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ACCELERATED COMPRESSION SET PROPERTIES OF FOURTEEN ELASTOMERS

by C. D. HOOPER AND J. T. SCHELL
Propulsion and Vehicle Engineering Laboratory

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George C. Marshall Space Flight Center, Huntsville, Alabama

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ABSTRACT

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Fourteen types of synthetic elastomers, from which O-rings and other gaskets might be fabricated, were investigated for their compression set properties. Each compound was tested at a minimum of three temperatures [ranging from 70°C (158°F) to 250°C (482°F)] during various periods of time up to 32 days.

Results showed that most elastomers have reasonably good compression set properties at room temperature (25°C) but that many of these might have critical limitations when subjected to the same compression at an elevated temperature. These tests, like other accelerated tests, were not expected to indicate the small differences that might be encountered in actual service; however, they do provide a practical evaluation of the properties that are useful where a high degree of precision is not expected.

A summary of the data obtained from this study is presented in graphical form, illustrating the characteristics and limitations of each compound tested.

Author

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MATERIALS DIVISION
PROPULSION AND VEHICLE ENGINEERING LABORATORY
RESEARCH AND DEVELOPMENT OPERATIONS

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SUMMARY

Compression set is an important property that must be considered in the selection of an elastomeric material for gasketing in space vehicle applications. In this study, 14 types of synthetic rubber, from which O-rings and other gaskets might be fabricated, were investigated for their compression set properties over a temperature range of 70°C (158°F) to 250°C (482°F) for periods of time to 32 days.

Each compound was tested at a minimum of three temperatures, and the data obtained were extrapolated to room temperature (approximately 25°C, 77°F). Results showed that most elastomers have reasonably good compression set properties at room temperature but that many of them have acute limitations for gasketing usage at some higher temperature. Compounds such as silicones, Viton-A, and fluoro-silicone may be subjected to temperatures as high as 200°C (392°F) for a limited period of time; however, others such as Thiokol and urethane, should not be considered for applications above 70°C (158°F).

A summary of the data obtained from this study is presented in graphical form in FIG 1 - 34. The test apparatus and illustrations of test specimens are shown in FIG 35.

INTRODUCTION

Currently, at least 14 types of elastomers are available for meeting highly specialized requirements in the aviation and rocket industries. Several of the elastomers are so new that very little is known about their properties.

In missiles and spacecraft, compression set is one of the properties that is vitally important to successful sealing by resilient type gaskets. This property is more critical where high or low temperatures, high pressures, and the effects of chemicals, solvents, fuels, hydraulic media, or storage time are to be considered.

The results obtained by accelerated compression set testing of various elastomers have been expressed in numerous scientific publications; however, there has been no order or system. Most of the published data offer only limited useful information regarding compression set; therefore, this study was an obvious necessity for this Center.

This study consisted of a systematic testing program for 14 elastomers over a broad range of conditions that were sufficient to make the interpolation of intermediate values convenient. Test procedures were consistent with ASTM D-395 Method "B", with the exception of time and temperature.

DISCUSSION

Compression set is defined as the percent of permanent deformation resulting when a rubber specimen has been subjected to a specified constant deflection, time interval, and temperature. A high numerical value of compression set indicates poor compression set quality.

Fourteen standard rubber formulations of 60 Shore-A hardness were tested in accordance with ASTM D-395-55 Method "B" (FIG 35) with the exception of time and temperature variations. This procedure measures the ability of the compound to retain its elastic properties during prolonged compressive stress. Each compound was tested at a minimum of three temperature levels in precision controlled ovens for periods up to 32 days or until the compound approached 90 percent compression set, whichever occurred first. A maximum of 90 percent compression set was established as an arbitrary limit for all compounds at the beginning of the study.

Seven temperatures [70°C (158°F), 100°C (212°F), 125°C (257°F), 150°C (302°F), 175°C (347°F), 200°C (392°F), and 250°C (482°F)] were selected for this study, and each of the 14 types of elastomers was tested at a minimum of three of these temperatures, depending on the type of rubber. Each elastomer was measured at intervals of 1, 2, 4, 8, 16, and 32 days, and compression set versus time plots were prepared for selected temperatures (FIG 1 - 14). Bar graphs that compared the compression set results of the several types of elastomers for 32 days were prepared for all temperature ranges in which they were tested (FIG 15 - 21). Another group of graphs (FIG 22 - 34) was prepared to illustrate the relationship of compression set to temperatures over an interval of 32 days.

CONCLUSIONS AND RECOMMENDATIONS

Results indicated that vulcanized silicone, Viton-A, fluorosilicone, Kel-F, and acrylic elastomers have excellent compression set at elevated temperatures. Silicone, Viton-A, and fluorosilicone may be used in temperatures as high as 200°C (392°F); however, the maximum temperature applied to Kel-F should not exceed approximately 150°C (302°F). Acrylic rubber should not be placed in service where the temperature will exceed 125°C (257°F), and only for a short time even at this level.

Hypalon, natural, Neoprene, NBR, and SBR have a reasonably good compression set property at accelerated conditions, but they should not be placed in service where the temperature may exceed 100°C (212°F). Thiokol, urethane, and butyl have the poorest compression set property at elevated temperatures of all the compounds tested and should not be used in areas where the temperature may exceed 70°C (158°F).

It was observed that no two types of elastomers reacted in the same manner under compression at elevated temperatures. Two or more compounds may have had similar compression set characteristics at room temperature for 32 days and may have followed the same pattern of behavior at 100°C (212°F) for one, two, or four days; however, at the end of eight days, these characteristics differed noticeably. This indicates that extended time may affect each compound to some degree at room temperature; however, this factor is magnified considerably at higher temperature levels.

These tests, like other accelerated tests, were not expected to indicate accurately the small differences that may be encountered in actual service; however, they do provide a practical evaluation of the properties that are useful where a high degree of precision is not required.

Each compound that was tested is listed below. The time and temperature levels in which the elastomers may be used as a gasket or sealant may be determined from the results of the test.

1. Thiokol (Polysulfide) (FIG 1)

This compound had the poorest compression set properties of all compounds tested. At 70°C (158°F), the lowest temperature range of the study, the compound reached 70 percent set in one day, 89 percent in two days, and exceeded the 90 percent limit (placed on all tests as an arbitrary limit) in four days (95 percent).

2. Urethane (Polyurethane) (FIG 2 and 22)

Urethane may be placed in service at 70°C (158°F) for a period of 32 days or more (38 percent in 32 days), but it should not be placed in areas (under compression) where the temperature exceeds 70°C (158°F) for an extended length of time. At 100°C (212°F), the amount of compression set noted in a 16-day test had reached the 90 percent limit, and extrapolation of the data to 32 days indicated approximately 103 percent.

3. Butyl (Isobutylene-Isoprene Copolymer)

At 70°C (158°F) for 32 days, butyl rubber is well within the 90 percent limit (70 percent set). At 100°C (212°F), the amount recorded in 16 days was 86 percent and had exceeded the limit at 32 days (95 percent).

4. Hypalon (Chlorosulfonated Polyethylene) (FIG 3, 4, 23, and 24)

The highest temperature at which this compound should be placed in service for any extended period of time is 100°C (212°F) (72 percent compression set in 32 days); however, it may be used at approximately 120°C (248°F) for shorter duration of time. After 16 days at this temperature, the amount of compression set noted was 78 percent and increased to 105 percent at the end of the 32-day period.

5. Neoprene (Polychloroprene) (FIG 5 and 25)

Neoprene should not be placed in service at temperatures exceeding 100°C (212°F) for extended periods of time. At the end of 32 days at 70°C (158°F), the compression set recorded was 27.5 percent, and although no actual measurements were made at 100°C (212°F), the compression set had increased to 61 percent in 16 days at 125°C (257°F) and was over the 90 percent limit in 32 days (96 percent).

6. Natural (Polyisoprene) (FIG 6 and 26)

At 100°C (212°F), the amount of compression set recorded at 32 days was 78 percent. At 125°C (257°F), this rate increased to 70 percent in four days, 82 percent in eight days, and over the 90 percent limit in 16 days (97.5 percent).

7. SBR (Styrene Butadiene Copolymer) (FIG 7 and 27)

This compound was placed in service at temperatures as high as 125°C (257°F) for a period of 32 days; however, at the end of this period, it was very close to the 90 percent limit. At 150°C (302°F), this limit was approached more rapidly (89 percent in four days and over 100 percent in eight days).

8. NBR (Butadiene Acrylonitrile Copolymer) (FIG 8 and 28)

The highest temperature at which this compound should be used for a 32-day period is 125°C (257°F). At this time and temperature, the compression set was 88 percent. When the temperature was increased to 150°C (302°F), the rate increased much more rapidly. After four days at 150°C (302°F), the compression set was 90 percent and had increased to 98 percent in eight days.

9. LS (Methyl Trifluoropropyl Silicone) (FIG 9 and 29)

This compound was well under the 90 percent limit after 32 days at 125°C (257°F) (26 percent compression set), but it seemed to be greatly affected when the temperature was raised to 150°C (302°F). At 150°C (302°F), the compression set had increased to 65 percent in eight days, 85 percent in 16 days, and 95 percent in 32 days.

10. Acrylic (Acrylic Ester Copolymer) (FIG 10 and 30)

At 125°C (257°F), acrylic rubber may be placed in service for a period of 32 days or more (73 percent set in 32 days); however, the compression rate is greatly increased when the temperature is raised to 150°C (302°F). At this temperature, the compression set had increased to 83 percent in 16 days and was 97.5 percent at 32 days.

11. Kel-F (Copolymer of Vinylidene Fluoride and Chlorotrifluoroethylene) (FIG 11 and 31)

The highest temperature to which this compound was subjected was 150°C (302°F). At this level, compression set was 80 percent in 32 days, which is well inside the 90 percent limit. At 125°C (257°F), the rate was reduced to 26 percent for the 32-day period.

12. Viton-A (Copolymer of Vinylidene Fluoride and Hexafluoropropylene) (FIG 12 and 32)

The highest temperature to which this compound should be subjected for extended periods is 175°C (347°F). At 150°C (302°F), the compression set was 64 percent at the end of 32 days, and at 200°C (392°F), it was 81 percent in four days and 94 percent in eight days.

13. Silicone RA-28 (Methyl Vinyl) (FIG 13 and 33)

The highest temperature to which this compound should be subjected for an extended period is 175°C (347°F); however, this temperature may be raised for short durations. At 175° C (347°F), the compression set was 58 percent in 32 days. At 200°C (392°F), the rate had rapidly increased to 89 percent in 16 days, and,at 250°C (482°F),it exceeded the 90 percent limit in two days.

14. Silicone RA-31 (Methyl Phenyl Vinyl) (FIG 14 and 34)

This is the only compound tested that was still under the 90 percent limit in 32 days at 200°C (392°F). At this point, the compression set was 76.5 percent; however, like RA-28, the compression was over the limit in two days at a temperature of 250°C (482°F).

FUTURE WORK

In addition to this short-time, elevated-temperature program, a study is now being concluded on the long-term compression set properties of selected elastomers. During this study, specimens were exposed to uniform loading for protracted periods of up to three years. A report which describes this program is being prepared.

