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COLLECTOR SEALANTS AND BREATHING

Final Report, September 25, 1978—December 31, 1979

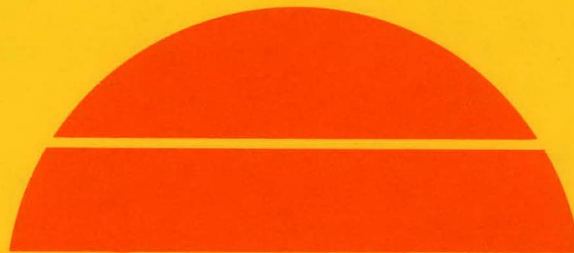
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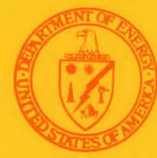
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Work Performed Under Contract No. AC04-78CS15362

Westinghouse Electric Corporation
Research and Development Center
Pittsburgh, Pennsylvania



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Solar Energy

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COLLECTOR SEALANTS AND BREATHING

FINAL REPORT

25 SEPTEMBER 1978 - 31 DECEMBER 1979

M. A. MENDELSON
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F. A. YEOMAN
F. W. NAVISH, JR.

FEBRUARY 20, 1980

WORK PERFORMED UNDER CONTRACT

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WESTINGHOUSE ELECTRIC CORPORATION
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ABSTRACT

An extensive literature survey followed by laboratory screening tests was used to select candidate Class PS (preformed rubber seals) and Class SC (sealing compounds or caulks) elastomers for more comprehensive laboratory testing to assess their performance capabilities under the harsh environment of a thermal solar collector cell. Tests employed included thermal aging in air, hydrolytic stability, weathering resistance, corrosion of metallic substrates, ozone resistance and fungal susceptibility. Since the most hostile factor in the solar collector cell environment is long-term exposure to elevated temperatures in air, the thermal stability testing was extended to the monitoring of change in several physical properties during thermal aging in air. These included compression set, tensile characteristics, hardness and weight loss.

Although none of the PS elastomers tested was found to be entirely satisfactory, the fluorocarbon (Viton) displayed the best durability and thermal stability overall. The silicones were second best. Unfortunately, the fluorocarbons tend to exhibit excessive low temperature compression set, a characteristic which could be a serious problem in geographic zones having relatively cold winters. The silicones show very poor resistance to compression set on thermal aging and, while the fluorocarbon is considerably superior in this respect, it nevertheless displays undesirably high values.

The polyacrylate and acrylic copolymers and one of the ethylene-propylene terpolymers (EPDM - Nordel) were the best of the intermediate temperature elastomers. Except for resistance to compression set, these materials were inferior to the silicones in thermal stability. The other EPDM compounds and butyl rubber were considerably inferior to the three compositions just mentioned.

The only Class SC compositions which retained moderate physical integrity on thermal aging were the silicones. Outgassing studies showed that, as a group, the Class SC elastomers generate considerably more volatiles (and condensables) during thermal aging than do the Class PS elastomers.

Five collectors, which had been in service at three different locations, were examined with regard to their design and quality of fabrication. Two of the collectors were poorly sealed and three were moderately to well-sealed. Problems associated with the collectors consisted of corrosion, glaze deposits, and degradation of sealants and absorber plate coatings. Only one of the collectors appeared to be in very good condition.

The use of an externally attached bladder to compensate for breathing caused by pressure-temperature change is feasible as long as considerable space is available underneath the collector. This approach will not solve problems associated with organic vapors from the sealants.

Silica gel and activated carbon appear to provide the most efficacious adsorbents for removing moisture and organic vapors, respectively. A duo-adsorbent system utilizing a combination of both materials would provide a superior means for removal of water and the organic volatile outgassing products of the sealants.

1.0 SUMMARY

1.1 Literature Survey of Physical and Aging Properties of Sealants (Task 4.1)

An extensive literature survey designed to summarize the properties of all categories of commercially available elastomers was completed. This survey was concentrated on characteristics which are considered pertinent to long-term durability and to cost effectiveness in the flat-plate solar collector application. Information developed indicated that silicone and fluorocarbon elastomers were the most viable high temperature rubbers. Fluorosilicone and phosphonitrilic rubbers were eliminated on the basis of high cost.

Candidates in the somewhat lower temperature service category ($\sim 125^{\circ}\text{C}$) consisted of polyacrylic, ethylene propylene terpolymer (EPDM), ethylene acrylic, butyl, chlorosulfonated polyethylene and epichlorohydrin.

The literature survey included computer printouts incorporating several hundred references. Under separate cover, the assemblage was submitted to the Solar Heating and Cooling Research and Development Branch, Office of Conservation and Solar Applications, Los Alamos Scientific Laboratory, U.S. Department of Energy.

1.2 Evaluation by Supplemental Laboratory Tests (Task 4.2)

A series of Class PS (extrusion grade gasket type sealants) elastomers representative of each category chosen from results of the literature survey or because of extensive use in the solar collector cell industry was evaluated by selected tests taken from National Bureau of Standards NBSIR 77-1437 and ANSI/ASTM D-3667-78, Standard Specification for Rubber Seals Used in Flat-Plate Solar Collectors. Three silicones, three EPDM rubbers, two fluorocarbons, three epichlorohydrin rubbers, one ethylene acrylic elastomer (VAMAC), one polyacrylic rubber, one

chlorosulfonated polyethylene, one bromobutyl rubber and two butyl rubbers were studied in these screening tests. Properties measured in these tests included compression set after 70 hr at 150°C, compression set after 166 hr at -10°C, ultimate elongation, tensile strength and hardness and changes in these three properties after aging 166 hr at 150°C and volatiles lost during the latter aging period.

As a consequence of this screening, three silicones, one fluorocarbon, one ethylene propylene terpolymer, one ethylene acrylic, one polyacrylic and one butyl rubber were selected for more extensive testing.

Since our market survey disclosed only a very limited number of viable candidates in the Class SC (caulks) category, comparable screening tests were not performed in this area. Six candidates were made available for the more extensive testing. Three of these were silicones, one an acrylic, one a butyl and one a chlorosulfonated polyethylene. Following the screening tests, the candidates selected were subjected to more extensive testing in the following test categories. A very brief summary of test results is presented under each category.

1.2.1 Compression Set

Change in compression set of Class PS elastomers was monitored during thermal aging. This property is essentially a measure of the ability of an elastomer to maintain a tight seal as a gasketing material by retention of its resilience. Low test values are desirable. We found compression set retention of all elastomers during thermal aging to be disappointingly poor in terms of actual thermal stresses which may be encountered in the solar collector cell application. The fluorocarbon elastomer exhibited by far the best performance, but is still regarded as being deficient for the temperature requirements of the application. Despite its clear superiority in the high temperature compression set testing, performance of the fluorocarbon on the low temperature compression set test included in the screening test was poorer than that of nearly

all materials tested. The silicones were particularly disappointing in the high temperature compression set tests.

1.2.2 Corrosion

Elevated temperature corrosive properties of the several elastomers were observed by clamping sheets of the elastomers between metal plates of galvanized iron, mill finish aluminum or anodized aluminum and observing changes of the metal surfaces as thermal aging progressed. None of the materials tested attacked anodized aluminum and only the Class PS butyl attacked mill finish aluminum. Galvanized iron was attacked by Class PS butyl, Class PS EPDM (Nordel), Class PS acrylic and Class PS Hypalon.

1.2.3 Thermal Aging of Tensile, Weight Loss and Hardness Specimens

Thermal aging of compression set specimens in air as already discussed constitutes an important criterion of thermal stability in air. Since retention of properties during thermal aging in air is of such vital importance, further testing was carried out with both Class PS and Class SC materials. This consisted of aging tensile specimens and hardness and weight loss specimens in air and monitoring changes in these characteristics. It was found, not surprisingly, that the best overall performer in these tests was the fluorocarbon rubber followed by the silicone elastomers. There were considerable differences within the group of silicones, however. The intermediate temperature Class PS polymers, polyacrylic, ethylene acrylic and EPDM comprise a group whose performances are rather similar, but inferior to the silicones. The acrylics outperform the EPDM in terms of retention of tensile properties during thermal aging, but weight loss and hardness data show performances of all three to be roughly comparable. The butyl rubbers (both Class PS and Class SC) are clearly inferior to all other compositions tested. The Class SC caulks lose their elastomeric properties, as indicated by loss of elongation, quite rapidly during aging at relatively low temperatures.

1.2.4 Hydrolytic Aging of Tensile and Adhesion-in-Peel Specimens

Resistance of elastomers to degradation in hydrolytic environments was studied by aging of tensile specimens of both Class PS and Class SC materials under total water immersion at a series of aging temperatures and intervals and by aging adhesion-in-peel specimens of the Class SC compositions under similar conditions. In the tensile tests, performance of the EPDM (Nordel) was excellent and that of the fluorocarbon (Viton) quite good. Butyl rubber was also good, but its chemical structure had led us to expect better performance than proved to be the case. Performance of the acrylic and ethylene-acrylic polymers (Hycar and Vamac) was somewhat inferior to that of the silicones, but better than had been expected owing to the presence of potentially hydrolyzable ester groups in these materials. The silicones ranged from fair to good in performance but were nevertheless considered disappointing.

Adhesion-in-peel hydrolytic aging data were difficult to interpret because of great variability, because of variations in initial strengths and because most failures were a composite of cohesive failure of the elastomer and failure in adhesion to the substrate. The silicones were generally superior to the completely organic caulks after hydrolytic aging, due in major degree to their greater initial strengths. Butyl and Hypalon did not appear to undergo any significant degradation during the hydrolytic aging, but were so weak initially that testing was difficult.

1.2.5 Accelerated Weathering

Tensile specimens of both Class PS and Class SC elastomers were aged in an Atlas Weather-O-Meter with a carbon arc as ultraviolet source and intermittent water spray to simulate rain. Aging was performed at about 60°C. Changes in tensile properties were then observed. The Class PS elastomers in general were essentially unaffected by this treatment. The acrylic caulk (Tremco Mono) lost its elongation completely during the weathering exposure. Weathering effects on the Class SC silicone caulks were not severe. The Class SC butyl and Hypalon compositions proved too fragile to test by this procedure.

1.2.6 Fungus Resistance

Tensile specimens were exposed simultaneously to an aqueous emulsion of five different fungal organisms by an outside contractor, Microbac Laboratories. Visual observations of fungal growth and measurements of tensile properties were carried out after two aging periods. In terms of fungus growth, silicones were found to be generally more resistant than other compositions. The butyl compositions in general exhibited more fungal growth than any of the other materials tested. The fluorocarbon (Viton) exhibited more fungal growth than expected although it was more resistant than the butyl compositions. Changes in tensile properties as a result of fungal exposure were found to be quite small in general and in most cases, fungal attack should not present major problems.

1.2.7 Ozone Resistance

Tapered strip specimens of both Class PS and Class SC elastomers were maintained at 15% elongation during an exposure of about one week to ozone at 40°C and 100 pphm concentration. The samples were observed daily for such visual signs of deterioration as cracking. This work was performed by the Ozone Research and Equipment Corporation of Phoenix, AZ. With the exception of butyl rubber, all materials tested appeared to display good resistance to ozone. Physical properties of the acrylic caulk and the two butyl caulks did not permit them to be tested by this procedure.

1.2.8 Chemical Degradation and Outgassing Studies

Outgassing studies have shown that Class SC elastomers produce considerably larger quantities of volatiles than the Class PS materials. Fluorocarbon elastomers showed the least outgassing followed by certain Class PS silicone rubbers, acrylic elastomers and EPDM (Nordel) in that order.

TABLE 1.2.9

| CODE | MATERIAL IDENTIFICATION | | | SUMMARY OF MATERIAL EVALUATION ^{a,b} | | | | | | | |
|------|--------------------------|-------|-------------------------------|---|---|--|------------------------------------|-----------------------------------|----------------------------------|---------------------|----------------------------------|
| | SUPPLIER DESIGNATION | CLASS | TYPE | LOW TEMPERATURE COMPRESSION SET ^d | COMPRESSION SET ON THERMAL AGING ^d | RETENTION OF TENSILE PROPERTIES ON THERMAL AGING | OUTGASSING-SEALANTS ^{e,f} | HYDROLYTIC STABILITY ^g | CORROSION OF METALLIC SUBSTRATES | RESISTANCE TO OZONE | RESISTANCE TO FUNGI ^h |
| L | 31-323-0731A | PS | Fluorocarbon | 1 | 3 | 4 | 4 | 3 | 4 | 4 | 3 |
| G | SE-7550 | PS | Silicone | 4 | 1 | 3 | 4 | 3 | 4 | 4 | 4 |
| I | HS-70 | PS | Silicone | 4 | 1 | 3 | 4 | 3 | 4 | 4 | 4 |
| Q | NPC 80/40 | PS | Silicone | --- | 1 | 3 | 4 | 3 | 4 | 4 | 4 |
| J | 3300-12A, Vamac | PS | Ethylene-acrylic | 1 | 1 | 2 | 2 | 3 | 4 | 4 | 3 |
| K | 210-108-35-1, Hycar 4054 | PS | Acrylic | 3 | 2 | 2 | 4 | 2 | 3 | 4 | 4 |
| N | 3300-11, Nordel | PS | EPDM | 4 | 2 | 2 | 3 | 4 | 3 | 4 | 4 |
| CC | Vistalon 78E-09-28-2 | PS | EPDM | 1 | 1 | --- | --- | --- | --- | --- | --- |
| II | E-633 | PS | EPDM | 1 | 1 | --- | --- | --- | --- | --- | --- |
| EE | Hydrin 100 | PS | Epichlorohydrin | 4 | 1 | --- | --- | --- | --- | --- | --- |
| FF | Hydrin 200 | PS | Epichlorohydrin | 4 | 1 | --- | --- | --- | --- | --- | --- |
| GG | Hydrin 400 | PS | Epichlorohydrin | 4 | 1 | --- | --- | --- | --- | --- | --- |
| BB | Hypalon-3300-10 | PS | Chlorosulfonated Polyethylene | 1 | 1 | --- | --- | --- | --- | 4 | --- |
| 7a P | 8EX-123 (butyl 100) | PS | Butyl | 1 | 1 | 1 | 2 | 3 | 1 | 1 | 1 |
| O | SR 35020 | PS | Butyl | 4 | 1 | --- | 3 | --- | --- | --- | --- |
| DD | 8EX-122 | PS | Bromobutyl | 1 | 1 | --- | --- | --- | --- | --- | --- |
| A | DC 732 (white) | SC | Silicone | --- | --- | 3 | 2 | 3 | 4 | 4 | 4 |
| B | DC 790 | SC | Silicone | --- | --- | 3 | 3 | 3 | 4 | 4 | 3 |
| C | RTV 103 | SC | Silicone | --- | --- | 3 | 2 | 3 | 4 | 4 | 4 |
| D | Mono | SC | Acrylic/terpolymer | --- | --- | 1 | 1 | 2 | --- | --- | 3 |
| E | Eternaflex | SC | Chlorosulfonated Polyethylene | --- | --- | 1 | 1 | 2 | 1 | --- | 4 |
| F | Tremco butyl sealant | SC | Butyl | --- | --- | 1 | 1 | 2 | 4 | --- | 4 |

a = Ratings: 4 = excellent, 3 = good, 2 = fair, 1 = poor

b = Most materials that failed the screening test (Table 3, p86) were not subjected to most of the above tests.

c = Further descriptions of materials are presented in Tables 1 and 2

d = Compression set measurements were performed only on class PS materials.

e = Several materials received late in the program were not evaluated except for outgassing characteristics. This data is reported in Tables 19 - 21, and Figs. 58 - 66.

f = Outgassing ratings of 4, 3, 2, and 1 refer to total volatiles evolved on aging for 216 hours at 150°C of <0.5%, 0.5-2%, 2-6%, and >6% respectively.

g = Hydrolytic stability ratings of Class SC compounds were based upon retention of tensile properties and adhesion-in-peel strength retention.

h = Ratings for resistance to fungi are based upon both observations of fungal growth and effect on tensile properties.

1.3 Modes of Degradation (Task 4.3)

Information developed in the supplemental laboratory tests (Item 1.2 above) serves to illuminate deterioration of specific physical properties during exposure to environmental stresses. Chemical modes of degradation were observed through determination of the chemical nature of the thermal outgassing products from candidate elastomers. A newly developed test provides data on the rate and degree of outgassing of a material and also an infrared analysis of the evolved products. Results show that most silicone sealants give off a low molecular weight alkyl siloxane very similar to dimethyl silicone. The other sealants examined usually evolved mixed processing oils of the naphthenic type and/or stearic acid or metal stearate accelerators and activators. In general, the Class SC elastomers show higher outgassing than the Class PS elastomers.

1.4-1.5 Long-Term Durability and Recommendations on Limitations of Elastomers (Tasks 4.4 and 4.5)

Where the criterion of life at a selected aging temperature is taken as exceeding a compression set value of 50%, it was found that no elastomer tested was completely adequate. The best material, the fluorocarbon elastomer, Viton, provided data which extrapolated to a 510 day life for continuous exposure at 150°C. This is roughly equivalent to four years service life under conditions of maximum severity and possibly three or four times that long under more normal service conditions. The best silicone by the compression set criterion was NPC 80/40 which failed after 100 days at 125°C and extrapolated to a 315 day life at 110°C. It thus has questionable life at 110°C, and could not be used at temperatures higher than that. All other materials tested were not as good. It is evident that satisfactory service life can be obtained in the flat-plate solar collector application with presently available rubbers only if the collector unit is so designed that its performance does not depend upon low compression set values in order to maintain a seal.

Where retention of tensile properties during thermal aging in air is the criterion of service life, the fluorocarbon elastomer, Viton, is again the best performer. Failures did not occur with this material at the aging times and temperatures of our tests, but we judge that it would perform satisfactorily at temperatures as high as 175°C if compression set is not a consideration. The Class PS silicones were the next best performers by the tensile criterion and we believe that they are acceptable for service at 150°C or slightly lower, if low compression set is not required. The intermediate temperature elastomers, Hycar (polyacrylic), Vamac (ethylene acrylic) and Nordel (EPDM), are regarded as serviceable at 125°C or slightly lower. The Class PS butyl is the least stable and is not considered useful at temperatures above 100°C. The Class SC silicone caulks are considered to be about as good as the Class PS silicones, but the Class SC organic caulks, Mono (acrylic), Hypalon (chlorosulfonated polyethylene) and butyl are much less stable and lose their elastomeric properties quite rapidly at temperatures in the 100°C to 125°C range.

Corrosion of substrates, hydrolytic stability, resistance to weathering, resistance to fungal organisms and to ozone were not, in general, found to be major problems. Corrosion problems become largely nonexistent if either anodized aluminum or mill finish aluminum is used for frame material rather than galvanized iron. It is suggested that fungicides be incorporated into fluorocarbon or butyl rubber formulations, but other elastomers in general possess adequate resistance to fungal organisms. Only the butyl rubber was found to have inadequate resistance to ozone and only the Class SC acrylic caulk appeared to be affected appreciably by the accelerated weathering exposure used. The Class SC organic caulks, Mono (acrylic), Hypalon (chlorosulfonated polyethylene) and butyl were found to evolve unacceptably high quantities of condensables and, in consequence, should not be used in locations where they have direct communication with the interior of the solar collector cell. This consideration also applies to some Class PS rubbers, primarily in the butyl and EPDM categories. A similar problem exists with the Class SC silicone

caulks which evolve relatively large quantities of low molecular weight cyclic polysiloxanes which condense upon the glazing and are ultimately converted into an opaque white deposit of silica.

1.6 Recommendations for Selection of Polymers and Compounding Formulations (Task 4.6)

Fluorocarbon and silicone elastomers in that order were found to possess the best elevated temperature life characteristics. The fluorocarbon (Viton) is marginally acceptable in its present formulation for resistance to elevated temperature compression set, and the silicones need substantial improvement. Reformulation of both categories to improve this characteristic is indicated and moderate increase in degree of cross-linking as well as increase in filler loading constitute reasonable approaches to the problem.

An additional weakness of most fluorocarbon elastomers is excessive compression set at low temperatures. Correction of this shortcoming could involve reformulation to decrease regularity in the polymer chains, thus lowering glass transition temperature and decreasing low temperature crystallinity. The same end might be achieved by incorporation of high molecular weight plasticizers, particularly if they can be grafted onto the polymer chains.

Review of rubber formulations in general with a view to minimizing quantity of volatile additives present is desirable. Functional additives should be incorporated only when specifically needed and then selected for minimum volatility. Optimization of rubber compositions not specifically formulated for the solar cell application is definitely indicated and is also desirable where materials formulated for this application were not optimized.

1.7 Breathing Survey (Task 4.7)

A computerized literature search on solar collector breathing was performed using the Lockheed Information Retrieval System and the Department of Energy (DOE) RECON System. This search provided very little useful information. The main source of information proved to be the solar collector manufacturers. A tabulation of the available information from seventeen manufacturers of liquid collectors showed that sixteen employed vented units. Only four manufacturers used a desiccant to dry the air. Silica gel was used by each of these four; however, one of the manufacturers also included a molecular sieve.

1.8 Effects of Breathing -- Analysis of Solar Collectors (Task 4.8)

Five solar collectors, which had been in service at three different locations, were examined with regard to their design and quality of fabrication. Particular attention was given to problem areas such as leakage, corrosion, glaze deposits, quality of coatings, etc.

Two PPG (Pittsburgh Plate Glass) collectors from the Towns Elementary School, Atlanta, GA were very well sealed and only small amounts of rain water had penetrated into the interiors of the units. The two PPG collectors from the National Bureau of Standards were, in contrast, extremely leaky, and large quantities of rain water had passed through them. Although the same butyl sealant had been used in the units from both locations, the sealing technique used on the NBS collectors was apparently inferior.

The collector received from Los Alamos, which was manufactured by the Turbo Refrigerating Co., Denton, Texas, was found to be in excellent condition. No evidence of gross migration of rain water into the interior of the collector could be found.

1.9 Desiccants and Organic Adsorbents (Task 4.9)

The literature survey on desiccants for moisture and adsorbents for organic vapors has been completed.

Ten solid desiccants have been identified; however, only three of these are suitable for use in solar collectors. These are silica gel, molecular sieves and activated alumina.

Silica gel is the preferred desiccant due to its high moisture capacity (~ 42%) and its ease of regeneration. Silica gel can be regenerated within the operational temperature range of the solar collector, and thus active desiccant is essentially always available.

Most desiccants will also adsorb organic vapors; however, activated carbon is the only desiccant that has a preference for organic vapors.

A duo-adsorbent system consisting of silica gel and activated carbon is recommended for evaluation in solar collectors.

1.10 Recommendations for Improved Materials and Collector Design (Task 4.10)

The use of an externally attached bladder to compensate for breathing during temperature/pressure changes is a feasible approach as long as adequate space underneath the collector is available. It should be noted, however, that this approach reduces only the chance for water condensation within the collector. The problems associated with organic vapors will still be present.

Due to the relatively short service life predictions in terms of resistance to compression set at elevated service temperatures, obtained for all categories of elastomers, it is recommended that solar collector units be designed to provide continuous pressure by some type of spring loading against gaskets intended to seal the unit from water in the surrounding environment.

Because organic caulks such as acrylic or butyl lose their elastomeric properties rather rapidly on exposure to even rather mildly elevated temperatures in air, their use in solar collectors is not recommended. The Class SC silicones retain elongation very well, but are mechanically weaker than the Class PS silicones. They may be used where they are not subject to mechanical stress and where they have no direct communication with the interior of the solar collector to permit deposition of volatile cyclic polysiloxane decomposition products on the glazing or the absorber plate.

If adsorbents are to be used to limit deposition of condensables inside solar collector units, it is recommended that a duo-adsorbent, incorporating both silica gel and activated carbon, be chosen. The former adsorbent preferentially adsorbs water and the latter organic condensables. Since effective circulation of the solar collector atmosphere through the adsorbent to remove condensables before they deposit on the glazing or absorber plate is a significant problem, it is still vital to select elastomers which evolve minimum amounts of condensables.

2.0 RECOMMENDATIONS FOR FOLLOW-UP STUDIES

We recommend a program to develop improved elastomeric gasketing materials for use in thermal solar collectors. These sealants should be designed to provide superior long-term retention of physical properties in the hostile environment of the solar collector, without causing side effects deleterious to the efficacy of the collector unit.

Two tasks are outlined for accomplishment of this objective. The first involves identifying the types of polymers which provide the best overall performance when used in thermal environments of 400°F, 300°F and 200°F, respectively. The second task consists of developing optimum chemical formulations which incorporate the polymers identified in the first task.

The materials developed should provide high thermal stability and very good resistance to attack by oxygen, ozone, moisture, and weathering. Efforts should also be directed towards reducing the compression set which elastomers normally display upon thermal aging under compression, and reducing the formation of volatile materials during aging, particularly those which are likely to condense on the glazing or absorber plate.

3.0 INTRODUCTION

The objectives of this program were: (1) to investigate the pertinent properties of a variety of possible sealants for solar collectors and identify the most promising candidates, and (2) to study the effect of breathing in flat-plate, thermal solar collector units. The study involved two types of sealants, Class PS which includes preformed seals or gaskets and Class SC which includes sealing compounds or caulks. It was the intent of the study to obtain data regarding initial properties of candidate elastomers from manufacturers and from the technical literature and to use those sources to provide data pertaining to endurance of these materials under environmental service conditions.

Where necessary, these data were augmented by experimental measurements. Environmental stresses evaluated by these measurements included elevated temperatures, moisture, ultraviolet light, ozone and oxygen, and fungus.

The second major area of the work involved a study of the effects of materials used and design on the durability of solar collectors. Factors such as design, fabrication, materials of construction, seals and sealing techniques and absorber plate coatings were observed on actual field units removed from service. Such phenomena as leakage, corrosion and formation of deposits on glazing and absorber plate were noted.

An evaluation of the properties of several desiccants was made in order to provide means to mitigate the deleterious effects of water on collector life. Adsorbents for organic degradation products of sealants were also investigated in order to protect the glazing and absorber plate from deposited coatings. Since adsorbents and desiccants in general tend to take up both water and organic decomposition products, relative affinities of a number of these agents for water and for organic compounds were determined.

4.0 TASKS

4.1 Physical and Aging Properties of Sealants

A survey report which summarizes the properties of all categories of commercially available elastomers was completed. This document places particular emphasis upon characteristics which are pertinent to long-term service under the environmental stresses encountered in the flat-plate solar collector application. Considerable data are presented in such areas as initial physicals, properties at both low and elevated temperatures, aging in air at elevated temperatures, resistance to ozone and weathering, hydrolytic stability and water absorption, compression set and stress relaxation. Cost effectiveness was a major consideration in making selections of candidate elastomers. Information developed indicated that silicone and fluorocarbon elastomers were the most viable high temperature rubbers. Fluorosilicone and phosphonitrilic rubbers were eliminated on the basis of high cost.

Candidates in the somewhat lower temperature service category ($\sim 125^{\circ}\text{C}$) consisted of polyacrylic, ethylene propylene terpolymer (EPDM), ethylene acrylic, butyl, chlorosulfonated polyethylene and epichlorohydrin.

The literature survey included computer printouts incorporating several hundred references. Under separate cover, the assemblage was submitted to the Solar Heating and Cooling Research and Development Branch, Office of Conservation and Solar Applications, Los Alamos Scientific Laboratory, U.S. Department of Energy.

4.2-4.4 Tasks 4.2 Supplemental Laboratory Tests, 4.3 Modes of Degradation, and 4.4 Long-Term Durability

For purposes of clarity, since these tasks are intertwined, they are combined and subheadings are employed to provide details of the particular area studied.

Since test data in many critical areas, both from the technical literature and from manufacturers, have been found to be quite incomplete, we have been obliged to supplement these sources extensively with our own laboratory measurements. We believe that this procedure provides the substantial advantage of insuring that test data from different materials are more rigorously directly comparable because sample testing conditions are known to be identical.

The thermal endurance of the sealants has been examined by studying the effects of thermal aging in air on retention of tensile properties, compression set, change of hardness and loss of weight.

Early in the thermal aging, the elastomers generally experience an increase in degree of cure. This results in an overall improvement of their tensile properties (Fig. 1). Simultaneously, low molecular weight materials, such as plasticizers and processing aids, diffuse out of the polymer matrix. This has the overall effect of increasing the tensile strength while reducing the elongation. In many cases, additional cross-linking continues throughout much or all of the aging period with the effect of increasing the modulus and eventually embrittling the polymer. However, some polymers undergo essentially the opposite effect -- that of chain scission which reduces the strength and frequently the ultimate elongation of the material. (The overall effects of these processes are illustrated in Fig. 2.) Further complicating the picture is that both processes occur simultaneously in many elastomers and that some of the reactions are essentially pyrolytic and others oxidative in nature. The degradation process becomes even more complex when the elastomer consists of copolymers or terpolymers since the different types of chain units can degrade by different mechanisms, can interact and also influence the reactivity of their neighbors.

Simultaneous cross-link formation and chain scission which occur during the thermal-oxidative degradation of many elastomers transform the polymer into a harder but weaker material having a consistency that can best be described as cheesy.

As linkages in the primary chains are being broken and low molecular weight chain fragments and oxidized degradation products are diffusing out of the polymer, the material experiences a loss in weight. In an actual collector, the very low molecular weight products will escape from its confines; however, the less volatile degradation products can coat the absorber plate and the glazing, thus decreasing the efficiency of the collector. (This aspect will be discussed in detail in the section on outgassing.)

When elastomers are subjected for extended periods of time to a compressive load while being permitted to distort at elevated temperatures, they will not recover to their original dimensions when the load is removed. Measuring the compression set of compressed elastomers during thermal aging is a very useful and realistic means for evaluating materials for gasket applications.

Permanent set or distortion of a polymer can be an essentially physical or combination of chemical and physical phenomena. Under the application of heat and pressure, the polymer chains rearrange to accommodate the applied forces. When the pressure is released, the newly formed alignments undergo some shifting, but nevertheless generally do not permit a complete recovery of the material to its original configuration. Chemical changes frequently accompany the shifting of the polymer chains. Cross-links are broken under the combined influence of thermal stress and the induced shear forces, and are often reformed in a manner so as to accommodate the newly formed alignment of the polymer chains. When cross-links break and reform to an appreciable extent, polymers will exhibit very poor or perhaps no recovery.

a. Thermal Aging Screening Tests

Data accumulated during our literature survey were used to select categories of rubbers which we felt offered most promise for use in the flat-plate solar collector application. This list was supplemented by other rubbers suggested by personal contacts within the rubber industry as being potentially suitable for this application as well as by materials known to be in current use by solar collector cell fabricators. The next step involved screening tests designed to select materials for more detailed studies.

ANSI/ASTM D-3667-78, Standard Specification for Rubber Seals Used in Flat-Plate Solar Collectors, is a series of tests for the purpose of qualifying both Class PS and Class SC elastomers for service in the solar collector application. This standard proposes performance requirements to qualify either type of elastomer for sealing flat-plate solar collectors. The entire specification is reproduced in Appendix I. In our screening tests for Class PS materials, all tests described in Table 1 of the ANSI/ASTM D-3667-78 specification with the exception of condensable volatiles, ozone resistance and low temperature resistance were performed. Our market survey indicated that only a relatively small number of viable candidates were commercially available in the Class SC (caulking type sealant) category and, in consequence, we decided not to perform a comparable series of screening tests on these compositions. Suitable candidates were evaluated directly in the more exhaustive tests which followed. Table 1 identifies these Class SC materials by code number and supplier.

Elastomers evaluated in the Class PS screening tests included three silicones, three ethylene propylene terpolymers (EPDM), two fluorocarbons, three epichlorohydrins, one ethylene acrylic, one polyacrylic, one chlorosulfonated polyethylene, one bromobutyl and two butyls. These compositions are identified by code number, supplier and chemical composition in Tables 1 and 2.

Results of the screening tests are summarized qualitatively in Table 3. Of the three silicone elastomers tested, Silastic 747 was dropped from consideration for the more stringent testing because its drop in tensile strength during aging and its weight loss were excessive. Elastomers SE-7550 and HS-70 performed very well in all categories. A fourth silicone elastomer, NPC 80/40, was not yet available at the time of the original screening, but was added to the list of materials for exhaustive testing. Nordel 3300-11 was the only EPDM rubber of the three formulations tested which exhibited good retention of physical properties on aging at 150°C. The two fluorocarbon elastomers screened appear to be essentially identical in all respects. Their performance was excellent in all categories except for low temperature compression set. We selected Viton 31323-0731 for the extended testing, due primarily to its immediate availability. None of the three epichlorohydrin elastomers performed well enough to merit further testing, and, in consequence, the entire category was eliminated from further consideration. Although the ethylene acrylic, Vamac 3300-12, did not perform well on the screening tests, the supplier, DuPont, indicated that the initial batch provided had been undercured and that a second batch 3300-12A, which they later provided, would be far superior. This second batch was placed in the list of compositions for more exhaustive testing. The one chlorosulfonated polyethylene screened, Hypalon 3300-10, showed poor compression set at both low and high temperatures. Although it was eliminated from further testing, it performed very well in areas other than compression set and merits consideration in designs where good compression set characteristics are not needed. The one polyacrylic elastomer screened performed excellently in all respects save for ultimate elongation where it failed by a small margin. It was selected for further testing. The bromobutyl elastomer screened performed poorly and was eliminated from further testing. However, the better butyl rubber (8EX-123), even though it failed by a substantial margin in low temperature compression set and in total volatiles, was continued into the final testing because it is known that butyl rubbers have found extensive application in the solar collector

industry. It should be pointed out that the EPDM elastomer, Nordel 3300-11, and the butyl rubber, 8EX-123, were specifically formulated to meet requirements of the flat-plate solar collector application and are not representative of these categories of elastomers generally.

b. Compression Set Aging Tests

For Class PS or gasketing type sealants, compression set may well be the most pertinent property for the flat-plate solar collector cell application. A gasket material which undergoes severe compression set will not maintain pressure against the collector frame to provide an effective seal in many collector unit designs. Compression set measurements were carried out by the method of ASTM D-395-69, Method B. This procedure involves aging of the sample under constant deflection. Aging times and temperatures selected for these tests are not those suggested in the ASTM procedure, but rather periods and temperatures chosen to provide Arrhenius data which can be used to predict durability in a variety of thermal environments. The test, briefly, consists of compression of a cylindrical rubber specimen in a metal jig to the thickness of a metal spacer. This compression is approximately 25% of the original thickness. Thickness of the rubber specimen is measured after release of compressive load at each of several aging intervals, at a series of test temperatures. Compression set is calculated by the formula: $C = 100 \times (t_o - t_f) / (t_o - t_s)$ where C is compression set expressed in percent, t_o is the original thickness of specimen, t_f is the final specimen thickness under no-load conditions, and t_s is the thickness of the metal spacer bar. In the numerical data obtained for two of the silicone elastomers, NPC 80/40 and SE 7550, compression set values greater than 100% were observed. This is attributed to essentially complete loss of resilience by the specimen combined with an additional reduction in sample thickness due to shrinkage associated with thermal degradation.

