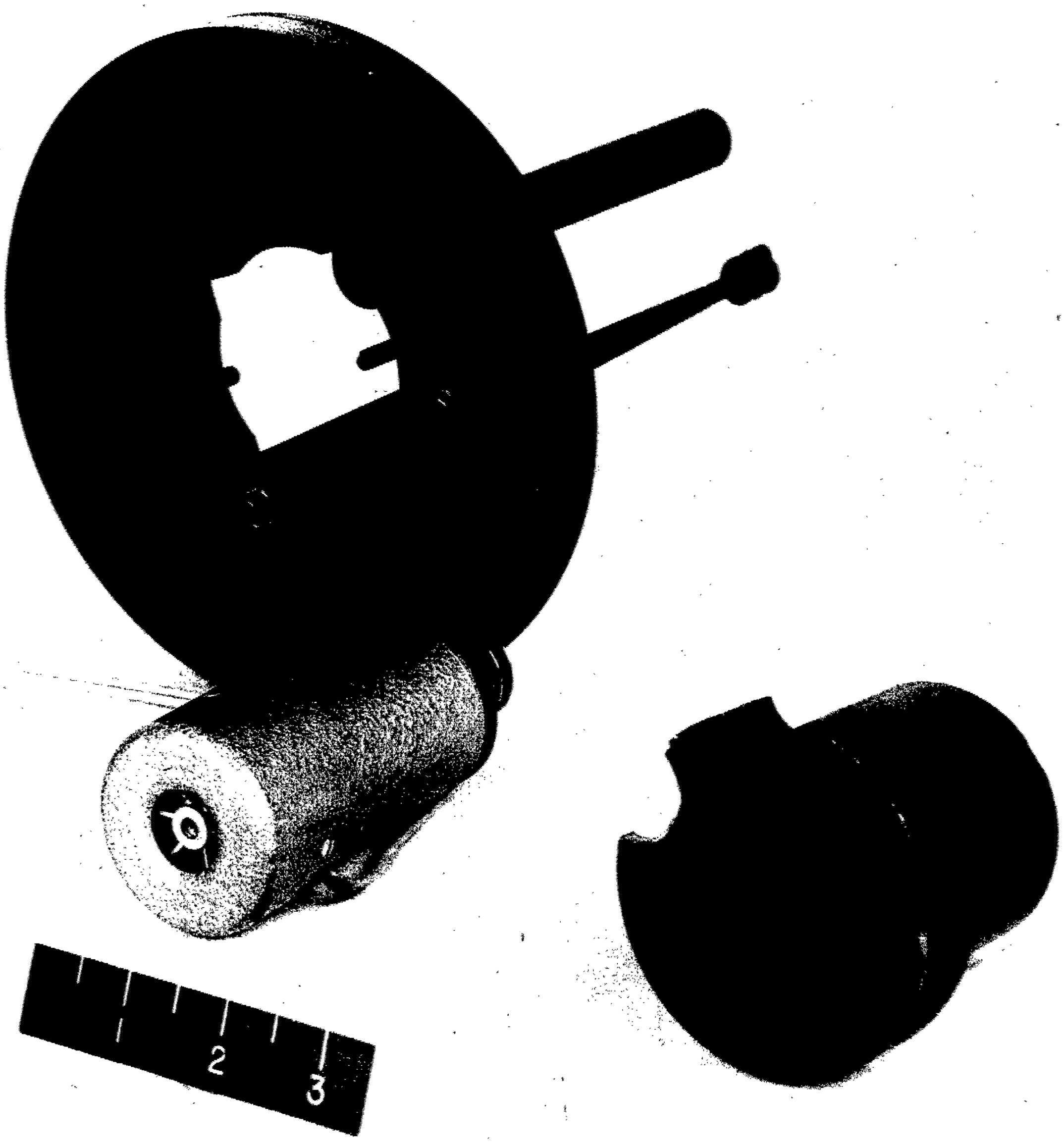

Figure 16. The Main Lid and Smaller Lid (Tube of Propeller Shaft is Removed)