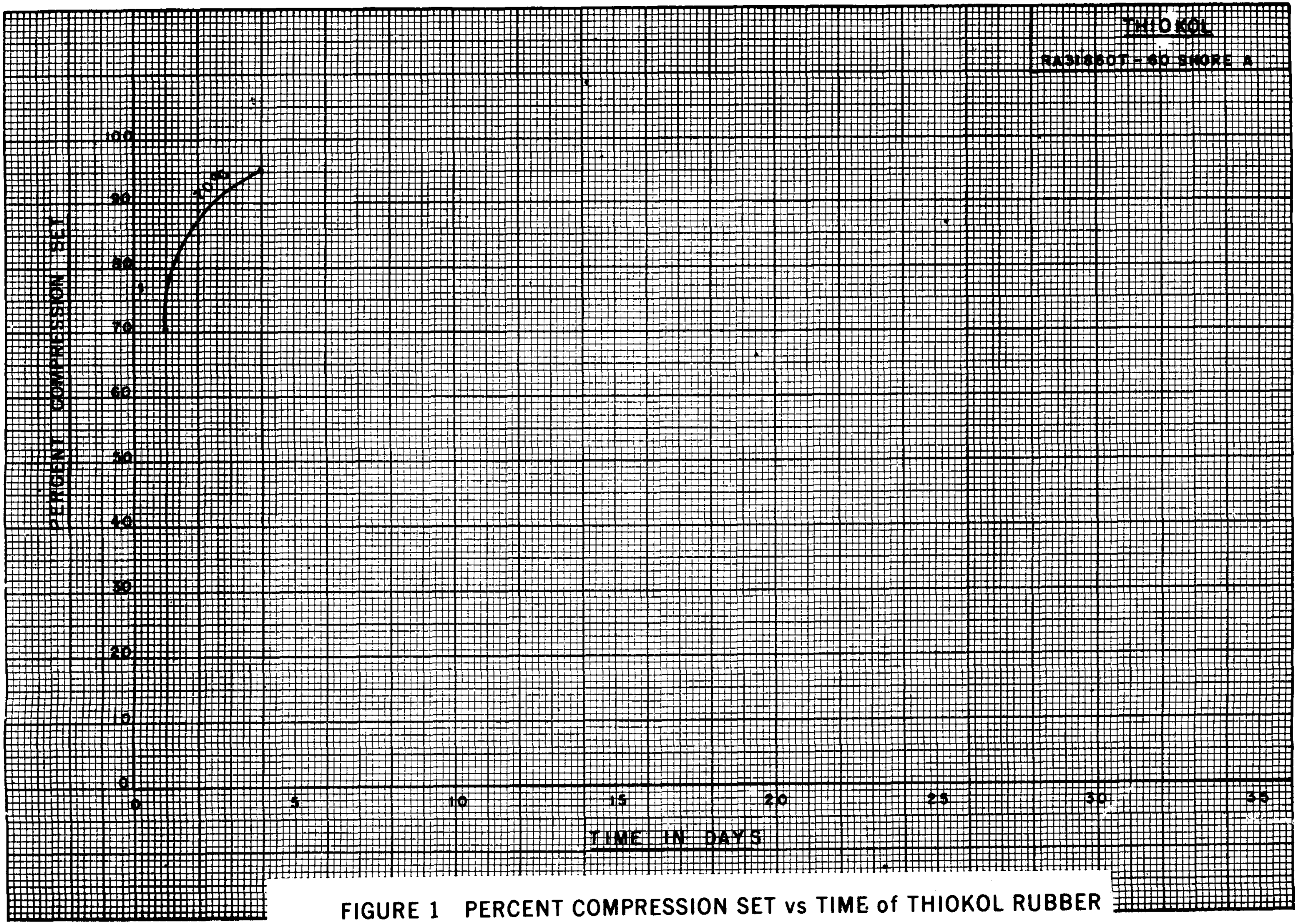


FIGURE 1 PERCENT COMPRESSION SET vs TIME of THIOKOL RUBBER

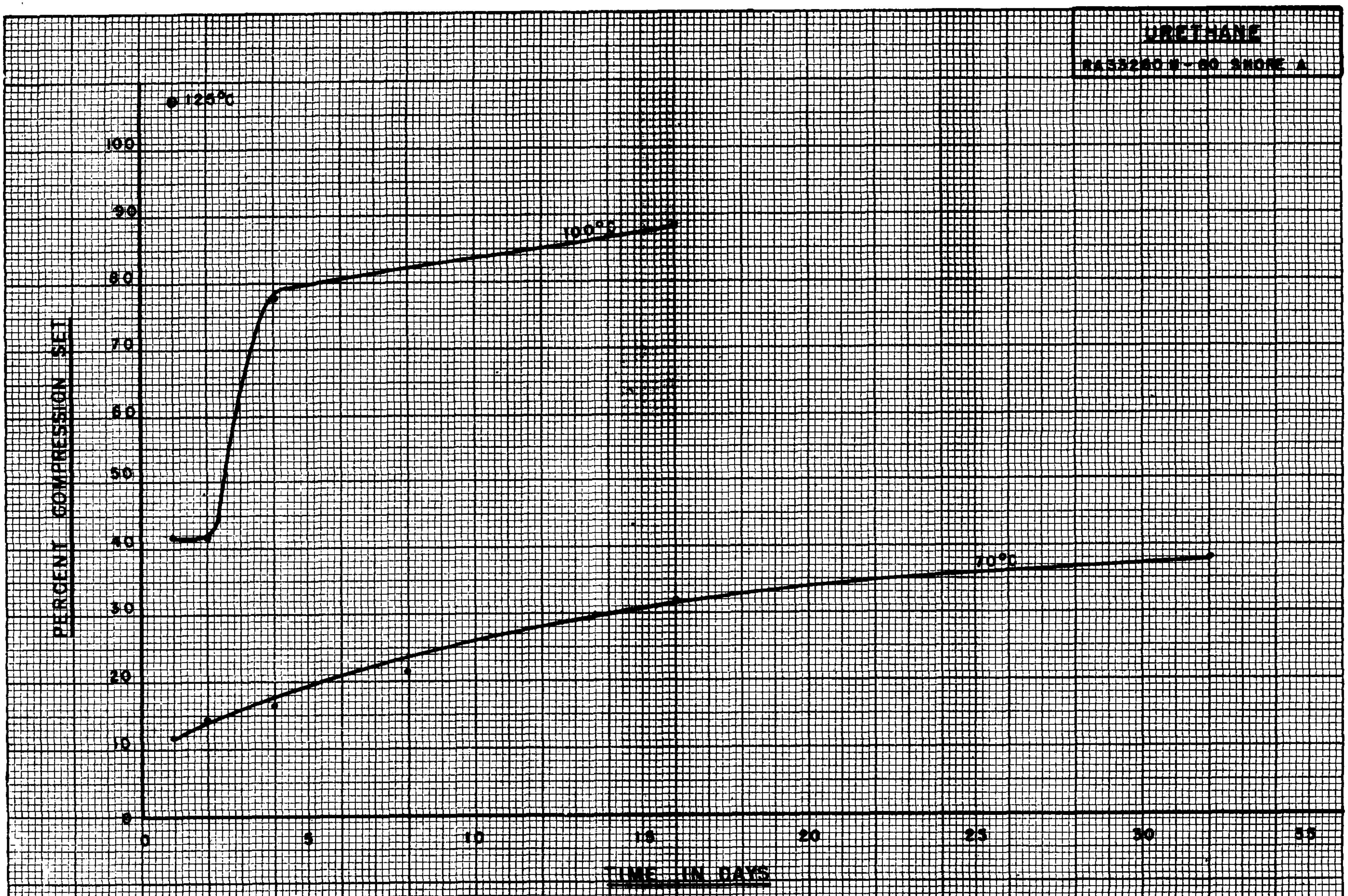


FIGURE 2 PERCENT COMPRESSION SET vs TIME of URETHANE RUBBER

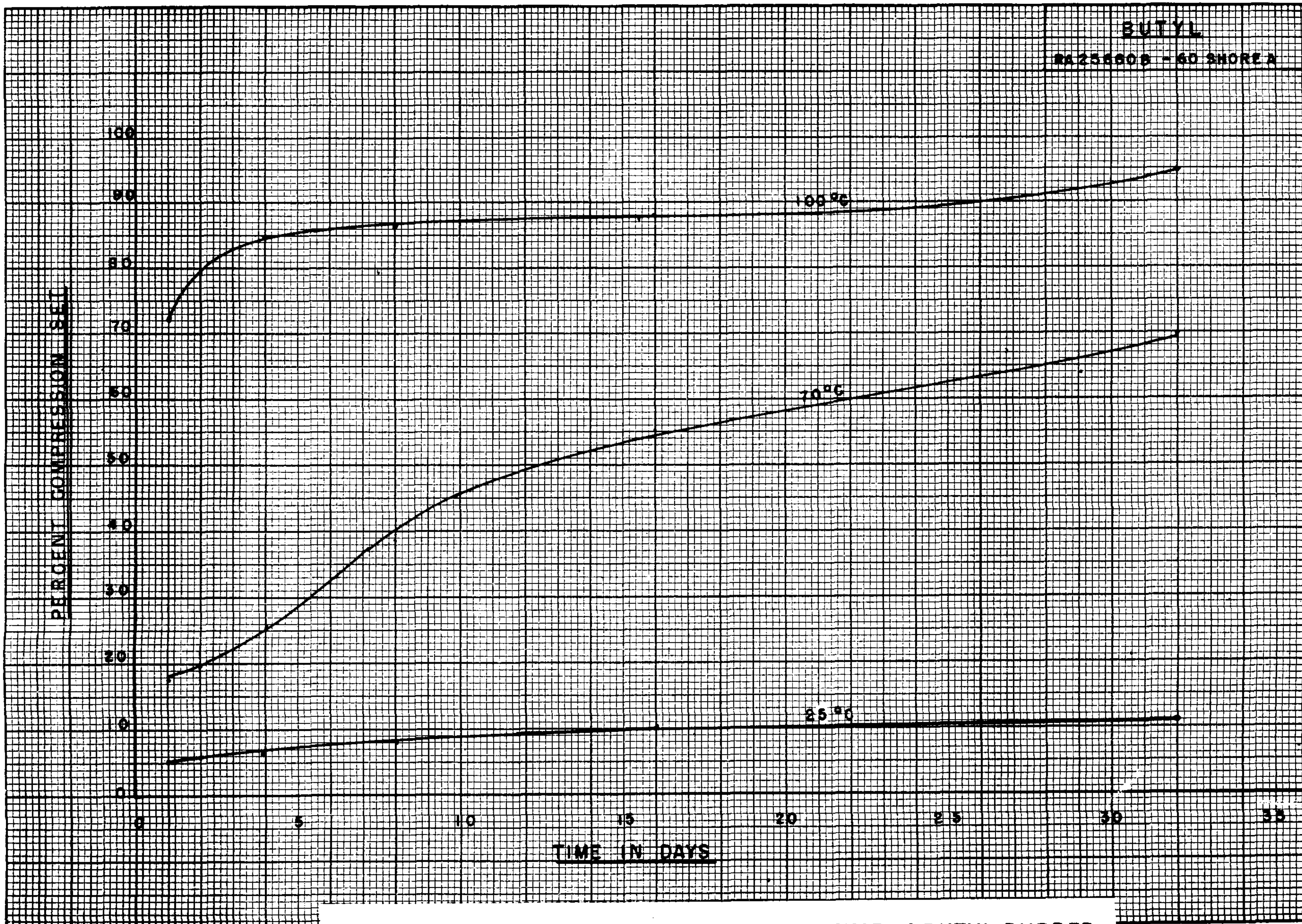


FIGURE 3 PERCENT COMPRESSION SET vs TIME of BUTYL RUBBER

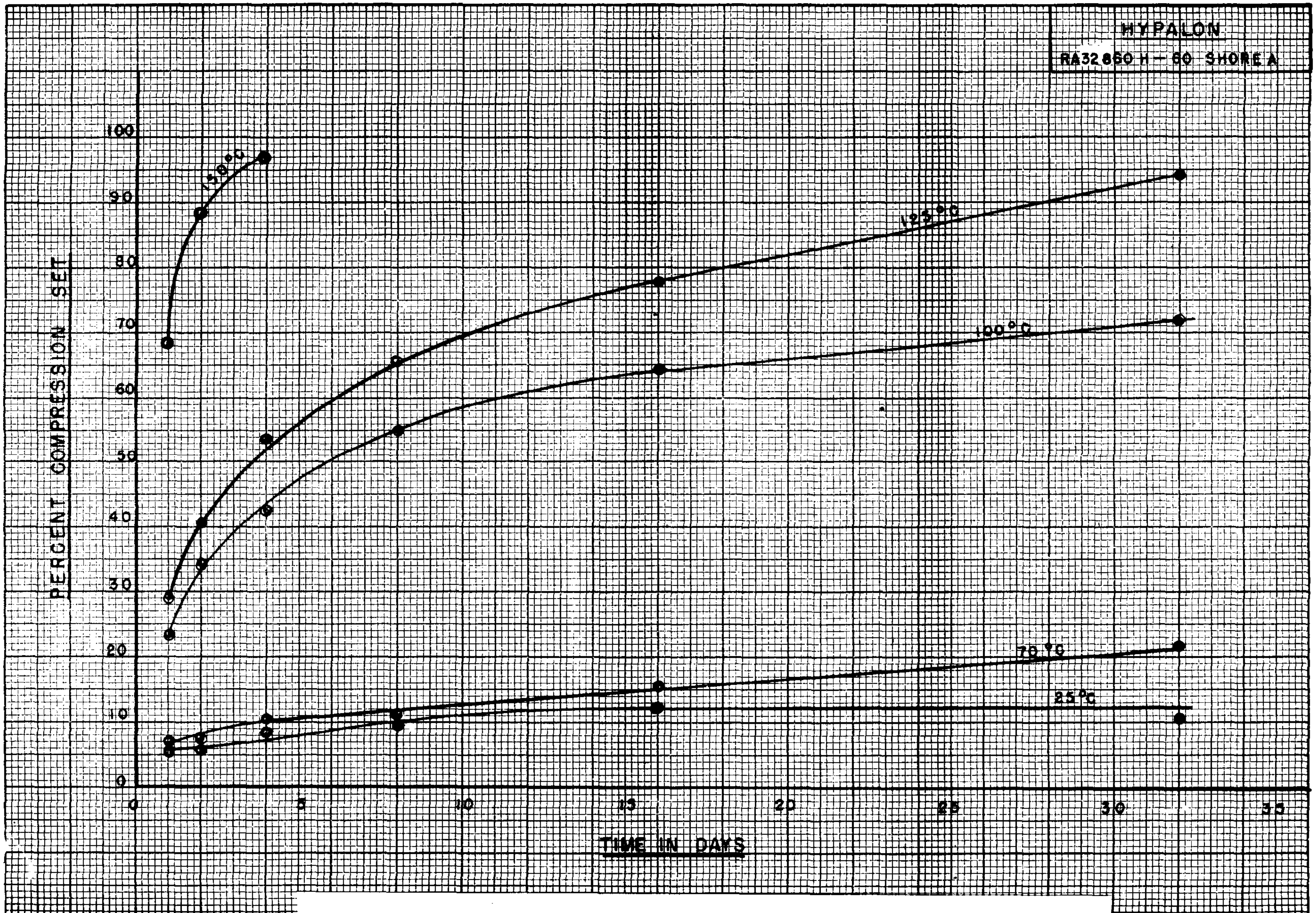


FIGURE 4 PERCENT COMPRESSION SET vs TIME of HYPALON RUBBER

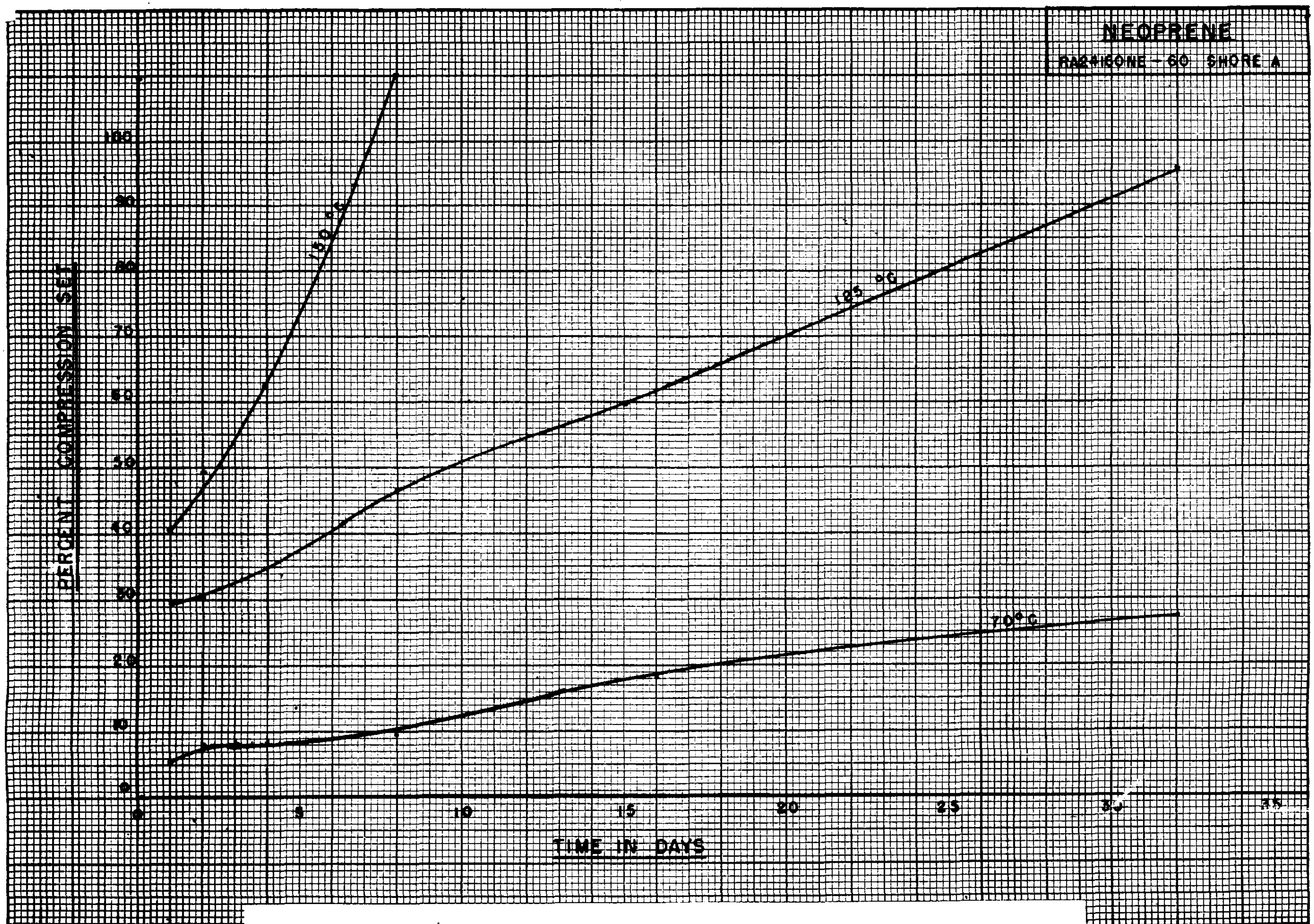


FIGURE 5 PERCENT COMPRESSION SET vs TIME of NEOPRENE RUBBER

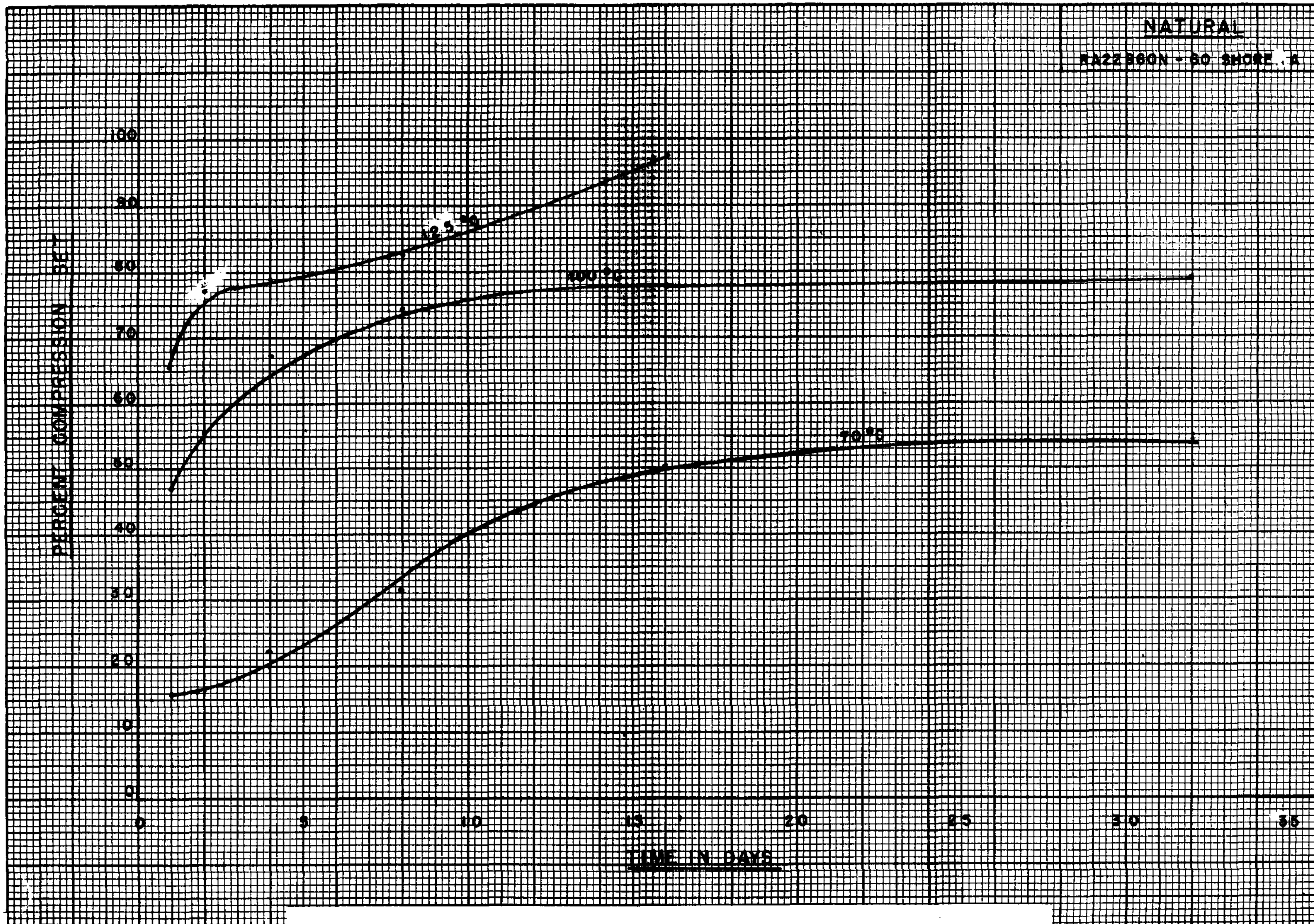