Compression set samples used in these studies were stacks of 1 in. diameter disks which were die cut from PS elastomer sheets provided by rubber suppliers. The sheet stock from which these disks were cut varied in thickness from 65 mils to 80 mils. Each compression set sample consisted of a stack of these disks approximately 500 mils in total thickness. Compression set values reported are average values obtained from testing of four specimens. These data are displayed in Table 4 and Figs. 3-11. The compression set data which we have obtained reflect far poorer performance than we had anticipated on the basis of conversations with manufacturers of solar collector units. All three silicones tested exhibit compression sets of the order of 100% after only one day of aging at either 250°C or 225°C. The best of the three, NPC 80/40, showed compression set values of 94% after one day at 225°C and 89% after 28 days at 175°C. We feel that a sealant should not exceed 50% in compression set if it is to retain its ability to maintain an adequate seal. We do not believe that the disappointing performances observed for the silicones can be explained on the basis of inadequate post-cures. If this were the case, aging at lower temperatures would be expected to produce an initial rapid rise in compression set followed by a substantial leveling off of the curve. Although some leveling off does indeed occur at the lower test temperatures, the rise continues at such a rate as to produce high compression set values in unacceptably low aging times. The compression set values displayed in Table 4 for NPC 80/40, HS-70 and SE-7550, aged at 150°C and 140°C, illustrate this point, as do the curves in Figs. 3, 4 and 5. Performance of the fluorocarbon is far superior with compression set values of 79% after 28 days at 250°C and 32.9% after 46 days at 175°C.

Table 5 displays aging times required at 175°C, 150°C and 125°C for the eight elastomeric compositions tested to reach compression set values of 50%. These data were obtained by interpolation of the compression set aging curves and were used to plot the Arrhenius curves displayed in Fig. 11. The Arrhenius plots again show that the fluorocarbon elastomer far outperforms all other materials tested. It is of interest to note

that the plots for the EPDM, the polyacrylic and the best silicone (NPC 80/40) cross at about 125°C. The indication is that both the EPDM and the polyacrylic will outperform the silicone at temperatures higher than 125°C with the EPDM being considerably superior to the polyacrylic, and that the silicone will perform better than these two organic materials at temperatures below 125°C. The superior performance of the organics in the higher temperature region is, of course, supported by actual data in the compression set aging curves. The remaining two organic elastomers, ethylene-acrylic and butyl, perform more poorly than EPDM and polyacrylic. The Arrhenius plot for butyl crosses that for SE-7550 at about 140°C, indicating that it will have greater (but very short) life at temperatures above that level. Below about 140°C, the butyl is the poorest composition tested. The ethylene-acrylic plot crosses that for the silicone, HS-70, at 125°C. Hence, its performance is better than that of the poorer silicones at temperatures above that level, but poorer below. The ethylene-acrylic must be considered the second poorest composition tested from the standpoint of compression set retention.

It may be of interest to point out that extrapolation of the Arrhenius plot for the EPDM rubber shows it crossing the plot for the fluorocarbon elastomer at about 225°C, a circumstance which would indicate that the EPDM should outperform the fluorocarbon at temperatures higher than 225°C. We know that this is not true and the circumstance is pointed out merely to illustrate fallacies which may be encountered by reliance on Arrhenius extrapolation over too great a temperature range. Conclusions drawn from Arrhenius data treatment are valid only so long as the chemical reactions involved in the polymer degradation under consideration remain the same. In the case of EPDM degradation, new degradation mechanisms would become significant before the 225°C temperature level was reached, thus invalidating the extrapolation.

c. Corrosion Tests

Corrosion tests also were performed concurrently with the compression set aging studies. Small panels of three substrates, mill finish aluminum, anodized aluminum and galvanized iron were clamped in contact with compression set buttons of rubber compositions and aged at both 125°C and 150°C. Test specimens were visually examined at a series of aging intervals. Results are displayed in Table 6.

Of the thirteen compositions tested, only the Hypalon (chloro-sulfonated polyethylene), the polyacrylic (210-108-35-1), the Nordel (EPDM) and the butyl (8EX-123) attacked any of the three substrates tested. Hypalon produced severe corrosion of the galvanized iron, but did not attack either aluminum composition. The polyacrylic produced mild corrosion of the galvanized iron and did not attack either of the other substrates. Nordel produced moderate corrosion of the galvanized iron, but attacked neither of the aluminum substrates, and the butyl rubber produced mild corrosion of the mill finish aluminum and moderate corrosion of the galvanized iron. It did not attack the anodized aluminum. It should be noted that none of the elastomers evaluated attacked the anodized aluminum after 229 days of exposure.

d. Thermal Stabilities of Class PS and Class SC Materials

A general discussion of the several phenomena which take place during thermal aging of elastomers in air was presented (c.f., pp. 17 and 18). Pertinence of the study of these phenomena as they apply to sealants for flat-plate solar collector cells is evident from our knowledge of the high temperatures encountered in this application. Even though these sealants are normally employed in the peripheral locations of the solar collector units, temperatures encountered are believed to be relatively high. The extent of the temperature elevation encountered is dependent on the particular design of any single type of unit, and it has been our experience that manufacturers in general do not know accurately what temperatures are actually reached by sealants.

Experimental evaluation of thermal stabilities of elastomers in air was based upon monitoring of deterioration of tensile properties during thermal aging of ASTM D-412 tensile specimens in air and upon weight loss and changes in hardness during this thermal aging. Retention of compression set during thermal aging is obviously a part of this picture, but was treated in Sections 4.2-4.4 (b. Compression Set Aging Tests).

d (1). Thermal Aging of Class PS Tensile Specimens. Tensile specimens of Class PS elastomers for use in these tests were prepared from sheets of post-cured, vulcanized elastomers of approximately 70 mils in thickness which had been provided to us by the several rubber companies involved. Exceptions were silicone rubbers HS-70 and NPC 80/40 which were supplied to us by North American Reiss as calendared rolls and required a 4 hr cure at 200°C before cutting of the tensile specimens. Test specimens were die cut from the elastomeric sheets with Die C as described in the procedures of ASTM D-412-75.

Test specimens were aged in air at temperatures ranging from 225°C to 125°C in 25° intervals. In general, not more than three aging temperatures were used for any one composition and aging temperature selection was based upon knowledge of the thermal stability range of each material. Testing began after one day of aging and aging exposures were doubled with each successive interval up to 64 days. Final aging interval was 100 days.

Aging was accomplished by suspending specimens in groups of 20-25 in open one-half gallon paint cans which were placed in forced air circulation ovens. The tensile specimens were tested in accordance with the ASTM D-412-75 procedures for rubber properties in tension and tensile strength; ultimate elongation and modulus at 100% extension were measured. Values reported are averages for three specimens.

d(2). Discussion and Results of Thermal Aging Tensile and Elongation Tests

Effects of thermal aging in air on retention of TS* and E* are shown in Tables 7 and 8 and Figs. 12-21. Whenever feasible, the retention of either TS or E was subjected to an Arrhenius treatment in order to obtain predictions of the lives of the materials at temperatures other than those employed in the tests. This treatment of data is especially useful for making predictions of the longevity of materials at temperatures lower than the elevated test temperatures and thus provides a means of accelerated testing.

The logarithm of the time required for the percent retention of the TS or E of the sealant to drop to 60% of its original value is plotted against the reciprocal of the absolute temperature of the test (Figs. 22-26). We feel that a 40% decline of either tensile property indicates that the sealant has undergone substantial degradation. Nevertheless, in some cases, Arrhenius plots were also made for 50% retention. When the TS and E underwent comparable declines, both properties were subjected to an Arrhenius treatment; otherwise the property which underwent the most serious decline was employed to evaluate the thermal endurance of the sealant.

Generally, data points at at least two test temperatures are required to draw the straight line for the Arrhenius plot. Some investigators will employ a single point and assume a slope based on results from a chemically similar material undergoing the same process. Feeling that such an approach is too speculative, we did not employ it in this investigation.

The Arrhenius treatment provides the most reliable predictions when the length of extrapolation is minimized, data are collected at several temperatures, and the points fall on a straight line. When

* TS = tensile strength; E = ultimate elongation

the points do not fall on a straight line, those at the lowest temperatures are weighted the most heavily in drawing the plots. For example, in cases where the graph contains three points essentially equidistant in temperature, we arbitrarily (based on many years of experience) weight the highest temperature point 50% as high as the middle temperature point and draw the line through the lowest temperature point and the weighted average of the two upper temperature points.

The validity of the Arrhenius treatment is based upon the assumption that the activation energy for the rate limiting step in the degradation process is essentially independent of temperature. Since the mechanism of the degradation process can change with temperature, the validity of this assumption decreases as the temperature range of the study and the extrapolation become larger. Thus, points on an Arrhenius plot occasionally deviate too far from a straight line to permit any reasonable linear extrapolation. For example, it was not feasible to apply the Arrhenius treatment to silicone HS-70 since the mechanism for its degradation process appears to differ appreciably between 225°C and 175°C, thus giving three log time versus reciprocal absolute temperature points (corresponding to 225°C, 200°C and 175°C) which deviate considerably from a straight line. In Figs. 23-25 where only two points were employed to define the straight line, there is an obvious risk in these extrapolations. Such results should only be considered as "ball park" estimates.

In addition to the silicone HS-70, several other materials were excluded from the Arrhenius treatment for the following reasons: (1) initial properties on the material were too poor; (2) monitored properties of the material did not exhibit any clearly defined base points (ex. some caulking compounds continued to give off solvents and other volatile compounds during much of the aging period); (3) ultimate elongation exceeded the capability of the testing apparatus; (4) material did not exhibit failure at at least two test temperatures.

In any continuation of this work, it is suggested that experimental procedures be modified so as to accommodate most of the excluded materials. Such modifications can involve items such as selection of other test temperatures, altering the testing apparatus or departing from the shape of the ASTM D-412 dumbbell specimen in order to improve the capability to measure the extremely highly extendable, low modulus elastomers, and assuring the loss of essentially all of the volatile materials in the caulking compound prior to commencing with thermal aging in order to establish a base for comparison.

The results of the Arrhenius treatments are shown in Table 9. As expected, the life of the butyl rubber held continuously at 125°C (252°F) is extremely short -- 49 days. The EPDM is considerably better, showing a life of 180 days. Next are the acrylic and ethylene-acrylic having estimated lives of 480 and 790 days, respectively. These are followed by a silicone which displays an extrapolated life of 4300 days. The fluorocarbon, which was not subjected to this treatment because it did not exhibit failure on testing, is undoubtedly superior. At a temperature of 150°C (302°F), which is readily attainable in many collectors, the order remains the same; however, the thermal endurances of the sealants are quite poor. Butyl gives 7 days; EPDM, 30 days; acrylic, 72 days; ethylene-acrylic, 82 days; and the silicone, 430 days.

d (3). Thermal Aging of Class SC Tensile Specimens. It was necessary to cast sheets of the Class SC elastomers (caulks) from which the tensile specimens could be cut. The mold employed was fabricated from neoprene, Mylar and glass in the following manner. Four rectangular cavities 5-1/2 in. x 1 in. x 1/16 in. were cut from 7 in. x 7 in. x 1/16 in. neoprene sheets. The neoprene sheet was then coated with a silicone mold release agent and laid down on a 7 in. x 7 in. sheet of 2 mil Mylar which had been coated first with a silicone and then a fluorocarbon mold release agent. This assembly was then clamped to a 7 in. x 7 in. x 1/8 in. glass plate to rigidify the structure.

A caulking compound was inserted into the mold cavities and spread evenly with a single-edged razor blade until it conformed to the dimensions of the cavity. Each casting was permitted to cure at room temperature for 4 to 6 weeks. Tensile specimens were die cut from the room temperature cured castings in the same manner as were the Class PS specimens.

Code* A, B and C specimens (silicone caulks) were aged at 175°C, 200°C and 225°C. In general, aging intervals began at one day and doubled with each consecutive exposure up to 64 days. Final aging interval was 100 days. Intermediate intervals were omitted in some cases when it appeared that information gained would be of limited value. Prior to initiation of the aging exposures, all tensile specimens were post-cured 24 hr at the temperatures at which they were to be aged. Aging was carried out in the same manner utilized for the Class PS tensile specimens. Triplicate tensile specimens were aged for each aging temperature-aging interval combination.

The Code D (acrylic terpolymer), E (Hypalon) and F (butyl) tensile specimens were post-cured 24 hr prior to initiation of thermal aging at the temperatures at which they were to be aged as had been done with the silicone caulks. Aging temperatures selected were 100°C, 125°C and 150°C, reflecting the known lower thermal stabilities of these materials. Purpose of the initial post-cures was to minimize the very large changes that occur at the outset of elevated temperature exposure as volatiles (present in large quantities in caulking formulations) are driven off. Aging intervals for the D, E and F materials were identical to those used for the silicone caulks.

Where possible, the tensile specimens were evaluated by measurement of tensile strength, ultimate elongation and tensile modulus at 100% elongation by the procedures of ASTM D-412-75, Rubber Properties in Tension. Values reported represent averages for three specimens.

* Refer to Table 1 for descriptions of codes for materials.

Performance of caulking compositions during thermal aging is very difficult to interpret, due in major degree, to the rather large quantities of volatile materials normally employed in these compositions to provide suitable working properties prior to cure. These volatiles remain present in substantial quantity even after long curing at room temperature. As a consequence, considerable amounts of volatile components continue to evolve during the early portion of any thermal aging program. Loss of these volatiles, primarily plasticizers and solvents, has the effect of increasing tensile strength and modulus quite sharply while reducing ultimate elongation. Reversion of the polymer, involving degradation of polymer chains into shorter segments by random chain scission, results in reduction of tensile strength and rigidity. It is evident that this latter process which proceeds simultaneously with loss of volatiles produces opposing effects. In consequence, it is not easy to determine to what extent an observed change in properties is due to relatively small progress in one process or to substantial progress in both with one predominating. The picture is further complicated by the presence of a third process which proceeds concurrently with the two just discussed. This is additional cure, an inevitable consequence of the fact that primary cure is carried out at room temperature. Further cure will result in increased cross-linking, a structural change whose effects reinforce those of loss of volatiles, i.e., increased tensile strength and modulus and lowered extensibility.

In view of this complexity, it seems appropriate to concentrate interpretation of the test data on actual properties at the longest aging period. This appears to provide the most realistic assessment of practical performance which is available. The General Electric silicone caulk, RTV-103, easily outperforms the two Dow Corning silicones in tensile properties after 100 days at 225°C. DC-732 and DC-790 follow in that order. The same order of performance is maintained at the two lower aging temperatures, but at 175°C, the DC-732 and DC-790 have become roughly comparable. Although RTV-103 remains the best performer at 175°C,

its performance is closer to that of the Dow Corning resins than at the higher temperatures. It is unfortunate that these data are not suitable for the Arrhenius treatment since such a treatment would provide slopes which could be used to predict aging curve intersections. Since this information is not available, we can only suggest that it is possible that the observed sequence in performance of individual compositions might change at lower aging temperatures.

The Code D, E and F materials, acrylic terpolymer, Hypalon (chlorosulfonated polyethylene) and butyl, respectively, are in a lower thermal stability category than the silicones, as might be expected of completely organic elastomers. The silicones show better tensile properties after 100 days aging at 175°C than does the acrylic, the best of the organic Class SC elastomers, after 100 days aging at 150°C. The acrylic caulk still retains respectable tensile strength after 100 days at 150°C (well above the initial tensile strength, although it appears to be declining), but its elongation has declined almost to zero, indicating loss of elastomeric properties. The butyl rubber showed zero elongation at the start of the test at all aging temperatures, indicating that even the 24-hour post-cure at 100°C had been sufficient to cause it to lose its elastomeric properties. Hypalon performed better, but was markedly inferior to the acrylic. Its ultimate elongation had declined to zero after only four days of aging at 150°C, and near to zero after 64 days at 125°C. This compares to 80% elongation for the acrylic after 100 days at 125°C. After 100 days at 100°C, the acrylic exhibited 210% ultimate elongation while the Hypalon had declined to 60%.

d (4). Thermal Aging of Class PS and SC Weight Loss and Hardness Specimens

Class PS weight loss and hardness specimens were 3-1/2 in. x 2-1/4 in. strips cut from rubber sheets of nominal 70 mils thickness provided by the several rubber suppliers. Post-cure of these materials is covered in Section d (1). Class SC weight loss and hardness specimens were prepared in the same manner as the Class SC tensile specimen blanks

described in Section d (3), with the exceptions that the mold cavity was 3-1/2 in. x 2-1/4 in. and that a 4 in. putty knife was used in place of the razor blade to spread the caulk sealant into the cavity.

Duplicate specimens of both the Class PS and Class SC materials were aged in air in the same manner as were the tensile specimens covered in Sections d (1) and d (3). They were removed periodically from the aging ovens for weighing and hardness measurement. All measurements were performed after the samples had cooled to room temperature. Since these tests are non-destructive, consecutive tests were performed on the same specimens. Hardness was determined on samples used for the weighings with a Shore A Durometer. Aging temperatures and intervals varied with the composition being aged, but were adjusted to the known relative thermal stabilities of the formulations being studied. The actual aging temperatures, test intervals and results are displayed in Table 10 and Figs. 27-44. Data reported in this table represent the average of measurements on two specimens.

Weight loss which occurs during thermal aging of elastomeric systems in air is subject to alternative interpretations. A relatively high weight loss indicates either that volatile components are being lost (as would be the case initially with caulking formulations) or that the polymer is undergoing degradation to form volatile decomposition products. Low weight loss, however, must not be regarded as final proof of high thermal stability since degradation (in the sense of loss of elastomeric properties) could occur by cross-linking rather than by chain scission, and no volatiles would be formed. Oxidation by atmospheric oxygen can occasionally result in retention of oxygen by the polymer with a consequent weight gain although, more frequently, volatile oxidation products are split off and a weight loss takes place. Despite the complexity of the overall picture, high weight loss in most instances is associated with poor thermal stability and low weight loss with good thermal stability. As indicated in the foregoing discussion, low weight loss is the more ambiguous phenomenon and such data need to be reinforced

by data from other tests in which changes in mechanical properties with aging are monitored before any final judgment of thermal stability is made. High weight loss, on the other hand, in conjunction with visual observation of changes in sample appearance may be sufficient evidence in itself to disqualify a material. Because it is inexpensive to run and does provide preliminary information quickly, weight loss is generally regarded as a useful screening test.

This type of measurement is very pertinent to the flat-plate solar collector application because volatiles, whether originally present as plasticizers or whether pyrolytic or oxidative degradation products, may deposit on the glazing or on the absorber plate, thereby decreasing the efficiency of the unit. This effect will be discussed in detail in the section on outgassing studies, part 1.

Change in hardness during thermal aging is also indicative of ongoing degradation. As is the case with weight loss, however, failure of the hardness to change does not provide rigorous proof of thermal stability. For example, hardness effects resulting from concomitant formation of cross-links in one type of chain segment and chain scission in a different type of chain segment in a copolymer chain could essentially cancel each other.

d (5). Discussion of Class PS Weight Loss and Hardness Data. No attempt was made to apply an Arrhenius treatment to any of the weight loss data because it is regarded primarily as a screening test. Observation of the weight loss data for the higher temperature materials (silicones and fluorocarbon) reveals that the fluorocarbon is poorer than the two better silicones at 250°C, but the order of performance inverts as aging temperature is lowered so that the fluorocarbon becomes far superior to all of the silicones in weight loss performance at 175°C. Interestingly, the weight loss performance of HS-70 remains the best of all three of the Class PS silicones at all aging temperatures from 250°C to 150°C. NPC 80/40 is very poor at the higher aging temperatures, but outperforms SE-7550 at 175°C and below. Resistance of the three silicones to weight loss at 150°C is in the following order: HS-70 > NPC 80/40 > SE-7550.

Among the intermediate temperature Class PS elastomers, Hycar (polyacrylic), Vamac (ethylene-acrylic) and Nordel (EPDM) rather closely parallel each other in performance on weight loss. Nordel is best by a small margin on short-term aging at 150°C with Vamac showing the highest weight loss. On longer term aging at 150°C, the order is changed with the Hycar polyacrylic showing the highest weight loss and the Vamac and Nordel being nearly identical. Examination of the 125°C data suggests that the Nordel would be somewhat superior. The butyl rubber was clearly inferior to all three of the other intermediate temperature Class PS elastomers on weight loss performance at all aging temperatures.

The fluorocarbon rubber, Viton, displays the best resistance to change in hardness on thermal aging at every aging temperature employed. The silicones, SE-7550 and HS-70, display comparable hardness increases in general upon thermal aging with the exception that HS-70 appears to be slightly more stable at 250°C. At 250°C and 225°C the silicone, NPC 80/40, is affected more severely than the other silicones. This gap narrows at 175°C, and at 150°C its performance is essentially identical to that of the other silicones.

The four intermediate temperature elastomers display roughly comparable and moderate increases in hardness at 125°C and 100°C. At 150°C, however, their performances are markedly inferior to those of the silicones. The small hardness changes observed with the butyl rubber are undoubtedly misleading since this material undergoes chain scission at these temperatures with a consequent softening effect which negates the hardening effect from other concurrent processes of degradation.

d (6). Discussion of Class SC Weight Loss and Hardness Data. Short-term weight loss of caulking compositions is an ambiguous phenomenon since loss of large quantities of such volatiles as solvents, plasticizers and volatile products of the curing reaction occurs concurrently with weight losses due to degradation of the polymer. Our use of long curing periods at room temperature, prior to initiation of aging, was designed to minimize this

problem, but cannot eliminate it. With these compositions, thermal stability is judged on the basis of rate of weight loss after considerable preliminary thermal aging.

The silicone caulks are in considerable degree less subject to the ambiguities of the preceding discussion than are the lower temperature organic caulks. The silicone caulks are formulated from low molecular weight silicone intermediates and as a consequence, have quite low viscosities as compounded and therefore do not require the addition of any substantial quantity of solvents or plasticizers.

Examination of the weight loss curves, Figs. 35-37 and Table 10, for the three silicone caulks indicates that performance of the RTV-103 and DC-732 compositions are about equivalent with the DC-732 appearing to be slightly superior at the higher aging temperatures and appreciably better at the lower temperatures. DC-790 is markedly inferior to both.

Among the lower temperature caulking compounds, Hypalon (chlorosulfonated polyethylene), Tremco "Mono" (acrylic) and Tremco butyl, the acrylic displayed the lowest weight losses. Next came the butyl caulk, and Hypalon was the highest.

Just as with weight loss, the DC-790 caulk exhibited the greatest increase in hardness at the three test temperatures, 225°C, 200°C and 175°C. The RTV-103 displayed better resistance to hardness change at 225°C than did DC-732. The DC-732, however, was superior at the two lower test temperatures, 200°C and 175°C.

The lower temperature caulking compounds experienced far greater changes in hardness during thermal aging than did the silicones. The butyl caulk exhibited a very sharp rise in hardness during the first day due to initial loss of solvent and plasticizer, and then the remainder of the weight loss plot was nearly flat. Limitation of the rate of rise of hardness during the later phases of thermal aging to a very low value is attributed to chain scission which tends to negate other processes

which might increase hardness. Both Hypalon and the Tremco acrylic showed substantial increases in hardness as the aging progressed.

e. Hydrolytic Stability Tests

The flat-plate solar collector cell service environment is likely to involve not only intermittent exposure to high moisture levels (from high atmospheric humidities, from dew or from rain), but also concurrent elevated temperatures as the collector unit warms up immediately after a period of exposure to high moisture levels. It, therefore, becomes important to compare hydrolytic stabilities of sealants regarded as candidates for this application.

Elastomeric systems immersed in hot water can, depending upon their chemical and physical characteristics, undergo several simultaneous processes, all of which can alter the mechanical properties of the system. These processes, all involving different chemical or physical mechanisms, may, in some cases, reinforce each other in causing a particular effect and in other cases compete with each other because they tend to produce conflicting effects. During the early stages of exposure to the elevated temperatures, many polymers experience an increase in tensile strength and elongation because of further polymerization to increase their state of cure (Figs. 1, 2 and 45). As water permeates into the polymer matrix, it behaves as a plasticizer causing a diminution in tensile strength and often an increase in ultimate elongation. Concurrently, plasticizers and processing aids are leached out causing a decrease in elongation and an increase in tensile strength. Which of the preceding two effects predominates is dependent upon the nature of the polymer and the other ingredients in the compound. All of the formulations tested (Figs. 46-54) appeared to have exhibited, at least during the early stages of the test, increases in tensile strength (percent retention values > 100%) which is attributed primarily to further curing, but which in some cases may be augmented by the leaching out

of plasticizers. Later, as water continues to enter the interface between the polymer and reinforcing filler, the tensile strength of the system is reduced.

The most harmful hydrolytic effect arises when the polymer contains an appreciable concentration of such hydrolyzable linkages as ester, urethane, or siloxane. Cleavage of hydrolyzable cross-links manifests itself as a drop in tensile strength and modulus and an increase in ultimate elongation eventually leading to a loss of thermosetting characteristics. Hydrolytic cleavage of linkages or segments in the primary chain can lead to a catastrophic drop in tensile strength and, in some cases, ultimate elongation.

Hydrolytic stability testing was carried out by immersion of mechanical test specimens of elastomers in water at a series of elevated test temperatures for selected test intervals and monitoring deterioration of mechanical properties as the test exposures proceeded. Tensile specimens (ASTM D-412) of all Class PS compositions evaluated and of as many of the Class SC materials as possible were aged hydrolytically and deterioration of tensile properties was monitored. Data acquired in this program is presented in Tables 11 and 12. Hydrolytic stability and particularly stability of elastomer to substrate bonds under hydrolytic conditions, was further studied with the Class SC elastomers by hydrolytic aging of ASTM C-794 peel strength specimens.

e(1). Tensile Properties as a Criterion of Hydrolytic Stability. To assist in the establishment of suitable initial water immersion temperatures and test intervals for the extended testing of ASTM D-412 specimens, a limited number of preliminary screening tests were carried out in which tensile specimens of several compositions were exposed to immersion in 150°C water for relatively short periods. Changes in weight, hardness and modulus at 100% elongation were observed. This work led to selection of water immersion temperatures of 125°C, 100°C, 83°C and 67°C for the extended testing. It was decided that initial exposures in each case would be one day and that total exposure would be doubled

with each succeeding test interval until total exposure at the lower test temperatures had reached 128 days. Triplicate tensile specimens of each composition were prepared as described in the procedures of Section d ("Thermal Stabilities of Class PS and Class SC Materials") and were tested for each combination of water immersion temperature and aging interval. Tensile strength, ultimate elongation and tensile modulus at 100% extension were monitored as the hydrolytic aging progressed. Prior to these measurements and subsequent to removal from water immersion, samples were air dried 16 hr at 80°C.

A brief summary of the performance of each composition* evaluated by hydrolytic aging of tensile specimens is presented in the following paragraphs. Details are presented in Figs. 46-54 and Tables 11 and 12. Individual compositions represented by the code letters are identified in Table 1 and formulations of each composition, to the extent that they are known to us, are presented in Table 2.

Fluorocarbon - Viton 31-323-0731A (L)

After an early enhancement of cure, the TS** and E** appeared to drop slowly, perhaps due to a slight hydrolysis effect; however, the relatively greater drop of TS suggests that plasticization by water is occurring.

Silicone - NPC 80/40 (Q)

At 125°C, there is a moderate drop of both TS and E which indicates hydrolysis of the polymer. This decrease of tensile properties is considerably slower at 100°C. At 83°C, the TS rises slightly for the first 64 days, indicating that cross-linking or chain-forming reactions dominate over hydrolysis. This effect is even greater at 67°C.

* The acrylic (D), Hypalon (E) and butyl (F) caulking compounds were too weak and tacky to permit this type of testing. An attempt was made to test silicone caulk DC-790 (B); however, its great elongation exceeded the capability of the testing apparatus (Table 11).

** TS = tensile strength; E = ultimate elongation

Silicone HS-70 (Q)

The tensile properties decreased rapidly at 125°C and progressively less rapidly at the lower temperatures indicating a significant susceptibility to hydrolytic degradation.

Silicone SE-7550 (G)

The effect of hydrolytic aging was not plotted for this material since its high elongation was beyond the capability of the testing machine during the early aging period. Nevertheless, hydrolysis is indicated by the overall drop of TS and E at 125°C and 100°C. At the lower temperatures, the material appeared to exhibit good stability.

Silicone Caulking Compound RTV-103 (C)

At 125°C, E increases while TS decreases indicating hydrolysis of cross-links and a possible plasticizing effect by water. This compound exhibits good stability at the lower temperatures.

Silicone Caulking Compound DC-732 (A)

After an initially sharp rise in E and TS at 125°C, indicating additional cure, both tensile properties dropped precipitously, indicating considerable hydrolysis of the material. The stability at the lower temperatures was much better.

Butyl 8EX-123 (P)

The E and TS exhibited essentially the same characteristics, both dropping slowly at 125°C and 100°C after an initial increase. This behavior is consistent with an initial increase in degree of cure followed by plasticization by water.

Ethylene-Acrylic Vamac 3300-12A (J)

At 125°C, the E drops sharply, whereas the TS rises and then also exhibits a sharp drop. It appears that the polymer undergoes additional cross-linking followed by hydrolysis. On immersion at 100°C, the TS increases while the E decreases indicating that cross-linking is the dominant phenomena for the 64 day period at this

temperature. At the lower temperatures, E drops steadily while the TS rises for 80-100 days and then begins to drop slowly indicating that additional cross-linking predominates initially and that hydrolysis later becomes the controlling factor.

Acrylic, Hycar 4054, 210-108-35-1 (K)

This material was not tested at 125°C because a screening test indicated that it would fail almost immediately. However, it displayed good stability at the three lower temperatures. At 100°C, the increase of TS and concomitant decrease of E indicated that cross-linking reactions associated with continuing thermal exposure were quite important.

Ethylene-Propylene Terpolymer, EPDM, Nordel 3300-11 (N)

This material displayed excellent resistance to hot water.

In summation, most materials displayed good to fair resistance to hydrolytic degradation. The EPDM Nordel was excellent and the Viton quite good. The acrylic and ethylene-acrylic displayed better hydrolytic stability than expected because of their potentially hydrolyzable ester groups. The butyl rubber showed good resistance to hot water; however, because of its chemical structure, it was expected to perform even better. The silicones ranged from good to fair; however, on an overall basis, their performance was considered disappointing.

e(2). Peel Strength as a Criterion of Hydrolytic Stability for Class SC Materials. The ASTM C-794-75 test, "Adhesion-in-Peel of Elastomeric Joint Sealants" utilizes a test specimen in which a glass fabric-reinforced strip of the sealant is adhered to a substrate. The reinforcing fabric constitutes a single layer of fabric between two layers of the sealant so that none of the reinforcement is present at the interface between sealant and substrate. The sealant is cured while in contact with both the substrate and the reinforcing fabric so that wetting by the sealant at the interface with the substrate and the interface with the fabric is

of optimal quality. Our hydrolytic stability testing was carried out primarily with glass as the substrate. Glass panels for the test specimens were cut from 1/8 in. window glass. Where glass was used as the substrate material, no primers were utilized with any of the elastomeric compositions tested. The test specimens were prepared in essential accordance with the method described in the procedures of ASTM C-794-75, with the exception of the curing schedule. All specimens were allowed to cure one month under bench-top conditions in the laboratory before initiation of the hydrolytic aging exposures.

Hydrolytic aging temperatures were identical with those used for aging of the ASTM D-412 tensile specimens. In general, aging intervals between monitoring tests were also the same, except that some of the shorter and intermediate test intervals were omitted at the lower aging temperatures in the interest of conserving samples for the longer test intervals. Following removal from the hydrolytic aging environment and prior to testing, all test specimens were oven-dried at 80°C for 16 hr. Testing involved use of a tensile test machine to pull the glass fabric reinforced strip of elastomeric composition back upon itself at an angle of 180° with the glass substrate. Force required to accomplish this was measured. Where the strip did not separate cleanly from the substrate, a razor blade was used to accomplish this separation and pulling was restarted. It was, however, quite common for failure to continue within the body of the elastomer rather than at the elastomer-substrate interface, indicating that cohesive strength of the elastomer was smaller than the strength of adhesion to the substrate. Four test specimens were measured for each aging temperature-aging interval combination. Results of these tests are reported in Table 13A.

Adhesion of the Class SC caulks to metal substrates was also examined, but in a less extensive and more qualitative manner. Metal substrates studied were galvanized steel and mill finish aluminum. In those instances where the supplier of the caulk recommended use of a primer on the metallic substrate, the recommended primer was used. The combinations examined are tabulated below.

Caulk-Metallic Substrate-Primer Combinations Examined

| Elastomer Code No. | Class | Metallic Substrate | |
|-----------------------|-------|---------------------------------|-------------------------------------|
| | | Galvanized Steel Primer Used | Mill Finish Aluminum Primer Used |
| C | SC | SS-4044 ¹ | SS-4044 |
| D | SC | None | None |
| B | SC | 1200 ² | None |
| E | SC | None | None |
| F | SC | 1200 | None |
| A | SC | None | 1200 |

¹General Electric primer SS-4044.

²Dow Corning primer 1200.

All of the ASTM C-794-75 specimens prepared with metallic substrates were aged by immersion in water at 100°C for 109 days. Following removal from the hydrolytic environment, all samples were permitted to air-dry at room temperature on the laboratory bench top for a period of one week. They were then evaluated qualitatively by observation of their behavior when pulled apart manually. Judgments as to the relative adhesive and cohesive strengths of each caulk-substrate-primer combination are tabulated in Table 13B.

The adhesion of all the caulking compounds (SC) to the glass and metal panels was adversely affected by immersion in hot water (Tables 13A and 13B). Careful examination of the modes of failure revealed that some failures were adhesive in nature; that is, the caulk pulled away from the panel, and others cohesive in which the caulk easily tore leaving a significant residue on the panel. Generally the failures displayed a combination of both mechanisms.