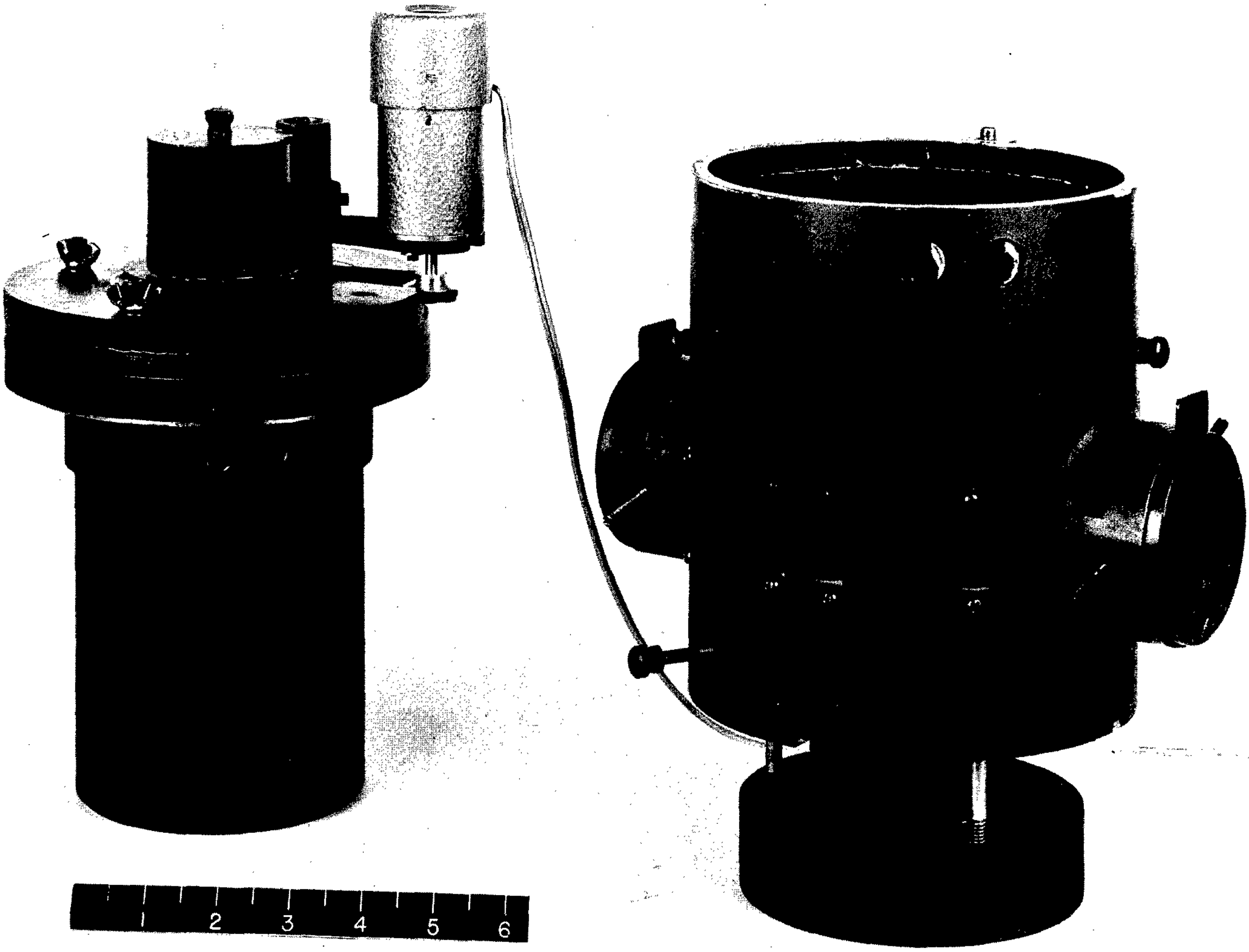

Figure 17. The Lid on Top of the Cell Adapter and the Housing

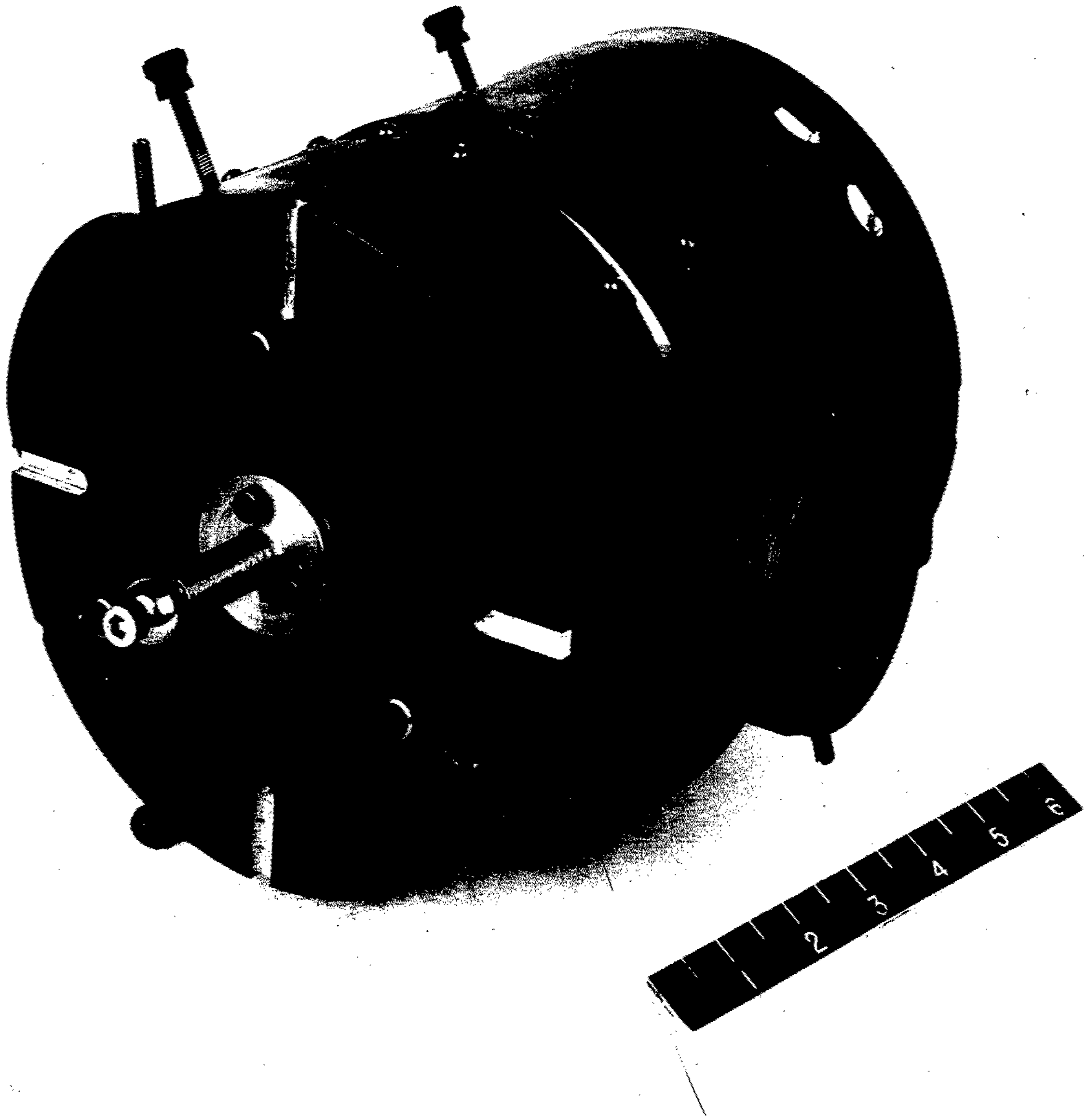

Figure 18. View of the Lower Part of the Housing

UNCLASSIFIED
Security Classification

DOCUMENT CONTROL DATA - R&D		
(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)		
1. ORIGINATING ACTIVITY *(Corporate author)* Air Force Materials Laboratory Wright-Patterson Air Force Base, Ohio	2a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED	
	2b. GROUP	
3. REPORT TITLE New Thermostat for High Temperature Light Scattering Measurements		
4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*		
5. AUTHOR(S) *(Last name, first name, initial)* Gehatia, Matatiahu; Gibbs, William E.		
6. REPORT DATE March 1968	7a. TOTAL NO. OF PAGES 30	7b. NO. OF REFS 3
8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S) AFML-TR-67-426	
b. PROJECT NO. 7342		
c. Task No. 734203	9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*	
d.		
10. AVAILABILITY/LIMITATION NOTICES This document may be further distributed by any holder only with specific prior approval of the Air Force Materials Laboratory (MANP), Wright-Patterson Air Force Base, Ohio 45433. In DDC.		
11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY Air Force Materials Laboratory Wright-Patterson Air Force Base, Ohio	

13. ABSTRACT

A new thermostat for high temperature light scattering measurements has been constructed. It is composed of the following major parts: (1.) metal housing; (2.) glass container with fused optical windows to contain the bath liquid; and (3.) cell adapter which also serves as a heat exchanger.