FIGURE 6 PERCENT COMPRESSION vs TIME of NATURAL RUBBER

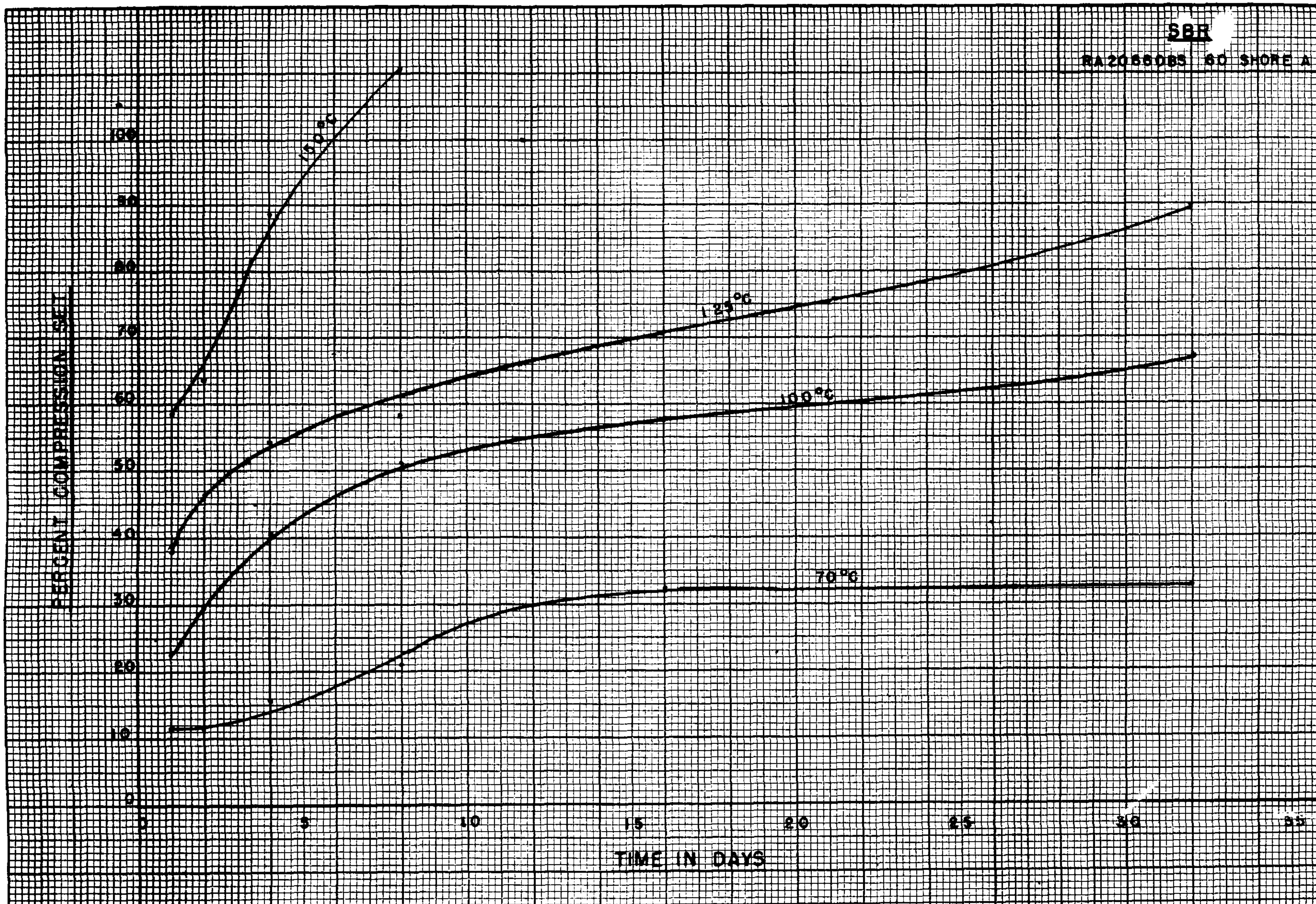


FIGURE 7 PERCENT COMPRESSION SET vs TIME of SBR RUBBER

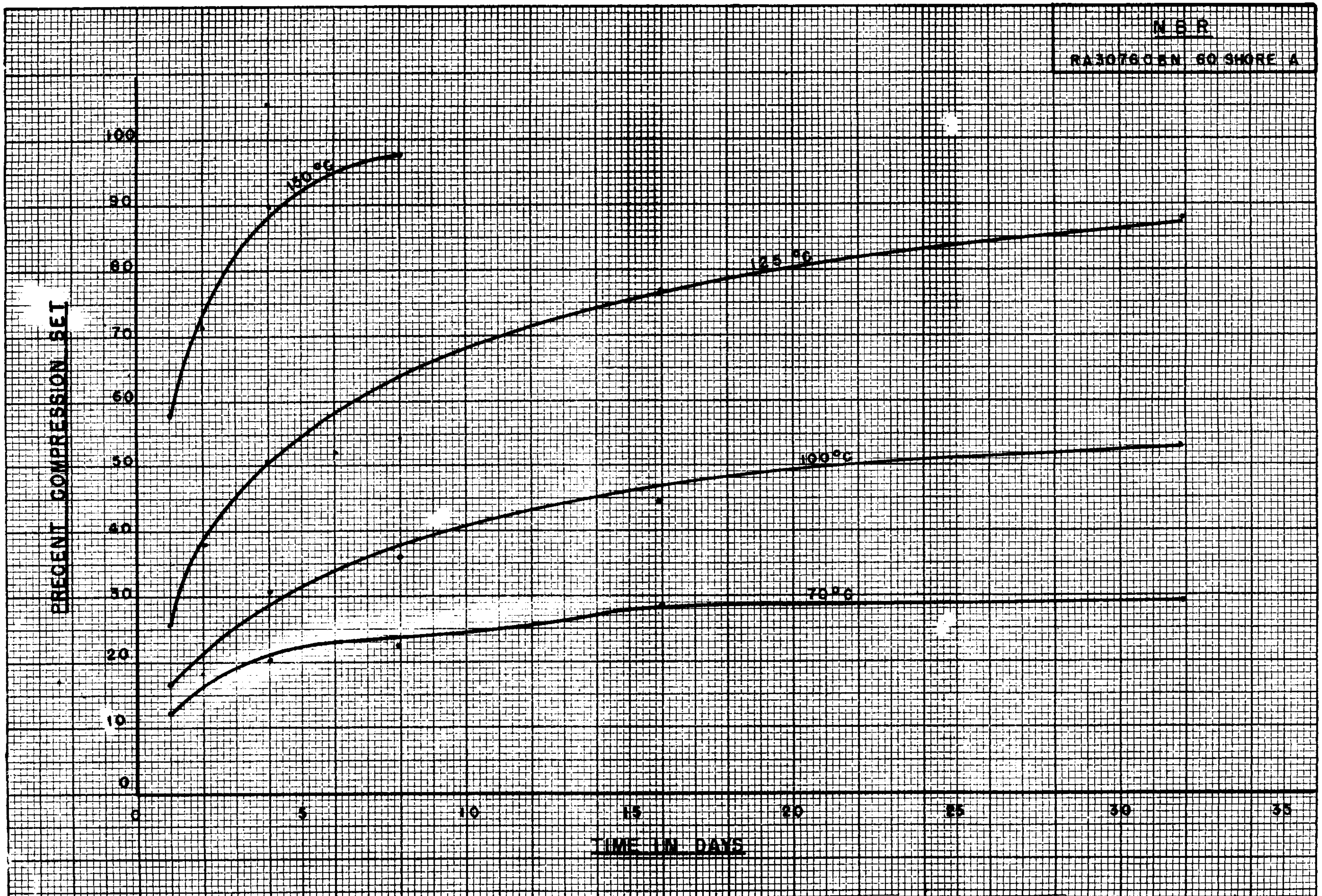


FIGURE 8 PERCENT COMPRESSION SET vs TIME of NBR RUBBER

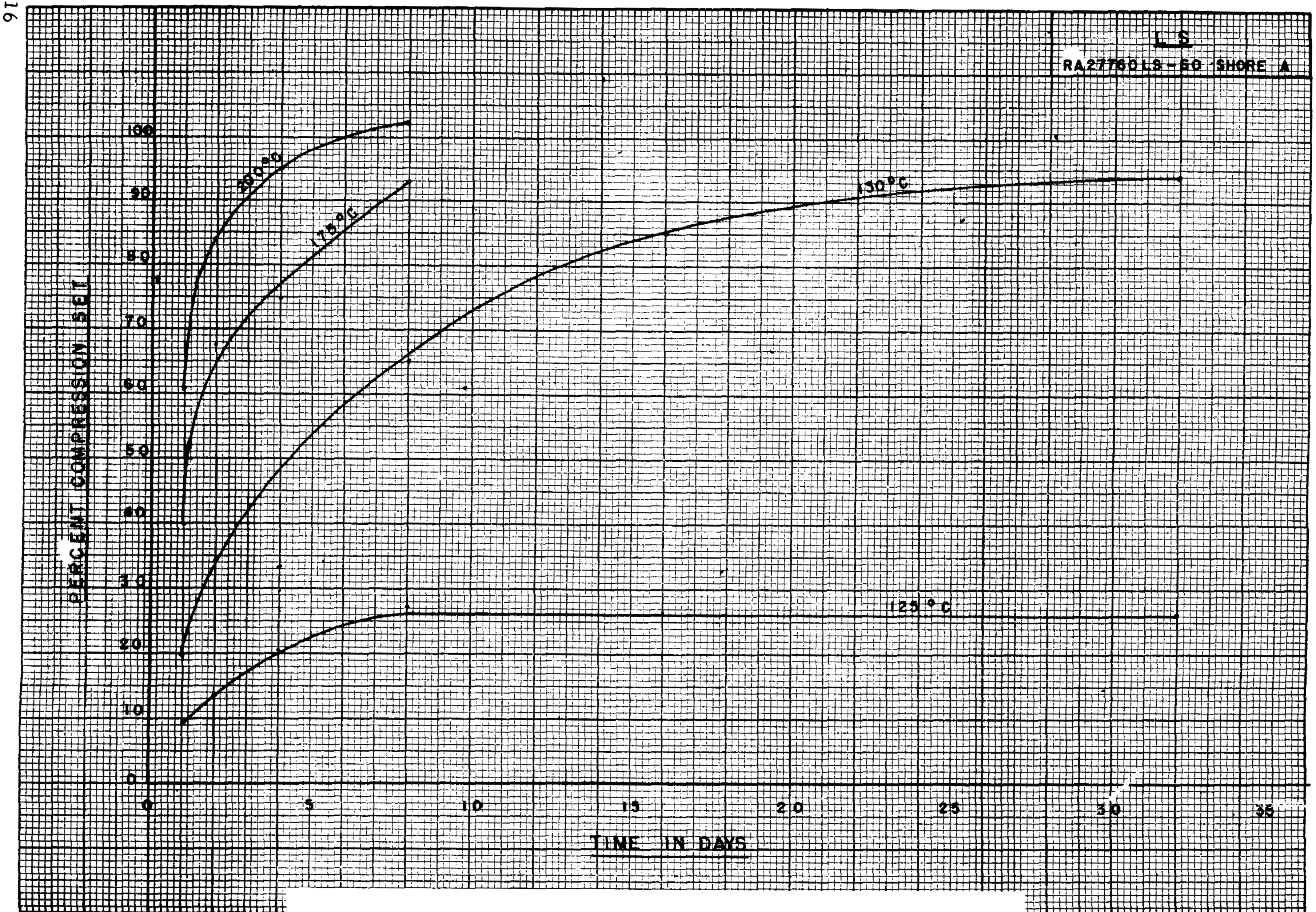


FIGURE 9 PERCENT COMPRESSION SET vs TIME of LS RUBBER

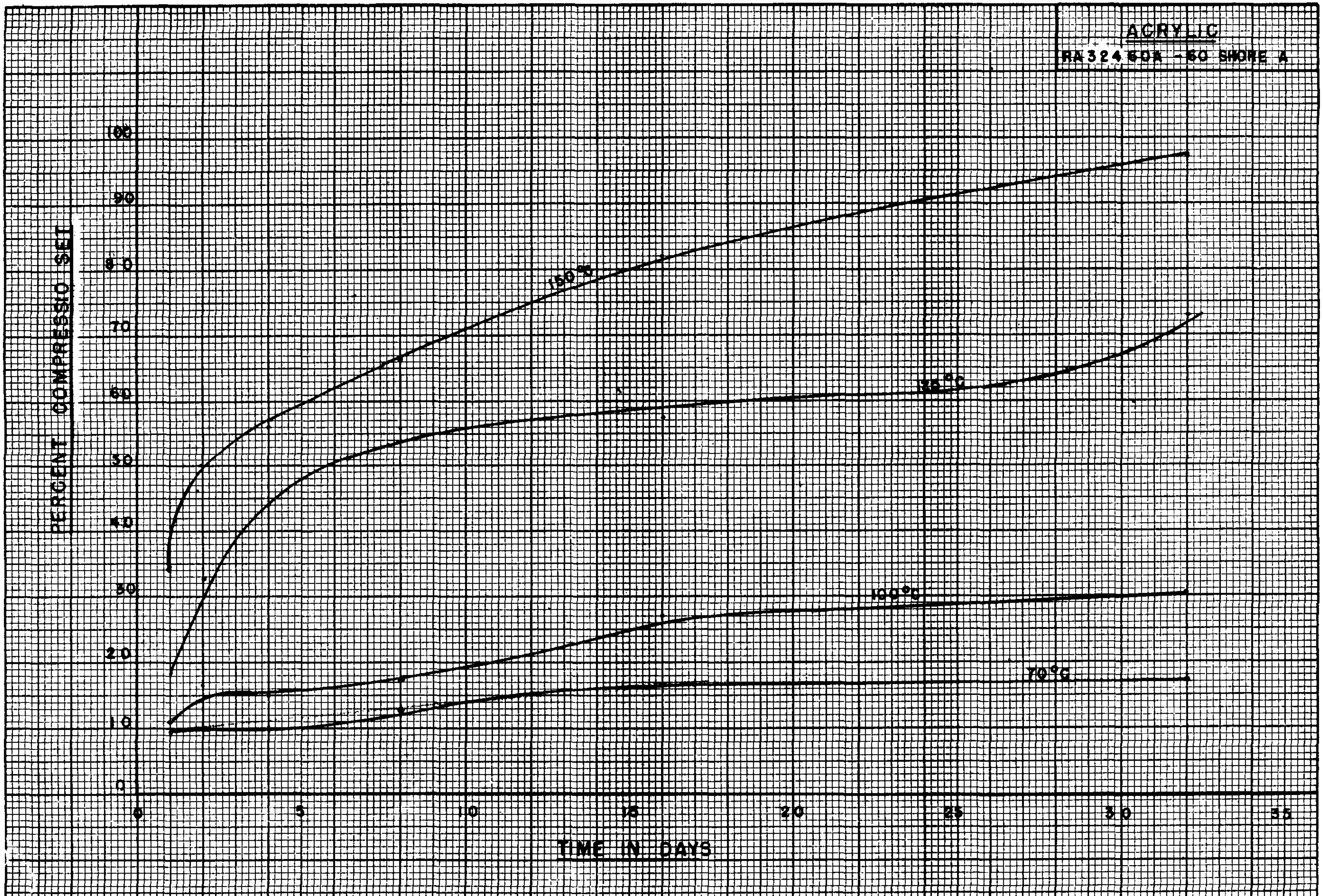


FIGURE 10 PERCENT COMPRESSION SET vs TIME of ACRYLIC RUBBER

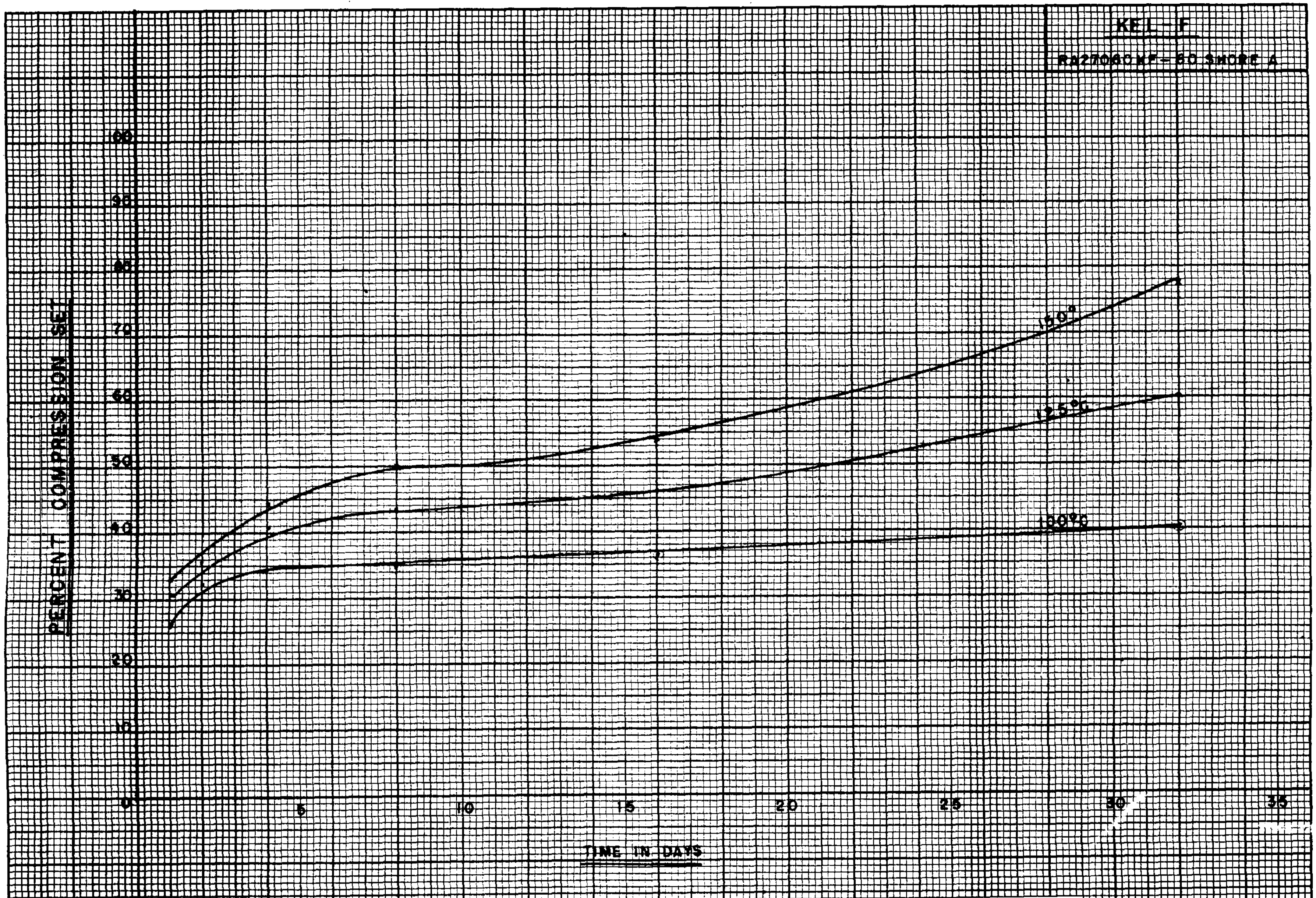


FIGURE 11 PERCENT COMPRESSION SET vs TIME of KEL-F RUBBER