Under conditions of this test, several phenomena occur simultaneously. The elevated temperature promotes the loss of low molecular weight components such as residual solvent, plasticizer and polymer fragments, and in some cases enhances the state of cure.

Increased curing and loss of volatiles tend to improve the strength of the material. This is consistent with the improvements in peel strength shown by several of the compounds. As water enters the polymer matrix, it leaches out some of the low molecular weight compounds and also behaves as a plasticizer. In addition, it may weaken bonding between the reinforcing fillers and the polymer matrix. Both this and its plasticizing effects cause a diminution in the cohesive strength of the material. As the water displaces the polymeric material from the interface with the glass or metal panel substrate, a marked drop in adhesive strength results. Superimposed on all of these phenomena is the susceptibility of some polymers to undergo chain scission resulting from hydrolysis of primary chain linkages or cross-links. In time, hydrolytic degradation will lead to catastrophic failure of such materials.

It is very difficult to compare the effects of hot water on retention of adhesive peel strengths of different caulking compounds since in addition to considering the percent retention of original peel strength, one must also take into account the absolute magnitude of the initial strength. For example, since silicone RTV-103 (code C) has an initial peel strength fifteen times greater than Hypalon (code E) and almost eight times greater than butyl (code F), a far greater drop in the percent retention of the strength of RTV-103 can be tolerated.

Of the three caulking compounds which displayed relatively high initial strengths, RTV-103, 15 lb; acrylic (code D), 11.5 lb; and silicone EC-790 (code B), 9.4 lb; and the intermediate strength silicone DC-732 (code A), 4.3 lb, only the DC-790 exhibited good properties after four days in water at 125°C. The DC-732 exhibited the earliest evidence of catastrophic failure (Table 13A). On immersion at 100°C, DC-790 exhibited good property retention for 8-16 days. The RTV-103 was next best displaying a low but significant peel strength over the same period of time. The DC-790 was also the best performer on the 83°C test giving adhesive peel values close to 10 lb and 4 lb after

64 and 128 days, respectively. After an initial decline in strength, the acrylic showed good resistance to 83°C water, gradually improving to give a peel strength of ~ 7 lb after 64 days. The RTV-103 displayed a moderate value of 5 lb after 64 days and a low strength of 2 lb after 128 days. Compound DC-732 retained slightly over 2 lb peel strength after 16 days and 0.4 lb after 128 days. After 128 days, at 67°C, the DC-790 did not appear to have undergone significant degradation. The DC-732 which had lagged behind the RTV-103 at the higher temperatures, surpassed it at 67°C on a percent retention of initial property basis. It retained approximately 61% and 32% of its peel strength after 64 and 128 days, respectively, whereas the RTV-103 over the same time periods exhibited retentions of 20% and 17%, respectively. However, because of its greater initial strength, the RTV-103 displayed a higher peel strength than the DC-732 after 128 days.

The butyl and Hypalon were very difficult to evaluate because of their very low initial peel strengths of 1.9 lb and 1.0 lb, respectively. In addition, as a result of their weakness and tackiness, it was very difficult to handle and measure these materials. Despite handling problems, the data indicate that these two compounds did not undergo any significant degradation on immersion in water at temperatures as high as 100°C.

The scatter in the peel resistance data precluded the preparation of meaningful graphical representations of the decay in the combination of adhesive and cohesive strength of the specimens on hydrolytic aging. Some, but not all, of this difficulty can be attributed to the complexity resulting from dual modes of failure. A larger statistical sampling would render the data more interpretable; however, the inherently large scatter in the results, which were obtained under carefully controlled laboratory conditions, suggests that considerable variations in the quality of the caulking application would result in actual factory and field applications.

In use in actual collector systems, movement resulting from thermal expansion and contraction could cause failure; however, this is much less severe than the 180° peeling employed in these tests. Nevertheless, it appears important to design collectors so as to minimize dependency on retention of the adhesive and cohesive strength of the caulking compound.

f. Accelerated Weathering

Outdoor weathering as encountered in the flat-plate solar collector cell application consists primarily of a combination of ultraviolet light, moisture and elevated temperatures. Obviously, exposure to oxygen or ozone and to pollutants comprise a portion of the environmental picture, but test methods for accurate and simultaneous simulation of all factors involved are not available. ASTM C-793-75 is an established test procedure for caulking type elastomers (Class SC) which provides ultraviolet exposure, intermittent simulation of rain and a moderate degree of temperature elevation. The National Bureau of Standards has described a modification of ASTM C-793-75 test (Stiehler, R. D. et al., "Solar Energy Systems -- Standards for Rubber Seals," NBSIR 77-1437, March 1978)¹ in which specimens are thermally aged in air at a series of elevated temperatures prior to exposure to accelerated weathering. Exposure to ultraviolet light from a carbon arc for 1000 hr is proposed in this modification, but no mention is made of either simulation of rain or use of elevated temperature during the irradiation. Since extensive evaluations of the effects of exposure to elevated temperatures and ozone are being performed separately in our program, we prefer to limit accelerated weathering testing to essentially the conditions described in the original version of ASTM C-793. These conditions involve no prior thermal aging and 250 hr exposure to ultraviolet light from a carbon arc with eighteen minutes of water spray during each 2 hr of ultraviolet irradiation and a chamber temperature of 60°C during exposure.

The ASTM C-793-75 procedure utilizes a 1/2 in. mandrel test for evaluation of weathering specimens. We felt that this test was too qualitative and could possibly be misleading since occasional Class SC elastomers may undergo depolymerization during weathering as opposed to the more normal cross-linking reactions, and, as a consequence of the concurrent softening, actually show improved performance on the mandrel test even though severely degraded. In the hope of obtaining more quantitative and less ambiguous results, we exposed ASTM D-412 tensile specimens to weathering in place of the specimen prescribed by the ASTM C-793-75 procedure. Tensile specimens for this study were prepared as described in the procedures for Section d, "Thermal Stabilities of Class PS and Class SC Materials".

For mounting in the Weather-O-Meter, the samples were suspended from a 10 mil aluminum strip to which each was attached by a loop of bare copper wire. Each sample had been punched in the grip section of the dog bone to provide a 1/16 in. diameter hole for insertion of the wire. The wire loop which held the tensile specimen was suspended from the horizontal aluminum strip by having one end bent into a hook which passed over the top of the strip. Three specimens of each composition were suspended from a single strip of aluminum and a similar mounting was attached to the lower ends of the specimens to keep them properly extended for exposure to the ultraviolet light source. One sample assembly of this type for each composition to be tested was hung from the top rack of the Weather-O-Meter.

All Class SC specimens had received a four to six week cure at room temperature prior to mounting. It was found that two of these materials, Tremco Mono (acrylic) and Hypalon (chlorosulfonated polyethylene) could not sustain the weight of the lower aluminum strip at room temperature. Tensile specimens of both failed during overnight suspension in the Weather-O-Meter before the machine had been started. An attempt was made to salvage the test by post-curing fresh tensile specimens of both compositions first for 1 hr at 80°C and then for 2 hr at 100°C before

mounting. This expedient made it possible to secure test data on Tremco Mono, but both Tremco butyl sealant and the Gibson-Homans Hypalon failed to retain sufficient integrity during the weathering test to provide meaningful data.

An Atlas Electric Device Weather-O-Meter, Model XW-WR, was used to provide the accelerated weathering test exposures on the ASTM D-412 tensile specimens. This machine employs a carbon arc in conjunction with a Corex D filter to provide the ultraviolet exposure. The energy distribution curves of Fig. 55 compare this light with natural sunlight at Chicago. The conditions of the weathering test exposure described in the ASTM C-793-75 procedures have already been outlined. In practice, it was found that the exposure chamber temperature dropped to an average of 52°C during each 18 min spray period. During the 102 min between spray periods, average chamber temperature was 57°C. Relative humidity in the exposure chamber ranged between 97% and 100% during the spray cycles and remained at about 89% during the dry cycles.

Following the accelerated weathering exposure, the tensile specimens were evaluated by measurement of ultimate tensile strength, ultimate elongation and tensile modulus at 100% elongation. These measurements were performed in accordance with the procedures of ASTM D-412.

Table 14 displays the data obtained from the tensile specimens before and after the exposure to simulated weathering. It was found that these elastomers in general were relatively unaffected by the exposure used. The ethylene-acrylic polymer (Vamac) appears to show somewhat greater change than the other Class PS elastomers tested. Its increase in elongation and decrease in modulus at 100% extension suggest that some depolymerization has occurred with consequent reduction in degree of cross-linking. Performance of the Class SC caulks is less consistent. These compositions might be expected to become increasingly rigid during the weathering exposure due in part to continued loss of solvent and

plasticizer present in the material as received and in part to continuing cure. This would be expected to result in lower ultimate elongation and in higher modulus at 100% extension. The two Dow Corning silicone caulks and the acrylic conform to this prediction from the standpoint of decrease in ultimate elongation, but DC-732 does not show an increase in tensile modulus. Since the acrylic (Tremco Mono) lost its extensibility completely, its modulus could not be measured. Behavior of this acrylic is quite surprising since acrylics in general possess excellent weatherability. Since the formulation of this composition is proprietary, we are not able to speculate on the cause of this essentially complete loss of extensibility other than to suggest that exposure to ultraviolet light causes it to develop an unusually high degree of cross-linking. The tensile specimens of this particular composition were unusually thin, a circumstance which would tend to cause increased susceptibility to the action of the ultraviolet light source.

We believe that, to the extent that the ASTM C-793-75 test may be considered an adequate weathering exposure, all compositions tested, with the exception of the acrylic (Tremco Mono), show acceptable weatherability. It must be recognized that longer exposures to accelerated weathering would be likely to show significant differences in weathering resistance between compositions which appear equivalent on the test actually carried out.

g. Fungus Resistance

Resistance to attack by fungus is a meaningful consideration because collector assemblies may be stored for long periods of time prior to installation. Even though temperatures under service conditions are sufficiently high to kill the fungus organisms, sealants susceptible to attack by fungus could be degraded before the collector is installed.

ASTM D-412 specimens of both Class PS and Class SC sealants were exposed to fungal attack by an outside contractor, Microbac Laboratories, Inc. of Pittsburgh, PA. The fungal exposure employed was that defined by Military Specification, MIL-F-13927A (ORD) and in

more detail by Military Standard 810-C with the exception that one of the five organisms specified, *penicillium lilacinum*, was not available. This organism was replaced by *chaetomium globosum*. The other four specified organisms, *aspergillus flavus*, *aspergillus niger*, *penicillium citrinum* and *trichoderma* were utilized as directed.

The degree of growth of fungal organisms on sealant sample surfaces was observed after 30-day and 60-day exposure intervals by Microbac personnel. Their observations and ratings are presented in Table 15. The rating scale used is that established in ANSI/ASTM G-21-70.

Following both the 30-day and 60-day exposure periods, the specimens were characterized by measurements of tensile strength, ultimate elongation and tensile modulus at 100% elongation. Shore A hardness was also measured. Table 16 presents this data in comparison with values from tensile specimens not exposed to fungus. The data reported represent average values from three specimens. Fig. 56 pictures fungal growth on specimens after the 30-day exposure interval.

The visual observations on extent of fungal growth on rubber specimens indicate that silicone elastomers in general are more resistant than other types. The Hypalon (chlorosulfonated polyethylene) is also very resistant due presumably to inherent fungicidal properties associated with the sulfonic acid or with the hydrochloric acid which would be liberated when the aqueous fungal suspension is brought into contact with the elastomer. The relatively high resistance of the Tremco butyl rubber at 30 days is almost certainly due to presence of additives which exhibited fungicidal properties since the other two butyl rubbers performed poorly. The ethylene-propylene terpolymer, Nordel 3300-11, and the polyacrylic, 210-108-35-1, exhibited only light fungal growth and are probably acceptable from this standpoint. Despite visual indications of susceptibility to fungus with a substantial proportion of the formulations tested, change in physical properties, in general, was relatively small. Increase in tensile strength or modulus is very probably due to development

of increased cross-linking during the fungal exposures which are carried out in the temperature range of 80° to 85°F. The Class SC materials normally contain a volatile solvent which is incorporated to provide a workable viscosity at the time of application of the caulk. Escape of this solvent during the fungal exposure provides an additional mechanism for increase in tensile strength and modulus as well as in hardness. It would appear from the generally small observed physical changes that degradation due to fungus is not a major problem. The fungal exposure provided in the test is certainly far more severe than any which would be encountered in storage of collector units. It does appear that a fungicide should be incorporated into any butyl rubber formulations which are used and this observation applies also to the fluorocarbon elastomer which exhibited an unexpectedly large amount of fungal growth.

h. Ozone Resistance

Exposure to atmospheric ozone results initially in cracking and crazing of elastomers. Since defects of this type, once initiated, can propagate to result in mechanical failure under conditions of stress, it becomes important to compare ozone resistance of candidate sealants for the solar collector cell application.

Test specimens of the same Class PS and Class SC elastomers evaluated for resistance to fungus attack were exposed to ozone at the Ozone Research and Equipment Corporation of Phoenix, AZ in accordance with the procedures of ASTM D-1149-55T (Mod.) and ASTM D-518-Procedure C. The ASTM specimens were so mounted that they were held at 15% elongation during an exposure of 166 hr at 40°C in an atmosphere containing 100 ppm ozone. These stretched samples were observed daily under 7X magnification for visual signs of deterioration. Results are summarized in Table 17.

Tremco's Mono (acrylic) and butyl caulk compounds and the Gibson-Homans butyl caulk were not evaluated because physical integrity of the specimens was not adequate to sustain the 15% elongation. The

Tremco butyl SC compound, having a consistency like cardboard, was too rigid for the 15% stretch mounting, and the acrylic and the Gibson-Homans butyl SC compounds remained too soft and tacky to handle even after a 60-day exposure to the room environment.

With the exception of the butyl rubber, all of the materials evaluated appeared to display good resistance to ozone. It is suggested that antiozonants be incorporated into butyl rubber formulations, especially in applications where the butyl compound is exposed to the atmosphere or is in proximity to large electrical equipment.

i. Chemical Degradation and Outgassing Studies

The outgassing of preformed seals (PS) and seal caulks (SC) is of high concern to solar collector manufacturers. The evolved materials usually condense on the cooler glass or plastic glaze surfaces, either in the form of a liquid or a low melting solid. These deposits significantly reduce the transmittance of solar light (Table 18) through the glass and thereby reduce the efficiency of the collector.

Inside each solar collector two types of dew (condensation) points exist. One is a moisture dew point and the other an organic hydrocarbon vapor dew point. We are all familiar with water vapor dew points as a function of temperature. Organic dew points are analogous to the moisture dew point. The amount of a given organic vapor present in the air at any specific pressure/temperature condition is a function of the volatility of that particular organic material at those conditions.

Naturally, air will hold more vapor, whether it be moisture or organic, at a high temperature than it will at a low temperature. As the temperature decreases to a point below the moisture or organic vapor dew point, condensation will occur on the coolest surfaces in the collector which usually include the glass or plastic glaze.

As the temperature of the collector rises above the dew point, the condensate on the glass glaze will evaporate. The evaporation of moisture is clean and complete, and no residues or deposits are left on

the glass. However, over long periods of time, moisture condensates can slowly leach out sodium or other metal salts from the glass and a white salt deposit will slowly form. These salt deposits also greatly reduce the transmittance of solar light through the glass.

Organic condensates result from the diffusive loss of low molecular weight compounds originally present in the compounded elastomer seal and of products formed when the sealant or coating is degraded or depolymerized as a result of exposure to elevated temperatures, water, ultraviolet light, oxygen, ozone, etc. The organic condensates do not evaporate from the glass in a clean manner and usually leave either a colored oily residue, a white powdery deposit or a continuous solid film. These organic residues result from further chemical reactions of the liquid organic condensate through a thermal, oxidative or ultraviolet induced mechanism. During the many condensation/evaporation cycles that a solar collector experiences, these condensate coatings slowly build up, producing an adverse effect upon light transmittance. The sources of organic outgassing consist of the preformed seals, caulk seals, thermal insulation, organic coatings and organic polymeric materials used in structural applications.

A test was developed to study the degradation and outgassing of seals and coatings. This test method provides data on the rate and degree of outgassing of a material, and also provides an analysis of the composition of the evolved products.

In this test a sample of the material is placed in the bottom of a test tube, and a sodium chloride crystal is mounted in the open end of the tube and held in place by a series of dimples in the glass wall of the tube. This assembly (Fig. 57) is then positioned vertically into a closely fitting hole in the top of an oven so that only the lower two-thirds of the tube is inside the oven. Most of the condensable outgassing products condense on the bottom surface of the sodium chloride crystal, with a small amount condensing on the glass tube area

surrounding the crystal. Condensable volatiles are observed as weight increase of the crystal and noncondensables by the difference between this and the total weight loss of the test sample. The coated sodium chloride crystal is placed directly into the infrared spectrophotometer for determination of the chemical nature of the condensable products.

Many sealant compositions were characterized by this test (Table 1). The preformed sealants (PS) were evaluated as received. The seal caulks (SC) compounds were cast in approximately 1/16 in. thick sheets on Mylar film, which had been treated with a Teflon mold release agent. All of the SC compounds were room temperature vulcanized for four to six weeks prior to testing.

In the standardized outgassing test, the oven was operated at 150°C for a period of nine days. The sodium chloride crystal reached an equilibrium temperature of $65 \pm 2^\circ\text{C}$ during these tests. The compositions of the condensable products were identified by infrared analysis using a Perkin-Elmer, Model 700 Infrared Spectrophotometer.

The results of these outgassing studies are displayed in Tables 19, 20 and 21, and Figs. 58 through 66.

The silicone seal caulks (Table 19 and Fig. 58) showed varying degrees of volatility. Compounds B and W (DC-790 and DC-795) produced the lowest amount of volatiles (1.3% and 1.7%, respectively), while Compound V (G.E. 2400) produced the greatest amount of volatiles (7.8%). All of the other silicone seal caulks fell within these extremes. (Materials described here which were not subjected to the other tests were received too late for those tests.)

The preformed silicone seals produced less volatiles than the silicone seal caulks (Table 19 and Fig. 59). Compound H (Silastic 747) produced the most volatiles (1.1%) in this group, while Compound G (G.E. silicone SE-7550) produced the lowest (0.04%).

The infrared spectra obtained on all of the silicone sealants showed the condensables to be predominantly low molecular weight linear or cyclic alkyl polysiloxanes (Table 19). The alkyl polysiloxanes are characterized by the three major absorption bands at 800, 1000-1100 and 1260 cm^{-1} (Fig. 60, Spectrum 2). The condensables from Compound A (DC-732 white) also possessed infrared absorption bands at 1380 and 1460 cm^{-1} (Fig. 60, Spectrum 1). These absorption bands, plus the bands between $2800\text{--}3000\text{ cm}^{-1}$ indicate the presence of a paraffinic oil. Compound H (DC Silastic 747) shows additional absorption bands at 1490, 1590 and 1720 cm^{-1} (Fig. 60, Spectrum 3). These bands are characteristic of aromatic type esters.

The intermediate temperature caulk sealants produced a large quantity of volatiles (Fig. 61). Compound D (Tremco Mono) produced 4.4% volatiles, Compound E (Gibson-Homans Hypalon) gave 28.4% and Compound F (Tremco butyl sealant) produced 14.5% volatiles (Table 20). The infrared spectra of the condensables obtained from these materials are shown in Fig. 62. The condensable volatiles from Compound D are low molecular weight acrylic fragments or oxidized paraffinic oil. Compound E liberated condensables which appeared to contain an alkyl sulfonic acid ester. The condensables from Compound F are either oxidized paraffinic processing oil or oxidized low molecular weight butyl fragments or a combination of both.

The preformed intermediate temperature sealants produced considerably less volatiles than their seal caulk counterparts (Fig. 63). The two fluorocarbon sealants (Compounds L and M) showed less than 0.02% volatiles, while Compound J (ethylene/acrylic) produced 3.89% (Table 20). The infrared spectra of some of these compounds are shown in Fig. 64. Compound J produced volatiles consisting of low molecular weight ethylene/acrylic fragments. Compounds O and R, which are butyl sealants, produced volatiles consisting of oxidized processing oil or oxidized low molecular weight butyl fragments. Compound P, which is also a butyl sealant, was found to give off stearic acid.

The data obtained on several ethylene-propylene terpolymers (EPDM) is displayed in Table 21 and Figs. 65 and 66. Compound N (DuPont Nordel 3300-11) exhibited low volatility (0.8%). Compound S, an extruded EPDM absorber plate (Sola Roll) manufactured by Bio-Energy Systems, produced 3.8% volatiles after nine days and 6.0% after 16 days of testing (Table 21). Compound T consisted of three molded sheets of EPDM 18464 and also was supplied by Bio-Energy Systems. These sheets were numbered upon receipt, as 1, 2 and 3, for our identification purposes. Test specimens were taken only from sheets 1 and 2 for the outgassing test. As seen by the data (Table 21 and Fig. 65), a great difference in volatility was found between sheets 1 and 2. Sheet 1 (Compound T, #1) produced high volatility ranging between 3.6-4.7%. Sheet 2 (Compound T, #2) only had a volatility of 1.1-1.3%. After testing for 16 days, the difference had increased to 5.3-6.0% for T, #1 versus 1.2-1.5% for T, #2.

The condensable product from Compound N is a naphthenic processing oil as characterized by the hydrocarbon absorption bands at 1260, 1380, 1460 and 2800-3000 cm^{-1} , and the aromatic unsaturation band (C=C) at 1600 cm^{-1} (Table 21 and Fig. 66). The spectra for the other EPDM samples are essentially identical and the condensables have been identified as being either oxidized paraffinic oil or oxidized low molecular weight chain scission fragments or a combination of both.

As shown in Fig. 65, the outgassing of EPDM compounds S and T is proceeding at a fairly uniform rate. A constant rate of volatile formation is a good indicator of depolymerization or reversion of the polymer. The infrared analyses have not shown any significant change in composition of the condensables since the beginning of the test. Therefore, one would have to conclude that these condensables are essentially oxidized low molecular weight EPDM depolymerization fragments.

The quantity of very low molecular weight noncondensables evolved is less than 0.4% for most of the test specimens. It is believed that a large portion of the noncondensable fraction is moisture, which had been adsorbed by the specimens. Silicones C, U and V produced high amounts of noncondensables which consisted primarily of low molecular weight alkyl siloxanes, acetic acid and/or other products formed during room temperature vulcanization. Butyl sealants, O and R, also produced a substantial quantity of noncondensables which were identified as low molecular weight butyl polymer, organic solvents and moisture.

4.5 Recommendations on Limitations of Elastomers

When all property requirements are considered, one must conclude that there are no presently available sealants which meet all requirements of the thermal solar collector application. Limitations of the several available types of materials will be discussed by property category.

4.5.1 Compression Set

Test exposures upon which data extrapolations are based are continuous exposures and lead to thermal life predictions based on continuous exposure. In actual service, life will be longer since exposure to top service temperatures is intermittent. The factor by which service life will exceed continuous thermal exposure life will range from about three up to far higher values depending upon geographical location and positioning of the collector. We feel that an elastomer used as a gasket must not exceed 50% in compression set in order to retain reasonable ability to maintain a seal in a fixed gap. Our life and service temperature estimates are therefore based upon time required to reach 50% compression set at a selected aging temperature.

The best candidate to meet reasonable service requirements is the fluorocarbon rubber (Viton). Its 510-day extrapolated life at 150°C indicates a four year service life under conditions of maximum severity

and perhaps several times that long under normal conditions. The best silicone in the compression set service category, NPC 80/40, exhibits a life of only 100 days at 125°C and extrapolates to a life of about 315 days at 110°C. This is not considered adequate for acceptable service life and yet all other compositions tested save the Viton were poorer than this.

Despite these data, Viton cannot be unequivocally recommended. Its low temperature compression set is unacceptably high. We must conclude then, that if selection of a gasketing elastomer is limited to presently available formulations, it becomes necessary to redesign the thermal solar collector unit in such a way that the frames which must be sealed are spring-loaded and apply continuous pressure against the gasket.

4.5.2 Corrosion

None of the compositions tested attacked anodized aluminum, the solar collector frame material used by most manufacturers, and, under the conditions of our tests, only butyl attacked mill finish aluminum. Hypalon, butyl, the Hycar polyacrylic and the Nordel EPDM all attacked galvanized iron. It, thus, appears that galvanized iron is not the frame material of choice for the flat-plate solar collector unit application and that corrosion by elastomeric sealants will not be a problem if the collector cell frames are fabricated of anodized aluminum.

4.5.3 Thermal Stability by Elevated Temperature Aging of Tensile, Weight Loss, and Hardness Specimens in Air

Monitoring of tensile strength, ultimate elongation, weight and hardness as these properties changed during thermal aging in air was utilized as a further indication of rubber degradation even though hardness might be regarded as the only one of these characteristics having real relevance to performance in the solar collector unit. The Arrhenius treatment was applied only to the Class PS tensile specimens and even there it was found not to be suitable for all compositions tested.

Because failures (decline of tensile properties to 60% of initial values) were not experienced with the fluorocarbon (Viton) tensile specimens, no Arrhenius plots could be made, but it would appear from the data that the material would be useful at a temperature of at least 175°C. The Class PS silicones would perform acceptably well at 150°C or slightly below. The Hycar polyacrylic, the Vamac ethylene-acrylic and the Nordel EPDM should be useful at 125°C or slightly lower. The order of performance as indicated by thermal aging of tensile specimens in air is ethylene acrylic > polyacrylic > EPDM. Butyl exhibits the poorest stability of all and appears by the tensile life criterion not to be useful at any temperature above 100°C. Butyl rubber is very extensively used by some manufacturers of flat-plate solar collector cells because of its unique low impermeability to water and other vapors. The wisdom of this policy is elusive since nearly all collector units manufactured are vented to provide breathing and thus are not hermetic. We suspect that many manufacturers, particularly the smaller operations, may be unaware of the poor thermal stability of the butyl rubber.

The Class SC organic caulks, Hypalon (chlorosulfonated polyethylene), Mono (acrylic) and butyl are all formulated with relatively large amounts of such processing aids as solvents and plasticizers and, because of the volatility of these components, should not be used in locations where there is communication with the interior of the solar collector cell. Such use is almost certain to result in deposition of condensable products on glazing and collector plates with lowering of solar cell unit efficiency. These materials tend to lose their elastomeric properties, particularly ultimate elongation, quite rapidly, even at temperatures as low as 125°C and 100°C, so that their utility in the solar collector cell application is questionable at best. The Class SC silicone caulks, on the other hand, appear comparable to the best of the Class PS silicones in performance in retention of tensile strength and ultimate elongation during thermal aging and would be useful at 150°C. RTV-103 and DC-732 are superior to DC-790.

It should be pointed out that these materials are intrinsically rather weak initially, even though they resist deterioration with aging, and, in consequence, should not be used in locations where there is appreciable physical stress.

4.5.4 Hydrolytic Stability

The Nordel EPDM rubber was found to be the best in hydrolytic stability. The fluorocarbon (Viton) and the silicones were somewhat disappointing due presumably to a plasticizing action by water in the former case and to hydrolytic chain scission in the latter case. The acrylics, Hycar and Vamac, were relatively good, and also better than expected since they incorporate hydrolyzable groups. The Class PS butyl (8EX-123) also exhibited good hydrolytic stability, although its chemical structure led us to expect better performance than was actually observed. It is quite possible that the changes in tensile characteristics of the Class PS butyl were due to the thermal rather than the hydrolytic environment.

It is important to realize that the total immersion in elevated temperature water conditions which were used as the hydrolytic environments in our test program are useful only to compare hydrolytic stabilities of different materials. These conditions cannot be extrapolated to solar collector cell service conditions and used to predict life because the service environment permits water to be alternately adsorbed and then expelled on a cyclic basis. Actually we do not regard hydrolytic stability as more than a minor problem at worst since the hydrolytic stabilities observed in the water immersion tests lead us to believe that no material tested would fail in service from this cause prior to the occurrence of other modes of failure.

It is interesting to observe that DC-790 far outperforms DC-732 and RTV-103, the other two silicone caulks studied, on the adhesion-in-peel testing as contrasted with the thermal aging in air studies where it is by far the poorest of the three. In locations where

retention of adhesion is important, DC-790 must be considered the best caulk tested. The Class SC acrylic, Mono, shows better initial adhesion on this test than the other two organic caulks, Hypalon and butyl, but deteriorates rapidly at 100°C and falls close to the level of the other two at the two lower aging temperatures after long exposure. When the severity of these tests is considered, it seems reasonable to predict that the acrylic would retain better adhesion during its service life. It must be kept in mind, however, that all Class SC caulks tested lose their elastomeric properties quite rapidly during thermal aging, even at low aging temperatures and that even the acrylic, the most thermally stable of the three, would not be useful on a long-term basis above about 100°C.

4.5.5 Accelerated Weathering

At the Weather-O-Meter exposure used, effects on tensile properties of the elastomers tested proved to be quite small with the exception of Mono, a Class SC acrylic. This material lost its extensibility completely, suggesting that the exposure to ultraviolet light caused a large increase in cross-linking. It should be noted that the Class PS acrylic did not exhibit this effect. If the Weather-O-Meter exposure used in these tests is adequate to expose weathering problems, we must conclude that weathering is not of more than minor concern. Since the test exposure has been standardized as an ASTM test, it is to be regarded as having considerable authenticity, but results of longer test exposures would be interesting.

4.5.6 Fungus Resistance

Although the test exposures to fungus resulted in limited fungal growth on most of the elastomeric compositions tested, changes in tensile properties were generally small. Fungal exposure is not regarded as a significant problem except for those few compositions where relatively large amounts of fungal growth were observed. It would appear that fungicides should be incorporated into butyl or fluorocarbon rubber formulations.

4.5.7 Ozone Resistance

With the exception of the Class PS butyl rubber, all compositions tested exhibited good resistance to ozone and, generally, this environmental factor is not regarded as a severe problem. Poor resistance of the butyl rubber should be correctable by incorporation of an antiozonant into the formulation. The three organic Class SC materials, Mono (acrylic), Hypalon (chlorosulfonated polyethylene) and butyl could not be tested because these materials failed quickly when mounted at 15% elongation.

4.5.8 Outgassing

The outgassing studies have shown the Class SC elastomers to produce considerably more volatiles than the Class PS elastomers. Fluorocarbon PS elastomers produced the smallest amount of volatiles and were rated the best in this property. Certain silicone PS elastomers were also found to produce low volatiles and would be our second choice. The acrylics and Nordel (EPDM) also showed relatively low volatiles and would be our third and fourth recommendations, respectively.

It must be realized that these results were obtained in the laboratory through testing at only one test temperature (150°C). The actual initial rates at which volatiles are given off in an operating solar collector could, therefore, be higher or lower depending on its operating temperature. It is difficult to relate accurately the effect of condensables on the actual solar light transmittance through the collector glaze over the life of the collector. Laboratory tests have shown, however, that condensables do significantly reduce solar light transmittance and are one of the major factors in reducing solar collector efficiency.

It, therefore, becomes important that only those elastomers that show low volatility be selected for collector applications.

4.6 Recommendations for Selection of Polymers and Compounding Formulations

We have tabulated below the elastomer types which, based upon the results of this study, we believe to be viable candidates for the flat-plate solar collector cell application in both the high and intermediate temperature service categories. These selections are based upon consideration of physical properties, availability and economics.

1. High temperature
 - Fluorocarbons
 - Silicones
2. Intermediate temperature
 - Acrylics
 - Acrylic copolymers
 - EPDM

The one physical property which we observed to limit service lives of elastomers most severely is elevated temperature compression set. Most promising approaches toward improving this characteristic involve moderate increase of cross-link density and increase of filler content. The fluorocarbon rubber and the polyacrylic in particular suffer from excessive compression set at low temperatures. This difficulty is associated with high glass transition temperatures and thus with development of crystallinity during low temperature exposure. A promising approach to correction of this problem involves modification of the polymer chains in such a manner as to decrease regularity in the chain. This objective might be achieved by introduction of an additional monomer which can be copolymerized into the growing chain at random intervals. Other possibilities involve use of polymer blends or alloys and IPN (interpenetrating networks). An approach certain to be effective at reducing low temperature crystallinity involves incorporation of certain high molecular weight plasticizers into the composition. Ideally these molecules would be grafted onto the primary chains of the polymer to prevent their volatilization. If this cannot

be accomplished, then great care must be exercised in selection of materials introduced in order to minimize volatilization.

Rubber formulations in general are quite complex as illustrated by the compositions whose formulations are displayed in Table 2. Functional additives and processing aids such as solvents, plasticizers and lubricants must be selected with great care and with a view to minimizing volatility. The weaknesses of each type of elastomer must be considered carefully in designing a formulation which utilizes that elastomer. Functional additives such as antioxidants, antiozonants, UV stabilizers and fungicides must be incorporated when necessary to improve the performance of a particular elastomeric system, but are better omitted when not really needed.

Optimization of the better formulations which we have evaluated is an additional important consideration. In obtaining compositions for testing, we requested suppliers to provide formulations which they considered best adapted to the solar cell application. It is likely that the materials provided, although selected with flat-plate solar collector cell environment requirements in mind, were not in most cases actually formulated for that specific application. Even in those isolated cases where they were so formulated, it is unlikely, due to the rather small volume of the solar cell market at this time, that any real attempt at optimization was made.

4.7 Breathing Survey

A computerized literature search on solar collector breathing was made of the following data bases.

1. Lockheed Information Retrieval System
 - NTIS - National Technical Information System
 - U.S. Patents
 - Chemical Abstracts
 - Engineering Index

2. Department of Energy (DOE) System - RECON

- Energy Research Abstracts
- Nuclear Science Abstracts

The keywords which were used in various combinations in this search were solar collector(s), flat-plate collector(s), flat-plate solar collector(s), breathing, venting, vent, pressure, seal, desiccant(s), moisture, adsorbent(s), and outgassing.

Approximately one hundred print-out references were obtained from this search; however, only three of these, one German² and two United States patents,^{3,4} provided any useful information.

The best source of information proved to be solar collector manufacturers. Many manufacturers and suppliers were contacted for information on their solar collectors and 35 replies were obtained. In Table 22 are tabulated the results of this survey on 17 manufacturers of solar collectors.

A summary of the data reveals that 16 of these solar collectors are identified as being either vented, breathing or self-draining units. One collector made by Energy Systems, Inc. is described as being hermetically sealed. Only four manufacturers reported using a desiccant in their collector, and in all except one case, this was silica gel. One manufacturer uses a duo-adsorbent (Davison 801) consisting of molecular sieve and silica gel.