Previously designed thermostats consisted only of a metal cylinder containing a constant temperature liquid and a cell with the sample under investigation. The optical windows were glued or cemented to the wall. Such a thermostat cannot be used for experiments at high temperatures because no known chemical adhesive can resist hot corrosive liquids used as constant temperature liquids. Therefore, in the new thermostat the liquid container was constructed as a separate part, fabricated entirely from glass without any glue or cement, while other parts were fabricated from metal. This new assembly can be used for light scattering measurements up to 350°C.

This abstract may be further distributed by any holder only with specific prior approval of the Air Force Materials Laboratory (MANP), Wright-Patterson Air Force Base, Ohio 45433.

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Polymers — Molecular Weight, High Temperature						
Light Scattering Photometer						

INSTRUCTIONS

1. ORIGINATING ACTIVITY: Enter the name and address of the contractor, subcontractor, grantee, Department of Defense activity or other organization *(corporate author)* issuing the report.

2*a*. REPORT SECURITY CLASSIFICATION: Enter the overall security classification of the report. Indicate whether "Restricted Data" is included. Marking is to be in accordance with appropriate security regulations.

2*b*. GROUP: Automatic downgrading is specified in DoD Directive 5200.10 and Armed Forces Industrial Manual. Enter the group number. Also, when applicable, show that optional markings have been used for Group 3 and Group 4 as authorized.

3. REPORT TITLE: Enter the complete report title in all capital letters. Titles in all cases should be unclassified. If a meaningful title cannot be selected without classification, show title classification in all capitals in parenthesis immediately following the title.

4. DESCRIPTIVE NOTES: If appropriate, enter the type of report, e.g., interim, progress, summary, annual, or final. Give the inclusive dates when a specific reporting period is covered.

5. AUTHOR(S): Enter the name(s) of author(s) as shown on or in the report. Enter last name, first name, middle initial. If military, show rank and branch of service. The name of the principal author is an absolute minimum requirement.

6. REPORT DATE: Enter the date of the report as day, month, year; or month, year. If more than one date appears on the report, use date of publication.

7*a*. TOTAL NUMBER OF PAGES: The total page count should follow normal pagination procedures, i.e., enter the number of pages containing information.

7*b*. NUMBER OF REFERENCES: Enter the total number of references cited in the report.

8*a*. CONTRACT OR GRANT NUMBER: If appropriate, enter the applicable number of the contract or grant under which the report was written.

8*b*, 8*c*, & 8*d*. PROJECT NUMBER: Enter the appropriate military department identification, such as project number, subproject number, system numbers, task number, etc.

9*a*. ORIGINATOR'S REPORT NUMBER(S): Enter the official report number by which the document will be identified and controlled by the originating activity. This number must be unique to this report.

9*b*. OTHER REPORT NUMBER(S): If the report has been assigned any other report numbers *(either by the originator or by the sponsor)*, also enter this number(s).

10. AVAILABILITY/LIMITATION NOTICES: Enter any limitations on further dissemination of the report, other than those imposed by security classification, using standard statements such as:

(1) "Qualified requesters may obtain copies of this report from DDC."

(2) "Foreign announcement and dissemination of this report by DDC is not authorized."

(3) "U. S. Government agencies may obtain copies of this report directly from DDC. Other qualified DDC users shall request through ______________."

(4) "U. S. military agencies may obtain copies of this report directly from DDC. Other qualified users shall request through ______________."

(5) "All distribution of this report is controlled. Qualified DDC users shall request through ______________."

If the report has been furnished to the Office of Technical Services, Department of Commerce, for sale to the public, indicate this fact and enter the price, if known.

11. SUPPLEMENTARY NOTES: Use for additional explanatory notes.

12. SPONSORING MILITARY ACTIVITY: Enter the name of the departmental project office or laboratory sponsoring *(paying for)* the research and development. Include address.

13. ABSTRACT: Enter an abstract giving a brief and factual summary of the document indicative of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.

It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as *(TS)*, *(S)*, *(C)*, or *(U)*.

There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.

14. KEY WORDS: Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, rules, and weights is optional.