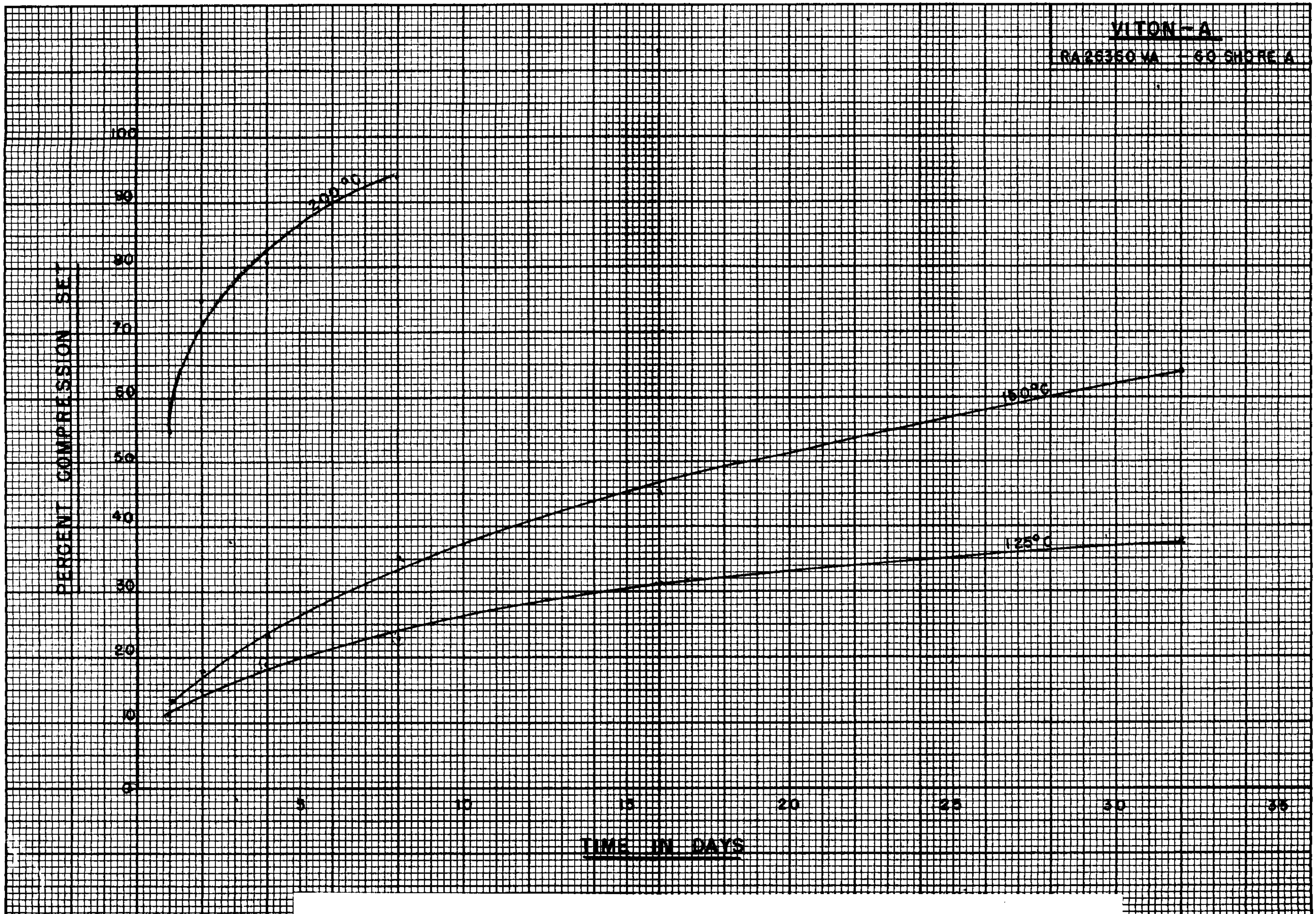


FIGURE 12 PERCENT COMPRESSION SET vs TIME of VITON-A RUBBER

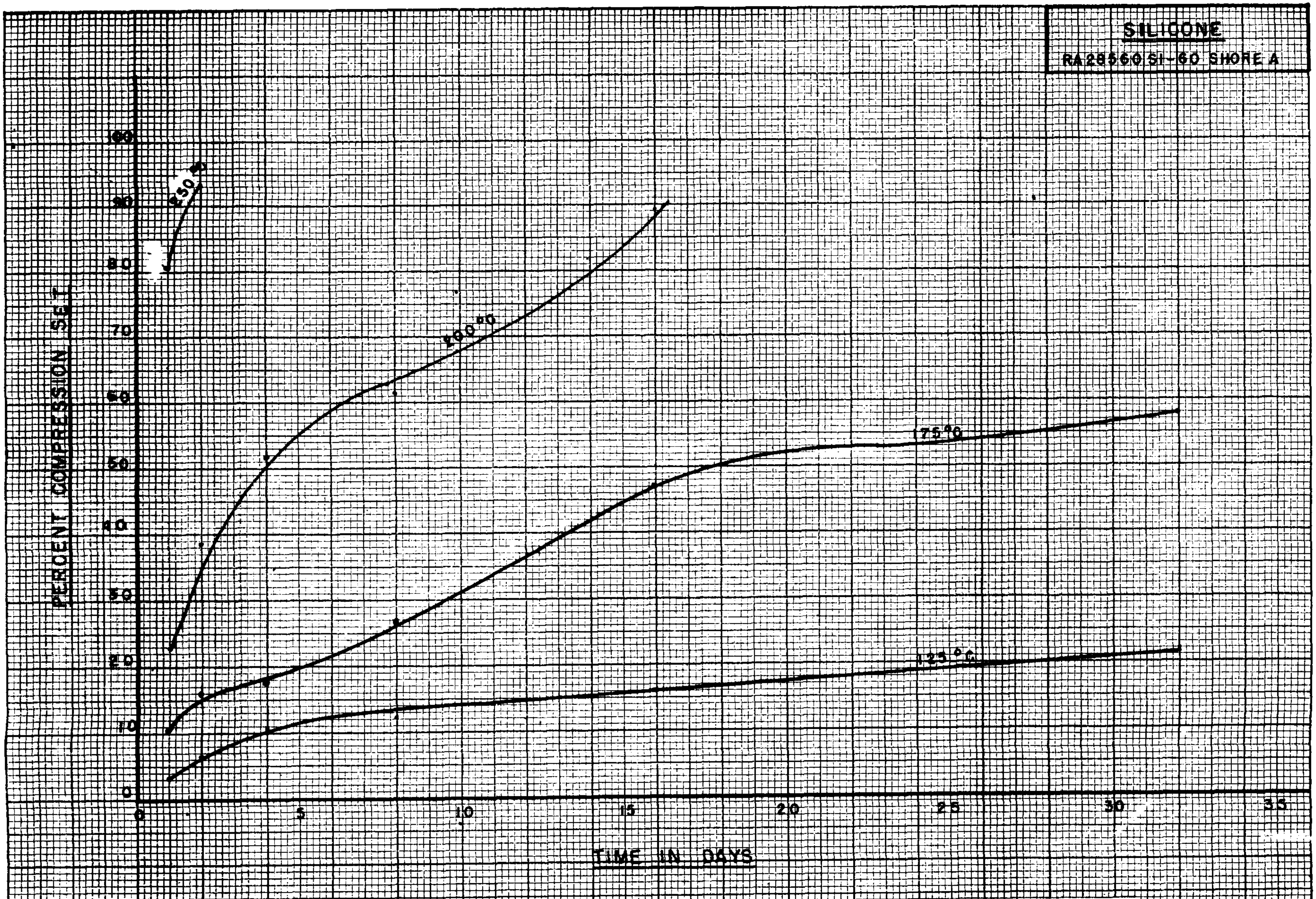


FIGURE 13 PERCENT COMPRESSION SET vs TIME of SILICONE RUBBER

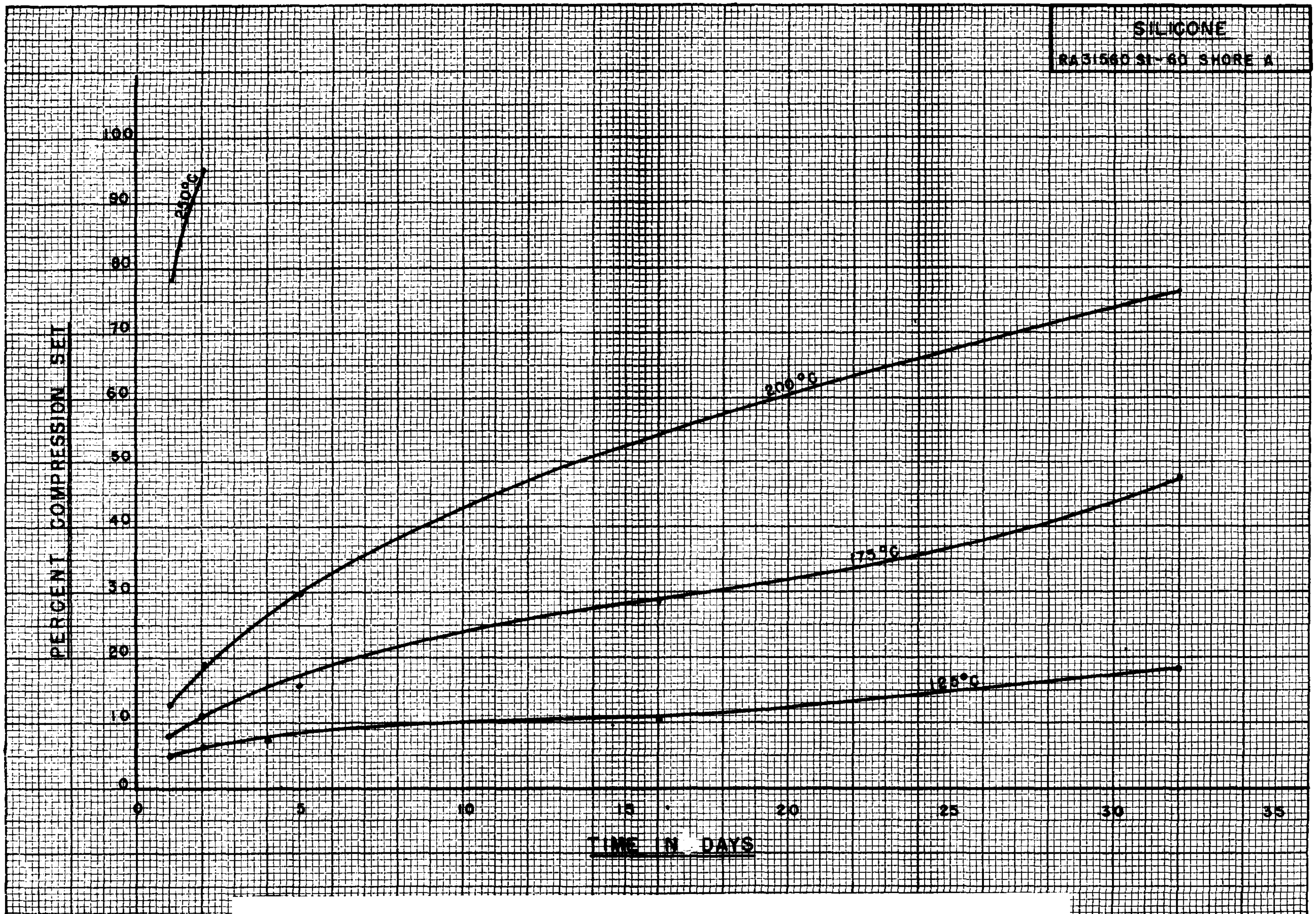


FIGURE 14 PERCENT COMPRESSION SET vs TIME of SILICONE RUBBER

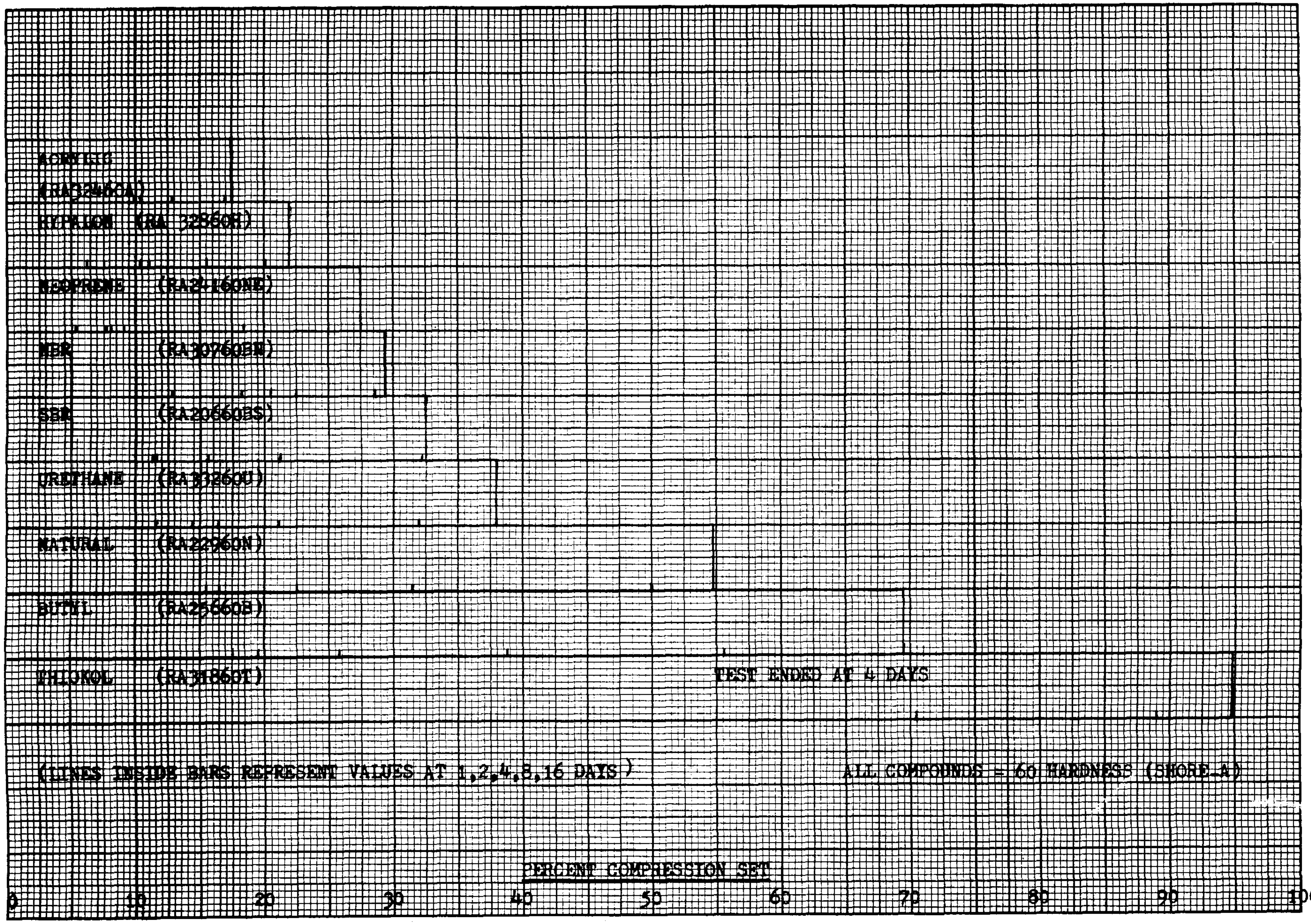


FIGURE 15 ULTIMATE COMPRESSION SET FOR 32 DAYS AT 70°C (9 ELASTOMERS)

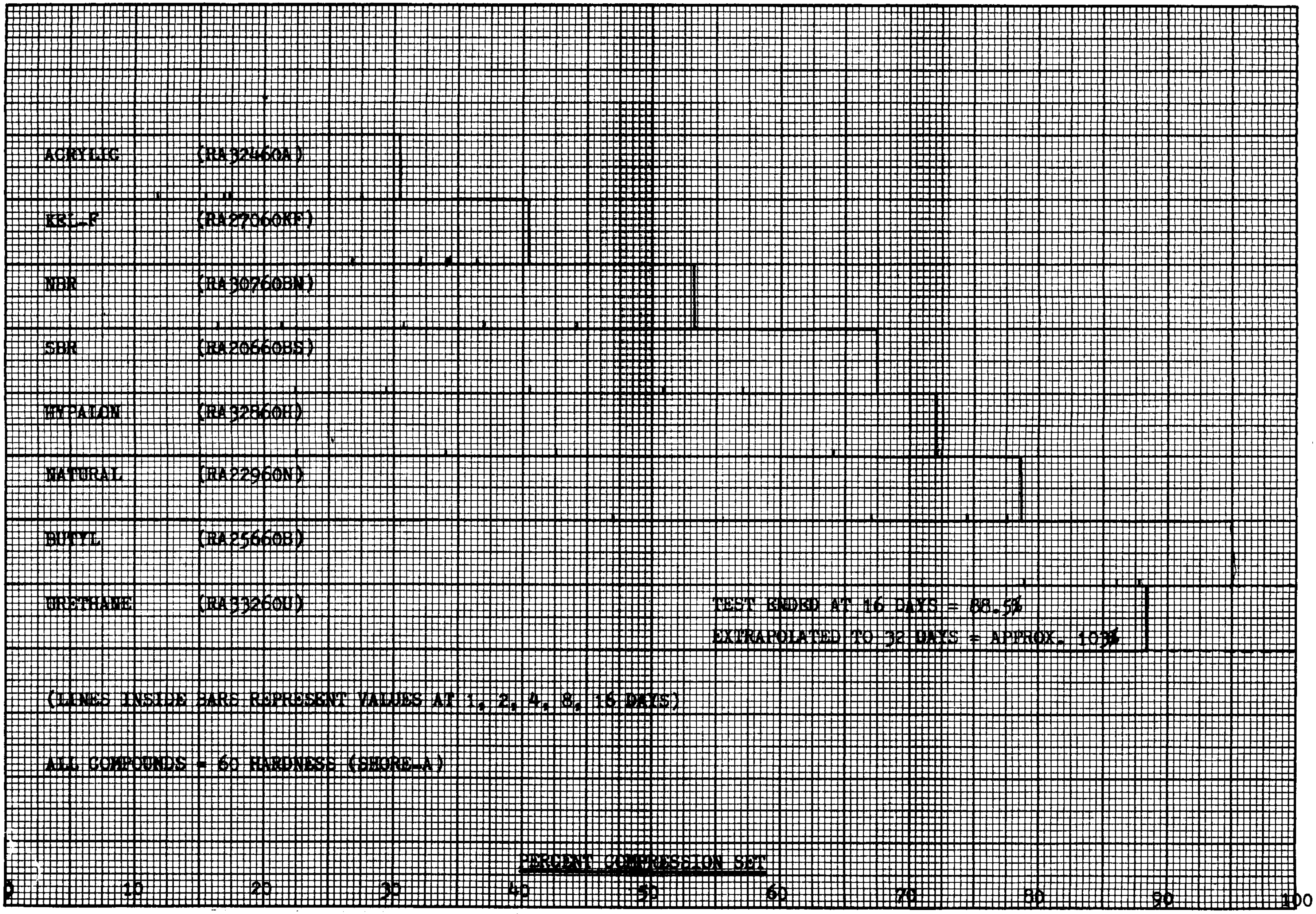


FIGURE 16 ULTIMATE COMPRESSION SET FOR 32 DAYS AT 100°C (8 ELASTOMERS)