Discussions with several solar collector manufacturers indicated that they did not feel that the moisture contained in the air as humidity was a serious problem in collector breathing. However, if the seals permitted leakage of gross amounts of rain water into the collector, this was considered to be a serious problem due to salts and corrosion products being deposited on the glazing.

The most serious problem appears to be the outgassing of the various polymeric materials used in the construction of a solar collector. The materials that have a potential for outgassing include

preformed rubber seals, caulk seals, absorber plate coatings, and insulation. They evolve products that can condense out on the cooler glass glaze, reducing the solar light transmittance and thereby lowering the efficiency of the collector.

Most of the solar collector breathing (moisture removal) techniques are derived from the multiple glazed breather window industry. Two patents^{5,6} were retrieved that describe these methods and techniques. Also, calculations were provided in these patents for the determination of the amount of desiccant required to remove moisture during the expected life of a multiple glazed window.

4.8 Effects of Breathing -- Analysis of Solar Collectors

Five solar collectors, which had been in service at three different locations, were obtained for analysis. These were examined with regard to their design, fabrication and materials of construction. Particular concern was given to problem areas such as leakage, corrosion, glaze deposits, deterioration of absorber plate coating, and degradation of sealants.

4.8.1 Towns Elementary School, Atlanta, Georgia

Two collectors were received from this location. These were manufactured by PPG Industries, Ford City, PA, and had been in service for three years and two months.

Construction. The overall view of this unit is shown in Fig. 67. A cross-sectional diagram of its construction is shown in Fig. 68. It is constructed using a double glaze of PPG Herculite tempered safety glass and an absorber plate made from roll bonded aluminum. The glazes and the absorber plate are separated by 1/2 in. galvanized iron alloy spacer tubes. These two spacers contain approximately 225 g of silica gel each. The spacer tubes have direct communication with the interior of the collector through a series of openings provided by a

crimp seal. The spacer tubes also communicate with the outside ambient through two 1/16 in. vent or breather tubes located at one corner of the spacer (Fig. 69).

The absorber plate coating was the Alcoa 655 process coating. This coating (proprietary) was prepared from an aluminum alloy/silicate mixture, which when heated to an elevated temperature, corroded the aluminum in such a way as to form a black finish.

The primary seal between the glazes, spacers and absorber plate was a bead of a butyl caulk seal material (Fig. 68). The secondary seal was formed by a 2 in. strip of 10 mil thick aluminum, coated on one side with 40-50 mil of the butyl sealant, and then formed around the edge of the assembly as shown in Fig. 68. A stainless steel channel placed over the secondary seal locks all the components into position by its squeezing action.

A 4 in. layer of fiberglass is then adhered to the back of the absorber plate to complete the unit.

Analysis. The first and most obvious observation one noted about these two collectors was the complete absence of a black absorber plate coating or finish. The absorber plate surface, which was initially black, now has the appearance of sandblasted gray aluminum. Discussions with Alcoa revealed that this coating (Alcoa #655 process) had poor stability and short life, and was no longer being used.

The double glazes showed no visual signs of haze and/or an opaque coating. Light transmittance values, using an incandescent light source and a photoelectric receiver (Fig. 70) showed no significant reduction. Fig. 71 shows the microampere values obtained at various positions on the glaze. Fig. 72 shows the correlation between microampere value and actual light transmittance loss, as measured between 400-950 nm, using a Coleman Spectrophotometer. Due to the shattering and fragmenting quality of tempered glass, pieces suitable for use in the spectrophotometer could not be obtained (see Appendix II).

The inner surface of the second glaze, facing the absorber plate, was coated with a very thin layer of a transparent material. Weight calculations indicated this coating to be present in the amount of 0.033 g/m^2 . Due to its transparency, it did not appear to reduce the light transmittance. An infrared spectra analysis of a sample of the coating removed with chloroform, indicated this material to be very similar to that obtained from a pyrolyzed sample of the butyl sealant. The main difference was a carbonyl (oxidation) adsorption band ($1725\text{--}1735 \text{ cm}^{-1}$) found in the glaze coating. It is hypothesized that the outgassing products from the butyl sealant condensed on the cooler glass glaze and were slowly oxidized by a thermal or an ultra-violet induced oxidation.

No water leakage or migration was observed through the primary seals between the glass glazes. However, both collector panels did show some slight seal failure and water migration into the space between the second glaze and the absorber plate. Rust was found in a few regions on the outer surfaces of the spacers and indicated some secondary seal failure (Fig. 73). Corrosion was also found between the spacer and absorber plate and indicated some primary seal failure.

A 1 in. wide band of white salts or corrosion products was found on the bottom of the absorber plate (Fig. 74). These materials were very slightly soluble in water, but were readily dissolved by dilute hydrochloric acid.

The seal failures occurred through one or two failure mechanisms. First, several small areas were found in which the butyl sealant did not adhere to the spacer or glass glaze. These areas did not show any corrosion or rust and thus, water had not leaked into them. These non-adhering areas were probably caused by an improper application of the secondary seal, or an oily smear on the spacer. Secondly, the loss of adhesion and leakage through the primary seal between the spacer and absorber plate could also have developed as previously mentioned.

Another possibility is that the opening could have resulted from a shearing action set up by the difference in the coefficient of thermal expansion between the aluminum absorber plate and the iron alloy spacer.

4.8.2 National Bureau of Standards, Gaithersburg, Maryland

Two collectors were also received from this location. These also were manufactured by PPG Industries, but possessed certain differences from the other previously discussed PPG solar collectors. These units were operated from March 1975 to May 1976.

Construction. An overall view of this unit is shown in Fig. 75. A cross-sectional diagram of its construction is shown in Fig. 76.

In these units, there were no primary seals between the glazes, spacers and absorber plate, and the secondary seal was only present at the outer edges of the outer glaze and the absorber plate. These collectors contained a steel enclosure which was fastened to the stainless steel channel with adhesive-backed aluminum tape. Thus, the fiberglass would not be exposed to installers and handlers.

Analysis. The most apparent problem with these collectors was their ability to leak rain water and the associated corrosion and rusting.

The sealing technique used in these units was grossly inferior to the other PPG units. As a result, the spacers are corroded over 60% of their surface area, the absorber plate has many corrosion sites (Fig. 77) and the glazes are coated with a white opaque discontinuous deposit of salts and corrosion products (Fig. 78). Weight measurements indicated these coatings to be present in the amount of 0.151 g/m^2 for the bottom side of the outer glaze to 0.237 g/m^2 for the upper side of the inner glaze. The light transmittance values as shown in Fig. 79 are significantly lowered by as much as 25% for the combined glazes.

No evidence of a butyl seal condensate coating was found on the inner glaze. However, a multicolored coating was found in several areas of the glaze (Fig. 80). This coating was identified by infrared analysis to be a polyacrylate.

Three heavy deposit streaks ran across each inner surface of the outer glazes. These indicate hotspot areas, and appeared to be caused by external metal cross-bars which were used to hold the collector in its service position (Fig. 81). The absorber plate coating was of the lacquer variety and was in excellent shape, and jet black. Infrared analysis indicated it to be based upon an acrylic type polymer.

The steel enclosure, isolating the fiberglass, was badly rusted. In many areas, particularly the corners and edges, it had corroded completely through (Fig. 82).

4.8.3 University of California Laboratory, Los Alamos, New Mexico

One solar collector was received from this location. This unit was manufactured by the Turbo Refrigerating Co., Denton, Texas. It had been in continuous operation between May 1, 1975 and February 9, 1979 (three years and nine months).

Construction. An overall view of this unit is shown in Fig. 83, and a cross-sectional diagram of its construction is shown in Fig. 84.

This unit is a double glaze collector using untempered, low iron glass. Each glaze consists of three individual glass panels, which are connected to each other through an H-shaped preformed silicone seal (Fig. 24). The glazes are sealed into the steel spacer by means of a C (channel) shaped silicone seal as shown in Fig. 24. The steel spacer is attached to the main steel frame by a steel locking sleeve which is riveted to the frame. A silicone caulk seal is applied over the rivet head and end. Also, a caulk seal is applied at the interface between the sleeve, outer glaze and its preformed silicone seal.

The absorber plate, as well as all other steel construction members, is coated with a black velvet coating. A polyurethane foam is used as the back insulation. No provision was made for venting, breathing or drying.

Analysis. This unit appeared to be well fabricated and sealed. No evidence of gross migration (leakage) of rain water into the interior of the collector could be found. A small amount of moisture had been present in the unit as evidenced by some droplet residue marks, and some water streaks on the inner glaze. Also, a narrow band of stain and discoloration was found on the bottom end of the absorber plate, which could indicate a small pool of water had been present.

The rubber preformed seals were identified by infrared analysis as silicones. The channel seal, used in sealing the glaze into the spacer, was designed so as to press upon the glaze. As shown in Fig. 24, the ends of the channel seal were thicker than the rest of the seal. When they are inserted into the spacer slot, more pressure is exerted on the ends and they are squeezed very tightly on the glass. The H-shaped seal, used to join the glaze panels, relies completely on a snug fit for its sealing action. Apparently, both of these seals were leak-proof.

The outer glass glaze was clear and clean, and showed no significant reduction in light transmittance. The inner glaze, however, did show a slight reduction in transmittance due to a white, powdery, and discontinuous coating on the glaze surface towards the absorber plate. Light transmittance values are shown in Fig. 85. Fig. 86 shows the blotchy nature of this coating. This coating was present in the amount of 0.129 g/m^2 and was insoluble in both water and dilute hydrochloric acid, thus indicating it was not a corrosion/salt type product. An infrared analysis showed it to be 100% silica, such as fumed or colloidal silica.

No evidence of corrosion or rusting was found in the interior, or for that matter, the exterior of the collector.

4.9 Desiccants

Desiccants are materials that remove or take up moisture. There are two classes of desiccants; these are solid desiccants and liquid desiccants.

There are three mechanisms through which desiccants take up moisture. These are:

1. Surface (physical) adsorption and capillary condensation
2. Chemical reaction
3. Chemical reaction followed by dissolution

In this study, we will limit ourselves to solid desiccants which remove moisture by either mechanism 1 or 2.

1. Silica gel⁷ is a porous, granular, amorphous form of silica. Its internal structure is a vast network of connected micropores. These pores attract and take up moisture by physical adsorption and capillary condensation. Silica gel will also adsorb alcohols, hydrocarbons and other organic compounds. However, silica gel preferentially adsorbs water vapor in the presence of organic vapors. In fact, water vapors will completely displace the organic vapors from silica gel saturated with organic vapors.

The moisture capacity of silica gel, as a function of relative humidity, is shown in Table 23. Its maximum capacity, in the static state, is about 42% @ 100% RH, and thus, silica gel is one of the most efficient and most widely used desiccants.

Another advantage of silica gel is its ease of regeneration. Moisture saturated silica gel starts to lose moisture at 200°F and requires only a few hours to be reactivated. No other desiccant can be reactivated at such a low temperature.

Silica gel is, therefore, an ideal desiccant for use in solar collectors.

2. Activated alumina⁸ is an aluminum oxide in a porous, amorphous form and having a high surface area. Activated alumina holds moisture by physical adsorption and capillary condensation. It has a preference for moisture, but will also adsorb organic vapors. As shown in Table 23, its moisture capacity, in the static state, is about 18% at 100% RH. Its capacity is about 60% less than that for silica gel. Activated alumina can be regenerated by heating to 350°F to 600°F.

Activated alumina, therefore, does not appear to be a good candidate desiccant for solar collectors because of its low moisture adsorption and its high regeneration temperature.

3. Alumina gel⁸ is an activated alumina with approximately 1.5 to 2.0% SiO₂ (silica gel). It is a porous material possessing high surface area. It holds moisture by physical adsorption and capillary condensation, and has a preference for water over organic compounds. Alumina gel will hold about 36% moisture at 80% RH and requires temperatures in excess of 350°F for regeneration.

The alumina portion of alumina gel, which is the major portion, would have great difficulty in being regenerated in a solar collector. Therefore, it is not considered as a viable candidate.

4. Calcium sulfate and Drierite⁹ are anhydrous calcium sulfates in a granular, porous form. Calcium sulfate takes up moisture by two types of phenomena. First, on exposure to moisture, it forms the hemihydrate ($\text{CaSO}_4 \cdot 1/2 \text{H}_2\text{O}$) and takes up about 6.6% water. Secondly, due to its porosity, it takes up an additional 5.4-7.4% moisture by physical adsorption and thus, has a total moisture pick-up of 12-14%. Its regeneration also takes place under two different heating regimes. At 250°F, the physically adsorbed moisture is driven off; however, it requires a temperature of 400-425°F to break up the hemihydrate.

Calcium sulfate type desiccants, therefore, would not be suitable for use in a solar collector.

5. Magnesium perchlorate⁹ [$\text{Mg}(\text{ClO}_4)_2$] has a high affinity for moisture and will absorb about 48.5% by weight. Its mechanism for drying is the formation of the hexahydrate $\text{Mg}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$. As the moisture absorption proceeds through the mono-, di-, tri- and penta- forms of the hydrate, its rate of reaction decreases and the desiccant becomes less efficient. In regeneration from the hexahydrate, the first hydrate can be removed at 275°F, while the remaining hydrates become increasingly more difficult and require temperatures in the range of 400-500°F.

Magnesium perchlorate is, therefore, a poor desiccant candidate for solar collectors.

6. Barium perchlorate⁹ [$\text{Ba}(\text{ClO}_4)_2$] also absorbs moisture through the hydrate formation. Barium perchlorate is reported to form only the trihydrate $\text{Ba}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}$; thus, its moisture capacity is only 16%. It can be regenerated at between 250-450°F.

This desiccant would also be a poor candidate for solar collectors.

7. Barium oxide⁹ (BaO) holds water due to the formation of barium hydroxide $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$. It has one of the highest drying capacities available and can theoretically take up nearly 100% of its weight in water. The final, completely water-saturated desiccant appears sticky and wet. It requires a chemical method for regeneration. Because of its final wet condition and requirement of chemical regeneration, barium oxide would be a poor desiccant candidate for solar collectors.

8. Calcium oxide⁹ (CaO) also absorbs moisture through the hydroxide formation. Its capacity is low due to the formation of carbonates (CaCO_3) on its surface, which will not convert to the hydroxide.

This desiccant also is not a suitable candidate.

9. Activated carbon¹⁰⁻¹³ adsorbs moisture through physical adsorption and capillary condensation. Its capacity is very similar to that for activated alumina (16%). Activated carbon, however, does not show preferential adsorption for water as do all of the other desiccants. In fact, water adsorbed on activated carbon is displaced by incoming organic vapors.

Activated carbon, therefore, would be more useful in adsorbing organic vapors evolved by solar collector seals and coatings.

10. Molecular sieves and zeolites^{14,15} are crystalline metal alumino-silicates. These materials hold moisture through physical adsorption and capillary condensation. These desiccants have a preference for water and will adsorb up to 23% @ 100% RH for the synthetic molecular sieves and 60-70% @ 100% RH for the naturally occurring zeolite. These materials are regenerated by heating to 250-600°F.

From the data available, it would have to be concluded that silica gel is the best desiccant for removing moisture during solar collector breathing. Its selection over the other desiccants is based upon its high water capacity, and its ease of regeneration. Both of these properties are highly desirable in a solar collector. Molecular sieves and activated alumina would be the second and third choices, respectively.

4.9.1 Adsorbents for Organic Vapors

The list of available adsorbents for organic vapors is shown in Table 24.

All of the adsorbents listed, with the exception of activated carbon, have a preference for water vapor. Even though these materials will adsorb organic vapors, when they are subjected to saturating water vapors, the adsorbed organic vapors will be displaced. Thus, the organic vapors are again available for condensing on the glass glaze.

Activated carbon preferentially adsorbs organic vapors which are not displaced by water vapors. Thus, once they are removed from the system, they cannot return. For this reason, activated carbon would be the adsorbent of choice for organic vapors.

4.9.2 Effectiveness of Adsorbents Towards Organic Vapors

A test was developed to compare the efficacies of various adsorbents for adsorption of organic vapors evolved during thermal degradation of sealants. This test procedure involves heating an elastomer specimen in a tube maintained at 150°C for 216 hr and permitting a sample of adsorbent, also maintained at 150°C in the same vessel, to adsorb the outgassing products generated by the pyrolyzing elastomer (Fig. 87). Weight loss of the elastomer is compared with weight gain of the adsorbent in order to obtain percentage adsorption.

As shown in Table 25, there appears to be very little difference in performance between the three adsorbents evaluated in this test. The outgassing products evolved from Compound P (8EX-123 butyl) were adsorbed equally well by the activated alumina (72.9%), activated carbon (70.4%) and the molecular sieve (75.0%). Undoubtedly, variations in particle size distribution, pore size and surface area affect the performance of adsorbents. In this screening study, no attempt was made to evaluate these effects. The outgassing products evolved from Compound C (silicone) were also equally well adsorbed by the activated alumina (84.0%), activated carbon (87.6%) and molecular sieve (86.1%). However, a higher proportion of the volatiles evolved from the silicone sealant are adsorbed by the adsorbents.

If the placement of the adsorbent is changed from that shown in Fig. 87, then less volatiles will be adsorbed. For example, if an equal weight of an adsorbent is contained in a 200 mesh screen tube (3/16 in. x 3 in.), which is then placed vertically inside the test tube containing the sealant, only 40-45% of the total volatiles will be adsorbed over the same test conditions. Thus, the amount of available

vapors removed is not only dependent on the temperature, the ratio of adsorbent to sealant, but more particularly on the location of the adsorbent with respect to the sealant and glass (or plastic glaze).

If all of the evolved vapors had to pass through an adsorbent bed, situated somewhere between the sealant and the glaze, the accumulation of condensate on the glaze would be minimized. However, if the adsorbent is only located at a vent or breather tube, the volatiles would have unrestricted passage from the sealant to the cooler glaze surface and condensation would ensue. This latter condition appears to exist in all current solar collectors.

Since it is impractical to have a layer of adsorbent located between all sealant sites and the glaze, it is, therefore, impossible to eliminate vapor condensation on the glaze through use of an adsorbent. Therefore, the best approach is to use only those sealant materials which exhibit a minimum of outgassing.

4.9.3 Duo-Adsorbent System

From this study, it is concluded that a duo-adsorbent system is preferred for a solar collector breathing system.

A duo-adsorbent system¹⁶ is currently used and recommended for multiple-glazed breather window systems for removing both moisture and organic vapors. The most widely used system is a mixture of silica gel and molecular sieve (nominal pore diameter 4 Å). The molecular sieve 4 Å shows selective adsorption for the water molecules and excludes all organics with a diameter greater than 4 Å. The silica gel portion of the duo-adsorbent has a larger pore size and will adsorb the larger organic vapor molecules. However, if the molecular sieve becomes completely saturated with water, and the silica gel is exposed to water vapors, the organic vapors will be displaced and will reenter the air space from which they were originally removed.

To remove organic vapors essentially irreversibly, a duo-adsorbent system composed of silica gel and activated carbon would be expected to provide excellent performance. The activated carbon would hold the organic vapors, while the silica gel would hold the moisture.

4.10 Ventilation of Solar Collector

Unless a solar collector is designed to function hermetically, mechanical provisions, such as vents and pressure relief devices, must provide that air be permitted to flow in and out of the collector at such a rate that excessive pressure will not develop. Normally, control of breathing in a flat-plate thermal solar collector is obtained with a desiccant to reduce the humidity of the air inside the collector space. In many locations, it is advisable to permit the air to enter through a filter or a bed of desiccant in order to remove water and particulate matter.

An unusual approach that will avoid the problems associated with breathing involves the use of an elastomeric bladder to control the internal pressure of the solar collector. An essentially hermetic collector can be constructed as shown in Fig. 88.

Investigation into the use of an internal elastomeric bladder (Fig. 89) indicated that quite a large bladder would be required to compensate for the temperature/pressure changes. Mathematical calculations have shown that over the temperature range of -20°C to $+100^{\circ}\text{C}$, an elastomeric bladder would have to be capable of expanding to a volume equivalent to 40% of the internal air-space volume within the collector. Between -40°C and $+150^{\circ}\text{C}$, the bladder expansion would have to be 67%. An internally contained elastomeric bladder would, therefore, produce a substantially larger and more costly solar collector.

The alternative would be an externally attached bladder as shown in Fig. 90. In this approach, the elastomeric bladder would be attached to a vent tube protruding out of the back of the collector. For example, it could be located in attic space just beneath the roof of

the structure. Using this approach, the size of the collector would not have to be increased and, therefore, costs would be essentially unchanged.

The small test cell shown in Fig. 90 demonstrated this concept to be feasible, provided adequate space was available behind the collector, to accommodate the expanded bladder.

It should be noted, however, that the bladder approach eliminates or greatly reduces only the chance of water condensation within the solar collector. The outgassing of organic vapors from the various components and sealants contained within the solar collector would still be a major problem. This unit would be essentially hermetically sealed and the problems associated with organic vapor condensation would be greatly increased since the organic vapors are completely trapped within the collector with no chance for escape to the atmosphere.

REFERENCES

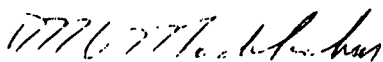
1. Stiehler, R. D., Hockman, A., Embree, E. J., and Masters, L. W., "Solar Energy Systems - Standards for Rubber Seals," NBSIR 77-1437, National Bureau of Standards Report (March 1978).
2. Cruchet, C. I., German Patent 2,522,222, May 26, 1976.
3. Cerra, O. J. and Patil, P. G., U.S. Patent 3,995,614, December 7, 1976.
4. Mazzoni, R. J. and Schutrum, L. F., U.S. Patent 3,990,431, November 9, 1976.
5. Patil, P. G., U.S. Patent 3,775,914, December 4, 1973.
6. Stewart, J. L. and Bowser, G. H., U.S. Patent 3,771,276, November 13, 1973.
7. Davison Chemical Division, W. R. Grace & Co., "Davison Silica Gels," Bulletin No. 1C-15-378.
8. Aluminum Company of America "Activated and Catalytic Alumina," Bulletin, Section GB2A.
9. Hougen, O. A. and Dodge, F. W., "The Drying of Gases," Edward Brothers, Inc., Ann Arbor, Michigan (1947).
10. American Norit Co., Inc., "Introduction to Activated Carbon," and "Norit Testing Methods."
11. Union Carbide Corp., Carbon Products Division, "The Capability of Carbon," Bulletin #CP 4462 5M975.
12. Hassler, J. W., "Activated Carbon," Chemical Publishing Co., Inc., New York, 1963.
13. Hassler, J. W., "Purification with Activated Carbon," Chemical Publishing Co., Inc., New York, 1974.

REFERENCES (Continued)

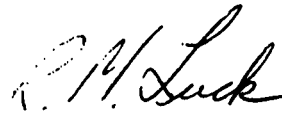
14. Union Carbide Corp., "Crystalline Molecular Sieves," Bulletin #F-109.
15. Breck, D. W., "Crystalline Molecular Sieves," J. Chem. Ed., 48, 678 (December 1964).
16. Davison Chemical Division, W. R. Grace & Co., "Guide to Insulating Glass Adsorbents," and "Part I: Insulating Glass Desiccants: Selection Based on Performance at Actual Use Conditions."

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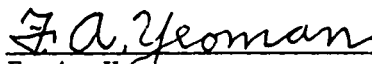
This work has been supported by the Solar Heating and Cooling Research and Development Branch, Office of Conservation and Solar Applications, U.S. Department of Energy.



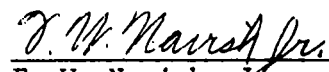
M. A. Mendelsohn
Insulation Department



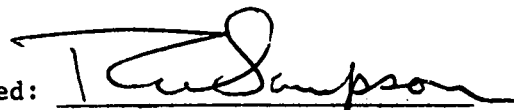
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TABLE 1
ELASTOMERS EVALUATED

| <u>Code</u> | <u>Supplier's Designation</u> | <u>Class*</u> | <u>Type Elastomer</u> | <u>Supplier</u> |
|-------------|---|---------------|---|--|
| A | Silicone rubber sealant SC (also known as DC 732 white) | SC | Silicone | Dow Corning |
| B | 790 building sealant (also known as DC 790) | SC | Silicone | Dow Corning |
| C | RTV 103 | SC | Silicone | General Electric |
| D | Mono | SC | Acrylic terpolymer | Tremco |
| E | Eternaflex Hypalon sealant | SC | Hypalon | Gibson-Homans |
| F | Tremco butyl sealant | SC | Butyl | Tremco |
| G | SE-7550 | PS | Silicone | General Electric |
| H | Silastic 747 | PS | Silicone | Dow Corning |
| I | HS-70 | PS | Silicone | Dow Corning polymer compounded by North American Reiss |
| J | 3300-12A, Vamac | PS | Ethylene/acrylic | DuPont |
| K | 210-103-35-1, Hycar 4054 | PS | Acrylic | Goodrich |
| L | 31-323-0731A, Viton | PS | Fluorocarbon | DuPont |
| M | PLV 1008, Viton | PS | Fluorocarbon | DuPont polymer compounded by Pelmor |
| N | 3300-11, Nordel | PS | Ethylene-propylene terpolymer (EPDM) | DuPont |
| O | SR 35020 | PS | Butyl | Stalwart |
| P | 8EX-123 (Butyl 100) | PS | Butyl | Polysar |
| Q | NPC 80/4C | PS | Silicone | Dow Corning polymer compounded by North American Reiss |
| R | -- | PS | Butyl | Obtained from a used Pittsburgh Plate Class collector |
| S | Sola Roll | PS | EPDM | Sample of gasket compound supplied by Bio-Energy Sys. |
| T | #18464 | PS | EPDM | Bio-Energy Systems |
| U | GE 1200 | SC | Silicone | General Electric |
| V | GE 2400 | SC | Silicone | General Electric |

* SC = sealing compound (caulking compound)

PS = preformed rubber seal (preformed rubber sheet or gasket)

TABLE 1 (Continued)

| <u>Code</u> | <u>Supplier's Designation</u> | <u>Class*</u> | <u>Type Elastomer</u> | <u>Supplier</u> |
|-------------|-------------------------------|---------------|-----------------------|-------------------------------|
| W | DC 795 | SC | Silicone | Dow Corning |
| X | DC 732 (black) | SC | Silicone | Dow Corning |
| Y | DC 93-076-2 | SC | Silicone | Dow Corning Aerospace Sealant |
| Z | DC 732 (clear) | SC | Silicone | Dow Corning |
| AA | 96-B-24 | PS | Silicone | Pawling Rubber Co. |
| BB | Hypalon 3300-10 | PS | Hypalon | DuPont |
| CC | Vistalon 78E-09-28-2 | PS | EPDM | Exxon |
| DD | Bromobutyl 8EX-122 | PS | Bromobutyl | Polysar |
| EE | Hydrin 100 (HM 13-27-1) | PS | Hydrin | Goodrich |
| FF | Hydrin 200 (HM 13-SEC-3-2) | PS | Hydrin | Goodrich |
| GG | Hydrin 400 (HM 14-10-1.) | PS | Hydrin | Goodrich |
| HH | Viton B1-323-0731 | PS | Fluorocarbon | DuPont |
| II | E-633 | PS | EPDM | Pawling Rubber Co. |

TABLE 2
CHEMICAL FORMULATIONS OF COMPOUNDED ELASTOMERS*

| Compound and Cure Conditions | Code | Parts by Weight | Ingredient | Supplier | Chemical Description | Function** |
|---------------------------------|------|--------------------|---------------------|-----------------------------|---|---|
| Viton. (31-323-0731A) | L | 94.4 | Viton E60 | DuPont | Fluoroelastomer | Base polymer |
| | | 25 | MT Black (N908) | Cabot | Medium thermal carbon black | Reinforcement |
| | | 3 | Mag D | Whittaker, Clark & Daniels | Magnesium oxide | Vulcanizing agent, stabilizer |
| <u>Cure Conditions</u> | | 6 | Ca(OH) ₂ | (Multiple source of supply) | Calcium hydroxide | Drying agent, acid acceptor |
| 24 hr/168°C | | 1.5 | VPA #1 | DuPont | (proprietary) | Viton processing aid |
| | | 1.7 | VC #20 | DuPont | 33% organophosphonium salt + 67% fluoroelastomer | Viton curative |
| | | 4 | VC #30 | DuPont | 50% dihydrooxyaromatic compound + 50% fluoro- elastomer | Viton curative |
| Viton (31-323-0731) | HH | 100 | Viton E60C | DuPont | | |
| | | 25 | MT (N908) | Cabot | Medium thermal carbon black | Reinforcement |
| | | 3 | Maglite D | Whittaker, Clark & Daniels | Magnesium oxide | Vulcanizing agent, stabilizer |
| <u>Cure Conditions</u> | | 6 | Ca(OH) ₂ | (Multiple source of supply) | Calcium hydroxide | Drying agent, acid acceptor |
| 10 min/168°C + 24 hr/171°C | | 1.5 | VPA #1 | DuPont | | Viton processing aid |
| Hypalon | BB | 100 | Hypalon 40 | DuPont | Chlorosulfonated polyethylene | Base polymer |
| | | 32 | TLD-90 | Wylough & Loser | 90% dispersion of lithurge | Polymeric carrier |
| <u>Cure Conditions</u> | | 2 | Polyethylene 617A | Allied Chemical | Low molecular wt. polyethylene | Processing aid |
| 30 min/152°C | | 35 | SRF-LM (N762) | Witco Chemical Co., Inc. | Semireinforcing furnace black | Reinforcement |
| | | 0.5 | MDTS | DuPont | Benzothiazole disulfide | Activator |
| | | 2 | Tetrone A | DuPont | Dipentamethylene thiuram tetrasulfide | Very active accelerator |
| | | 1 | NBC | DuPont | Active ingredient, nickel dibutylthiocarbamate | O ₃ , weathering, cracking inhibitor |
| Nordel (3300-11) | N | 100 | Nordel 1320 | DuPont | Ethylene propylene terpolymer (EPDM) | Base polymer |
| <u>Cure Conditions</u> | | 5 | Zinc oxide | American Zinc Sales Co. | Zinc oxide | Activator + reinforcement |
| 30 min/160°C | | 2 | Agerite Resin D | R.T. Vanderbilt | Polymerized trimethyl dihydroquinoline | Antioxidant |
| | | 60 | FEF (N550) | Cabot | Fast extruding furnace black | Reinforcement |
| | | 8 | DiCap 40C | Hercules Powder Co., Inc. | 40% dicumylperoxide on calcium carbonate | Nonsulfur vulcanizing cross-linking agent |
| | | 2 | HVA-2 | DuPont | N,N-m-phenylene dimaleimide | Curative |
| Vamac (3300-12A) | J | 124 | Vamac (N124) | DuPont | Ethylene-acrylic copolymer | Base polymer |
| | | 30 | SRF (N774) | Cabot | Semireinforcing black | Reinforcement |
| <u>Cure Conditions</u> | | 1.25 | MDA | DuPont | Methylene dianiline | Curing agent |
| 3 hr/177°C | | 4 | DPG | American Cyanamid Co. | Diphenyl guanidine | Accelerator |

* Formulations that were supplied by manufacturers are presented in this table. Suppliers of the other compounds viewed their formulations as proprietary and therefore did not reveal them.

** Most of the terminology used in describing the functions of the ingredients is that employed in the product descriptions by the suppliers.