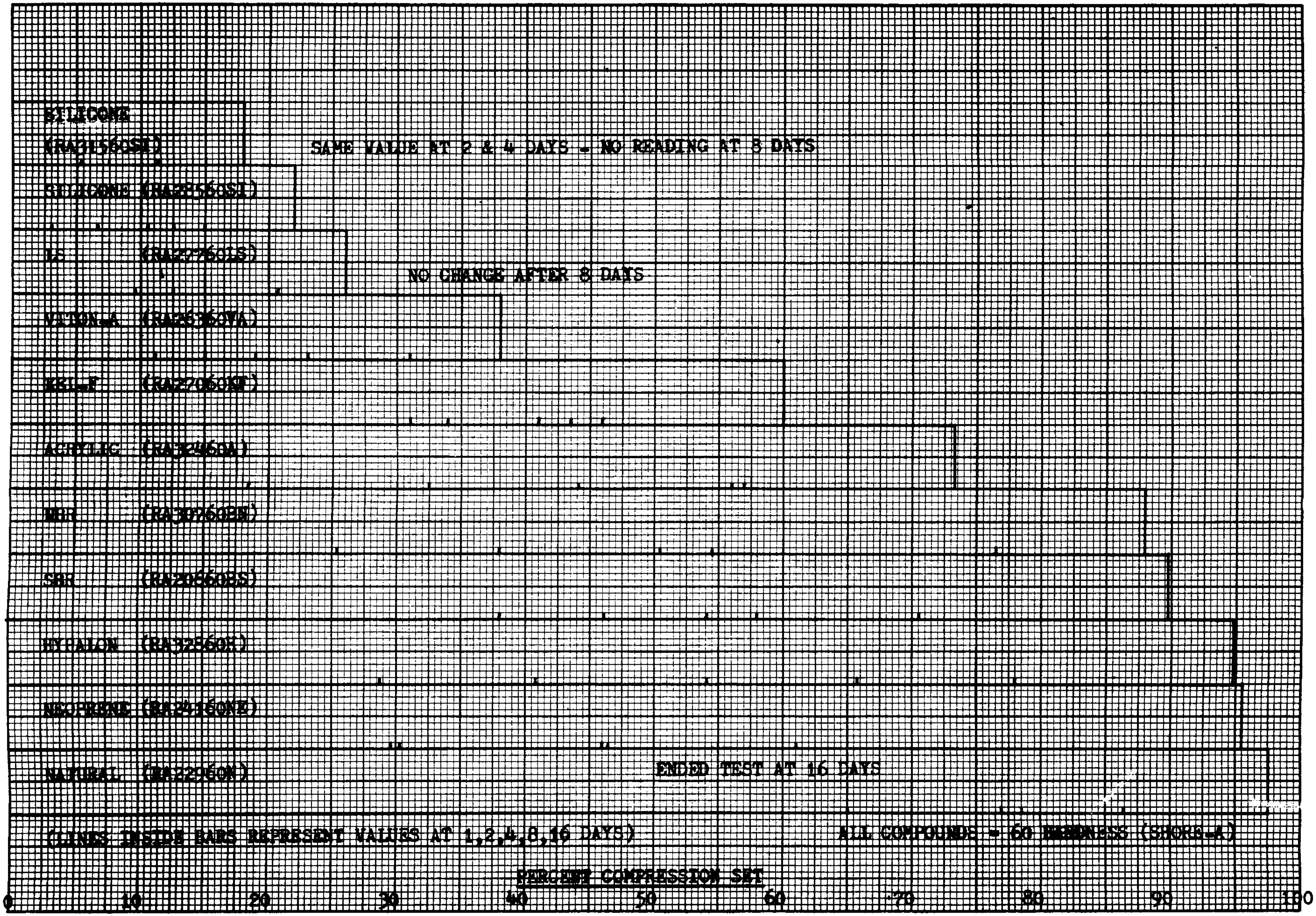


FIGURE 17 ULTIMATE COMPRESSION SET FOR 32 DAYS AT 125°C (11 ELASTOMERS)

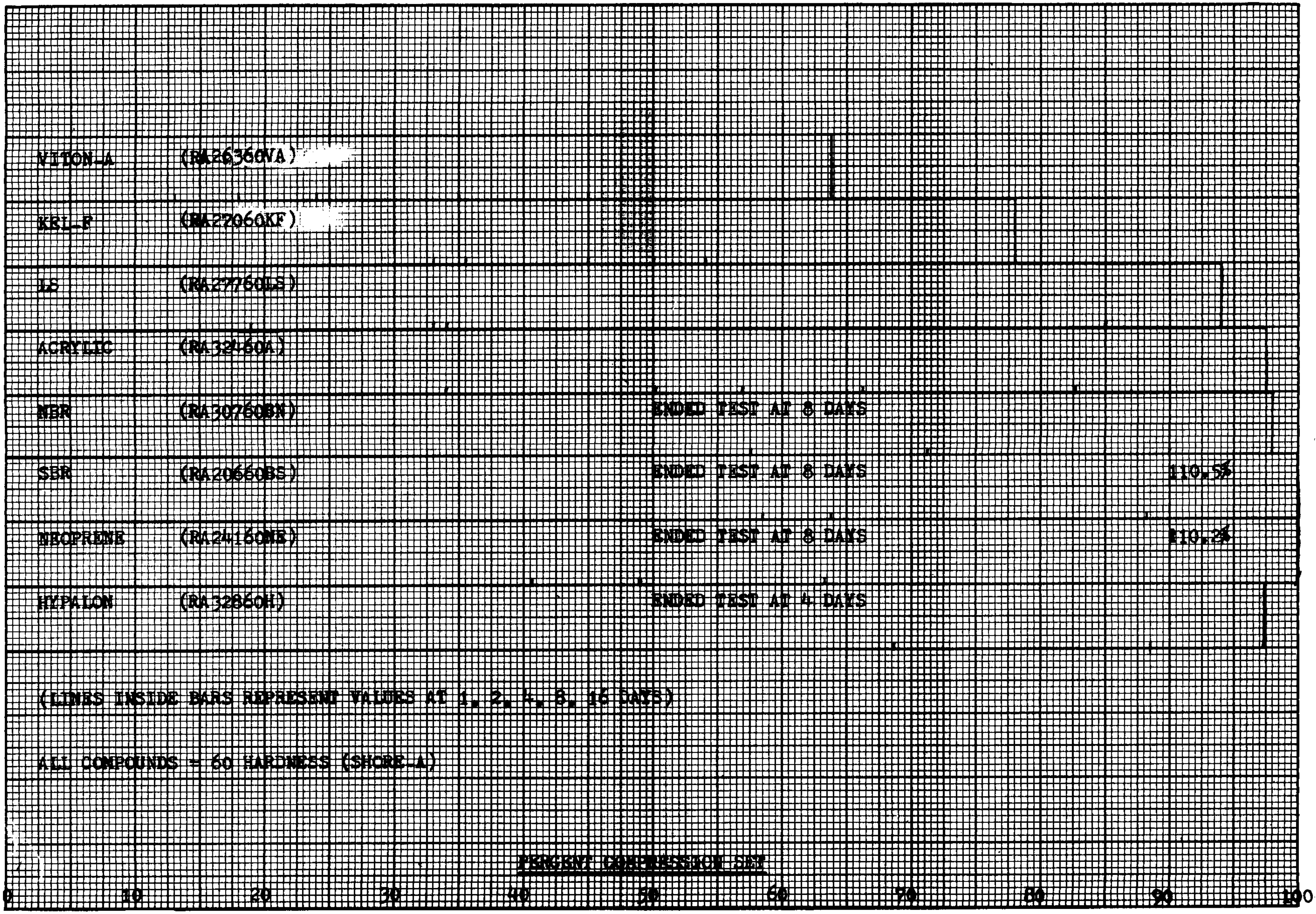


FIGURE 18 ULTIMATE COMPRESSION SET FOR 32 DAYS AT 150°C (8 ELASTOMERS)

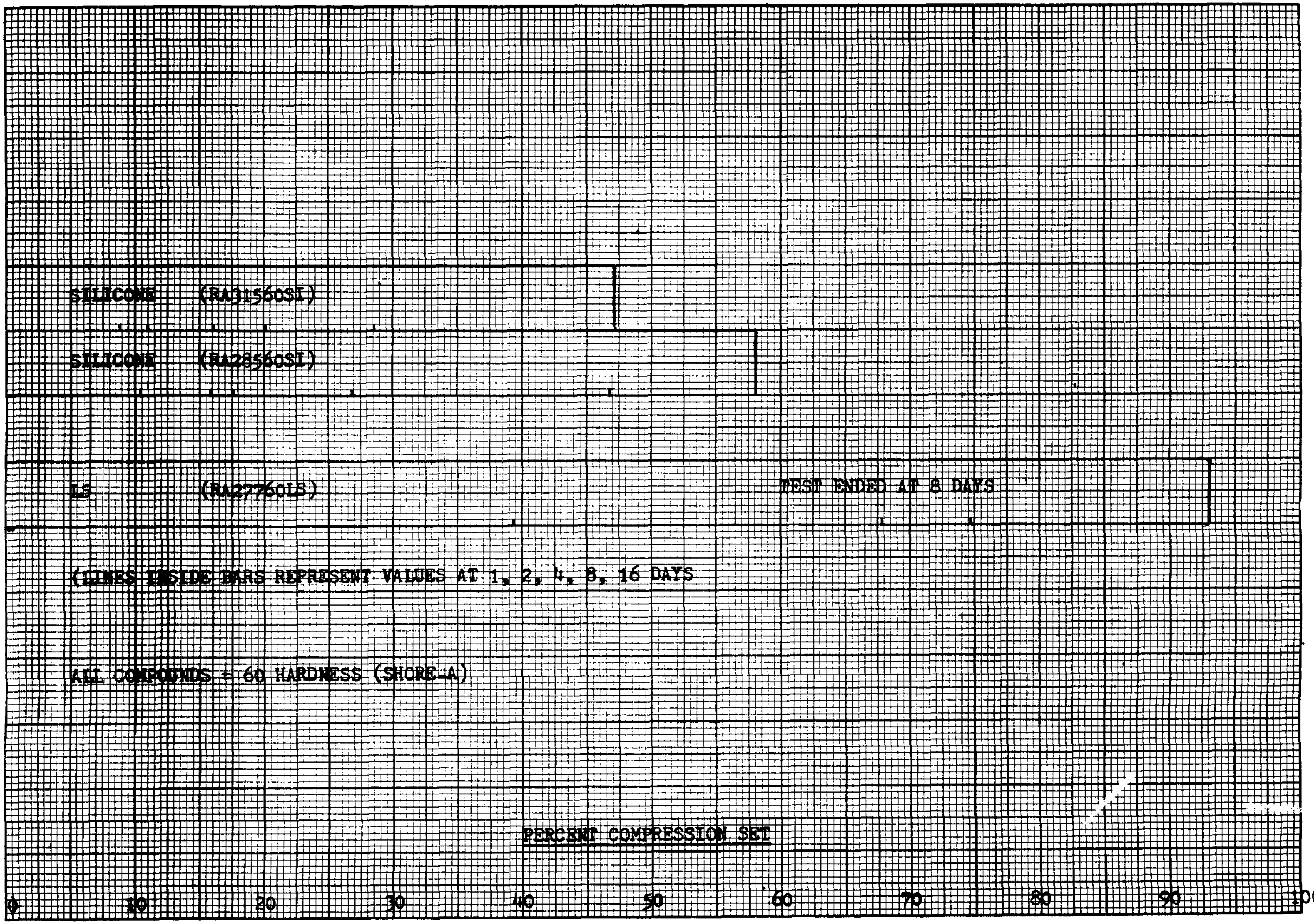


FIGURE 19 ULTIMATE COMPRESSION SET FOR 32 DAYS AT 175°C (3 ELASTOMERS)

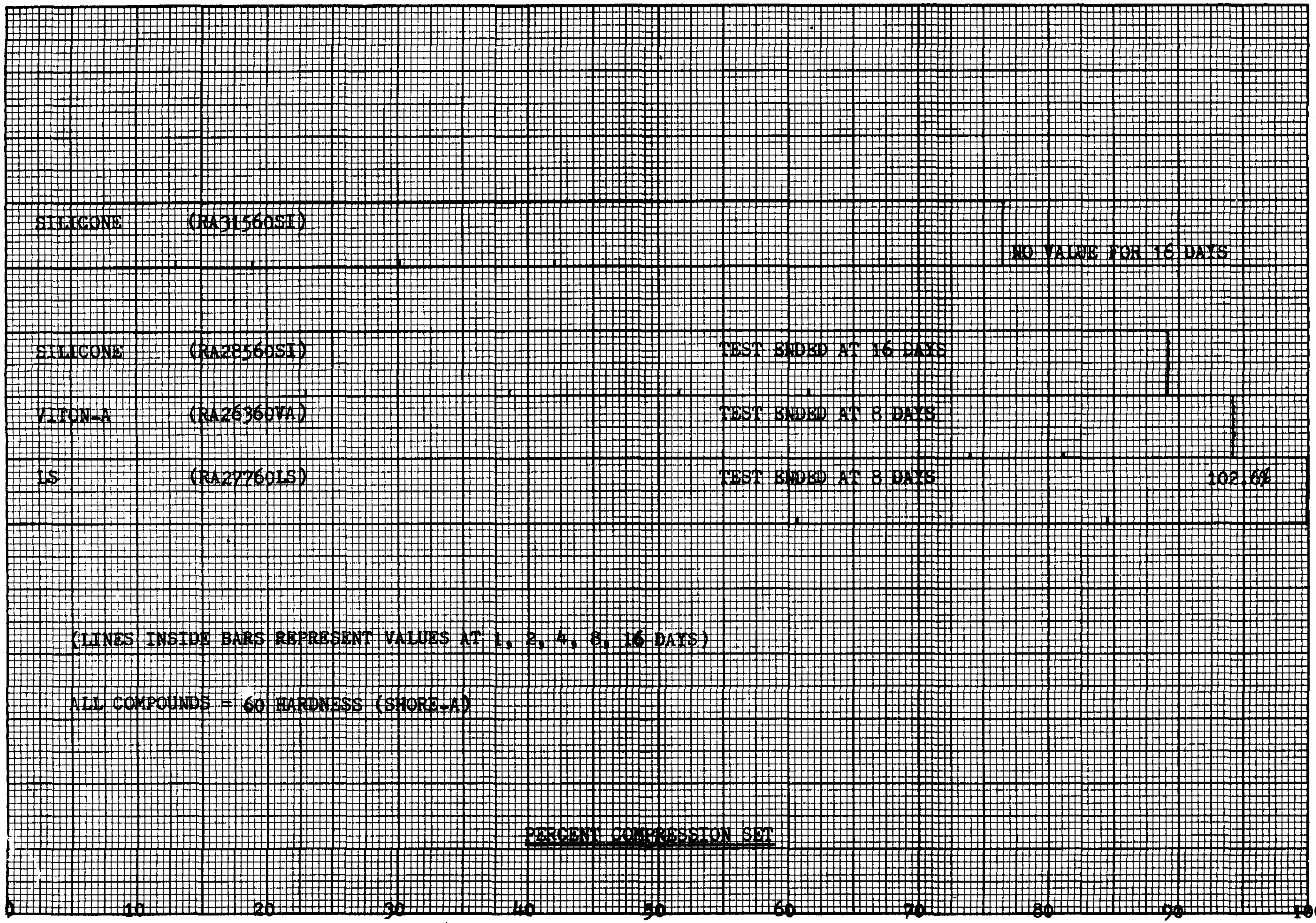


FIGURE 20 ULTIMATE COMPRESSION SET FOR 32 DAYS AT 200°C (4 ELASTOMERS)

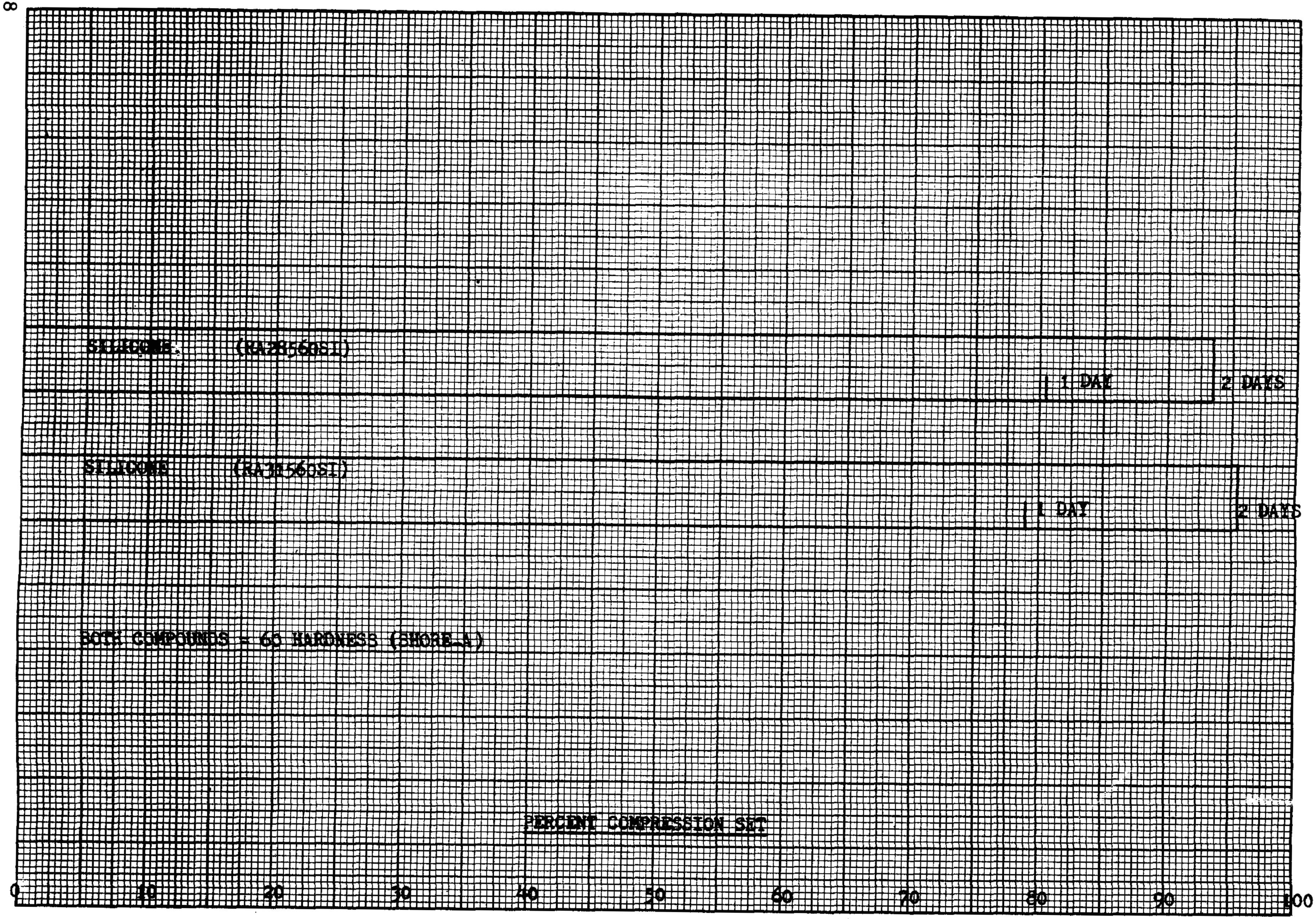


FIGURE 21 ULTIMATE COMPRESSION SET FOR 32 DAYS AT 250°C (2 ELASTOMERS)

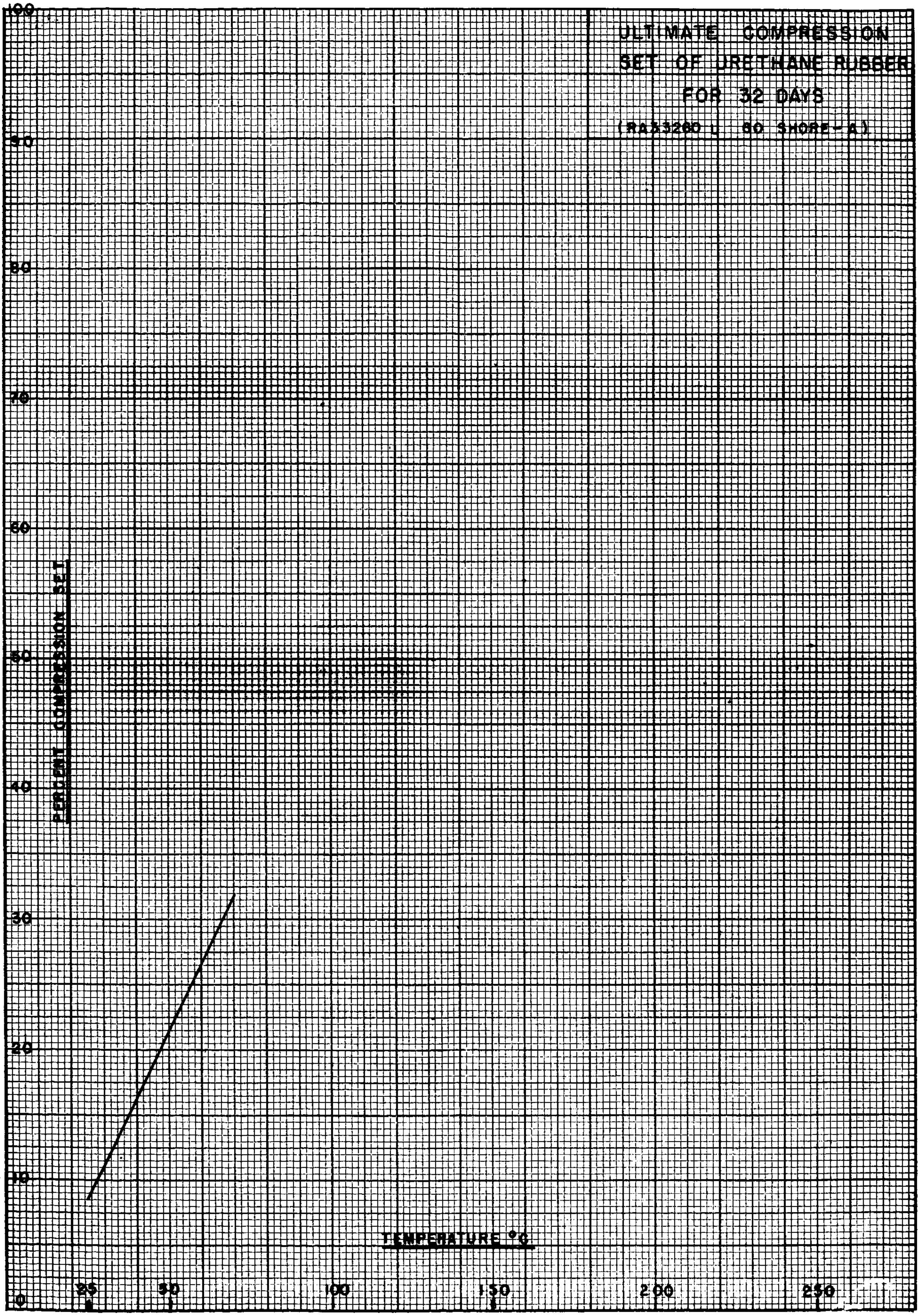


FIGURE 22 ULTIMATE COMPRESSION SET OF URETHANE RUBBER FOR 32 DAYS

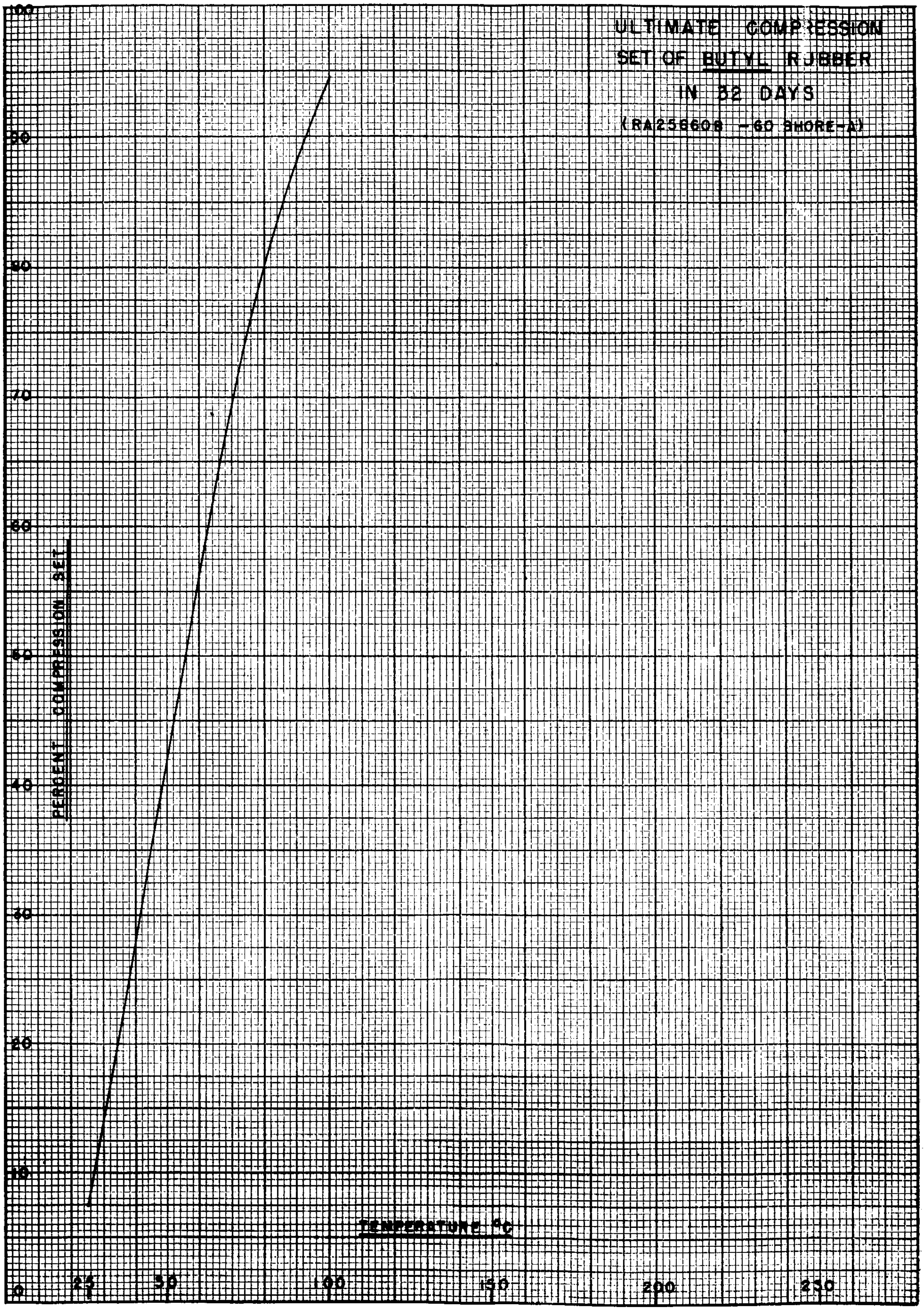


FIGURE 23 ULTIMATE COMPRESSION SET OF BUTYL RUBBER FOR 32 DAYS

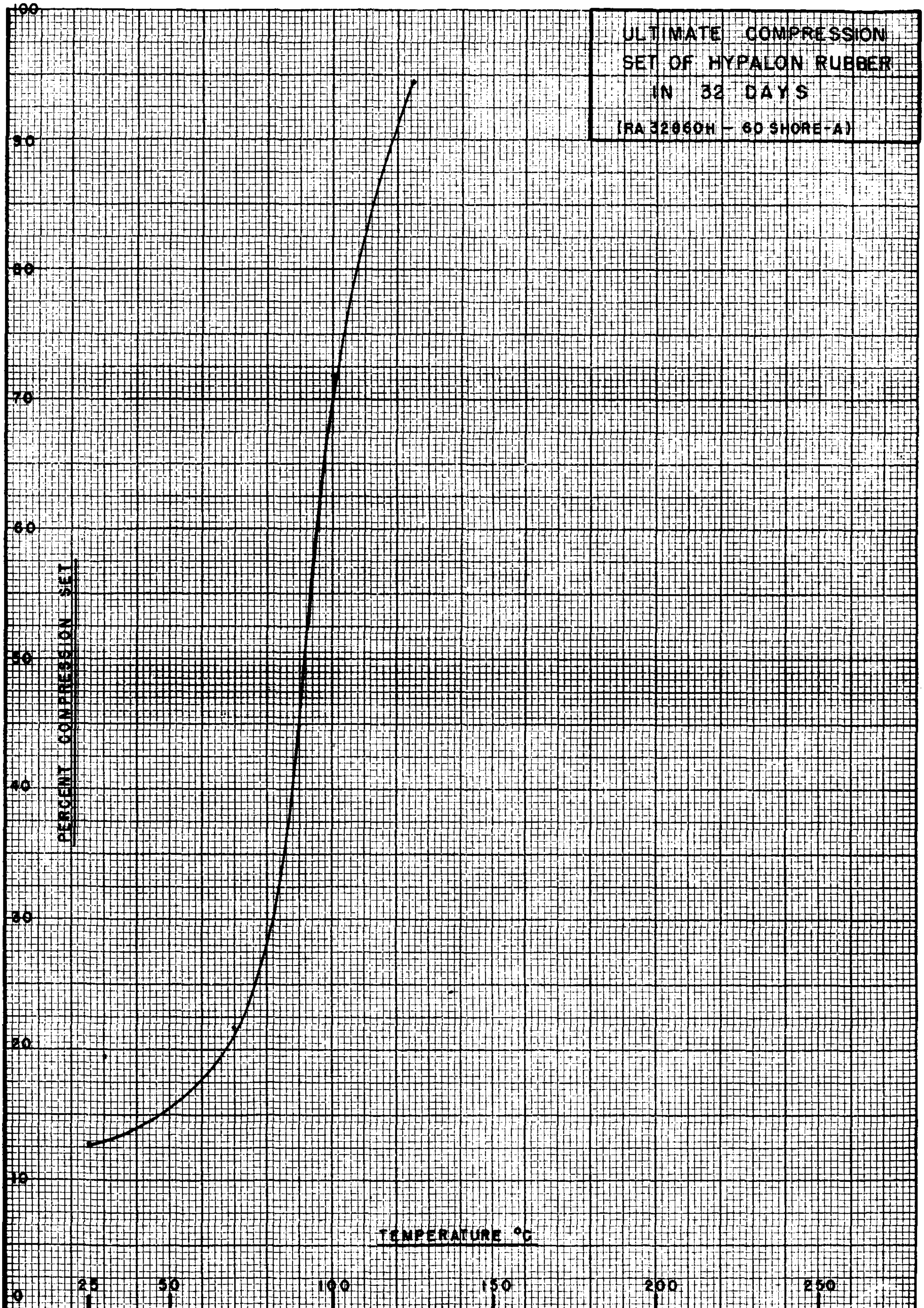


FIGURE 24 ULTIMATE COMPRESSION SET OF HYPALON RUBBER FOR 32 DAYS

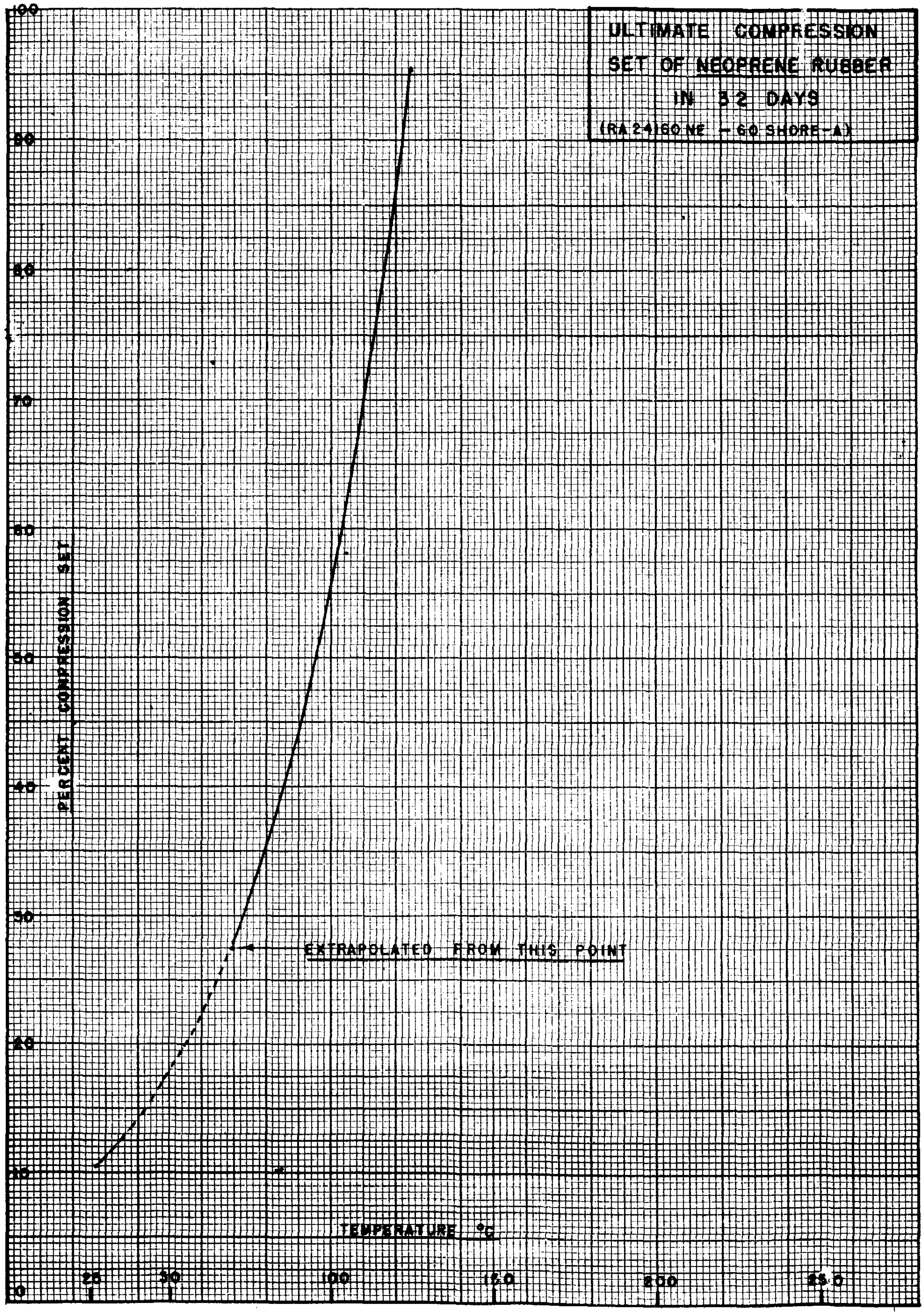


FIGURE 25 ULTIMATE COMPRESSION SET OF NEOPRENE RUBBER FOR 32 DAYS

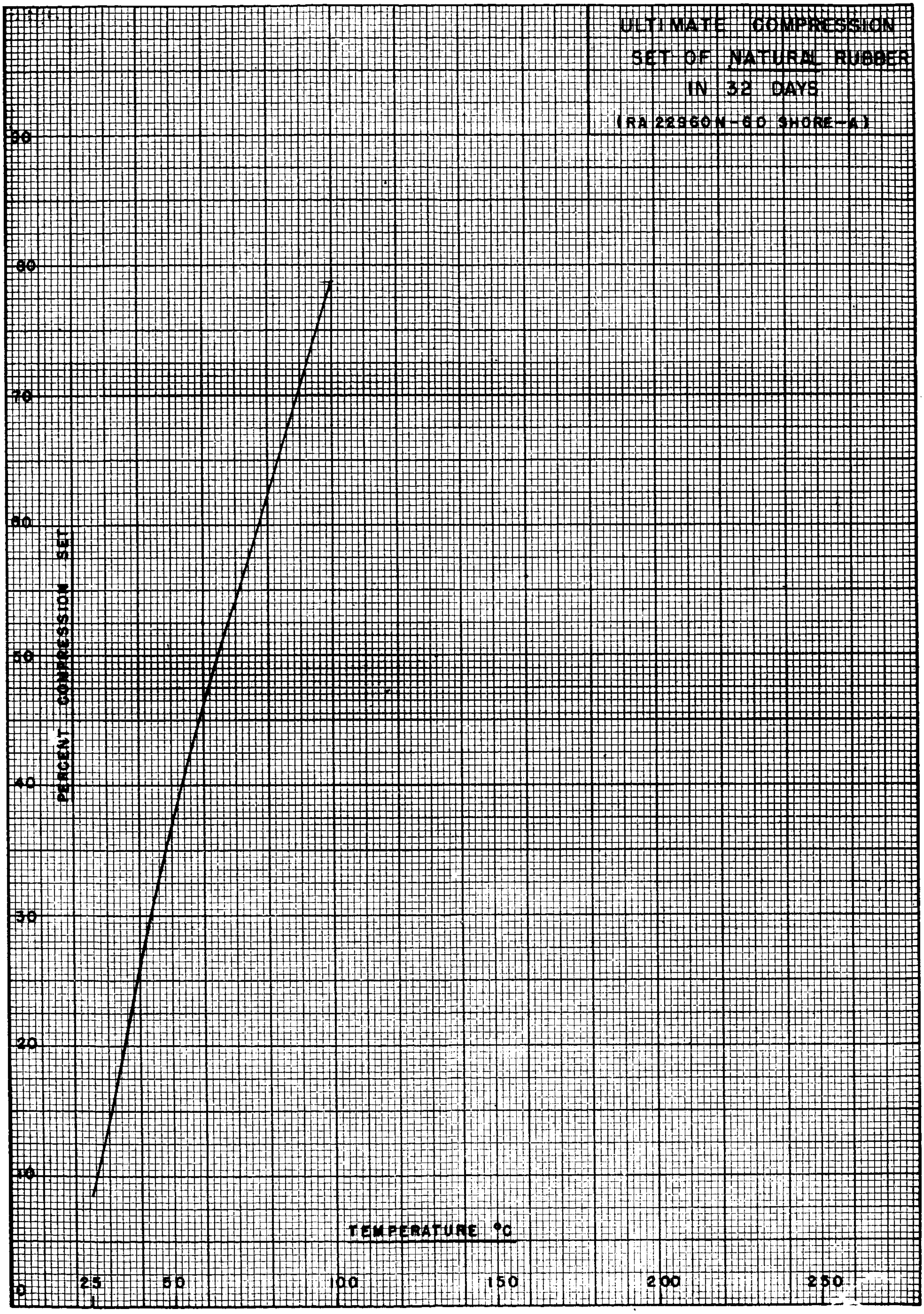


FIGURE 26 ULTIMATE COMPRESSION SET OF NATURAL RUBBER FOR 32 DAYS

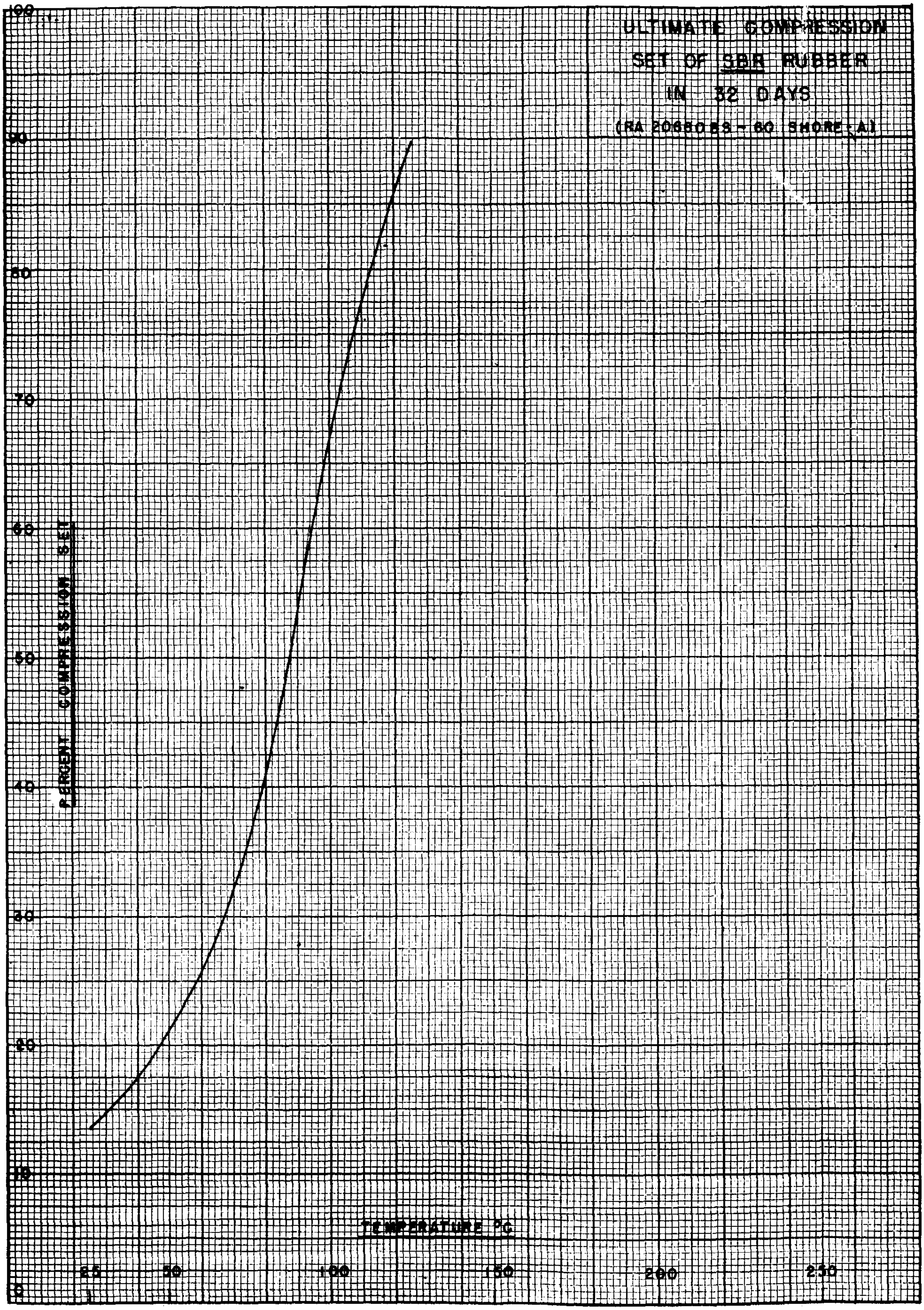


FIGURE 27 ULTIMATE COMPRESSION SET OF SBR RUBBER FOR 32 DAYS