TABLE 2 (Continued)

| Compound and Cure Conditions | Code | Parts by Weight | Ingredient | Supplier | Chemical Description | Function |
|--|------|--------------------|-------------------------------|--|--|--------------------------------|
| Vistalon (78E-09-28-2) <u>Cure Conditions</u> 20 min/160°C | CC | 100 | Vistalon 5600 | Exxon | Ethylene-propylene terpolymer | Base polymer |
| | | 100 | Carbon black N550 | Cabot | Fast extruding furnace black | Reinforcement |
| | | 95 | Cirosol 4240 oil | Sun Oil Co. | Naphthenic oil | Processing aid |
| | | 1.0 | Stearic acid | City Chemical Co. | Stearic acid | Primary activator, plasticizer |
| | | 5.0 | Zinc oxide | American Zinc Sales Co. | Zinc oxide | Activator, reinforcement |
| | | 1.5 | TMTDS | Monsanto | Tetramethylthiuram disulfide | Accelerator |
| | | 0.5 | MBT | DuPont | 2-mercaptobenzothiazole | Activator |
| | | 1.5 | Sulfur | Akron Chemical Co. | Sulfur | Vulcanizing agent |
| Bromobutyl (8EX-122) <u>Cure Conditions</u> 15 min/180°C | DD | 100 | Polysar bromobutyl X-2 | Polysar | Brominated butyl rubber | Base polymer |
| | | 1.0 | Stearic acid | City Chemical | Stearic acid | Primary activator, plasticizer |
| | | 1.0 | Maglite D | Whittaker, Clark & Daniels | Magnesium oxide | Vulcanizing agent, stabilizer |
| | | 10.0 | Heliozone wax | DuPont | Selected blend of petroleum waxes | Sun-checker, tracking resistor |
| | | 55.0 | N550 carbon black | Cabot | Fast extruding furnace black | Reinforcement |
| | | 10.0 | Sunpar 2280 | Sun Oil Co. | Paraffinic oil | Softener |
| | | 3.0 | Zinc oxide | American Zinc Sales | Zinc oxide | Activator, reinforcement |
| | | 0.3 | TMTD | Akron Chemical | Tetramethylthiuram disulfide | Accelerator |
| | | 2.0 | Permalax | DuPont | Active ingredient, di-ortho-tolylguanidine salt of dicatechol borate | |
| 78 Butyl (8EX-123) <u>Cure Conditions</u> 45 min/180°C | P | 100 | Polysar butyl 100 | Polysar | Butyl rubber | Base polymer |
| | | 1.5 | Stearic acid | City Chemical | Stearic acid | Primary activator, plasticizer |
| | | 4.0 | SP 1045 | Schenectady Chemical Inc. | Phenol formaldehyde resin | Curative |
| | | 10.0 | Heliozone wax | DuPont | Blend of petroleum waxes | Sun-checker, cracking resistor |
| | | 55.0 | N550 carbon black | Cabot | Fast extruding furnace black | Reinforcement |
| | | 7.0 | Sunpar 2280 | Sun Oil Co. | Paraffinic oil | Softener |
| | | 2.67 | Stannous chloride prespersion | Wara Chemical Corp. | Stannous chloride dihydrate (75% in 25% inert oil) | Catalyst |
| Acrylic (210-108-35-1) <u>Cure Conditions</u> 4 min/170°C + 8 hr/175°C | K | 100 | Hycar 4054 | B. F. Goodrich | Polyethylacrylate elastomer | |
| | | 2.0 | Acrawax C | Charles L. Husking Co., Inc. (Glycol Chem. Div.) | Synthetic wax | Finishing dusting agent |
| | | 2.0 | TE-80 | Tecnical Processing, Inc. | (Proprietary) | Lubricant processing aid |
| | | 60.0 | Phil black N550 | Phillips Petroleum Co. | Fast extruding furnace black | Reinforcement |
| | | 3.0 | Witco sodium stearate | Witco Chemical | Sodium stearate | Curative, dispersing agent |
| | | 2.0 | Adogen 345D | Ashland Chemical Co. | Hydrogenated tallowamine | Curative |
| | | 1.0 | NBS stearic acid | City Chemical Co. | Stearic acid | Activator, processing aid |

TABLE 2 (Continued)

| Compound and Cure Conditions | Code | Parts by Weight | Ingredient | Supplier | Chemical Description | Function |
|---------------------------------|------|--------------------|---------------------------------------|---------------------------|--|---|
| Hydrin 100 (HM 13-27-1) | EE | 100 | Hydrin 100 | B. F. Goodrich | Epichlorohydrin based elastomer | |
| | | 5.0 | Red lead | Eagle Picher Co. | > 90% red lead oxide | Activator, vulcanizer |
| <u>Cure Conditions</u> | | 1.0 | NBC | DuPont | Active ingredient, nickel dibutyl dithiocarbamate | O ₃ , weathering, cracking inhibitor |
| 30 min/175°C | | 40.0 | N550, FEF black | Cabot | Fast extruding furnace black | Reinforcement |
| | | 1.0 | TE-70 | Technical Processing Co. | | Lubricant, processing oil |
| | | 1.5 | 2-mercapto- imidazoline (NA-22) | DuPont | 2-mercapto-imidazoline | Fast general purpose accelerator |
| Hydrin 200 (HM 13-SEC-3-2) | FF | 100 | Hydrin 200 | B. F. Goodrich | Epichlorohydrin based elastomer | |
| | | 1.0 | Zinc stearate | Witco Chemical Co. | Zinc stearate | Activator, dusting agent |
| | | 5.0 | Red lead | Eagle-Picher Co. | > 90% red lead oxide | Activator, vulcanizer |
| <u>Cure Conditions</u> | | 1.0 | NBC | DuPont | Active ingredient, nickel dibutyl dithiocarbamate | O ₃ , weathering, cracking inhibitor |
| 30 min/175°C | | 40.0 | N550, FEF black | Cabot | Fast extruding furnace black | Reinforcement |
| | | 1.0 | ZO-9 | Yerzley Co. | Blend of waxes | Mold lubricant, plasticizer |
| | | 1.5 | 2-mercapto- imidazoline | DuPont | 2-mercapto-imidazoline | Fast general purpose accelerator |
| Hydrin 400 (HM 14-10-1) | GC | 100 | Hydrin 400 | B. F. Goodrich | Epichlorohydrin based elastomer | |
| | | 0.8 | Stearic acid | City Chemical Co. | Stearic acid | Primary activator, plasticizer, softener |
| <u>Cure Conditions</u> | | 2.0 | Dyphos | National Lead | Dibasic lead phosphite | Heat, light, weathering, chemical stabilizer |
| 20 min/160°C | | 0.9 | NBC | DuPont | Active ingredient, nickel dibutyl dithiocarbamate | O ₃ , weathering, cracking inhibitor |
| | | 0.5 | Methyl niclate | R. T. Vanderbilt | Methyl niclate | Accelerator |
| | | 20.0 | N326 black | Cabot | High abrasion furnace black, low structure | Reinforcement |
| | | 30.0 | N550 black | Cabot | Fast extruding furnace black | Reinforcement |
| | | 10.0 | DOP | Monsanto | Diocetyl phthalate | Plasticizer, softener |
| | | 0.3 | ZO-9 | Yerzley Co. | Blend of waxes | Mold lubricant, plasticizer |
| | | 2.5 | SR-350 | Sartomer Corp. | Polyfunctional methacrylate | Auxiliary cure material |
| | | 3.5 | Di-Cup 40C | Hercules Powder Co., Inc. | ~ 40% dicumylperoxide on calcium carb nate | Nonsulfur, vulcanizing, cross- linking agent |

TABLE 3
CLASS PS ELASTOMER SCREENING TESTS

| Class PS Material | Hardness ¹ Grade | Ultimate Elongation | Compr. Set 70 h, 150°C ² | Compr. Set 166 h, 10°C | Hardness Change ² | Ultimate Elongation Change ² | Tensile Strength Change ² | Volatiles Lost ² |
|--------------------------------------|--------------------------------|------------------------|--|---------------------------|---------------------------------|---|--|--------------------------------|
| <u>Silicone</u> | | | | | | | | |
| SE-7550 | 5 | P ⁺ | P ⁺ | P ⁺ | P ⁺ | P ⁺ | P ⁺ | P ⁺ |
| Silastic 747 | 7 | P | P ⁺ | P ⁺ | P ⁺ | P | F | F |
| HS-70 | 7 | P ⁺ | P | P ⁺ | P ⁺ | P ⁺ | P ⁺ | P ⁺ |
| <u>EPDM</u> | | | | | | | | |
| E-633 | 7 | P ⁺ | F ⁻ | F ⁻ | P | F ⁻ | F | F ⁻ |
| 78E-09-28-2 | 6 | P ⁺ | F ⁻ | F ⁻ | F ⁻ | F ⁻ | P ⁺ | F |
| Nordel 3300-11 | 8 | P ⁺ | P ⁺ | P ⁺ | P ⁺ | P ⁺ | P ⁺ | F |
| <u>Fluorocarbon</u> | | | | | | | | |
| Viton 31323-0731 | 8 | P ⁺ | P ⁺ | F ⁻ | P ⁺ | P ⁺ | P ⁺ | P ⁺ |
| Viton PLV 1008 | 8 | P ⁺ | P ⁺ | F ⁻ | P ⁺ | P ⁺ | P ⁺ | P ⁺ |
| <u>Epichlorohydrin</u> | | | | | | | | |
| HM 14-10-1 | 6 | P ⁺ | F ⁻ | P ⁺ | P | P | P | F ⁻ |
| HM 13-SEC3-2 | 7 | P ⁺ | F | P ⁺ | P | P | P ⁺ | F |
| HM 13-27-1 | 8 | P ⁺ | F | P ⁺ | F | F | P | F ⁻ |
| <u>Ethylene Acrylic</u> | | | | | | | | |
| Vamac 3300-12 | 7 | P ⁺ | F | F ⁻ | P ⁺ | F | P ⁺ | F ⁻ |
| <u>Chlorosulfonated Polyethylene</u> | | | | | | | | |
| Hypalon 3300-10 | 7-8 | P ⁺ | F ⁻ | F ⁻ | P ⁺ | P | P ⁺ | P ⁺ |
| <u>Polyacrylic</u> | | | | | | | | |
| 210-108-35-1 | 6-7 | F | P ⁺ | P | P ⁺ | P ⁺ | P ⁺ | P ⁺ |
| <u>Bromobutyl</u> | | | | | | | | |
| 8EX-122 | 6 | P | F | F | P ⁺ | F | P | F ⁻ |
| <u>Butyl</u> | | | | | | | | |
| 8EX-123 | 5-6 | P ⁺ | P ⁺ | F ⁻ | P ⁺ | P ⁺ | P ⁺ | F ⁻ |
| SR 35020 | 5 | P ⁺ | F ⁻ | P ⁺ | P ⁺ | P | F ⁻ | F ⁻ |

¹(Hardness grade x 10) + 5 = Shore A durometer hardness

²Materials exposed to 150°C/70 h

P = pass by relatively small margin

P⁺ = pass by substantial margin

F = fail by relatively small margin

F⁻ = fail by substantial margin

TABLE 4

EFFECT OF THERMAL AGING ON COMPRESSION SET^a

| Code | Material Type | Class | Temp. (°C) | Percent Compression Set | | | | | | | | | | | |
|------|----------------------|-------|------------|-------------------------|----------|----------|----------|-----------|-----------|-----------|----|----|-----|-----|-----|
| | | | | Days Aged | | | | | | | | | | | |
| | | | | 1 | 2 | 4 | 8 | 16 | 28 | 46 | 63 | 81 | 122 | 179 | 263 |
| G | Silicone | PS | 250 | 113 | | | | | | | | | | | |
| I | Silicone | PS | 250 | 100 | | | | | | | | | | | |
| L | Fluorocarbon | PS | 250 | 32 | 42 | 52 | 62 | 72 | 79 | | | | | | |
| Q | Silicone | PS | 250 | 105 | | | | | | | | | | | |
| G | Silicone | PS | 225 | 108 | | | | | | | | | | | |
| I | Silicone | PS | 225 | 99 | | | | | | | | | | | |
| L | Fluorocarbon | PS | 225 | 20 | -- | 33 | 42 | 52 | 60 | 68 | 72 | 77 | | | |
| Q | Silicone | PS | 225 | 94 | 104 | | | | | | | | | | |
| G | Silicone | PS | 200 | 88 | 103 | | | | | | | | | | |
| I | Silicone | PS | 200 | 78 | 91 | 100 | | | | | | | | | |
| L | Fluorocarbon | PS | 200 | 12 | -- | 21 | 28 | 36 | 44 | 51 | -- | 59 | 67 | 75 | 83 |
| Q | Silicone | PS | 200 | 65 | 81 | 95 | 103 | | | | | | | | |
| G | Silicone | PS | 175 | 37 | 55 | 73 | 86 | 97 | 101 | | | | | | |
| I | Silicone | PS | 175 | 37 | 51 | 67 | 81 | 94 | 100 | | | | | | |
| L | Fluorocarbon | PS | 175 | 10 | 11 | -- | 17 | 22 | 27 | 33 | -- | 40 | 48 | 56 | 66 |
| Q | Silicone | PS | 175 | 20 | 30 | 46 | 63 | 80 | 89 | 97 | | | | | |
| | | | | <u>1</u> | <u>2</u> | <u>4</u> | <u>8</u> | <u>15</u> | <u>32</u> | <u>53</u> | | | | | |
| J | Ethylene/ acrylic | PS | 175 | 33 | -- | 53 | 61 | 70 | 79 | | | | | | |
| K | Acrylic | PS | 175 | 25 | -- | 38 | 47 | 55 | 69 | 82 | | | | | |

^aOnly preformed sheets were tested; except for measurements, materials were held under compression continuously. Values are averages of four sets of data.

TABLE 4 (Continued)

| Code | Material Type | Class | Temp. (°C) | Percent Compression Set | | | | | | | | | | |
|------|----------------------|-------|---------------|-------------------------|----------|----------|-----------|-----------|-----------|-----------|------------|------------|------------|------------|
| | | | | Days Aged | | | | | | | | | | |
| | | | | 1 | 2 | 7 | 17 | 22 | 46 | 59 | | | | |
| N | EPDM | PS | 175 | 19 | 22 | 32 | 39 | | 69 | 89 | | | | |
| P | Butyl | PS | 175 | 38 | 45 | 62 | 74 | 78 | | | | | | |
| | | | | <u>1</u> | <u>5</u> | <u>9</u> | <u>30</u> | | <u>41</u> | <u>56</u> | | <u>72</u> | | |
| G | Silicone | PS | 150 | 21 | 44 | 56 | 76 | | | | | | | |
| I | Silicone | PS | 150 | 23 | 44 | 54 | 79 | | | | | | | |
| Q | Silicone | PS | 150 | 12 | 26 | 34 | 59 | | 67 | 73 | | 78 | | |
| | | | | <u>1</u> | <u>4</u> | <u>8</u> | <u>15</u> | | <u>32</u> | <u>53</u> | | <u>84</u> | | |
| J | Ethylene/ acrylic | PS | 150 | 27 | 41 | 51 | 59 | | 69 | 76 | | | | |
| K | Acrylic | PS | 150 | 16 | 26 | 32 | 40 | | 52 | 62 | | 71 | | |
| | | | | <u>1</u> | <u>2</u> | <u>4</u> | <u>11</u> | | <u>24</u> | <u>52</u> | | <u>85</u> | <u>175</u> | <u>207</u> |
| N | EPDM | PS | 150 | 15 | 16 | 21 | 29 | | 41 | 50 | | 56 | 75 | |
| P | Butyl | PS | 150 | 29 | 35 | 43 | 55 | | 64 | 73 | | 78 | | |
| | | | | <u>1</u> | <u>5</u> | <u>9</u> | <u>30</u> | <u>41</u> | <u>56</u> | <u>72</u> | <u>113</u> | <u>146</u> | <u>173</u> | <u>222</u> |
| G | Silicone | PS | 140 | 16 | 32 | 42 | 60 | 67 | 73 | 77 | | | | |
| I | Silicone | PS | 140 | 18 | 35 | 43 | 66 | 72 | 78 | | | | | |
| Q | Silicone | PS | 140 | 11 | 21 | 27 | 46 | 51 | 55 | 59 | 67 | 71 | 73 | 76 |
| | | | | <u>1</u> | <u>4</u> | <u>8</u> | <u>15</u> | <u>32</u> | <u>53</u> | <u>84</u> | <u>117</u> | <u>193</u> | <u>225</u> | |
| J | Ethylene/ acrylic | PS | 125 | 12 | 25 | 33 | 41 | 50 | 53 | 58 | 65 | 75 | | |
| K | Acrylic | PS | 125 | 12 | 16 | 22 | 28 | 38 | 43 | 47 | 52 | 64 | 68 | |
| | | | | <u>1</u> | <u>2</u> | <u>4</u> | <u>11</u> | <u>24</u> | <u>52</u> | <u>85</u> | <u>175</u> | <u>212</u> | | |
| N | EPDM | PS | 125 | 14 | 16 | 18 | 24 | 30 | 41 | 43 | 65 | 72 | | |
| P | Butyl | PS | 125 | 21 | 24 | 34 | 44 | 50 | 56 | 61 | 69 | 73 | | |
| | | | | <u>1</u> | <u>2</u> | <u>4</u> | <u>11</u> | <u>24</u> | <u>52</u> | <u>85</u> | <u>175</u> | <u>207</u> | | |
| N | EPDM | PS | 100 | 14 | 18 | 21 | 25 | 29 | 40 | 42 | | | | |
| P | Butyl | PS | 100 | 10 11 15 | 22 | 29 | 38 | 46 | 58 | 63 | | | | |

TABLE 5
SUMMARY OF COMPRESSION SET THERMAL AGING DATA

| <u>Material</u> | <u>Code</u> | Time (days) to Reach 50% Compression Set | | |
|------------------|-------------|---|--------------|--------------|
| | | <u>150°C</u> | <u>125°C</u> | <u>175°C</u> |
| Fluorocarbon | L | 510* | 7400* | 135 |
| Silicone | Q | 21 | 100 | 5 |
| Silicone | G | 7 | 50 | 2 |
| Silicone | I | 8 | 33 | 2 |
| EPDM | N | 50 | 103 | 27.5 |
| Acrylic | K | 28 | 105 | 10.5 |
| Ethylene-acrylic | J | 8 | 35 | 4 |
| Butyl | P | 8 | 23 | 4.5 |

* Extrapolation of an Arrhenius plot.

TABLE 6
CORROSION TESTS^{a,b,c}

| Code | Material Type | Class | Metallic Substrate | Aging Time and Temperature | | | | | | | | | |
|------------------------------------|--------------------|-------|--------------------|----------------------------|-------|---------|-------|---------|-------|----------|-------|----------|-------|
| | | | | 6 days | | 26 days | | 92 days | | 153 days | | 229 days | |
| | | | | 125°C | 150°C | 125°C | 150°C | 125°C | 150°C | 125°C | 150°C | 125°C | 150°C |
| A,B,C,F,G, I,J,L,Q ^d | ---See footnote--- | | Al | C | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | | | GalFe | C | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | | | AnAl | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| E | Hypalon | SC | Al | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | | | GalFe | 2 | 2 | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 |
| | | | AnAl | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| K | Acrylic | PS | Al | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | | | GalFe | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 |
| | | | AnAl | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| N | EPDM | PS | Al | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | | | GalFe | 0 | 0 | 0 | 0 | 0 | 2 | 0 | 2 | 0 | 2 |
| | | | AnAl | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P | Butyl | PS | Al | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| | | | GalFe | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| | | | AnAl | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |

^aSealants were sandwiched between metal panels; observations of corrosion are visual; material D was not evaluated because of excessive tack.

^bSymbols: Al - aluminum panels; GalFe - galvanized iron panels; AnAl - anodized aluminum panels.

^cRatings: 0 - no visual evidence of corrosion; 1 - relatively slight corrosion; 2 - relatively moderate corrosion; 3 - relatively severe corrosion.

^dCode = A - Silicone, Class SC; B - Silicone, Class SC; C - Silicone, Class SC; F - Butyl, Class SC; G - Silicone, Class PS; I - Silicone, Class PS; J - Ethylene/acrylic, Class PS; L - Fluorocarbon, Class PS; Q - Silicone, Class PS

TABLE 7
EFFECT OF AGING IN AIR ON TENSILE PROPERTIES

| Code | Material Type | Class ^b | Aging Temp. (°C) | Property ^a | Days Aged | | | | | | | | |
|------|---------------|--------------------|------------------|-----------------------|----------------|-------|-------|-----|-----|-----|-----|-----|-----|
| | | | | | 0 ^b | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 100 |
| A | Silicone | SC | 225 | Tensile strength | 220 | 210 | 170 | 160 | 170 | -- | 110 | 120 | 120 |
| | | | | Elongation | 360 | 440 | 400 | 380 | 440 | -- | 170 | 47 | 140 |
| | | | | 100% Modulus | 90 | 70 | 50 | 60 | 60 | -- | 70 | 90 | 100 |
| B | Silicone | SC | 225 | Tensile strength | 110* | 80* | 80* | 90 | -- | 150 | 170 | 210 | 150 |
| | | | | Elongation | 810* | 770* | 780* | 390 | -- | 20 | 30 | 20 | 20 |
| | | | | 100% Modulus | 30 | 20 | 20 | 50 | -- | ND | ND | ND | ND |
| C | Silicone | SC | 225 | Tensile strength | 400 | 430 | 490 | 420 | 480 | 380 | 300 | 280 | 280 |
| | | | | Elongation | 390 | 400 | 440 | 400 | 430 | 370 | 270 | 220 | 180 |
| | | | | 100% Modulus | 120 | 120 | 120 | 120 | 120 | 120 | 140 | 160 | 170 |
| G | Silicone | PS | 225 | Tensile strength | 1100* | 1100* | 1010* | 810 | 690 | 560 | 490 | 450 | -- |
| | | | | Elongation | 770* | 730* | 670* | 460 | 330 | 220 | 80 | 60 | -- |
| | | | | 100% Modulus | 120 | 170 | 170 | 220 | 240 | 290 | ND | ND | -- |
| I | Silicone | PS | 225 | Tensile strength | 1320 | 1140 | 990 | 890 | 730 | 640 | -- | -- | -- |
| | | | | Elongation | 680 | 520 | 450 | 320 | 230 | 160 | -- | -- | -- |
| | | | | 100% Modulus | 230 | 290 | 300 | 380 | 400 | 470 | -- | -- | -- |
| K | Acrylic | PS | 225 | Tensile strength | 1570 | 760 | 330 | 240 | 80 | -- | -- | -- | -- |
| | | | | Elongation | 170 | 90 | ~0 | ~0 | ~0 | -- | -- | -- | -- |
| | | | | 100% Modulus | 660 | ND | ND | ND | ND | -- | -- | -- | -- |

^a Tensile strength, units are lb/in²; elongation, percent ultimate elongation; 100% modulus, tensile modulus at 100% elongation (cannot be determined when ultimate elongation <100%).

^b All SC specimens (caulking compounds) were permitted to age for 4-6 wks at room temperature and then cured for 24 hrs at the aging temperature prior to testing. The PS specimens (preformed seals) were cured by their suppliers and tested on an as-received basis.

* Specimens did not break - elongation exceeded capability of testing machine. Thus, true values are greater than those indicated.

ND = Not determined (modulus values at 100% elongation cannot be determined when ultimate elongation <100%).
Values are arithmetic averages of three sets of data.

TABLE 7 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property ^a | Days Aged | | | | | | | | |
|------|---------------|-------|------------------|-----------------------|----------------|-------|-------|------|------|------|------|------|------|
| | | | | | 0 ^b | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 100 |
| L | Fluorocarbon | PS | 225 | Tensile strength | 700 | 760 | 770 | 830 | 780 | 760 | 710 | 790 | 850 |
| | | | | Elongation | 700 | 740 | 680 | 820 | 780 | 750 | 770 | 800 | 860 |
| | | | | 100% Modulus | 210 | 210 | 200 | 190 | 180 | 200 | 210 | 220 | 200 |
| Q | Silicone | PS | 225 | Tensile strength | 1030 | 970 | 1000 | 850 | 690 | 480 | 510 | 250 | 120 |
| | | | | Elongation | 250 | 210 | 200 | 150 | 100 | 10 | ~ 0 | ~ 0 | ~ 0 |
| | | | | 100% Modulus | 490 | 490 | 520 | 590 | 650 | ND | ND | ND | ND |
| A | Silicone | SC | 200 | Tensile strength | 250 | 220 | 200 | 210 | 200 | 180 | 180 | 160 | 170 |
| | | | | Elongation | 290 | 300 | 300 | 300 | 320 | 380 | 360 | 360 | 340 |
| | | | | 100% Modulus | 110 | 90 | 100 | 90 | 90 | 70 | 80 | 70 | 90 |
| B | Silicone | SC | 200 | Tensile strength | 120* | 110* | 100* | 80* | 80 | 90 | 50 | 60 | 60 |
| | | | | Elongation | 800* | 740* | 830* | 850* | 660 | 30 | 10 | 10 | 20 |
| | | | | 100% Modulus | 30 | 30 | 30 | 20 | 30 | ND | ND | ND | ND |
| C | Silicone | SC | 200 | Tensile strength | 540 | 490 | 500 | 490 | 420 | 450 | 480 | 440 | 400 |
| | | | | Elongation | 360 | 370 | 350 | 370 | 340 | 410 | 400 | 370 | 300 |
| | | | | 100% Modulus | 150 | 130 | 140 | 130 | 120 | 110 | 140 | 140 | 160 |
| G | Silicone | PS | 200 | Tensile strength | 1100* | 1000* | 1030* | 1010 | 1020 | 830 | 600 | 540 | 470 |
| | | | | Elongation | 770* | 770* | 760* | 750* | 670 | 60 | 34 | 23 | 16 |
| | | | | 100% Modulus | 120 | 130 | 140 | 150 | 180 | 210 | 270 | 340 | 420 |
| I | Silicone | PS | 200 | Tensile strength | 1320 | 1210 | 1120 | 1080 | 1060 | 940 | 770 | 730 | 720 |
| | | | | Elongation | 680 | 630 | 590 | 520 | 450 | 370 | 220 | 140 | 100 |
| | | | | 100% Modulus | 230 | 230 | 240 | 270 | 310 | 360 | 440 | 590 | 710 |
| L | Fluorocarbon | PS | 200 | Tensile strength | 1700 | 1710 | 1680 | 1800 | 1760 | 1640 | 1600 | 1770 | 1660 |
| | | | | Elongation | 210 | 200 | 210 | 190 | 200 | 190 | 190 | 220 | 180 |
| | | | | 100% Modulus | 700 | 740 | 680 | 820 | 780 | 750 | 770 | 800 | 860 |
| Q | Silicone | PS | 200 | Tensile strength | 1030 | 1100 | 1050 | 970 | 790 | 580 | 420 | 290 | 90 |
| | | | | Elongation | 250 | 290 | 270 | 220 | 160 | 90 | ~ 0 | ~ 0 | ~ 0 |
| | | | | 100% Modulus | 490 | 450 | 440 | 490 | 520 | | | | |
| A | Silicone | SC | 175 | Tensile strength | 210 | 240 | 230 | 230 | -- | -- | 210 | 220 | 210 |
| | | | | Elongation | 270 | 300 | 290 | 310 | -- | -- | 410 | 430 | 380 |
| | | | | 100% Modulus | 100 | 110 | 100 | 100 | -- | -- | 80 | 80 | 90 |

TABLE 7 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property ^a | Days Aged | | | | | | | | |
|------|------------------|-------|------------------|-----------------------|----------------|-------|-------|-------|-------|------|------|------|-----|
| | | | | | 0 ^b | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 100 |
| B | Silicone | SC | 175 | Tensile strength | 100* | 100* | 100* | 120* | -- | -- | 120 | 140 | 150 |
| | | | | Elongation | 780* | 810* | 800* | 780* | -- | -- | 610 | 490 | 380 |
| | | | | 100% Modulus | 30 | 30 | 30 | 30 | -- | -- | 20 | 30 | 40 |
| C | Silicone | SC | 175 | Tensile strength | 470 | 410 | 450 | 410 | -- | -- | 480 | 470 | 510 |
| | | | | Elongation | 350 | 300 | 330 | 300 | -- | -- | 370 | 370 | 360 |
| | | | | 100% Modulus | 140 | 150 | 140 | 150 | -- | -- | 150 | 140 | 160 |
| G | Silicone | PS | 175 | Tensile strength | 1100* | 1030* | 1050* | 1270* | 1130* | 1060 | 870 | 750 | 660 |
| | | | | Elongation | 770* | 720* | 740* | 740* | 730* | 680 | 510 | 330 | 250 |
| | | | | 100% Modulus | 120 | 150 | 130 | 170 | 160 | 170 | 210 | 280 | 310 |
| I | Silicone | PS | 175 | Tensile strength | 1320 | 1260 | 1260 | 1470 | 1240 | 1110 | 940 | 960 | 880 |
| | | | | Elongation | 680 | 620 | 630 | 610 | 570 | 500 | 270 | 180 | 140 |
| | | | | 100% Modulus | 230 | 230 | 230 | 290 | 280 | 280 | 440 | 620 | 700 |
| J | Ethylene/Acrylic | PS | 175 | Tensile strength | 2030 | 2180 | 2310 | 2060 | 1990 | 1200 | 1030 | -- | -- |
| | | | | Elongation | 420 | 410 | 450 | 390 | 340 | 170 | 20 | -- | -- |
| | | | | 100% Modulus | 430 | 430 | 440 | 410 | 470 | 580 | -- | -- | -- |
| K | Acrylic | PS | 175 | Tensile strength | 1210 | 1210 | 1240 | 1480 | 1140 | 810 | -- | 770 | -- |
| | | | | Elongation | 160 | 160 | 150 | 130 | 90 | ~0 | -- | ~0 | -- |
| | | | | 100% Modulus | 660 | 600 | 570 | 780 | ND | ND | ND | ND | -- |
| N | EPDM | PS | 175 | Tensile strength | 1950 | 1440 | 1620 | 1160 | 520 | 690 | -- | -- | -- |
| | | | | Elongation | 170 | 160 | 180 | 140 | 40 | 10 | -- | -- | -- |
| | | | | 100% Modulus | 800 | 740 | 710 | 710 | ND | ND | -- | -- | -- |
| P | Butyl | PS | 175 | Tensile strength | 1470 | 920 | 500 | -- | 10 | 40 | -- | -- | -- |
| | | | | Elongation | 500 | 660 | 630 | -- | 480 | 10 | -- | -- | -- |
| | | | | 100% Modulus | 150 | 100 | 90 | -- | 60 | ND | -- | -- | -- |
| Q | Silicone | PS | 175 | Tensile strength | 1030 | 1080 | 1070 | 1290 | 1060 | 980 | 980 | 920 | 840 |
| | | | | Elongation | 250 | 250 | 250 | 240 | 220 | 190 | 180 | 150 | 140 |
| | | | | 100% Modulus | 490 | 500 | 490 | 580 | 530 | 520 | 560 | 670 | 780 |
| D | Acrylic | SC | 150 | Tensile strength | 100 | 160 | 180 | 210 | 340 | -- | 710 | 1650 | 340 |
| | | | | Elongation | 280 | 200 | 170 | 130 | 100 | -- | 54 | 20 | 10 |
| | | | | 100% Modulus | 110 | 150 | 170 | 200 | 340 | -- | ND | ND | ND |

TABLE 7 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property ^a | Days Aged | | | | | | | | |
|------|------------------|-------|------------------|-----------------------|----------------|------|------|------|-------------------|------|------|------|------|
| | | | | | 0 ^b | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 100 |
| E | Hypalon | SC | 150 | Tensile strength | 110 | 210 | 250 | 380 | 790 | -- | -- | -- | -- |
| | | | | Elongation | 80 | 50 | 40 | ~ 0 | ~ 0 | -- | -- | -- | -- |
| | | | | 100% Modulus | ND | ND | ND | ND | ND | -- | -- | -- | -- |
| F | Butyl | SC | 150 | Tensile strength | 450 | 390 | 200 | 240 | Specimens cracked | | | | |
| | | | | Elongation | ~ 0 | ~ 0 | ~ 0 | ~ 0 | | | | | |
| | | | | 100% Modulus | ND | ND | ND | ND | | | | | |
| J | Ethylene/acrylic | PS | 150 | Tensile strength | 2030 | 2030 | 2570 | 2540 | 2570 | 2290 | 2350 | 2660 | 2000 |
| | | | | Elongation | 420 | 420 | 430 | 430 | 400 | 400 | 330 | 320 | 200 |
| | | | | 100% Modulus | 360 | 360 | 380 | 380 | 400 | 360 | 600 | 590 | 760 |
| K | Acrylic | PS | 150 | Tensile strength | 1210 | -- | -- | 1190 | 1290 | 1300 | 1240 | 1010 | 690 |
| | | | | Elongation | 160 | -- | -- | 170 | 180 | 170 | 160 | 110 | 50 |
| | | | | 100% Modulus | 660 | -- | -- | 480 | 570 | 650 | 610 | 880 | ND |
| N | EPDM | PS | 150 | Tensile strength | 1950 | 1950 | 2530 | 2100 | 1920 | 1700 | 1070 | 630 | 780 |
| | | | | Elongation | 170 | 170 | 180 | 180 | 160 | 160 | 110 | 40 | 10 |
| | | | | 100% Modulus | 800 | 890 | 1100 | 880 | 1000 | 940 | 930 | -- | -- |
| P | Butyl | PS | 150 | Tensile strength | 1470 | 1410 | 1630 | 1210 | 910 | 380 | -- | -- | -- |
| | | | | Elongation | 500 | 570 | 570 | 580 | 570 | 530 | -- | -- | -- |
| | | | | 100% Modulus | 150 | 120 | 160 | 140 | 120 | 90 | -- | -- | -- |
| D | Acrylic | SC | 125 | Tensile strength | 90 | 100 | -- | 130 | 110 | 160 | 290 | 430 | 570 |
| | | | | Elongation | 150 | 190 | -- | 300 | 300 | 270 | 170 | 100 | 80 |
| | | | | 100% Modulus | 60 | 80 | -- | 120 | 100 | 130 | 270 | 440 | ND |
| E | Hypalon | SC | 125 | Tensile strength | 70 | 80 | -- | 120 | 180 | 200 | 270 | 630 | -- |
| | | | | Elongation | 140 | 160 | -- | 110 | 90 | 80 | 60 | 10 | -- |
| | | | | 100% Modulus | 60 | 70 | -- | 110 | ND | ND | ND | ND | -- |
| F | Butyl | SC | 125 | Tensile strength | 480 | 450 | -- | 460 | 460 | 440 | 230 | 170 | 80 |
| | | | | Elongation | ~ 0 | ~ 0 | -- | ~ 0 | ~ 0 | ~ 0 | ~ 0 | ~ 0 | ~ 0 |
| | | | | 100% Modulus | ND | | -- | ND | ND | ND | ND | ND | ND |

TABLE 7 (Continued)

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TABLE 8

PERCENT RETENTION OF TENSILE PROPERTIES ON AGING IN AIR^{a,b}

| Code | Material Type | Class | Aging Temp. (°C) | Property | Percent Retention | | | | | | | |
|------|---------------|-------|------------------|-------------------------------|-------------------|-----|------|------|-------|-------|-------|-------|
| | | | | | Days Aged | | | | | | | |
| | | | | | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 100 |
| A | Silicone | SC | 225 | Tensile strength | 95 | 77 | 73 | 77 | -- | 50 | 55 | 55 |
| | | | | Elongation | 122 | 111 | 106 | 122 | -- | 47 | 47 | 39 |
| | | | | 100% Modulus | 78 | 56 | 67 | 67 | -- | 78 | 100 | 111 |
| B | Silicone | SC | 225 | Tensile strength ^c | NC | NC | < 82 | -- | < 136 | < 155 | < 191 | < 136 |
| | | | | Elongation | NC | NC | < 48 | -- | < 2.5 | < 3.7 | < 2.5 | < 2.5 |
| | | | | 100% Modulus | 67 | 67 | 167 | -- | ND | ND | ND | ND |
| C | Silicone | SC | 225 | Tensile strength | 108 | 123 | 105 | 120 | 95 | 75 | 70 | 70 |
| | | | | Elongation | 103 | 113 | 103 | 110 | 95 | 69 | 56 | 46 |
| | | | | 100% Modulus | 100 | 100 | 100 | 100 | 100 | 117 | 133 | 142 |
| G | Silicone | PS | 225 | Tensile strength | NC | NC | < 74 | < 63 | < 57 | < 45 | < 41 | -- |
| | | | | Elongation | NC | NC | < 60 | < 43 | < 29 | < 10 | < 8 | -- |
| | | | | 100% Modulus | 142 | 142 | 183 | 200 | 242 | ND | ND | -- |

^a Same footnotes as Table 7. Additional footnotes are shown below.^b % retention = (value at indicated time x 100)/value at zero time^c Retention values are not calculated when both original and aged specimens were off-scale. When aged results are on-scale and zero time results are off-scale, the retentions are calculated on a less than (<) basis since the true value at zero time is greater than the recorded value.

NC = Not calculated (c.f., footnote c).

ND = Not determined (modulus values at 100% elongation cannot be determined when ultimate elongation < 100%).