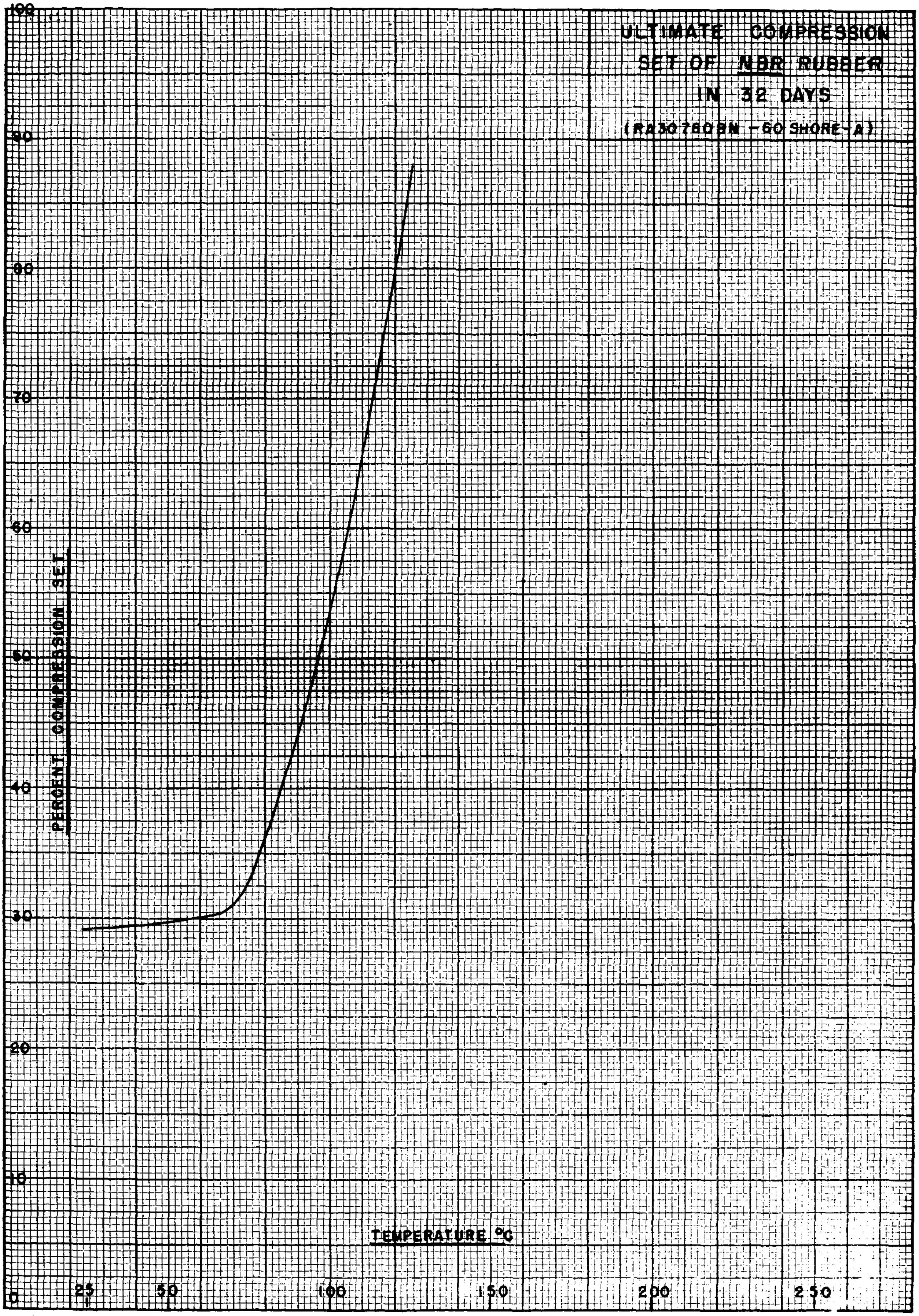


FIGURE 28 ULTIMATE COMPRESSION SET OF NBR RUBBER FOR 32 DAYS

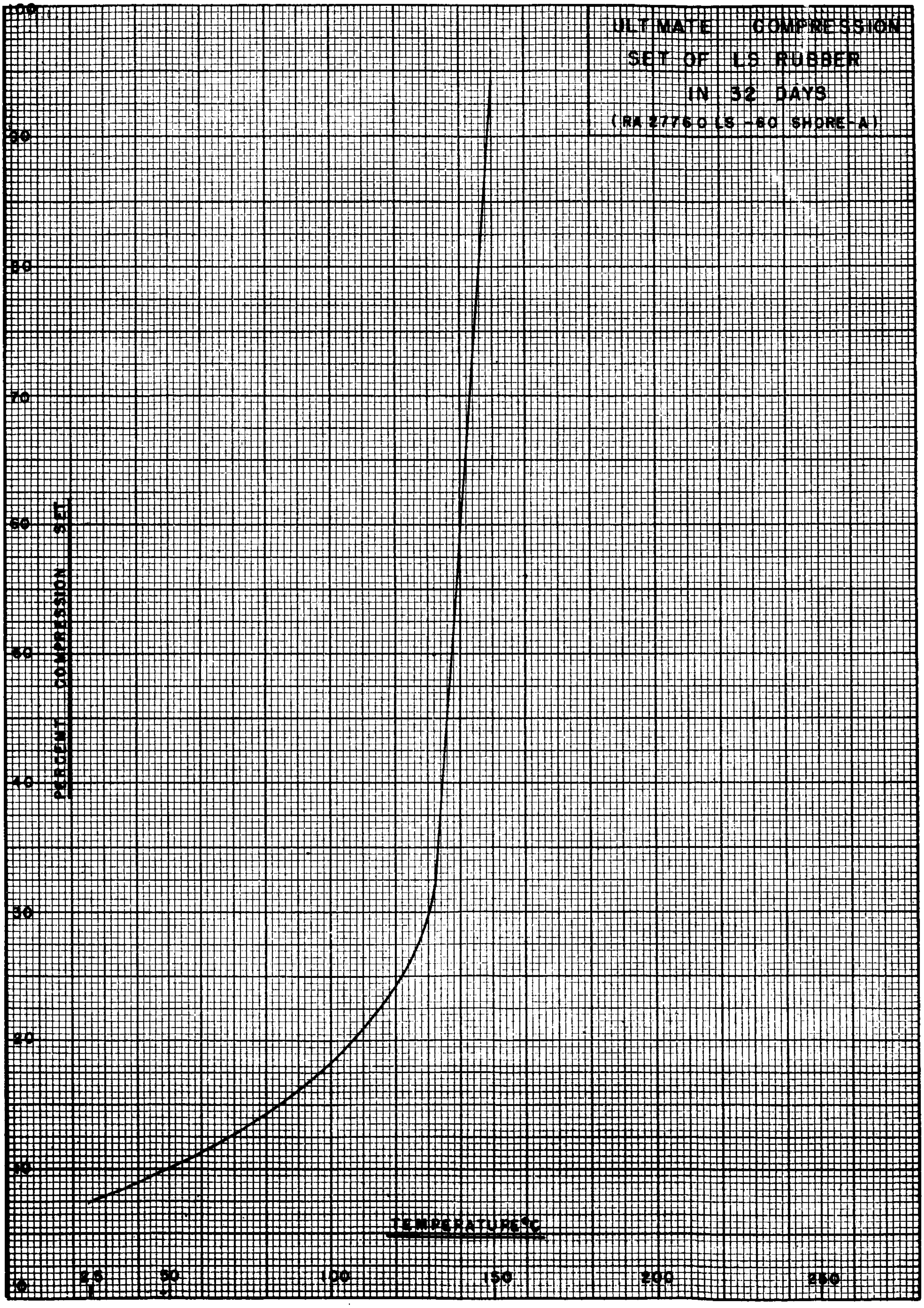


FIGURE 29 ULTIMATE COMPRESSION SET OF LS RUBBER FOR 32 DAYS

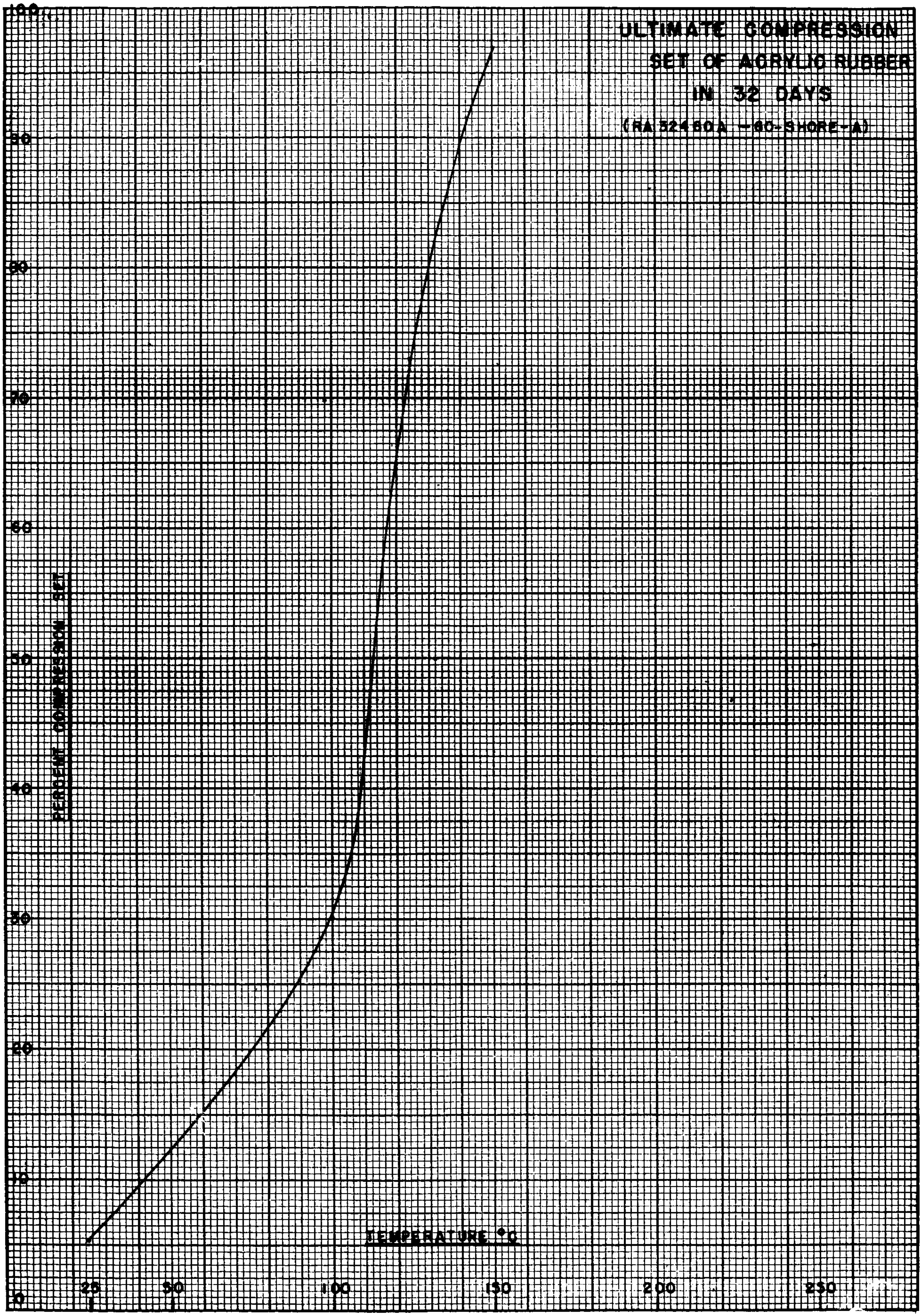


FIGURE 30 ULTIMATE COMPRESSION SET OF ACRYLIC RUBBER FOR 32 DAYS

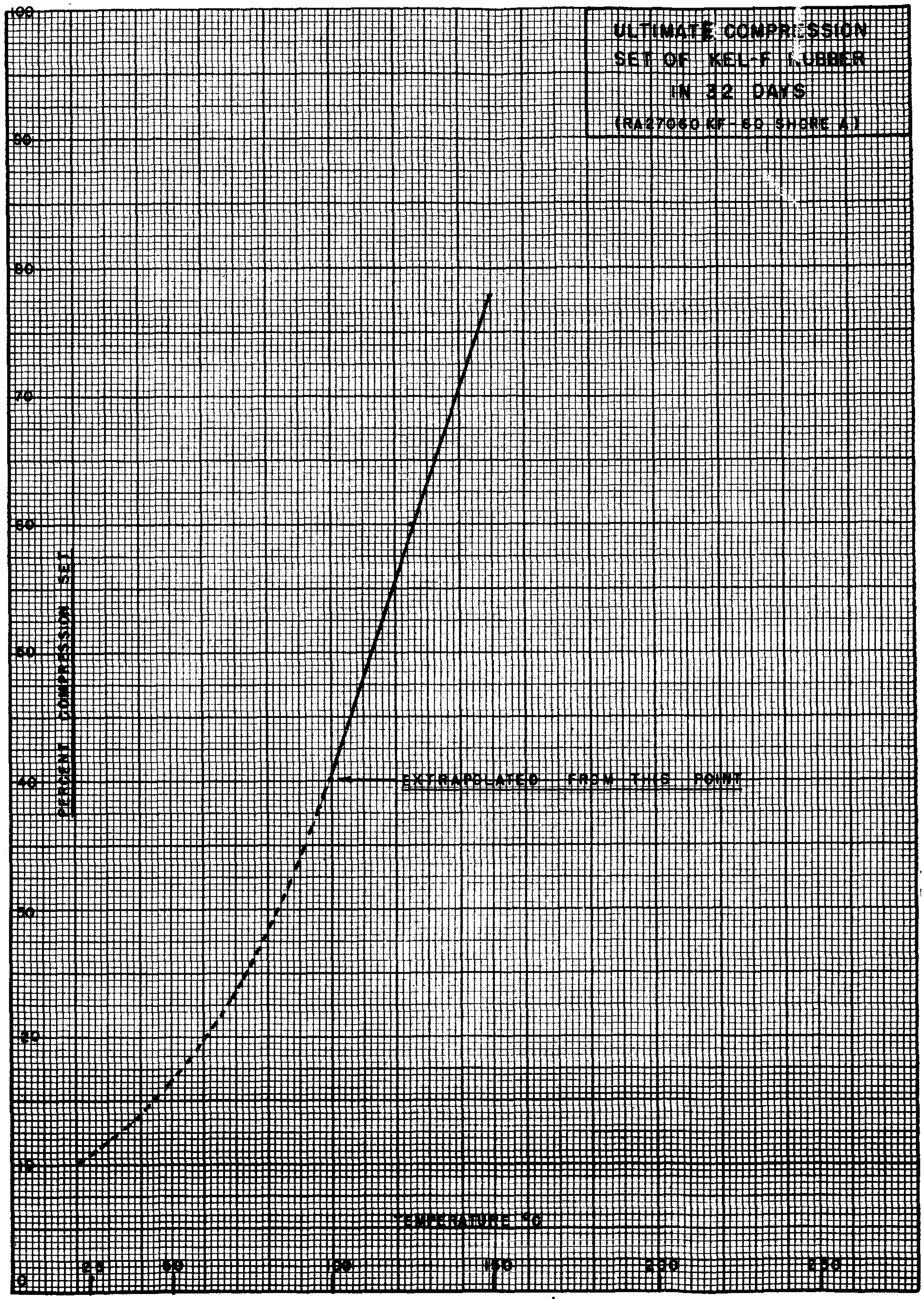


FIGURE 31 ULTIMATE COMPRESSION SET OF KEL-F RUBBER FOR 32 DAYS

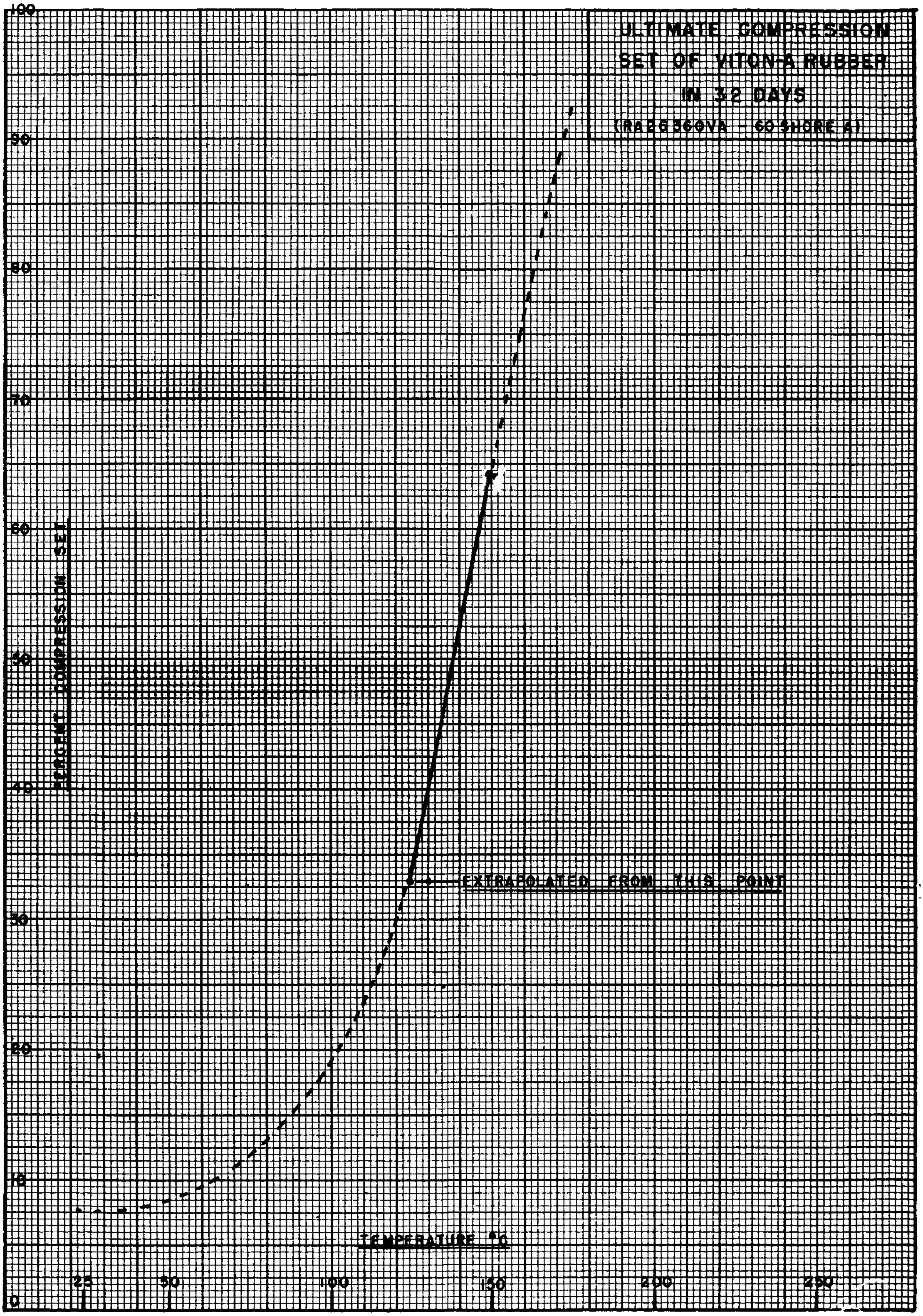


FIGURE 32 ULTIMATE COMPRESSION SET OF VITON RUBBER FOR 32 DAYS

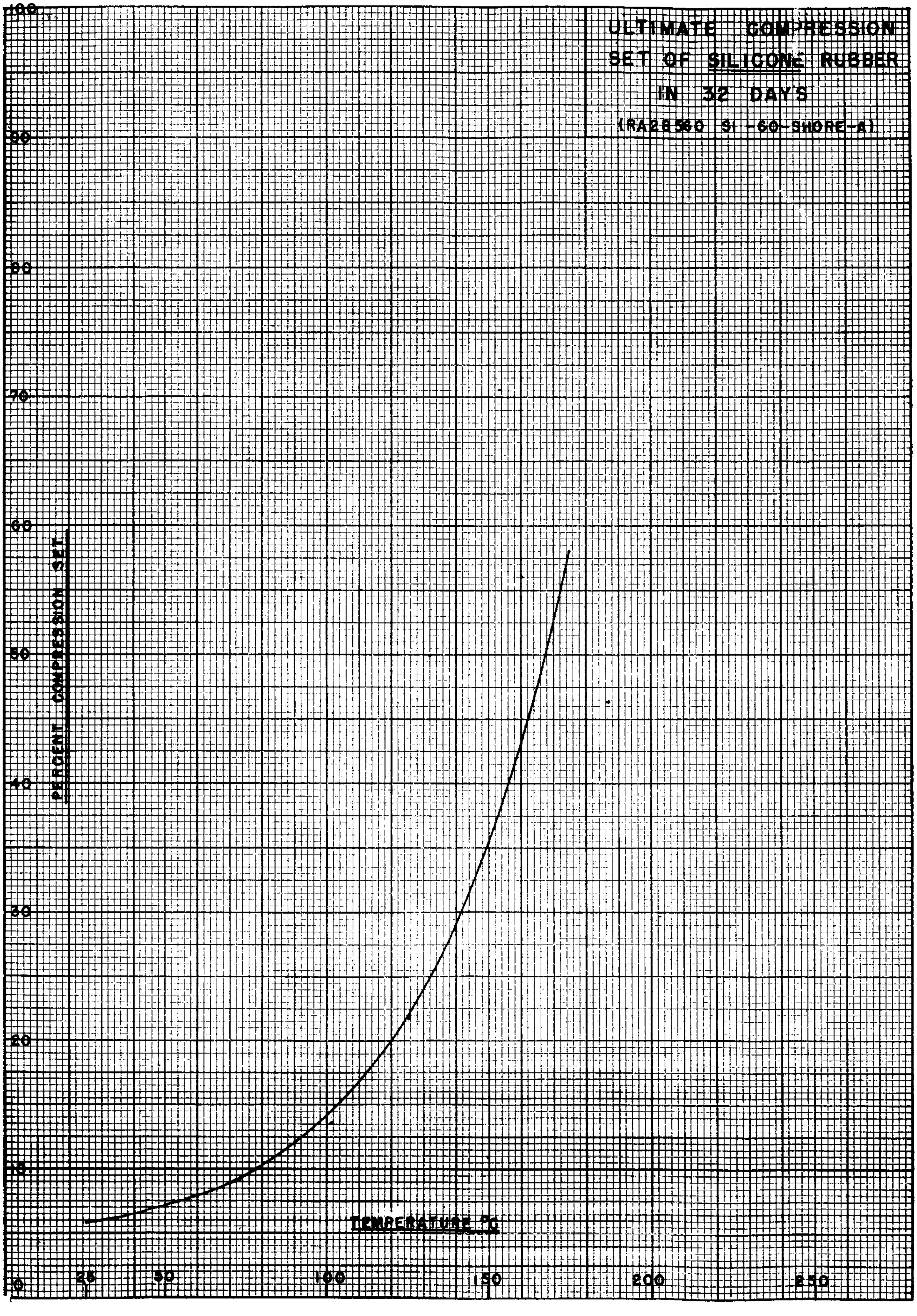


FIGURE 33 ULTIMATE COMPRESSION SET OF SILICONE RUBBER FOR 32 DAYS

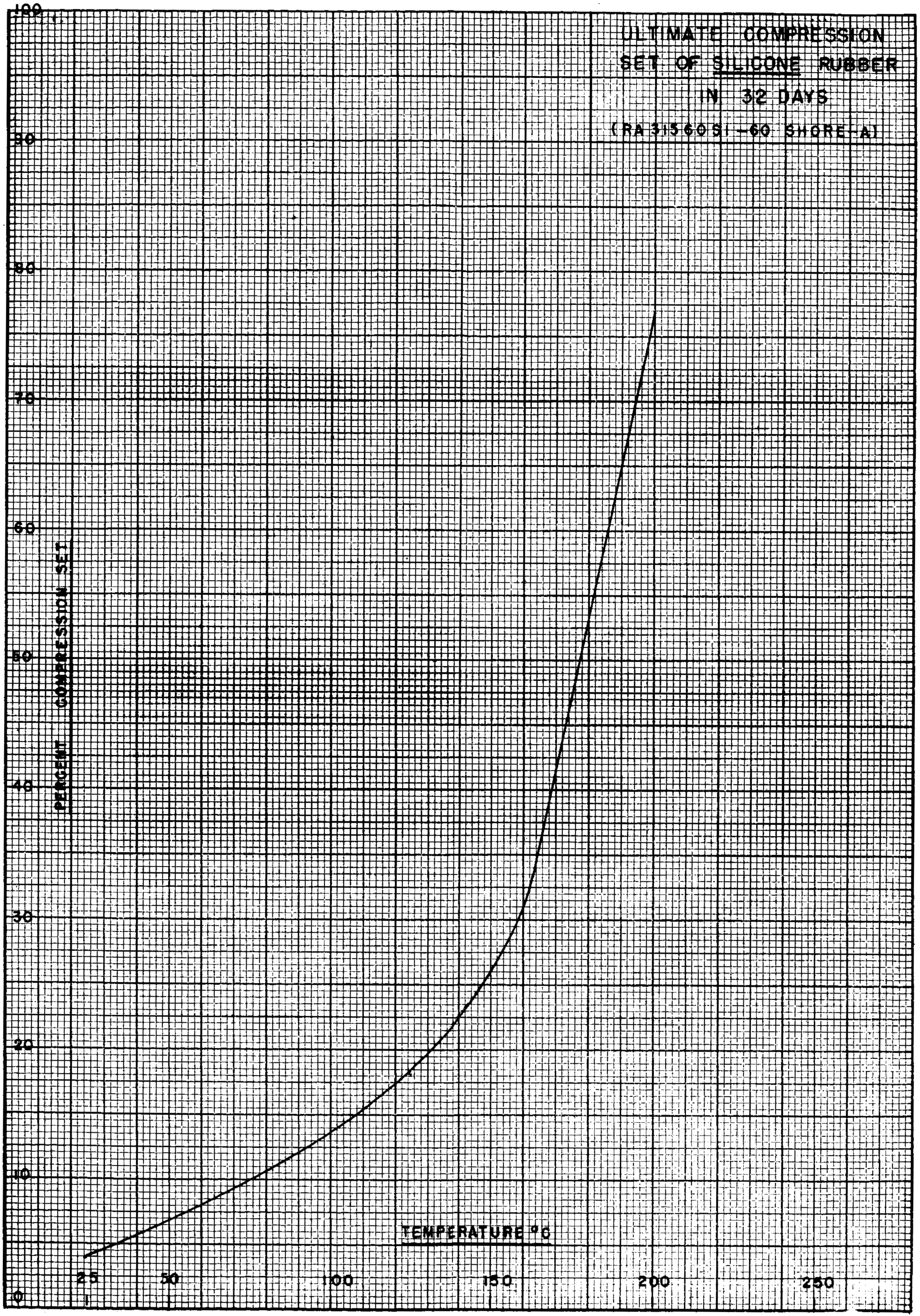