TABLE 8 (Continued)

TABLE 8

| Code | Material Type | Class | Aging Temp. (°C) | Property | Percent Retention | | | | | | | |
|------|---------------|-------|------------------|------------------|-------------------|-----|-----|------|-------|-------|-------|-------|
| | | | | | Days Aged | | | | | | | |
| | | | | | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 100 |
| I | Silicone | PS | 225 | Tensile strength | 86 | 75 | 67 | 55 | 48 | -- | -- | -- |
| | | | | Elongation | 76 | 66 | 47 | 34 | 24 | -- | -- | -- |
| | | | | 100% Modulus | 126 | 130 | 165 | 174 | 204 | -- | -- | -- |
| K | Acrylic | PS | 225 | Tensile strength | 48 | 21 | 15 | 5 | -- | -- | -- | -- |
| | | | | Elongation | 53 | ~ 0 | ~ 0 | ~ 0 | -- | -- | -- | -- |
| | | | | 100% Modulus | ND | ND | ND | ND | -- | -- | -- | -- |
| L | Fluorocarbon | PS | 225 | Tensile strength | 108 | 104 | 108 | 97 | 96 | 103 | 101 | 96 |
| | | | | Elongation | 100 | 95 | 90 | 86 | 95 | 100 | 105 | 95 |
| | | | | 100% Modulus | 106 | 97 | 117 | 111 | 107 | 110 | 114 | 123 |
| Q | Silicone | PS | 225 | Tensile strength | 94 | 97 | 83 | 67 | 47 | 50 | 250 | 120 |
| | | | | Elongation | 84 | 80 | 60 | 40 | 4 | 0 | 0 | 0 |
| | | | | 100% Modulus | 92 | 90 | 100 | 107 | ND | ND | ND | ND |
| A | Silicone | SC | 200 | Tensile strength | 88 | 80 | 84 | 80 | 72 | 72 | 64 | 68 |
| | | | | Elongation | 103 | 103 | 103 | 110 | 131 | 124 | 124 | 117 |
| | | | | 100% Modulus | 82 | 91 | 82 | 82 | 64 | 73 | 64 | 82 |
| B | Silicone | SC | 200 | Tensile strength | NC | NC | NC | < 67 | < 75 | < 42 | < 50 | < 50 |
| | | | | Elongation | NC | NC | NC | < 83 | < 3.8 | < 1.3 | < 1.3 | < 2.5 |
| | | | | 100% Modulus | 100 | 100 | 67 | 100 | ND | ND | ND | ND |
| C | Silicone | SC | 200 | Tensile strength | 91 | 93 | 91 | 78 | 83 | 89 | 81 | 74 |
| | | | | Elongation | 103 | 97 | 103 | 94 | 114 | 111 | 103 | 83 |
| | | | | 100% Modulus | 87 | 93 | 87 | 80 | 73 | 93 | 93 | 107 |
| G | Silicone | PS | 200 | Tensile strength | NC | NC | NC | 93 | 75 | 55 | 49 | 43 |
| | | | | Elongation | NC | NC | NC | < 87 | < 66 | < 34 | < 23 | < 16 |
| | | | | 100% Modulus | 108 | 117 | 125 | 150 | 175 | 225 | 283 | 350 |
| I | Silicone | PS | 200 | Tensile strength | 92 | 85 | 82 | 80 | 71 | 58 | 55 | 55 |
| | | | | Elongation | 93 | 87 | 76 | 66 | 54 | 32 | 21 | 15 |
| | | | | 100% Modulus | 100 | 104 | 117 | 135 | 157 | 235 | 257 | 301 |

TABLE 8 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Days Aged | | | | | | | |
|------|----------------------|-------|------------------|------------------|-----------|-----|-----|-----|------|-------|-------|-------|
| | | | | | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 100 |
| L | Fluorocarbon | PS | 200 | Tensile strength | 101 | 99 | 106 | 104 | 96 | 94 | 104 | 98 |
| | | | | Elongation | 95 | 100 | 90 | 95 | 90 | 105 | 86 | |
| | | | | 100% Modulus | 106 | 97 | 117 | 111 | 107 | 110 | 114 | 123 |
| Q | Silicone | PS | 200 | Tensile strength | 107 | 102 | 94 | 77 | 56 | 41 | 28 | 9 |
| | | | | Elongation | 116 | 108 | 88 | 64 | 36 | 0 | 0 | 0 |
| | | | | 100% Modulus | 92 | 90 | 100 | 107 | ND | ND | ND | ND |
| A | Silicone | SC | 175 | Tensile strength | 114 | 110 | 110 | -- | -- | 100 | 105 | 100 |
| | | | | Elongation | 111 | 107 | 115 | -- | -- | 152 | 159 | 141 |
| | | | | 100% Modulus | 110 | 100 | 100 | -- | -- | 80 | 80 | 90 |
| B | Silicone | SC | 175 | Tensile strength | NC | NC | NC | -- | -- | < 120 | < 140 | < 150 |
| | | | | Elongation | NC | NC | NC | -- | -- | < 78 | < 63 | < 49 |
| | | | | 100% Modulus | 107 | 100 | 107 | -- | -- | 107 | 100 | 114 |
| C | Silicone | SC | 175 | Tensile strength | 87 | 96 | 87 | -- | -- | 102 | 100 | 109 |
| | | | | Elongation | 86 | 94 | 86 | -- | -- | 106 | 106 | 103 |
| | | | | 100% Modulus | 100 | 100 | 100 | -- | -- | 67 | 100 | 133 |
| G | Silicone | PS | 175 | Tensile strength | NC | NC | NC | NC | < 97 | < 79 | < 68 | < 60 |
| | | | | Elongation | NC | NC | NC | NC | < 88 | < 66 | < 43 | < 32 |
| | | | | 100% Modulus | 125 | 108 | 142 | 133 | 142 | 175 | 233 | 258 |
| I | Silicone | PS | 175 | Tensile strength | 95 | 95 | 111 | 94 | 84 | 71 | 73 | 67 |
| | | | | Elongation | 91 | 93 | 90 | 84 | 74 | 40 | 26 | 21 |
| | | | | 100% Modulus | 100 | 100 | 126 | 122 | 122 | 191 | 270 | 304 |
| J | Ethylene/ acrylic | PS | 175 | Tensile strength | 107 | 114 | 101 | 98 | 59 | 51 | -- | -- |
| | | | | Elongation | 98 | 107 | 93 | 81 | 40 | 5 | -- | -- |
| | | | | 100% Modulus | 100 | 102 | 95 | 109 | 135 | ND | -- | -- |
| K | Acrylic | PS | 175 | Tensile strength | 100 | 102 | 122 | 94 | 67 | -- | 64 | -- |
| | | | | Elongation | 100 | 94 | 81 | 56 | ~ 0 | -- | ~ 0 | -- |
| | | | | 100% Modulus | 91 | 86 | 118 | ND | ND | -- | ND | -- |

TABLE 8 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Percent Retention | | | | | | | | |
|------|------------------|-------|------------------|------------------|---|-----|-----|-------------------|-----|-----|---------|-----|--|
| | | | | | Days Aged | | | | | | | | |
| | | | | | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 100 | |
| N | EPDM | PS | 175 | Tensile strength | 74 | 83 | 59 | 27 | 35 | -- | -- | -- | |
| | | | | Elongation | 94 | 106 | 82 | 24 | 6 | -- | -- | -- | |
| | | | | 100% Modulus | 800 | 93 | 89 | 89 | ND | -- | -- | -- | |
| P | Butyl | PS | 175 | Tensile strength | 63 | 34 | -- | 5 | 3 | -- | -- | -- | |
| | | | | Elongation | 132 | 126 | -- | 96 | 2 | -- | -- | -- | |
| | | | | 100% Modulus | 67 | 60 | -- | -- | -- | -- | -- | -- | |
| Q | Silicone | PS | 175 | Tensile strength | 105 | 104 | 125 | 103 | 95 | 95 | 89 | 82 | |
| | | | | Elongation | 100 | 100 | 96 | 88 | 76 | 72 | 60 | 44 | |
| | | | | 100% Modulus | 102 | 100 | 116 | 108 | 104 | 114 | 137 | 168 | |
| D | Acrylic | SC | 150 | Tensile strength | 160 | 180 | 210 | 340 | -- | 710 | -- | 340 | |
| | | | | Elongation | 71 | 61 | 46 | 36 | -- | 19 | -- | 4 | |
| | | | | 100% Modulus | 136 | 155 | 182 | 309 | -- | ND | -- | ND | |
| E | Hypalon | SC | 150 | Tensile strength | 190 | 230 | 350 | 720 | -- | -- | -- | -- | |
| | | | | Elongation | 63 | 50 | ~ 0 | ~ 0 | -- | -- | -- | -- | |
| | | | | 100% Modulus | ND | ND | ND | ND | -- | -- | -- | -- | |
| F | Butyl | SC | 150 | Tensile strength | 87 | 44 | 53 | Specimens cracked | | | | | |
| | | | | Elongation | The zero time and subsequent elongation values were ~ 0 | | | | | | | | |
| | | | | 100% Modulus | ND | ND | ND | -- | -- | -- | -- | -- | |
| J | Ethylene/acrylic | PS | 150 | Tensile strength | 100 | 127 | 125 | 127 | 113 | 116 | 131 | 99 | |
| | | | | Elongation | 100 | 100 | 102 | 95 | 95 | 79 | 76 | 48 | |
| | | | | 100% Modulus | 100 | 106 | 106 | 111 | 100 | 167 | 164 | 211 | |
| K | Acrylic | PS | 150 | Tensile strength | -- | -- | 98 | 107 | 107 | 102 | 83 | 57 | |
| | | | | Elongation | -- | -- | 106 | 113 | 106 | 100 | 69 | 31 | |
| | | | | 100% Modulus | -- | -- | 73 | 86 | 98 | 92 | 133 | ND | |
| N | EPDM | PS | 150 | Tensile strength | 100 | 130 | 108 | 98 | 87 | 55 | 32 | 40 | |
| | | | | Elongation | 100 | 106 | 106 | 94 | 94 | 65 | 24 | 6 | |
| | | | | 100% Modulus | 111 | 138 | 110 | 125 | 118 | 116 | E < 100 | -- | |

TABLE 8 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Percent Retention | | | | | | | |
|------|---------------|------------------|------------------|----------|-------------------|-----|-----|-----|-----|-----|-----|-----|
| | | | | | Days Aged | | | | | | | |
| | | | | | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 100 |
| 100 | P | Butyl | PS | 150 | Tensile strength | 96 | 111 | 82 | 62 | 26 | -- | -- |
| | | | | | Elongation | 114 | 114 | 116 | 114 | 106 | -- | -- |
| | | | | | 100% Modulus | 80 | 107 | 93 | 80 | 60 | -- | -- |
| | D | Acrylic | SC | 125 | Tensile strength | 111 | -- | 144 | 122 | 178 | 322 | 478 |
| | | | | | Elongation | 119 | -- | 200 | 200 | 169 | 113 | 67 |
| | | | | | 100% Modulus | -- | -- | -- | -- | -- | -- | 53 |
| | E | Hypalon | SC | 125 | Tensile strength | 114 | -- | 171 | 257 | 286 | 386 | 400 |
| | | | | | Elongation | 114 | -- | 79 | 64 | 57 | 43 | 7 |
| | | | | | 100% Modulus | ND | -- | ND | ND | ND | ND | ND |
| | F | Butyl | SC | 125 | Tensile strength | 106 | -- | 96 | 96 | 92 | 48 | 35 |
| | | | | | Elongation | ~0 | -- | ~0 | ~0 | ~0 | ~0 | ~0 |
| | | | | | 100% Modulus | ND | -- | ND | ND | ND | ND | ND |
| 100 | J | Ethylene/acrylic | PS | 125 | Tensile strength | -- | -- | -- | -- | 103 | 110 | 128 |
| | | | | | Elongation | -- | -- | -- | -- | 95 | 93 | 102 |
| | | | | | 100% Modulus | -- | -- | -- | -- | 95 | 134 | 111 |
| | K | Acrylic | PS | 125 | Tensile strength | -- | -- | -- | -- | 112 | 107 | 112 |
| | | | | | Elongation | -- | -- | -- | -- | 125 | 113 | 119 |
| | | | | | 100% Modulus | -- | -- | -- | -- | 67 | 80 | 92 |
| | N | EPDM | PS | 125 | Tensile strength | -- | -- | -- | 104 | 102 | 110 | 106 |
| | | | | | Elongation | -- | -- | -- | 112 | 106 | 106 | 106 |
| | | | | | 100% Modulus | -- | -- | -- | 99 | 105 | 111 | 126 |
| | P | Butyl | PS | 125 | Tensile strength | -- | -- | 98 | 100 | 90 | 78 | 48 |
| | | | | | Elongation | -- | -- | 130 | 132 | 128 | 124 | 124 |
| | | | | | 100% Modulus | -- | -- | 80 | 93 | 80 | 80 | 73 |
| 100 | D | Acrylic | SC | 100 | Tensile strength | -- | -- | -- | -- | -- | 210 | 290 |
| | | | | | Elongation | -- | -- | -- | -- | -- | 240 | 200 |
| | | | | | 100% Modulus | -- | -- | -- | -- | -- | 320 | 400 |

TABLE 8 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Percent Retention | | | | | | | |
|------|---------------|-------|------------------|------------------|-------------------|----|----|----|----|--|------|------|
| | | | | | Days Aged | | | | | | | |
| | | | | | 1. | 2 | 4 | 8 | 16 | 32 | 64 | 100 |
| E | Hypalon | SC | 100 | Tensile strength | -- | -- | -- | -- | -- | 670 | 1200 | 1570 |
| | | | | Elongation | -- | -- | -- | -- | -- | 130 | 100 | 86 |
| | | | | 100% Modulus | -- | -- | -- | -- | -- | ND | ND | ND |
| F | Butyl | SC | 100 | Tensile strength | -- | -- | -- | -- | -- | 87 | 124 | 59 |
| | | | | Elongation | -- | -- | -- | -- | -- | (Original and subsequent E values ~ 0) | | |
| | | | | 100% Modulus | -- | -- | -- | -- | -- | ND | ND | ND |

TABLE 9
ESTIMATED LIFE OF SEALANTS IN AIR

| Code | Material | Days to Reach 60% Retention of Original Tensile Property | | | Property** |
|------|----------------------|---|--------------|------------|---------------------|
| | | 100°C | 125°C | 150°C | |
| P | Butyl | 520 (650) | 49 (59) | 7 (8) | Tensile strength |
| N | EPDM | -- | 180 | 30 | Tensile strength |
| | | | 220 | 35 | Ultimate elongation |
| K | Acrylic | -- | 590 | 91 | Tensile strength |
| | | | 480 | 72 | Ultimate elongation |
| J | Ethylene- acrylic | -- | 790 (970)* | 82 (96)* | Ultimate elongation |
| Q | Silicone | -- | 4300 (6800)* | 430 (630)* | Ultimate elongation |

* () indicates time to reach 50% retention of original property value.
Values \geq 180 days were obtained by extrapolation of Arrhenius plots.

** Property studied is that which was considerably more severely affected by the thermal aging. Both properties are listed in cases where the extent of their decline was similar.

TABLE 10

EFFECT OF THERMAL AGING IN AIR ON WEIGHT LOSS AND HARDNESS

103

| Code | Material Type | Class | Aging Temp. (°C) | Property | Days Aged | | | | | | | | |
|------|---------------|-------|------------------|---------------|-----------|-------|-------|-------|-------|-------|-------|-------|-------|
| | | | | | 0 | 1 | 2 | 4 | 8 | 16 | 28 | | |
| G | Silicone | PS | 250 | % Weight Loss | | 2.35 | 2.60 | 2.89 | 3.46 | 4.37 | 6.02 | | |
| | | | | Hardness | 48-50 | 53-54 | 54-55 | 57-58 | 61-63 | 67-68 | 71-73 | | |
| I | Silicone | PS | 250 | % Weight Loss | | 1.29 | 1.67 | 2.05 | 2.48 | 3.16 | 4.10 | | |
| | | | | Hardness | 60-61 | 64-65 | 64-65 | 67-69 | 70-72 | 74-75 | 75-76 | | |
| L | Fluorocarbon | PS | 250 | % Weight Loss | | 0.54 | 0.90 | 1.51 | 2.72 | 4.85 | 7.88 | | |
| | | | | Hardness | 76-78 | 76-78 | 76-78 | 77-79 | 77-79 | 79-81 | 80-82 | | |
| Q | Silicone | PS | 250 | % Weight Loss | | 1.54 | 2.48 | 4.49 | 8.59 | 17.0 | 28.4 | | |
| | | | | Hardness | 70-71 | 74-76 | 75-76 | 78-79 | 83-84 | 91-93 | 95-96 | | |
| | | | | | 0 | 1 | 2 | 3 | 5 | 9 | 17 | 36 | 65 |
| A | Silicone | SC | 225 | % Weight Loss | | 2.23 | 2.23 | 2.53 | 2.59 | 2.78 | 5.07 | 7.43 | |
| | | | | Hardness | 32-34 | 32-34 | 32-34 | 32-34 | 32-34 | -- | 32-34 | 49-50 | |
| B | Silicone | SC | 225 | % Weight Loss | | 1.65 | 3.32 | 5.28 | 11.02 | 24.7 | 36.3 | 39.0 | |
| | | | | Hardness | 11-12 | 11-12 | 11-12 | 11-12 | 15-17 | 47-49 | 74-75 | 75-76 | |
| C | Silicone | SC | 225 | % Weight Loss | | 5.24 | 5.24 | 5.74 | 5.74 | 6.39 | 7.17 | 8.33 | 9.40 |
| | | | | Hardness | 31-32 | 33-34 | 33-34 | 33-34 | 33-34 | 33-34 | 34-36 | 39-41 | 40-41 |

Hardness - Shore A scale.

Weight loss and hardness values are averages of two sets of data.

TABLE 10 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Days Aged | | | | | | | | | | |
|------|---------------|--------------|------------------|----------|---------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| | | | | | 0 | 1 | 2 | 4 | 8 | 16 | 28 | 46 | 81 | 122 | 179 |
| 104 | G | Silicone | PS | 225 | % Weight Loss | | 2.16 | 2.24 | 2.31 | 2.55 | 2.83 | 3.19 | 3.67 | | |
| | | | | | Hardness | 48-50 | 52-53 | 52-53 | 54-55 | 56-58 | 59-61 | 60-62 | 63-64 | | |
| | I | Silicone | PS | 225 | % Weight Loss | | 1.06 | 1.15 | 1.37 | 1.59 | 1.90 | 2.34 | 2.87 | | |
| | | | | | Hardness | 60-61 | 62-63 | 62-64 | 64-66 | 66-68 | 69-71 | 70-71 | 72-74 | | |
| | L | Fluorocarbon | PS | 225 | % Weight Loss | | 0.23 | 0.38 | 0.46 | 0.84 | 1.37 | 2.18 | 3.37 | 5.65 | 9.17 |
| | | | | | Hardness | 76-78 | 76-78 | 76-78 | 76-78 | 77-79 | 78-80 | 78-80 | 79-81 | 80-82 | 81-83 |
| | Q | Silicone | PS | 225 | % Weight Loss | | 0.92 | 1.28 | 1.49 | 3.49 | 7.75 | 16.4 | 27.0 | | |
| | | | | | Hardness | 70-71 | 73-74 | 73-75 | 74-76 | 76-78 | 81-82 | 89-90 | 93-95 | | |
| | | | | | | 0 | 1 | 2 | 3 | 5 | 9 | 17 | 36 | 65 | |
| | A | Silicone | SC | 200 | % Weight Loss | | 1.86 | 1.86 | 2.25 | 2.25 | 2.25 | 2.44 | 2.95 | 3.27 | |
| | | | | | Hardness | 32-34 | 32-34 | 32-34 | 32-33 | 32-33 | 32-34 | 32-34 | 32-34 | 32-34 | |
| | B | Silicone | SC | 200 | % Weight Loss | | 1.20 | 1.89 | 2.55 | 5.00 | 12.3 | 29.9 | 42.8 | 44.5 | |
| | | | | | Hardness | 11-12 | 11-12 | 11-12 | 11-12 | 14-15 | 18-19 | 55-57 | 65-67 | 65-67 | |
| | C | Silicone | SC | 200 | % Weight Loss | | 4.75 | 4.75 | 5.21 | 5.21 | 5.66 | 6.12 | 7.34 | 8.17 | |
| | | | | | Hardness | 30-33 | 33-34 | 33-34 | 33-34 | 33-34 | 33-34 | 34-36 | 37-39 | 39-40 | |

TABLE 10 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Days Aged | | | | | | | | | | |
|------|---------------|----------|------------------|---------------------------|-----------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
| | | | | | 0 | 1 | 2 | 4 | 8 | 16 | 28 | 46 | 81 | 122 | 179 |
| G | Silicone | PS | 200 | % Weight Loss Hardness | 48-50 | 1.53 50-52 | 1.61 51-53 | 1.73 52-54 | 1.90 54-56 | 2.14 56-58 | 2.43 56-58 | 2.71 59-60 | | | |
| I | Silicone | PS | 200 | % Weight Loss Hardness | 60-61 | 0.66 61-62 | 0.79 61-62 | 0.97 63-65 | 1.28 64-66 | 1.45 66-68 | 1.71 66-68 | 1.89 69-71 | | | |
| L | Fluorocarbon | PS | 200 | % Weight Loss Hardness | 76-78 | 0.13 76-78 | 0.21 76-78 | 0.21 76-78 | 0.41 76-78 | 0.54 76-78 | 0.93 77-79 | 1.29 77-79 | 1.96 78-80 | 2.78 79-81 | 4.04 79-81 |
| Q | Silicone | PS | 200 | % Weight Loss Hardness | 70-71 | 0.56 72-73 | 0.81 72-74 | 1.69 73-75 | 1.89 74-76 | 3.63 76-77 | 7.37 80-81 | 11.7 83-84 | | | |
| 105 | A | Silicone | SC | % Weight Loss Hardness | 32-34 | 1.07 32-34 | 1.07 32-34 | 1.53 32-34 | 1.71 32-34 | 1.76 32-34 | 1.93 32-34 | 2.29 33-36 | 2.34 33-35 | 2.91 35-37 | |
| | B | Silicone | SC | % Weight Loss Hardness | 11-12 | 0.55 11-12 | 0.90 11-12 | 1.02 11-12 | 1.19 12-13 | 2.18 12-13 | 5.01 13-15 | 13.9 35-42 | 27.7 60-65 | 37.6 60-65 | |
| | C | Silicone | SC | % Weight Loss Hardness | 30-33 | 3.12 33-34 | 3.70 33-34 | 4.13 33-34 | 4.27 33-34 | 4.77 33-34 | 5.34 34-35 | 5.84 37-39 | 6.27 38-39 | 6.77 38-40 | |
| | | | | | 0 | 1 | 2 | 4 | 8 | 16 | 28 | 46 | 81 | 122 | 179 |
| G | Silicone | PS | 175 | % Weight Loss Hardness | 48-50 | 1.09 49-51 | 1.17 49-51 | 1.17 50-52 | 1.33 51-53 | 1.53 52-54 | 1.77 52-54 | 2.05 54-56 | | | |
| I | Silicone | PS | 175 | % Weight Loss Hardness | 60-61 | 0.26 60-61 | 0.26 60-61 | 0.39 61-63 | 0.47 61-63 | 0.64 64-66 | 0.90 64-66 | 1.19 66-68 | | | |

TABLE 10 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Days Aged | | | | | | | | | | |
|------|------------------|-------|------------------|---------------|-----------|-------|-------|-------|-------|-------|---------|-------|-------|---------|-------|
| | | | | | 0 | 1 | 4 | 8 | 15 | 32 | 53 | 84 | 117 | | |
| J | Ethylene/acrylic | PS | 175 | % Weight Loss | | 2.47 | 3.11 | 3.33 | 3.97 | 6.17 | 9.63 | 15.7 | 19.6 | | |
| | | | | Hardness | 56-68 | 69-71 | 70-72 | 71-73 | 71-73 | 77-78 | 87-90 | > 95 | | | |
| K | Acrylic | PS | 175 | % Weight Loss | | 1.05 | 1.68 | 2.15 | 3.42 | 6.88 | 14.8 | 26.2 | 32.7 | | |
| | | | | Hardness | 59-60 | 61-63 | 63-65 | 64-66 | 69-71 | 81-83 | > 90 | | | | |
| | | | | | 0 | 1 | 2 | 4 | 8 | 16 | 28 | 46 | 81 | 122 | 179 |
| L | Fluorocarbon | PS | 175 | % Weight Loss | | 0.05 | 0.05 | 0.05 | 0.13 | 0.13 | 0.18 | 0.28 | 0.46 | 0.59 | 0.92 |
| | | | | Hardness | 76-78 | 76-78 | 76-78 | 76-78 | 76-78 | 76-78 | 76-78 | 76-78 | 76-78 | 76-78 | 76-78 |
| Q | Silicone | PS | 175 | % Weight Loss | | 0.20 | 0.20 | 0.28 | 0.37 | 0.76 | 1.45 | 1.97 | | | |
| | | | | Hardness | 70-71 | 70-72 | 71-72 | 72-74 | 72-74 | 72-74 | 74-75 | 74-76 | | | |
| | | | | | 0 | 1 | 2 | 3 | 5 | 9 | 17 | 36 | 65 | 86 | |
| D | Acrylic | SC | 150 | % Weight Loss | | 1.77 | 2.83 | 3.32 | 3.38 | 4.49 | 5.54 | 7.47 | 9.41 | 10.7 | |
| | | | | Hardness | 51-54 | 56-59 | 56-59 | 60-62 | 64-66 | 64-66 | 74-76 | 82-84 | 86-87 | brittle | |
| E | Hypalon | SC | 150 | % Weight Loss | | 15.1 | 19.5 | 22.1 | 26.7 | 33.1 | 37.1 | 38.9 | 39.8 | 39.8 | |
| | | | | Hardness | 19-20 | 38-41 | 49-51 | 56-58 | 71-73 | 84-85 | brittle | | | | |
| F | Butyl | SC | 150 | % Weight Loss | | 7.01 | 7.59 | 8.12 | 9.37 | 13.2 | 20.0 | 28.7 | 35.6 | 38.4 | |
| | | | | Hardness | 56-59 | 76-79 | 76-79 | 76-79 | 76-79 | 76-79 | 78-81 | 83-85 | 86-89 | brittle | |
| | | | | | 0 | 1 | 5 | 9 | 30 | 41 | 56 | 72 | 113 | 146 | 173 |
| G | Silicone | PS | 150 | % Weight Loss | | 0.63 | 1.00 | 1.04 | 1.33 | 1.46 | 1.50 | 1.50 | | | |
| | | | | Hardness | 47-48 | 47-48 | 50-51 | 50-51 | 51-52 | 52-53 | 52-53 | 52-53 | | | |
| I | Silicone | PS | 150 | % Weight Loss | | 0.18 | 0.26 | 0.26 | 0.61 | 0.78 | 0.87 | 0.87 | | | |
| | | | | Hardness | 60-61 | 60-61 | 60-61 | 60-61 | 60-62 | 63-64 | 63-64 | 63-64 | | | |

TABLE 10 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Days Aged | | | | | | | | | | |
|------|------------------|-------|------------------|---------------|-----------|----------|----------|----------|-----------|-----------|-----------|-----------|------------|------------|------------|
| | | | | | 0 | 1 | 4 | 8 | 15 | 32 | 53 | 84 | 117 | 193 | |
| J | Ethylene/acrylic | PS | 150 | % Weight Loss | | 2.01 | 2.30 | 2.42 | 2.70 | 3.05 | 3.28 | 4.14 | 4.95 | 7.19 | |
| | | | | Hardness | 66-68 | 67-69 | 69-71 | 70-72 | 70-72 | 72-74 | 72-74 | 72-75 | 77-78 | 82-84 | |
| K | Acrylic | PS | 150 | % Weight Loss | | 0.60 | 0.76 | 0.92 | 1.31 | 1.90 | 2.55 | 4.12 | 5.91 | 12.2 | |
| | | | | Hardness | 59-60 | 60-62 | 61-63 | 62-63 | 63-64 | 66-67 | 68-69 | 72-74 | 79-82 | 92-93 | |
| | | | | | <u>0</u> | <u>1</u> | <u>2</u> | <u>4</u> | <u>11</u> | <u>24</u> | <u>52</u> | <u>85</u> | <u>175</u> | | |
| N | EPDM | PS | 150 | % Weight Loss | | 1.57 | 1.69 | 1.69 | 1.73 | 1.73 | 1.73 | 2.67 | 7.03 | | |
| | | | | Hardness | 76-78 | 77-79 | 77-79 | 78-80 | 79-81 | 81-82 | 85-86 | 91-93 | brittle | | |
| P | Butyl | PS | 150 | % Weight Loss | | 2.82 | 3.25 | 3.82 | 4.57 | 5.50 | 7.05 | 7.91 | | | |
| | | | | Hardness | 51-53 | 55-57 | 57-59 | 59-61 | 60-62 | 62-63 | 62-63 | 62-63 | | | |
| | | | | | <u>0</u> | <u>1</u> | <u>5</u> | <u>9</u> | <u>30</u> | <u>41</u> | <u>56</u> | <u>72</u> | <u>113</u> | <u>146</u> | <u>173</u> |
| Q | Silicone | PS | 150 | % Weight Loss | | 0.12 | 0.20 | 0.20 | 0.67 | 0.87 | 1.10 | 1.30 | 2.12 | 3.14 | 3.46 |
| | | | | Hardness | 70-71 | 70-71 | 70-71 | 71-72 | 71-73 | 72-74 | 72-74 | 72-74 | 72-74 | 76-78 | 76-78 |
| | | | | | <u>0</u> | <u>1</u> | <u>2</u> | <u>3</u> | <u>5</u> | <u>9</u> | <u>17</u> | <u>36</u> | <u>65</u> | <u>86</u> | |
| D | Acrylic | SC | 125 | % Weight Loss | | 1.23 | 1.68 | 1.94 | 2.13 | 2.39 | 2.90 | 3.69 | 4.20 | 4.78 | |
| | | | | Hardness | 51-54 | 51-54 | 56-59 | 56-59 | 60-62 | 60-62 | 61-64 | 73-75 | 73-74 | 74-80 | |
| E | Hypalon | SC | 125 | % Weight Loss | | 5.40 | 8.32 | 9.63 | 11.1 | 12.8 | 15.2 | 20.0 | 25.3 | 27.9 | |
| | | | | Hardness | 19-20 | 26-28 | 30-32 | 39-40 | 40-42 | 47-48 | 48-50 | 69-70 | 83-85 | 84-87 | |
| F | Butyl | SC | 125 | % Weight Loss | | 6.33 | 7.07 | 7.32 | 7.57 | 8.13 | 8.87 | 10.7 | 15.2 | 17.6 | |
| | | | | Hardness | 56-59 | 75-78 | 75-78 | 75-78 | 75-78 | 75-78 | 78-80 | 77-80 | 78-80 | 80-83 | |

TABLE 10 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Days Aged | | | | | | | | | |
|----------|-------------------|-------|------------------|------------------------|-----------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
| | | | | | 0 | 1 | 4 | 8 | 15 | 32 | 53 | 84 | 117 | 193 |
| J | Ethylene/ acrylic | PS | 125 | % Weight Loss Hardness | 66-68 | 0.93 66-68 | 1.45 67-69 | 1.57 69-71 | 1.92 69-71 | 2.21 71-73 | 2.26 71-73 | 2.61 72-73 | 2.61 72-73 | 2.67 72-73 |
| K | Acrylic | PS | 125 | % Weight Loss Hardness | 59-60 | 0.29 59-60 | 0.35 59-60 | 0.41 60-61 | 0.64 61-63 | 0.81 64-66 | 0.92 64-66 | 1.50 65-67 | 1.50 67-69 | 2.02 68-70 |
| | | | | | 0 | 1 | 2 | 4 | 11 | 24 | 52 | 85 | 175 | |
| N | EPDM | PS | 125 | % Weight Loss Hardness | 76-78 | 1.02 76-78 | 1.10 77-79 | 1.10 77-79 | 1.10 78-80 | 1.10 78-80 | 1.10 79-81 | 1.10 79-81 | 1.10 82-83 | |
| P | Butyl | PS | 125 | % Weight Loss Hardness | 51-53 | 1.17 52-54 | 1.30 53-55 | 1.74 54-56 | 2.23 56-58 | 2.87 58-60 | 3.41 59-60 | 3.58 59-60 | 4.02 65 | |
| | | | | | 0 | 1 | 2 | 3 | 5 | 9 | 17 | 36 | 65 | 86 |
| 801 D | Acrylic | SC | 100 | % Weight Loss Hardness | 51-54 | 0.91 51-54 | 1.18 55-57 | 1.24 55-57 | 1.43 59-60 | 1.43 59-60 | 1.62 59-62 | 2.01 66-69 | 2.21 67-69 | 2.34 69-71 |
| E | Hypalon | SC | 100 | % Weight Loss Hardness | 19-20 | 2.67 25-28 | 4.77 29-32 | 5.66 37-40 | 7.51 37-40 | 9.41 40-42 | 11.2 47-50 | 12.9 54-56 | 15.4 65-67 | 16.2 66-68 |
| F | Butyl | SC | 100 | % Weight Loss Hardness | 56-59 | 5.16 74-76 | 5.66 75-76 | 5.87 75-76 | 6.24 75-77 | 6.38 75-77 | 6.82 78-80 | 7.17 78-80 | 7.68 78-81 | 7.75 79-80 |
| | | | | | 0 | 1 | 2 | 4 | 11 | 24 | 52 | 85 | 175 | |
| N | EPDM | PS | 100 | % Weight Loss Hardness | 76-78 | 0.76 76-78 | 0.88 77-79 | 0.88 77-79 | 0.92 77-79 | 0.92 77-79 | 0.92 78-80 | 0.92 78-80 | 0.92 79-81 | |
| P | Butyl | PS | 100 | % Weight Loss Hardness | 51-53 | 0.39 51-53 | 0.43 52-54 | 0.69 52-54 | 1.29 53-55 | 2.11 56-57 | 2.75 56-58 | 2.97 56-58 | 3.40 62-63 | |

TABLE 11

EFFECT OF HYDROLYTIC AGING ON TENSILE PROPERTIES

| Code | Material Type | Class ^b | Aging Temp. (°C) | Property ^a | Days Aged | | | | | | | | |
|------|----------------------|--------------------|------------------|-----------------------|-----------|------|------|-------|---------------------------------|-------|------|----|-----|
| | | | | | 0 | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 128 |
| A | Silicone | SC | 125 | Tensile strength | 160 | 150 | 150 | 130 | 80 | 20 | -- | | |
| | | | | Elongation | 210 | 250 | 270 | 320 | 440 | 150 | -- | | |
| | | | | 100% Modulus | 100 | 90 | 80 | 60 | 40 | 20 | | | |
| B | Silicone | SC | 125 | Tensile strength | 60* | 60* | 50* | 30* | Specimens too tacky for testing | | | | |
| | | | | Elongation | 830* | 830* | 795* | 718* | | | | | |
| | | | | 100% Modulus | 20 | 30 | 30 | 10 | | | | | |
| C | Silicone | SC | 125 | Tensile strength | 340 | 420 | 390 | 330 | 310 | 220 | 150 | | |
| | | | | Elongation | 290 | 370 | 370 | 370 | 360 | 450 | 440 | | |
| | | | | 100% Modulus | 130 | 130 | 120 | 80 | 110 | 60 | 50 | | |
| G | Silicone | PS | 125 | Tensile strength | 880* | 960* | 990* | 1030* | 1350* | 1010* | 700 | | |
| | | | | Elongation | 750* | 730* | 750* | 720* | 710* | 700* | 510 | | |
| | | | | 100% Modulus | 120 | 150 | 140 | 150 | 190 | 170 | 180 | | |
| I | Silicone | PS | 125 | Tensile strength | 1280 | 1150 | 1260 | 1280 | 1430 | 940 | 610 | | |
| | | | | Elongation | 700 | 590 | 570 | 520 | 430 | 49 | 26 | | |
| | | | | 100% Modulus | 220 | 230 | 280 | 290 | 410 | 370 | 410 | | |
| J | Ethylene/ acrylic | PS | 125 | Tensile strength | 2080 | 2370 | 2360 | 1970 | 2500 | 2880 | 1140 | | |
| | | | | Elongation | 420 | 470 | 490 | 330 | 240 | 120 | ~0 | | |
| | | | | 100% Modulus | 380 | 330 | 330 | 430 | 850 | 2380 | ND | | |
| L | Fluorocarbon | PS | 125 | Tensile strength | 1690 | 1610 | 1550 | 1370 | 1770 | 1410 | 1280 | | |
| | | | | Elongation | 220 | 210 | 250 | 220 | 230 | 250 | 210 | | |
| | | | | 100% Modulus | 650 | 690 | 640 | 630 | 780 | 620 | 660 | | |
| N | EPDM | PS | 125 | Tensile strength | 2010 | 2020 | 2040 | 1960 | 2300 | 1980 | 1910 | | |
| | | | | Elongation | 180 | 170 | 170 | 180 | 170 | 200 | 180 | | |
| | | | | 100% Modulus | 800 | 880 | 840 | 790 | 970 | 680 | 770 | | |

* Specimens did not break - elongation exceeded capability of testing machine. Thus, true values are greater than those indicated.

^a Tensile strength, units are lb/in²; elongation, percent ultimate elongation; 100% modulus, tensile modulus at 100% elongation (cannot be determined when ultimate elongation < 100%).

^b All SC specimens (caulking compounds) were permitted to age for 4-6 wks at room temperature and then cured for 24 hrs at the aging temperature prior to testing. The PS specimens (preformed seals) were cured by their suppliers and tested on an as-received basis.

ND = Not determined (modulus values at 100% elongation cannot be determined when ultimate elongation < 100%). Values are arithmetic averages of three sets of data.

TABLE 11 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Days Aged | | | | | | | | | |
|------|----------------------|----------|------------------|------------------|------------------|------|------|-------|------|-------|-------|------|-----|-----|
| | | | | | 0 | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 128 | |
| 110 | P | Butyl | PS | 125 | Tensile strength | 1600 | 1520 | 1560 | 1630 | 2020 | 1430 | 1250 | | |
| | | | | | Elongation | 600 | 610 | 550 | 600 | 570 | 540 | 470 | | |
| | | | | | 100% Modulus | 130 | 120 | 140 | 110 | 190 | 130 | 130 | | |
| | Q | Silicone | PS | 125 | Tensile strength | 1060 | 1050 | 1000 | 1000 | 1010 | 750 | 710 | | |
| | | | | | Elongation | 280 | 250 | 250 | 240 | 190 | 190 | 180 | | |
| | | | | | 100% Modulus | 460 | 500 | 470 | 460 | 570 | 460 | 450 | | |
| | A | Silicone | SC | 100 | Tensile strength | 160 | -- | 230 | 170 | 220 | 180 | 170 | 150 | |
| | | | | | Elongation | 210 | -- | 240 | 270 | 410 | 360 | 310 | 300 | |
| | | | | | 100% Modulus | 100 | -- | 120 | 80 | 90 | 70 | 70 | 70 | |
| | B | Silicone | SC | 100 | Tensile strength | 60* | -- | 60* | 60* | 50* | 40* | 30* | 30 | |
| | | | | | Elongation | 830* | -- | 730* | 840* | 830* | 830* | 810* | 700 | |
| | | | | | 100% Modulus | 20 | -- | 20 | 20 | 20 | 20 | 10 | 20 | |
| | C | Silicone | SC | 100 | Tensile strength | 340 | -- | 520 | 410 | 390 | 370 | 370 | 340 | |
| | | | | | Elongation | 290 | -- | 350 | 350 | 360 | 320 | 390 | 350 | |
| | | | | | 100% Modulus | 130 | -- | 150 | 120 | 110 | 110 | 100 | 110 | |
| | G | Silicone | PS | 100 | Tensile strength | 880* | -- | 1170* | 930* | 1070* | 1090* | -- | 940 | 700 |
| | | | | | Elongation | 750* | -- | 650* | 730* | 740* | 730* | -- | 530 | 390 |
| | | | | | 100% Modulus | 120 | -- | 200 | 130 | 150 | 150 | -- | 210 | 230 |
| | I | Silicone | PS | 100 | Tensile strength | 1280 | -- | 1590 | 1230 | 1310 | 1210 | -- | 810 | 510 |
| | | | | | Elongation | 700 | -- | 600 | 600 | 580 | 510 | -- | 220 | 90 |
| | | | | | 100% Modulus | 220 | -- | 320 | 240 | 300 | 300 | -- | 470 | ND |
| J | Ethylene/ acrylic | PS | 100 | Tensile strength | 2080 | -- | 1970 | 1950 | 2010 | 1950 | 2200 | 2330 | | |
| | | | | Elongation | 420 | -- | 450 | 430 | 420 | 390 | 380 | 290 | | |
| | | | | 100% Modulus | 380 | -- | 400 | 400 | 430 | 450 | 530 | 790 | | |
| K | Acrylic | PS | 100 | Tensile strength | 1570 | -- | 1370 | 1310 | 1310 | 1290 | 1440 | 1770 | | |
| | | | | Elongation | 170 | -- | 190 | 190 | 180 | 170 | 160 | 130 | | |
| | | | | 100% Modulus | 510 | -- | 580 | 500 | 540 | 590 | 690 | 1350 | | |

TABLE 11 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Days Aged | | | | | | | |
|------|---------------|-------|------------------|------------------|-----------|------|------|------|------|------|------|------|
| | | | | | 0 | 2 | 4 | 8 | 16 | 32 | 64 | 128 |
| L | Fluorocarbon | PS | 100 | Tensile strength | 1690 | 2000 | 1600 | 1620 | 1420 | -- | 1480 | 1250 |
| | | | | Elongation | 220 | 210 | 230 | 220 | 250 | -- | 210 | 190 |
| | | | | 100% Modulus | 650 | 840 | 670 | 700 | 650 | -- | 740 | 720 |
| N | EPDM | PS | 100 | Tensile strength | 2010 | 2560 | 2130 | 2140 | 1840 | -- | 2000 | 2060 |
| | | | | Elongation | 180 | 180 | 180 | 180 | 170 | -- | 170 | 190 |
| | | | | 100% Modulus | 800 | 1120 | 870 | 840 | 900 | -- | 900 | 900 |
| P | Butyl | PS | 100 | Tensile strength | 1600 | 1940 | 1520 | 1660 | 1580 | -- | 1440 | 1160 |
| | | | | Elongation | 600 | 560 | 600 | 600 | 570 | -- | 490 | 450 |
| | | | | 100% Modulus | 130 | 160 | 100 | 110 | 120 | -- | 140 | 140 |
| Q | Silicone | PS | 100 | Tensile strength | 1060 | 1300 | 1000 | 1010 | 980 | -- | 970 | 930 |
| | | | | Elongation | 280 | 240 | 230 | 230 | 230 | -- | 190 | 180 |
| | | | | 100% Modulus | 460 | 620 | 480 | 500 | 520 | -- | 560 | 590 |
| A | Silicone | SC | 83 | Tensile strength | 160 | -- | 160 | 200 | 190 | -- | 180 | 140 |
| | | | | Elongation | 210 | -- | 200 | 280 | 290 | -- | 310 | 220 |
| | | | | 100% Modulus | 100 | -- | 90 | 100 | 90 | -- | 90 | 80 |
| B | Silicone | SC | 83 | Tensile strength | 60* | -- | 60* | 70* | 40* | 53* | 44 | |
| | | | | Elongation | 830* | -- | 800* | 830* | 790* | 790* | 800 | |
| | | | | 100% Modulus | 20 | -- | 20 | 30 | 20 | 20 | 20 | |
| C | Silicone | SC | 83 | Tensile strength | 340 | -- | 450 | 390 | 460 | -- | 380 | 430 |
| | | | | Elongation | 290 | -- | 350 | 350 | 380 | -- | 340 | 360 |
| | | | | 100% Modulus | 130 | -- | 130 | 120 | 120 | -- | 120 | 140 |
| G | Silicone | PS | 83 | Tensile strength | 880* | -- | 910* | 950* | 950* | -- | 1080 | 1120 |
| | | | | Elongation | 750* | -- | 740* | 730* | 730* | -- | 660 | 570 |
| | | | | 100% Modulus | 120 | -- | 120 | 130 | 120 | -- | 170 | 210 |
| I | Silicone | PS | 83 | Tensile strength | 1280 | -- | 1260 | 1230 | 1210 | -- | 1230 | 930 |
| | | | | Elongation | 700 | -- | 660 | 610 | 600 | -- | 460 | 320 |
| | | | | 100% Modulus | 220 | -- | 105 | 118 | 109 | -- | 155 | 191 |

TABLE 11 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Days Aged | | | | | | |
|------|------------------|-------|------------------|------------------|-----------|------|------|------|------|------|------|
| | | | | | 0 | 4 | 8 | 16 | 32 | 64 | 128 |
| J | Ethylene/acrylic | PS | 83 | Tensile strength | 2080 | 1900 | 1870 | 1870 | 2050 | 2270 | 2050 |
| | | | | Elongation | 420 | 410 | 380 | 380 | 370 | 370 | 250 |
| | | | | 100% Modulus | 380 | 400 | 510 | 440 | 480 | 620 | 780 |
| K | Acrylic | PS | 83 | Tensile strength | 1570 | 1280 | 1300 | 1280 | 1350 | 1380 | 1270 |
| | | | | Elongation | 170 | 190 | 180 | 180 | 170 | 170 | 120 |
| | | | | 100% Modulus | 510 | 480 | 680 | 530 | 600 | 710 | 1120 |
| L | Fluorocarbon | PS | 83 | Tensile strength | 1690 | 1670 | 1760 | 1590 | -- | 1450 | 1320 |
| | | | | Elongation | 220 | 210 | 230 | 240 | -- | 240 | 210 |
| | | | | 100% Modulus | 650 | 690 | 680 | 610 | -- | 650 | 670 |
| N | EPDM | PS | 83 | Tensile strength | 2010 | 2160 | 2020 | 2050 | -- | 2030 | 2200 |
| | | | | Elongation | 180 | 180 | 170 | 170 | -- | 180 | 190 |
| | | | | 100% Modulus | 800 | 900 | 860 | 840 | -- | 880 | 940 |
| P | Butyl | PS | 83 | Tensile strength | 1600 | 1610 | 1580 | 1540 | -- | 1370 | 1200 |
| | | | | Elongation | 600 | 610 | 620 | 600 | -- | 520 | 490 |
| | | | | 100% Modulus | 130 | 120 | 120 | 120 | -- | 140 | 140 |
| Q | Silicone | PS | 83 | Tensile strength | 1060 | 1030 | 1020 | 1030 | -- | 1050 | 1000 |
| | | | | Elongation | 280 | 250 | 230 | 240 | -- | 200 | 170 |
| | | | | 100% Modulus | 480 | 480 | 500 | 500 | -- | 580 | 630 |
| A | Silicone | SC | 67 | Tensile strength | 160 | 230 | 200 | 190 | -- | 210 | 160 |
| | | | | Elongation | 210 | 290 | 250 | 300 | -- | 280 | 230 |
| | | | | 100% Modulus | 100 | 100 | 110 | 90 | -- | 110 | 100 |
| B | Silicone | SC | 67 | Tensile strength | 60* | 80* | 70* | 50* | -- | 60 | 70 |
| | | | | Elongation | 830* | 820* | 840* | 850* | -- | 800 | 710 |
| | | | | 100% Modulus | 20 | 30 | 30 | 20 | -- | 20 | 20 |
| C | Silicone | SC | 67 | Tensile strength | 340 | 410 | 490 | 370 | -- | 490 | 360 |
| | | | | Elongation | 290 | 320 | 380 | 360 | -- | 370 | 280 |
| | | | | 100% Modulus | 130 | 140 | 140 | 120 | -- | 150 | 140 |

TABLE 11 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Days Aged | | | | | | |
|------|-------------------|-------|------------------|------------------|-----------|------|------|------|------|-------|------|
| | | | | | 0 | 4 | 8 | 16 | 32 | 64 | 128 |
| G | Silicone | PS | 67 | Tensile strength | 880* | 920* | 930* | 950* | -- | 1130* | 1200 |
| | | | | Elongation | 750* | 730* | 750* | 810* | -- | 720* | 620 |
| | | | | 100% Modulus | 120 | 130 | 130 | 120 | -- | 180 | 200 |
| I | Silicone | PS | 67 | Tensile strength | 1280 | 1280 | 1260 | 1260 | -- | 1420 | 1130 |
| | | | | Elongation | 700 | 650 | 650 | 640 | -- | 480 | 390 |
| | | | | 100% Modulus | 220 | 240 | 230 | 230 | -- | 370 | 360 |
| J | Ethylene/ acrylic | PS | 67 | Tensile strength | 2080 | 1920 | 2010 | 2000 | 2040 | 2000 | 1960 |
| | | | | Elongation | 420 | 420 | 430 | 410 | 420 | 400 | 320 |
| | | | | 100% Modulus | 380 | 380 | 370 | 390 | 420 | 470 | 550 |
| K | Acrylic | PS | 67 | Tensile strength | 1570 | 1320 | 1280 | 1310 | 1360 | 1370 | 1360 |
| | | | | Elongation | 170 | 180 | 180 | 180 | 170 | 180 | 160 |
| | | | | 100% Modulus | 510 | 500 | 520 | 530 | 580 | 640 | 710 |
| L | Fluorocarbon | PS | 67 | Tensile strength | 1690 | 1720 | 1710 | 1750 | -- | 1800 | 1480 |
| | | | | Elongation | 220 | 210 | 230 | 250 | -- | 240 | 230 |
| | | | | 100% Modulus | 650 | 700 | 660 | 640 | -- | 780 | 660 |
| N | EPDM | PS | 67 | Tensile strength | 2010 | 2050 | 2250 | 2040 | -- | 2510 | 2010 |
| | | | | Elongation | 180 | 170 | 190 | 190 | -- | 200 | 170 |
| | | | | 100% Modulus | 800 | 870 | 880 | 810 | -- | 1020 | 940 |
| P | Butyl | PS | 67 | Tensile strength | 1600 | 1640 | 1520 | 1530 | -- | 1830 | 1460 |
| | | | | Elongation | 600 | 620 | 580 | 630 | -- | 590 | 550 |
| | | | | 100% Modulus | 130 | 120 | 120 | 120 | -- | 160 | 130 |
| Q | Silicone | PS | 67 | Tensile strength | 1060 | 1020 | 1070 | 1000 | -- | 1110 | 1110 |
| | | | | Elongation | 280 | 230 | 260 | 250 | -- | 220 | 200 |
| | | | | 100% Modulus | 460 | 510 | 490 | 480 | -- | 560 | 630 |

TABLE 12

PERCENT RETENTION OF TENSILE PROPERTIES OF HYDROLYTICALLY AGED MATERIALS

| Code | Material Type | Class | Aging Temp. (°C) | Property | Percent Retention | | | | | |
|------|------------------|-------|------------------|------------------|-------------------|-----|-----|---------------------------------|-----|-----|
| | | | | | Days Aged | | | | | |
| | | | | | 1 | 2 | 4 | 8 | 16 | 32 |
| A | Silicone | SC | 125 | Tensile strength | 94 | 94 | 81 | 50 | 13 | |
| | | | | Elongation | 119 | 129 | 152 | 210 | 71 | |
| | | | | 100% Modulus | 90 | 80 | 60 | 40 | 20 | |
| B | Silicone | SC | 125 | Tensile strength | NC | NC | NC | Specimens too tacky for testing | | |
| | | | | Elongation | NC | NC | NC | | | |
| | | | | 100% Modulus | 150 | 150 | 50 | | | |
| C | Silicone | SC | 125 | Tensile strength | 124 | 115 | 97 | 91 | 65 | 44 |
| | | | | Elongation | 128 | 128 | 128 | 124 | 155 | 152 |
| | | | | 100% Modulus | 100 | 92 | 62 | 85 | 46 | 38 |
| G | Silicone | PS | 125 | Tensile strength | NC | NC | NC | NC | NC | NC |
| | | | | Elongation | NC | NC | NC | NC | NC | NC |
| | | | | 100% Modulus | 125 | 117 | 125 | 158 | 142 | 150 |
| I | Silicone | PS | 125 | Tensile strength | 90 | 98 | 100 | 112 | 73 | 48 |
| | | | | Elongation | 105 | 127 | 132 | 186 | 168 | 186 |
| | | | | 100% Modulus | 105 | 127 | 132 | 186 | 168 | 186 |
| J | Ethylene/acrylic | PS | 125 | Tensile strength | 114 | 113 | 95 | 120 | 138 | 55 |
| | | | | Elongation | 112 | 117 | 79 | 57 | 29 | ~0 |
| | | | | 100% Modulus | 87 | 87 | 113 | 224 | 126 | ND |

NC = Not calculated

ND = Not determined (modulus values at 100% elongation cannot be determined when ultimate elongation < 100%).

Retention values are not calculated when both original and aged specimens were off-scale. When aged results are on-scale and zero time results are off-scale, the retentions are calculated on a less than (<) basis since the true value at zero time is greater than the recorded value.

TABLE 12 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Percent Retention | | | | | | | |
|------|----------------------|-------|------------------|------------------|-------------------|-----|-----|-----|-----|-----|-------|------|
| | | | | | Days Aged | | | | | | | |
| | | | | | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 128 |
| L | Fluorocarbon | PS | 125 | Tensile strength | 95 | 92 | 81 | 105 | 83 | 76 | | |
| | | | | Elongation | 95 | 114 | 100 | 105 | 114 | 95 | | |
| | | | | 100% Modulus | 106 | 98 | 97 | 120 | 95 | 102 | | |
| N | EPDM | PS | 125 | Tensile strength | 100 | 101 | 98 | 114 | 99 | 95 | | |
| | | | | Elongation | 94 | 94 | 100 | 94 | 111 | 100 | | |
| | | | | 100% Modulus | 110 | 105 | 99 | 121 | 85 | 96 | | |
| P | Butyl | PS | 125 | Tensile strength | 95 | 98 | 102 | 126 | 89 | 78 | | |
| | | | | Elongation | 102 | 92 | 100 | 95 | 90 | 78 | | |
| | | | | 100% Modulus | 92 | 108 | 85 | 146 | 100 | 100 | | |
| Q | Silicone | PS | 125 | Tensile strength | 99 | 94 | 94 | 95 | 71 | 67 | | |
| | | | | Elongation | 89 | 89 | 86 | 68 | 68 | 64 | | |
| | | | | 100% Modulus | 109 | 102 | 100 | 124 | 100 | 98 | | |
| A | Silicone | SC | 100 | Tensile strength | -- | 144 | 106 | 138 | 113 | 106 | 94 | |
| | | | | Elongation | -- | 114 | 129 | 195 | 171 | 150 | 143 | |
| | | | | 100% Modulus | -- | 120 | 80 | 90 | 70 | 70 | 70 | |
| B | Silicone | SC | 100 | Tensile strength | -- | NC | NC | NC | -- | NC | < 50 | |
| | | | | Elongation | -- | NC | NC | NC | -- | NC | < 84 | |
| | | | | 100% Modulus | -- | 100 | 100 | 100 | 100 | 50 | 100 | |
| C | Silicone | SC | 100 | Tensile strength | -- | 153 | 121 | 115 | 109 | 109 | 100 | |
| | | | | Elongation | -- | 121 | 121 | 124 | 110 | 134 | 121 | |
| | | | | 100% Modulus | -- | 115 | 92 | 85 | 85 | 77 | 85 | |
| G | Silicone | PS | 100 | Tensile strength | -- | NC | NC | NC | NC | -- | < 107 | < 80 |
| | | | | Elongation | -- | NC | NC | NC | NC | -- | < 71 | < 52 |
| | | | | 100% Modulus | -- | 167 | 108 | 125 | 125 | -- | 175 | 192 |
| I | Silicone | PS | 100 | Tensile strength | -- | 124 | 96 | 102 | 95 | -- | 63 | 40 |
| | | | | Elongation | -- | 86 | 86 | 83 | 73 | -- | 31 | 13 |
| | | | | 100% Modulus | -- | 145 | 109 | 136 | 136 | -- | 214 | ND |
| J | Ethylene/ acrylic | PS | 100 | Tensile strength | -- | 95 | 94 | 97 | 94 | 106 | 112 | |
| | | | | Elongation | -- | 107 | 102 | 100 | 93 | 90 | 69 | |
| | | | | 100% Modulus | -- | 105 | 105 | 113 | 118 | 139 | 208 | |

TABLE 12 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Percent Retention | | | | | | |
|------|---------------|--------------|------------------|----------|-------------------|-----|-----|-----|-----|-----|-------|
| | | | | | Days Aged | | | | | | |
| | | | | | 2 | 4 | 8 | 16 | 32 | 64 | 128 |
| 116 | K | Acrylic | PS | 100 | Tensile strength | 87 | 83 | 83 | 82 | 92 | 113 |
| | | | | | Elongation | 112 | 112 | 106 | 100 | 94 | 76 |
| | | | | | 100% Modulus | 114 | 98 | 106 | 116 | 135 | 265 |
| | L | Fluorocarbon | PS | 100 | Tensile strength | 118 | 95 | 96 | 84 | -- | 88 |
| | | | | | Elongation | 95 | 105 | 100 | 114 | -- | 95 |
| | | | | | 100% Modulus | 129 | 103 | 108 | 100 | -- | 114 |
| | N | EPDM | PS | 100 | Tensile strength | 127 | 106 | 106 | 92 | -- | 100 |
| | | | | | Elongation | 100 | 100 | 100 | 94 | -- | 94 |
| | | | | | 100% Modulus | 140 | 109 | 111 | 113 | -- | 113 |
| | P | Butyl | PS | 100 | Tensile strength | 121 | 95 | 104 | 99 | -- | 90 |
| | | | | | Elongation | 93 | 100 | 100 | 95 | -- | 82 |
| | | | | | 100% Modulus | 123 | 77 | 85 | 92 | -- | 108 |
| 116 | Q | Silicone | PS | 100 | Tensile strength | 123 | 94 | 95 | 92 | -- | 92 |
| | | | | | Elongation | 86 | 82 | 82 | 82 | -- | 68 |
| | | | | | 100% Modulus | 135 | 104 | 109 | 113 | -- | 122 |
| | A | Silicone | SC | 83 | Tensile strength | -- | 100 | 125 | 119 | -- | 113 |
| | | | | | Elongation | -- | 95 | 133 | 138 | -- | 148 |
| | | | | | 100% Modulus | -- | 90 | 100 | 90 | -- | 90 |
| | B | Silicone | SC | 83 | Tensile strength | -- | NC | NC | NC | NC | < 73 |
| | | | | | Elongation | -- | NC | NC | NC | NC | < 96 |
| | | | | | 100% Modulus | -- | 100 | 150 | 100 | 100 | 100 |
| | C | Silicone | SC | 83 | Tensile strength | -- | 132 | 115 | 135 | -- | 112 |
| | | | | | Elongation | -- | 121 | 121 | 131 | -- | 117 |
| | | | | | 100% Modulus | -- | 100 | 92 | 92 | -- | 92 |
| 116 | G | Silicone | PS | 83 | Tensile strength | -- | NC | NC | NC | -- | < 123 |
| | | | | | Elongation | -- | NC | NC | NC | -- | < 88 |
| | | | | | 100% Modulus | -- | 100 | 108 | 100 | -- | 142 |

TABLE 12 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Percent Retention | | | | | |
|------|------------------|-------|------------------|------------------|-------------------|-----|-----|-----|-------|-------|
| | | | | | Days Aged | | | | | |
| | | | | | 4 | 8 | 16 | 32 | 64 | 128 |
| I | Silicone | PS | 83 | Tensile strength | 98 | 96 | 95 | -- | 96 | 73 |
| | | | | Elongation | 94 | 87 | 86 | -- | 66 | 46 |
| | | | | 100% Modulus | 105 | 118 | 109 | -- | 155 | 191 |
| J | Ethylene/acrylic | PS | 83 | Tensile strength | 91 | 90 | 90 | 99 | 109 | 99 |
| | | | | Elongation | 98 | 90 | 90 | 88 | 88 | 60 |
| | | | | 100% Modulus | 105 | 134 | 116 | 126 | 163 | 205 |
| K | Acrylic | PS | 83 | Tensile strength | 82 | 83 | 82 | 86 | 88 | 81 |
| | | | | Elongation | 112 | 106 | 106 | 100 | 100 | 71 |
| | | | | 100% Modulus | 94 | 133 | 104 | 118 | 139 | 220 |
| L | Fluorocarbon | PS | 83 | Tensile strength | 99 | 104 | 94 | -- | 86 | 78 |
| | | | | Elongation | 95 | 105 | 109 | -- | 109 | 95 |
| | | | | 100% Modulus | 106 | 105 | 94 | -- | 100 | 103 |
| N | EPDM | PS | 83 | Tensile strength | 107 | 100 | 102 | -- | 101 | 109 |
| | | | | Elongation | 100 | 94 | 94 | -- | 100 | 106 |
| | | | | 100% Modulus | 113 | 108 | 105 | -- | 110 | 118 |
| P | Butyl | PS | 83 | Tensile strength | 101 | 99 | 96 | -- | 86 | 75 |
| | | | | Elongation | 102 | 103 | 100 | -- | 87 | 82 |
| | | | | 100% Modulus | 92 | 92 | 92 | -- | 108 | 108 |
| Q | Silicone | PS | 83 | Tensile strength | 97 | 96 | 97 | -- | 99 | 94 |
| | | | | Elongation | 89 | 82 | 86 | -- | 71 | 61 |
| | | | | 100% Modulus | 104 | 109 | 109 | -- | 126 | 137 |
| A | Silicone | SC | 67 | Tensile strength | 144 | 125 | 119 | -- | 210 | 160 |
| | | | | Elongation | 138 | 119 | 143 | -- | 133 | 110 |
| | | | | 100% Modulus | 100 | 110 | 90 | -- | 110 | 100 |
| B | Silicone | SC | 67 | Tensile strength | NC | NC | NC | -- | < 100 | < 117 |
| | | | | Elongation | NC | NC | NC | -- | < 96 | < 86 |
| | | | | 100% Modulus | 150 | 150 | 100 | -- | 100 | 100 |

TABLE 12 (Continued)

| Code | Material Type | Class | Aging Temp. (°C) | Property | Percent Retention | | | | | |
|------|----------------------|-------|------------------|------------------|-------------------|-----|-----|-----|-----|-------|
| | | | | | Days Aged | | | | | |
| | | | | | 4 | 8 | 16 | 32 | 64 | 128 |
| C | Silicone | SC | 67 | Tensile strength | 121 | 144 | 109 | | 144 | 106 |
| | | | | Elongation | 110 | 131 | 125 | | 128 | 97 |
| | | | | 100% Modulus | 108 | 108 | 92 | | 115 | 108 |
| G | Silicone | PS | 67 | Tensile strength | NC | NC | NC | -- | NC | < 136 |
| | | | | Elongation | NC | NC | NC | -- | NC | < 83 |
| | | | | 100% Modulus | 108 | 108 | 100 | -- | 150 | 167 |
| I | Silicone | PS | 67 | Tensile strength | 100 | 98 | 98 | -- | 111 | 88 |
| | | | | Elongation | 93 | 93 | 92 | -- | 69 | 56 |
| | | | | 100% Modulus | 109 | 105 | 105 | -- | 168 | 164 |
| J | Ethylene/ acrylic | PS | 67 | Tensile strength | 92 | 97 | 96 | 98 | 96 | 94 |
| | | | | Elongation | 100 | 102 | 98 | 100 | 95 | 76 |
| | | | | 100% Modulus | 100 | 97 | 103 | 111 | 124 | 145 |
| K | Acrylic | PS | 67 | Tensile strength | 84 | 82 | 83 | 87 | 87 | 87 |
| | | | | Elongation | 106 | 106 | 106 | 100 | 106 | 94 |
| | | | | 100% Modulus | 98 | 102 | 104 | 114 | 125 | 139 |
| L | Fluorocarbon | PS | 67 | Tensile strength | 102 | 101 | 104 | -- | 107 | 88 |
| | | | | Elongation | 95 | 105 | 114 | -- | 109 | 105 |
| | | | | 100% Modulus | 108 | 102 | 98 | -- | 120 | 102 |
| N | EPDM | PS | 67 | Tensile strength | 103 | 112 | 101 | -- | 125 | 100 |
| | | | | Elongation | 94 | 106 | 106 | -- | 111 | 94 |
| | | | | 100% Modulus | 109 | 110 | 101 | -- | 128 | 118 |
| P | Butyl | PS | 67 | Tensile strength | 103 | 95 | 96 | -- | 114 | 91 |
| | | | | Elongation | 103 | 97 | 105 | -- | 98 | 92 |
| | | | | 100% Modulus | 92 | 92 | 92 | -- | 123 | 100 |
| Q | Silicone | PS | 67 | Tensile strength | 96 | 101 | 94 | -- | 105 | 105 |
| | | | | Elongation | 82 | 93 | 89 | -- | 79 | 71 |
| | | | | 100% Modulus | 111 | 107 | 104 | -- | 122 | 137 |

TABLE 13A

EFFECT OF HYDROLYTIC AGING ON ADHESION OF CAULKING COMPOUNDS

| Code | Material Type | Class | Temp. (°C) | Peel Resistance (lb) - Principal Mode of Failure* | | | | | | | | |
|------|---------------|-------|------------|---|-------------------------|--------|--------|-------------------------|--------|--------|--------|--------|
| | | | | Aging Time in Water (Days) | | | | | | | | |
| | | | | 0 | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 128 |
| A | Silicone | SC | 125 | 4.3 C | 0.7 A | 0.8 A | 1.0 A | Specimens disintegrated | | | | |
| B | Silicone | SC | 125 | 9.4 C | 11.0 C | 9.4 C | 5.8 C | -- | -- | 0.2 C | 0.1 AC | |
| C | Silicone | SC | 125 | 15.0 C | 9.5 A | 6.8 AC | 1.1 A | 0.8 A | 0.2 AC | 0.3 AC | 0.2 AC | |
| D | Acrylic | SC | 125 | 11.5 C | Specimens disintegrated | | | | | | | |
| E | Hypalon | SC | 125 | 1.0 CA | 0.8 A | 1.6 AC | 3.7 A | -- | -- | -- | -- | |
| F | Butyl | SC | 125 | 1.9 C | 4.5 C | 1.0 C | 1.3 C | 1.1 AC | 1.0 AC | 0.3 AC | 0.3 C | |
| A | Silicone | SC | 100 | 4.3 C | -- | 2.1 AC | 1.3 AC | 0.7 A | 0.1 AC | 0.1 C | 0.1 AC | |
| B | Silicone | SC | 100 | 9.4 C | -- | 12.0 C | 8.4 C | 8.9 C | 1.8 A | 0.7 A | 0.8 AC | |
| C | Silicone | SC | 100 | 15.0 C | -- | 8.4 C | 9.0 C | 6.2 C | 1.9 A | 1.0 A | 0.6 AC | |
| D | Acrylic | SC | 100 | 11.5 C | Specimens disintegrated | | | | | | | |
| E | Hypalon | SC | 100 | 1.0 CA | 2.1 AC | 2.1 AC | 2.7 AC | 2.1 A | 2.0 A | 1.7 A | 0.5 A | |
| F | Butyl | SC | 100 | 1.9 C | -- | 0.5 C | 2.7 C | 2.0 C | 1.8 AC | 1.3 C | 2.2 AC | |
| A | Silicone | SC | 83 | 4.3 C | -- | -- | 4.7 AC | 2.8 AC | 2.3 AC | 1.6 AC | 1.0 A | 0.4 A |
| B | Silicone | SC | 83 | 9.4 C | -- | -- | 13.0 C | 9.8 C | 11.3 C | 9.8 AC | 9.8 AC | 4.3 AC |
| C | Silicone | SC | 83 | 15.0 C | -- | -- | 9.4 C | 6.9 C | 6.0 C | 3.5 C | 4.8 A | 2.0 A |
| D | Acrylic | SC | 83 | 11.5 C | -- | -- | 3.0 A | 5.3 A | -- | 4.0 AC | 6.9 AC | -- |
| E | Hypalon | SC | 83 | 1.0 AC | -- | -- | 2.3 A | 3.1 AC | 2.1 AC | 1.1 A | 2.3 A | 2.2 AC |
| F | Butyl | SC | 83 | 1.9 C | -- | -- | 2.4 C | 2.1 C | 1.3 A | 0.8 AC | 1.7 C | 1.5 AC |
| A | Silicone | SC | 67 | 4.3 C | -- | -- | 5.0 C | 3.6 AC | 2.3 AC | 3.2 AC | 2.6 AC | 1.4 A |
| B | Silicone | SC | 67 | 9.4 C | -- | -- | 12.8 C | 9.0 C | 9.8 AC | 11.0 C | 9.5 C | 9.3 C |
| C | Silicone | SC | 67 | 15.0 C | -- | -- | 10.2 C | 10.0 C | 9.6 C | 7.8 C | 3.0 AC | 2.5 C |
| D | Acrylic | SC | 67 | 11.5 C | 11.3 C | 11.9 C | 10.2 C | 11.3 C | -- | 4.0 C | 2.3 A | |
| E | Hypalon | SC | 67 | 1.0 AC | -- | -- | 3.5 C | 3.1 AC | 0.9 A | 2.2 AC | 1.9 AC | 2.1 AC |
| F | Butyl | SC | 67 | 1.9 C | -- | -- | 2.3 C | 2.2 AC | -- | 1.1 AC | 1.9 AC | 0.5 AC |

* Specimens were adhered to glass panels.