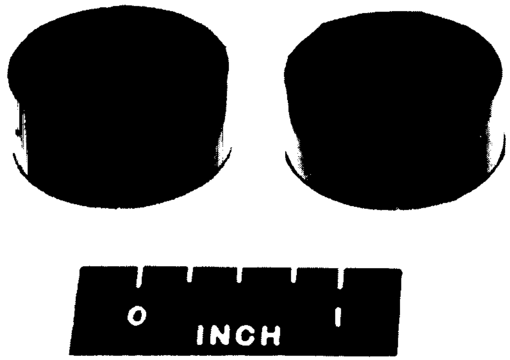
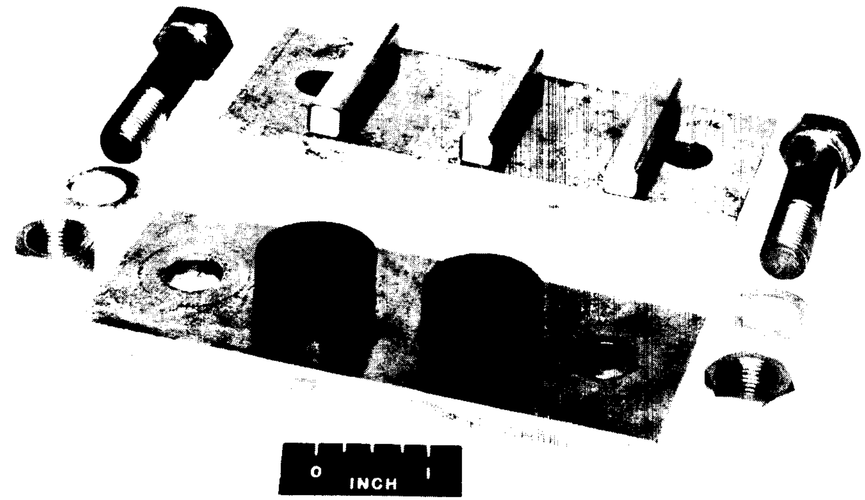


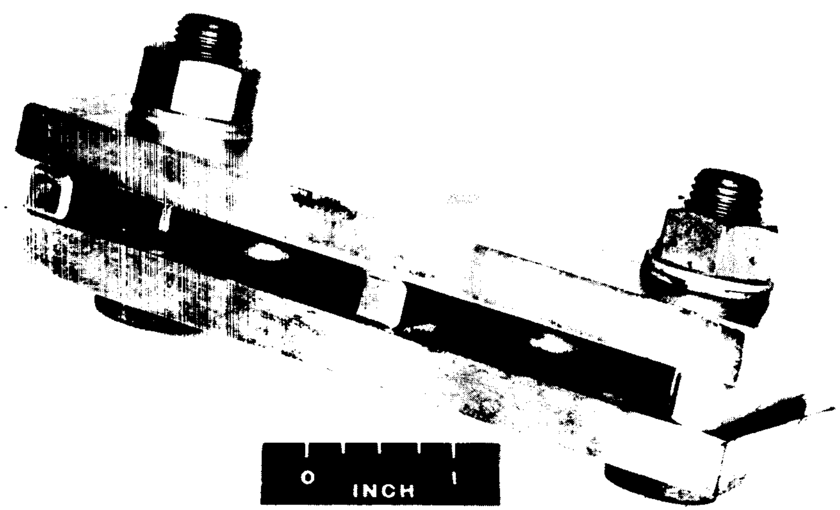
FIGURE 34 ULTIMATE COMPRESSION SET OF SILICONE RUBBER FOR 32 DAYS



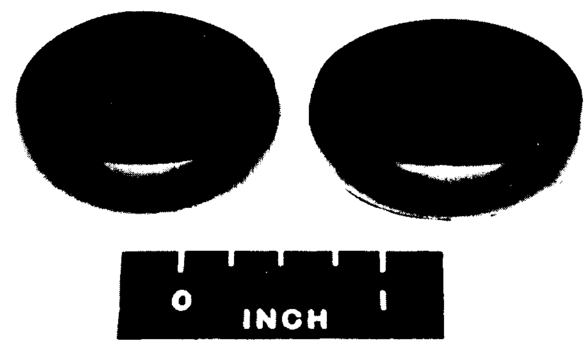
A. TEST SPECIMENS BEFORE COMPRESSION



B. PLATE ASSEMBLY WITH SPECIMENS AND SHIMS



C. SPECIMENS UNDER COMPRESSION



D. SPECIMENS AFTER COMPRESSION

FIGURE 35 - COMPRESSION SET FIXTURES

April 2, 1965 APPROVAL NASA TM X-53232

ACCELERATED COMPRESSION SET PROPERTIES OF FOURTEEN ELASTOMERS

By C. D. Hooper and J. T. Schell

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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