Modes of failure listed are based upon qualitative subjective judgments. C represents primarily cohesive failure; A refers to a primarily adhesive failure; AC indicates that both failure mechanisms were significant. Values are the average of the two highest of four specimens. Original values prior to exposure to water are listed as 0 days. The same set of data for original values was used for the four immersion temperatures.

TABLE 13B.

EFFECT OF HYDROLYTIC AGING ON ADHESION OF
CAULKING COMPOUNDS TO METAL PANELS.

| <u>Code</u> | <u>Material Type</u> | <u>Class</u> | <u>Adhesive Strength to Galvanized Steel</u> | <u>Adhesive Strength to Aluminum</u> | <u>Cohesive Strength</u> |
|-------------|--------------------------|--------------|--|--|------------------------------|
| A | Silicone | SC | Fair | Poor | Poor |
| B | Silicone | SC | Good | Excellent | Fair |
| C | Silicone | SC | Very Poor | Good | Poor |
| E | Hypalon | SC | Very Poor | Poor | Poor |
| F | Butyl | SC | Excellent | Excellent | Fair |

Qualitative evaluations were performed on composites that were immersed in water at 100°C for 109 days. (Acrylic caulk, Code D, could not be tested since it undergoes rapid degradation in 100°C water.)

TABLE 14

SIMULATED WEATHERING^{a,b}

| Code | Material Type | Class | Tensile Strength (lb/in ²) | | | Ultimate Elongation (%) | | | Modulus (lb/in ²) at 100% | | |
|------|----------------------|-------|--|-------|-------------------------|-------------------------|-------|-------------------------|---------------------------------------|-------|-------------------------|
| | | | 0 | 250 h | % Retention After 250 h | 0 | 250 h | % Retention After 250 h | 0 | 250 h | % Retention After 250 h |
| A | Silicone | SC | 230 | 160 | 70 | 290 | 230 | 79 | 100 | 100 | 100 |
| B | Silicone | SC | 60* | 90 | -- | 820* | 520 | | 20 | 30 | 150 |
| C | Silicone | SC | 350 | 440 | 126 | 320 | 360 | 113 | 130 | 130 | 100 |
| D | Acrylic | SC | 80 | 180 | 225 | 260 | 0 | 0 | -- | -- | -- |
| G | Silicone | PS | 910* | 880* | -- | 710* | 760* | -- | 120 | 120 | 100 |
| I | Silicone | PS | 1240 | 1290 | 104 | 650 | 720 | 111 | 220 | 220 | 100 |
| J | Ethylene/ acrylic | PS | 2030 | 2250 | 111 | 420 | 500 | 119 | 380 | 300 | 79 |
| K | Acrylic | PS | 1210 | 1300 | 107 | 160 | 180 | 113 | 510 | 530 | 104 |
| L | Fluorocarbon | PS | 1700 | 1620 | 95 | 210 | 220 | 105 | 650 | 650 | 100 |
| N | EPDM | PS | 1950 | 2060 | 106 | 170 | 170 | 100 | 800 | 860 | 108 |
| P | Butyl | PS | 1650 | 1560 | 95 | 560 | 600 | 107 | 130 | 130 | 100 |
| Q | Silicone | PS | 1110 | 1070 | 96 | 270 | 270 | 100 | 460 | 460 | 100 |

^a Tests were performed in an Atlas Electric Device Weather-O-Meter Model XW-WR.

^b Caulking compounds were permitted to age 4-6 weeks at room temperature prior to testing.

* Specimens did not break - elongation exceeded capability of testing machine; true values are greater than those indicated.

TABLE 15
FUNGUS RESISTANCE

Rating System

| <u>Observed Growth on Specimens</u> | <u>Rating</u> |
|---|---------------|
| None | 0 |
| Traces of growth (less than 10 percent) | 1 |
| Light growth (10-30 percent) | 2 |
| Medium growth (30-60 percent) | 3 |
| Heavy growth (60 complete) | 4 |

30 Day Report on Fungus Resistance

| | |
|----------------------------|--|
| <u>SE-7550</u> | No growth or damage noted. This sample is acceptable for fungus resistance. RATING = 0 |
| <u>Tremco Butyl</u> | No edge or surface growth, no damage noted. RATING = 0 |
| <u>Dow Corning 732</u> | Slight surface growth, no damage. This sample should be considered <u>NOT</u> acceptable for fungus resistance. RATING = 1 |
| <u>RTV-103</u> | No growth or damage noted. This sample is acceptable for fungus resistance. RATING = 0 |
| <u>Dow Corning 790</u> | Growth on the surface of sample. This is <u>NOT</u> acceptable for fungus resistance. RATING = 2 |
| <u>31-323-0731A</u> | Some edge growth, no damage. This sample should be considered <u>NOT</u> acceptable for fungus resistance. RATING = 2 |
| <u>NPC 80/40</u> | No surface growth, no damage. This sample is acceptable for fungus resistance. RATING = 0 |
| <u>3300-12</u> | Some edge growth on sample, no damage noted. This is <u>NOT</u> acceptable for fungus resistance. RATING = 2 |
| <u>HS-70</u> | Some edge growth, no damage noted. This sample should be considered <u>NOT</u> acceptable for fungus resistance. RATING = 2 |

TABLE 15 (Continued)

| | |
|------------------------|--|
| <u>210-108-35-1</u> | Moderate surface growth, no damage. This should <u>NOT</u> be acceptable for fungus resistance. RATING = 2 |
| <u>3300-11</u> | No surface growth or damage noted. This sample is acceptable for fungus resistance. RATING = 0 |
| <u>8EX-123</u> | Heavy surface growth noted, no damage. This sample is <u>NOT</u> acceptable for fungus resistance. RATING = 4 |
| <u>G&H Hypalon</u> | No surface growth or damage noted. This sample is acceptable for fungus resistance. RATING = 0 |
| <u>Tremco Mono</u> | Slight surface growth, some bubbling of surface. This is <u>NOT</u> acceptable for fungus resistance. RATING = 2 |
| <u>G&H Butyl</u> | Excessive surface growth on sample. This sample is <u>NOT</u> acceptable for fungus resistance. RATING = 4 |

60 Day and Final Ranking Report

(The following passed fungus resistance.)

| | |
|------------------------|---|
| <u>Tremco Butyl</u> | No growth. RATING = 0 |
| <u>RTV-103</u> | No growth. RATING = 0 |
| <u>NPC 80/40</u> | No growth. RATING = 0 |
| <u>G&H Hypalon</u> | No growth. Some damage of the surface of test sample. RATING = 0 |

(The following are marginal limits of resistance.)

| | |
|----------------------------|--------------------------|
| <u>SE-7550</u> | Light growth. RATING = 2 |
| <u>Dow Corning 732</u> | Light growth. RATING = 2 |
| <u>HS-70</u> | Light growth. RATING = 2 |
| <u>210-108-35-1</u> | Light growth. RATING = 2 |
| <u>3300-11</u> | Light growth. RATING = 2 |

TABLE 15 (Continued)

(The following failed fungus resistance.)

| | |
|----------------------------------|---------------------------|
| <u>Dow Corning</u> <u>790</u> | Medium growth. RATING = 3 |
| <u>31-323-0731A</u> | Medium growth. RATING = 3 |
| <u>3300-12</u> | Medium growth. RATING = 3 |
| <u>Tremco Mono</u> | Medium growth. RATING = 3 |
| <u>8EX-123</u> | Heavy growth. RATING = 4 |
| <u>G&H Butyl</u> | Heavy growth. RATING = 4 |

* Tests were performed by Microbac Laboratories, Inc., 4580 McKnight Road, Pittsburgh, PA 15237 (Phone 412-931-5851) according to MIL-F-13927A, references Military Standard 810-C, ANSI/ASTM G-21-70. This table is a verbatim copy of the reports dated July 5, 1979, submitted to Westinghouse by Mr. Albert Metro of Microbac Laboratories.

TABLE 16
EXPOSURE TO FUNGI^a

| | Code | Material Type | Class | Days Aged | | | | | | | | | | | |
|-------------|----------------------|---------------|-------|-----------------------|-------|------|------------|------|-----|-----------------|-----|-------|--------------------|-------|-------|
| | | | | Tensile Strength | | | Ultimate | | | Modulus at | | | Hardness (Shore A) | | |
| | | | | (lb/in ²) | | | Elongation | | | 100% Elongation | | | | | |
| | | | | 0 | 30 | 60 | 0 | 30 | 60 | 0 | 30 | 60 | 0 | 30 | 60 |
| 125 | A | Silicone | SC | 230 | 210 | 190 | 290 | 230 | 290 | 110 | 120 | 90 | 32-34 | 34-34 | 32-34 |
| | % Retention | | | | 91 | 83 | | 79 | 100 | | 109 | 82 | | | |
| | B | Silicone | SC | 60 | 70 | 60 | 820 | 760 | 810 | 30 | 30 | 30 | 11-12 | 11-12 | 11-12 |
| | % Retention | | | | 117 | 100 | | 93 | 99 | | 100 | 100 | | | |
| | C | Silicone | SC | 350 | 380 | 320 | 320 | 300 | 310 | 120 | 150 | 110 | 32-33 | 32-33 | 32-33 |
| | % Retention | | | | 109 | 91 | | 94 | 97 | | 125 | 92 | | | |
| | E | Hypalon | SC | 20 | 80 | 40 | 0 | 110 | 150 | -- | 90 | 20 | 23-24 | 38-40 | 38-40 |
| | % Retention | | | | 400 | 200 | | -- | -- | | -- | -- | | | |
| | F | Butyl | SC | 290 | 190 | 170 | 0 | 0 | 0 | -- | -- | -- | 56-59 | 72-75 | 73-75 |
| | % Retention | | | | 66 | 59 | -- | -- | -- | -- | -- | -- | | | |
| G | Silicone | PS | 910* | 1060* | 1070* | 710* | 670* | 720* | 120 | 150 | 170 | 47-48 | 47-48 | 47-48 | |
| % Retention | | | | | | | | | | 125 | 142 | | | | |
| I | Silicone | PS | 1240 | 1550 | 1330 | 650 | 550 | 840 | 220 | 330 | 290 | 59-60 | 60-61 | 60-61 | |
| % Retention | | | | 125 | 107 | | 85 | 83 | | 150 | 132 | | | | |
| J | Ethylene/ acrylic | PS | 2120 | 2060 | 1940 | 350 | 350 | 370 | 480 | 470 | 440 | 70-72 | 70-72 | 70-71 | |
| % Retention | | | | 97 | 92 | | 100 | 106 | | 98 | 92 | | | | |
| K | Acrylic | PS | 1310 | 1290 | 1130 | 180 | 170 | 180 | 520 | 530 | 450 | 59-60 | 59-60 | 59-61 | |
| % Retention | | | | 98 | 86 | | 94 | 100 | | 102 | 87 | | | | |
| L | Fluorocarbon | PS | 1710 | -- | 1400 | 210 | -- | 210 | 720 | -- | 620 | 76-78 | -- | 76-78 | |
| % Retention | | | | -- | 82 | | -- | 100 | | -- | 86 | | | | |

^a Caulking compounds were permitted to age 4 to 6 weeks at room temperature prior to testing.

* Specimens did not break - ultimate elongation exceeded capability of testing machine; true values are greater than indicated.

TABLE 16 (Continued)

| | | | Days Aged | | | | | | | | | | | |
|-------------|---------------|-------|--|------|------|-------------------------|-----|-----|--|-----|-----|--------------------|-------|-------|
| Code | Material Type | Class | Tensile Strength (lb/in ²) | | | Ultimate Elongation (%) | | | Modulus at 100% Elongation (lb/in ²) | | | Hardness (Shore A) | | |
| | | | 0 | 30 | 60 | 0 | 30 | 60 | 0 | 30 | 60 | 0 | 30 | 60 |
| | | | | | | | | | | | | | | |
| N | EPDM | PS | 2170 | 1600 | 2020 | 180 | 160 | 190 | 910 | 900 | 780 | 76-77 | 76-77 | 76-77 |
| % Retention | | | | 74 | 93 | | 89 | 106 | | 99 | 86 | | | |
| P | Butyl | PS | 1650 | 1910 | 1610 | 560 | 600 | 580 | 140 | 150 | 140 | 51-53 | 51-53 | 51-53 |
| % Retention | | | | 116 | 98 | | 107 | 104 | | 107 | 100 | | | |
| Q | Silicone | PS | 1110 | 1100 | 1080 | 270 | 200 | 240 | 510 | 590 | 520 | 70-71 | 72-73 | 72-73 |
| % Retention | | | | 99 | 97 | | 74 | 89 | | 116 | 102 | | | |

OREC

TABLE 17

OZONE RESISTANCE MEASUREMENTS(Copy of Report by Ozone
Research & Equipment Corp.)**OZONE RESEARCH AND EQUIPMENT CORPORATION**

3840 NORTH 40th AVENUE • PHOENIX, ARIZONA 85019 • AREA 602 — 272-2681

OZONE TEST REPORTOrder No. 34-JP-96199ADate 6/23/79

Company

Date Samples Shipped 4/19/79Westinghouse Electric Corporation
R & D Center
1310 Beulah Rd.
Pittsburgh, PA 15235
Invoice No. C2456Date Samples Received 4/26/79Date Samples Returned 6/28/79

TYPE OF TEST

- ☒
- Accelerated Ozone Test Chamber
-
- ☐
- Outdoor Exposure

TEST CHAMBER MODEL

- ☐
- OREC 0300
-
- ☐
- OREC 0300A
-
- ☒
- OREC 0600

TEST PERIOD

Date _____ Hour _____
Began 6/15/79 Began 4:10PM
Date _____ Hour _____
Completed 6/22/79 Completed 2:10PM

TEST SPECIFICATION

- ☒
- ASTM D1149-55T (Mod)
- ☐
- Customer
-
- ☐
- ASTM D470-54T
- ☐
- Provided
-
- ☐
- ASTM D1373-55T
- ☐
- Other
-
- ☐
- IPCEA, Para 4.4.1.5

REMARKS, TEST SPECIFICATION: Observations once daily except weekends(visual). with 7X magnification at end of test.

TEST CONDITIONS (For Outdoor Exposure Test Conditions see Supplement A):

Ozone Concentration 100 pphm/vol Number of Samples 26Test Temperature 40°C Sample Description See attached sheetOther Time: 166 hoursOther _____ Sample Identification See attached sheet

Other _____

TYPE OF STRETCH APPARATUS OR TECHNIQUE

- ☐
- ASTM D518-44, Method A
-
- ☐
- ASTM D518-44, Method B
-
- ☐
- ASTM D470-54T, Para 40(a)
-
- ☐
- ASTM D1373-55T, Para 20(b)
-
- ☐
- OREC Dynamic Stretch Apparatus
-
- ☒
- Other ASTM D518- Procedure "C"

DESCRIPTION OREC DYNAMIC STRETCHING
APPARATUSExtent of Stretch _____
Rate of Stretch 15%
Type of Stretch Static

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TABLE 17 (Continued)

DESCRIPTION OF "OTHER" STRETCHING APPARATUS OR TECHNIQUE: _____

DESCRIPTION OF OZONE TEST CHAMBER,
OREC 0600.

Illustration as per attached
brochure.
Oven Dimensions: 24"x18"x18"
Automatic Temperature Control
Automatic Ozone Concentration
Control.
Air Flow: 1 chamber change/min.
Air Velocity: Laterally, in excess
of 2 ft/sec.
Ozone Generator: Ultra Violet
Quartz Lamp.

DESCRIPTION OF OZONE TEST CHAMBER,
OREC 0600A or 0600C.

Illustration as per attached
brochure.
Oven Dimensions: 24"x18"x18"
Automatic Temperature Control
Automatic Ozone Concentration
Control.
Air Flow: See Test Conditions above
Air Velocity: Laterally, in excess
of 2 ft/sec.
Ozone Generator: Silent Arc
Discharge Generator.

EVALUATIVE TECHNIQUE: Visual daily (except weekends) See attached sheets
for daily reporting. 7X magnification as final evaluation.

SAMPLE EVALUATION: See attached Page 7

TEST(S) CONDUCTED BY: David H. Cross

The above is a true and exact test report in
certification of which is affixed the Seal of
Ozone Research & Equipment Corporation.
(Including the attached 7 pages)

TABLE 17 (Continued)

Westinghouse Electric Corporation

P.O. 34-JP-96199A/ Ozone Testing

Sample Description:

1. Dow Corning -732
2. 8EX123
3. NPC 80140
4. HS70
5. 330-12A
6. 210-108=35-1
7. SE 7550
8. Tremco Butyl (Not tested - see explanation Page 2 attached)
9. RTV 103
10. 3300-11
11. 31-323-0731A
12. Dow Corning 790
13. G&H Hypalon

Samples (two each of above 13 compounds) are numerically identified with A & B suffix.

TABLE L7 (Continued)

Final Observation - June 22, 1979
7X Magnification

| Sample ID | Remarks |
|-----------|--|
| 1A | After 96 hours, sample broke at narrow end of mount. No evidence of ozone degradation on the one-end mounted sample. |
| 1B | No evidence of ozone degradation. |
| 2A | Severe edge and surface cracking, particularly at narrow end mount. |
| 2B | Severe edge and surface cracking over 80% of sample. |
| 3A | No evidence of ozone degradation. |
| 3B | No evidence of ozone degradation. |
| 4A | No evidence of ozone degradation. |
| 4B | No evidence of ozone degradation. |
| 5A | No evidence of ozone degradation. |
| 5B | No evidence of ozone degradation. |
| 6A | No evidence of ozone degradation. |
| 6B | No evidence of ozone degradation. |
| 7A | No evidence of ozone degradation. |
| 7B | No evidence of ozone degradation. |
| 8A | Not tested - Sample hard and stiff; broke in attempting to mount. |
| 8B | Not tested - Sample hard and stiff; broke in attempting to mount. |
| 9A | No evidence of ozone degradation. |
| 9B | No evidence of ozone degradation. |
| 10A | No evidence of ozone degradation. |
| 10B | No evidence of ozone degradation. |
| 11A | Broke at narrow end of mount within 24 hours. No evidence of ozone degradation on the one-end mounted sample. |
| 11B | Broke at narrow end of mount within 24 hours. No evidence of ozone degradation on the one-end mounted sample. |
| 11C | After 48 hours into the test, this sample was mounted and exposed for the duration of the test. There was no evidence of ozone degradation |
| 12A | No evidence of ozone degradation. Some relaxation. |
| 12B | No evidence of ozone degradation. Some relaxation. |
| 13A | No evidence of ozone degradation. Extremely relaxed. |
| 13B | No evidence of ozone degradation. Extremely relaxed. |

TABLE 18
EFFECT OF GLAZE DEPOSITS
ON RELATIVE LIGHT TRANSMITTANCE

| <u>Deposit</u> | <u>Amount Grams/Sq. Meter</u> | <u>Relative Light Transmittance % Reduction</u> |
|----------------------------|-----------------------------------|---|
| Water Leaching of Glass | | |
| Salts | 0.151 | 20 |
| Salts | 0.237 | 34 |
| Silicone Rubber Fragments | | |
| Oil | 0.4 | 1 |
| Powder | 0.129 | 18 |
| Butyl Rubber Fragments | | |
| Oxidized | 0.033 | 4 |
| Acrylate Rubber Fragments | | |
| Oxidized | ~ 0.020 | ~ 1 |
| Stearic Acid from Rubber | | |
| Liquid | 0.4 | ~ 1 |
| Solid | 0.4 | 5-6 |
| Processing Oil from Rubber | | |
| Liquid | 0.4 | ~ 1 |
| Oxidized | 0.2 | ~ 10 |

* Values obtained using the incandescent light transmittance measurement apparatus (Appendix II).

TABLE 19
OUTGASSING OF SILICONE SEALANTS

| SC Compounds | Percent (%) | | Infrared Spectrophotometric Analysis of Condensables | |
|-----------------|---------------|------------------|--|---|
| | Condensables* | Noncondensables* | Major Bands - Wave Number cm^{-1} | Interpretation |
| A | 2.7 | 0.4 | 800, 1000-1100, 1260, 1380, 1460, 2800-3000 | Low molecular weight cyclic or linear alkyl polysiloxane and a processing oil |
| B | 1.0 | 0.3 | 800, 1000-1100, 1260, 2800-3000 | |
| C | 1.6 | 2.4 | 800, 1000-1100, 1260, 2800-3000 | |
| U | 1.2 | 3.1 | 800, 1000-1100, 1260, 2800-3000 | |
| V | 1.6 | 6.2 | 800, 1000-1100, 1260, 2800-3000 | |
| W | 1.0 | 0.4 | 800, 1000-1100, 1260, 2800-3000 | |
| X | 1.4 | 0.3 | 800, 1000-1100, 1260, 2800-3000 | |
| Y | 2.6 | 0.8 | 800, 1000-1100, 1260, 2800-3000 | |
| Z | 0.9 | 0.9 | 800, 1000-1100, 1260, 2800-3000 | |
| PS | | | | |
| Compounds | | | | |
| G | ~ 0.01 | ~ 0.03 | 800, 1000-1100, 1260, 2800-3000 | Alkyl polysiloxane |
| H | 0.9 | 0.2 | 800, 1000-1100, 1260, 1490, 1590, 1720, 2800-3000 | Alkyl polysiloxane plus an aromatic ester |
| I | 0.3 | 0.2 | 800, 1000-1100, 1260, 2800-3000 | Alkyl polysiloxane |
| Q | ~ 0.01 | ~ 0.05 | 800, 1000-1100, 1260, 2800-3000 | Alkyl polysiloxane |
| AA | 0.3 | 0.2 | 800, 1000-1100, 1260, 2800-3000 | Alkyl polysiloxane |

* Test conditions were 9 days @ 150°C.

TABLE 20

OUTGASSING OF INTERMEDIATE TEMPERATURE SEALANTSInfrared Spectrophotometric Analysis of Condensables

| SC Compounds | Percent (%) | | Infrared Spectrophotometric Analysis of Condensables | |
|-----------------|---------------|------------------|--|---|
| | Condensables* | Noncondensables* | Major Bands - Wave Number cm ⁻¹ | Interpretation |
| D | 0.4 | 4.0 | 1160, 1220, 1380, 1460, 1720, 2800-3000 | Acrylate or oxidized oil |
| E | 13.0 | 15.4 | 1180, 1240, 1380, 1460, 1720, 2800-3000 | Alkyl sulfonic acid ester |
| F | 6.5 | 8.0 | 1160, 1220, 1380, 1460, 1700, 2800-3000 | Oxidized processing oil or butyl fragments |
| PS Compounds | | | | |
| J | 1.77 | 2.1 | 1380, 1420, 1710, 2800-3000 | Acrylate/ethylene fragments |
| K | 0.3 | 0.09 | Very weak bands | NI |
| L | ~ 0.01 | ~ 0.01 | Very weak bands | NI |
| M | ~ 0.01 | ~ 0.01 | Very weak bands | NI |
| O | 0.6 | 0.2 | 1380, 1460, 1710, 2800-3000 | Oxidized processing oil |
| P | 2.3 | 0.3 | 1300, 1460, 1700, 2800-3000 | Stearic acid |
| R | 1.9 | 1.0 | 1260, 1360, 1460, 1710, 2800-3000 | Oxidized processing oil or low molecular weight butyl fragments |

* Test conditions were 9 days @ 150°C

NI = Not interpreted, insufficient sample

TABLE 21

OUTGASSING OF ETHYLENE-PROPYLENE TERPOLYMER SEALANTS

| Compound | Percent (%) | | Infrared Spectrophotometric Analysis of Condensables | |
|----------|--------------|-----------------|--|--|
| | Condensables | Noncondensables | Major Bands - Wave Number cm^{-1} | Interpretation |
| N* | 0.5 | 0.3 | 1260, 1380, 1460, 1600, 2800-3000 | Processing oil (naphthenic) |
| S* | 3.2 | 0.6 | 1280, 1380, 1460, 1700, 2800-3000 | Oxidized paraffinic oil or oxidized EPDM fragments or both |
| T #1* | 4.4 | 0.3 | 1280, 1380, 1460, 1700, 2800-3000 | |
| T #1* | 3.1 | 0.5 | 1280, 1380, 1460, 1700, 2800-3000 | |
| T #2* | 0.8 | 0.3 | 1280, 1380, 1460, 1700, 2800-3000 | |
| T #2* | 0.8 | 0.5 | 1280, 1380, 1460, 1700, 2800-3000 | |
| S** | 4.3 | 0.6 | 1270, 1380, 1460, 1700, 2800-3000 | Oxidized paraffinic oil or oxidized EPDM fragments or both |
| T #1** | 5.7 | 0.3 | 1290, 1380, 1460, 1700, 2800-3000 | |
| T #1** | 4.8 | 0.55 | 1290, 1380, 1460, 1700, 2800-3000 | |
| T #2** | 0.9 | 0.3 | 1270, 1380, 1460, 1700, 2800-3000 | |
| T #2** | 1.0 | 0.5 | 1270, 1380, 1460, 1700, 2800-3000 | |

* Test conditions were 9 days @ 150°C.

** Test conditions were 16 days @ 150°C.

TABLE 22
SOLAR COLLECTORS

| <u>Manufacturer</u> | <u>Model #</u> | <u>Type</u> | <u>Back Insulation</u> | <u>Side Insulation</u> | <u>Glaze Seals</u> |
|--|-------------------------|--|--|---|--|
| Overly Greensburg, PA | -- | Vented, desiccant | Fiberglass; no lubricant or binder | -- | Silicone rubber seal |
| *Pittsburgh Plate Glass Pittsburgh, PA | A441 | Breathing, silica gel | Fiberglass | None | Butyl sealant (prim.) Twindow. (sec.) |
| | Types 1, 2 & 3 | Twindow, Hermetic breathing absorber plate, silica gel | Fiberglass | Formerly used 'Marinite'; not employed in later models | Butyl sealant (sec.) Butyl sealant (prim.) |
| Solar Dev. Inc. Riviera Beach, FL | SD-5 & SD-6 | -- | Thermax R=16 (Celotex) (Glass reinforced polyisocyanurate/Al) | Thermax R=8 (Celotex) (Gl. rein. polyisocyan./Al) | -- |
| Reynolds Aluminum Richmond, VA | Series 1400 and 1500 | Self-draining | Closed cell foam/Al facing | -- | -- |
| Solar Energy Prod., Inc. Gainesville, FL | CA Series SC Series | Seal out moisture | Glass reinforced polyisocyanurate/Al Unbnd. borosilicate glass on above | Gl. rein. polyisocyan./Al | Ethylene propylene diene monomer (EPDM) extruded channel |
| Libby Owens Ford Toledo, OH | Series 100 and 200 | Vented, press. equal. passage, no need for desiccants | Low binder fiber- glass | -- | Molded or extruded rubber seal |
| Sunburst Solar Energy, Inc. Menlo Park, CA | BG, BGC, BEAC, BEA | -- | Rigid urethane foam | -- | -- |

*Pittsburgh Plate Glass has recently discontinued manufacture of solar collectors.

TABLE 22
SOLAR COLLECTORS (Continued)

| Manufacturer | Model # | Type | Back Insulation | Side Insulation | Glaze Seals |
|---|-----------------------------------|---|--|---|---|
| Revere Rome, NY | Sun-Aid | -- | High temp. fiber glass | -- | -- |
| Lennox Ind. | LSC-18-1.5 LSC-18-1 | Weep holes for ventilation and moisture removal | Semi-rigid fiber glass board, silicone mount. pads | Fiberglass | PVC weather strip |
| Solarnetics El Cajon, CA | EC-2 | -- | Polyurethane | Polyurethane | -- |
| Cimarron Solar Ind. Okla City, OK | -- | Vented for moisture relief | Special fiberglass | Foamglas ^(R) | -- |
| Dixon Energy Systems Hadley, MA | Yankee | -- | Spinglas ^(R) | Insulating board | Silicone gaskets |
| Chamberlain Manufacturing Corp. Elmhurst, IN | 711101, 711201, 712101, 712201 | Passive drying system, regenerated, vented, silica gel | Fiberglass (high temp.) | Compressed high temp. fiberglass | EPDM rubber weather seal-EPDM foam |
| Solar Innovations Lakeland, FL | SC-107, SC-208 SC-300, SC-400 | -- | Polyurethane Dyplast urethane Type I | -- | -- |
| Daystar Corp. Burlington, MA | 21-C, 21-B | -- | High temp. isocyanurate foamed in place | High temp. isocyanurate foamed in place | PVC extrusion silicone sealant |
| Energy Systems Inc. San Diego, CA | 1111S 1111D | Desiccant absorbent 801 (Davidson Chem) hermetically sealed | Glass reinforced rigid Thermax foam/Al facing | -- | Rhodorsil 3B Novatherm 415 (hot butyl sealant) |
| Champion Dryden, MI | -- | Air collector | Urethane foam | -- | Rubber gasket |

TABLE 23

DESICCANTS

| Desiccant | Static (Equilibrium) Water Capacity @ 25°C | Drying Mechanism | How Regenerated | Change with Temp. | | |
|--|---|--|--|--|------------------|--------------|
| | | | | H ₂ O Partial Pressure, Torr | Temp. °F | % Water |
| Silica gel (SiO ₂) | 22% @ 40% RH 35% @ 60% RH 42% @ 100% RH | Capillary adsorption | Heat @ 200°F and higher | 20 20 20 | 79 120 140 | 16 9 4 |
| Activated alumina (Al ₂ O ₃) | 12% @ 40% RH 15% @ 60% RH 17% @ 80% RH | Capillary adsorption | Heat @ 350°F-600°F | | | |
| Alumina gel (Al ₂ O ₃ +1.0-1.5% SiO ₂) | 17% @ 40% RH 24% @ 60% RH 36% @ 80% RH | Capillary adsorption | Heat @ 350°F-600°F | | | |
| 137 Calcium sulfate (Drierite) (CaSO ₄) | 6.6% 5.4-7.4% Total 12-14% | CaSO ₄ ·1/2 H ₂ O Capillary adsorption | Heat @ 400°F-425°F Heat @ 250°F | | | |
| Magnesium perchlorate [Mg(ClO ₄) ₂] | 48.4% | Mg(ClO ₄) ₂ ·6H ₂ O Hydrate formation | Heat-vacuum First hydrate + @ 275°F Rest + @ 400°F-500°F | | | |
| Barium perchlorate [Ba(ClO ₄) ₂] | 16.1% | Ba(ClO ₄) ₂ ·3H ₂ O Hydrate formation | Heat @ 250°F and up | | | |
| Barium oxide (BaO) | Approaches 100% | Ba(OH) ₂ ·8H ₂ O Hydroxide formation | Chemically | | | |
| Calcium oxide (CaO) | Low capacity | CaCO ₃ ·7H ₂ O Hydroxide formation | Chemically | | | |
| Activated carbon (C) | 14-16% | Capillary adsorption | Heat | | | |

TABLE 23
DESICCANTS

(Continued)

| Desiccant | Static (Equilibrium) Water Capacity @ 25°C | Drying Mechanism | How Regenerated | Change with Temp. | | |
|---|---|----------------------|-------------------------|--|------------------|------------------|
| | | | | H ₂ O Partial Pressure, Torr | Temp. °F | % Water |
| Zeolite (Natural) $\text{CaAl}_2\text{Si}_4\text{O}_{12} \cdot 6\text{H}_2\text{O}$ | 60-70% | Capillary adsorption | Heat @ 300°F and higher | | | |
| Molecular sieve $\text{Na}_{12}[(\text{AlO}_2)_2(\text{SiO}_2)_2]_{12} \cdot 27\text{H}_2\text{O}$ | 21% @ 40% RH 22% @ 60% RH 23% @ 80% RH | Capillary adsorption | Heat @ 250°F-600°F | 20 20 20 | 77 120 140 | 22 21.5 21 |

TABLE 24
ADSORBENTS FOR ORGANICS

| <u>Adsorbent</u> | <u>Adsorption Mechanism</u> | <u>Relative Order of Adsorbability</u> | <u>How Regenerated</u> |
|-------------------|---|---|------------------------|
| Silica gel | Capillary adsorption | Water > alcohols > aromatics > di-olefins > olefins > paraffins (least readily adsorbed) | By desorbing agent |
| Activated alumina | Capillary adsorption | Water >> organics | Heat 350°F to 600°F |
| Alumina gel | Capillary adsorption | Water >> organics | Heat 350°F to 600°F |
| Activated carbon | Capillary adsorption + chemisorption | In general organics > water | High heat and steam |
| Zeolite (natural) | Capillary adsorption | Water >> organics | High heat |
| Molecular sieve | Capillary adsorption | Water >> organics Unsaturated hydro. > saturated hydro. | High heat |

TABLE 25

EFFECTIVENESS OF ADSORBENTS TOWARDS ORGANIC VAPORS

| <u>Adsorbent</u> | <u>Adsorbent Weight</u> | <u>Sealant</u> | <u>Sealant Weight</u> | <u>Total Volatiles, g (Wt Loss of Sealant)</u> | <u>Percent Volatiles Adsorbed</u> |
|--------------------|-------------------------|----------------|-----------------------|--|-----------------------------------|
| Activated Alumina* | 1.5056 | Compound P | 1.0152 | 0.029 | 72.85 |
| Activated Carbon** | 1.5067 | " | 1.0689 | 0.030 | 70.42 |
| Molecular Sieve*** | 1.4998 | " | 1.0419 | 0.031 | 75.0 |
| Activated Alumina* | 1.5128 | Compound C | 1.8522 | 0.053 | 84.0 |
| Activated Carbon** | 1.5013 | " | 1.1550 | 0.033 | 87.6 |
| Molecular Sieve*** | 1.6613 | " | 1.1902 | 0.034 | 86.1 |

| <u>Bulk Density lbs/cu ft</u> | <u>Surface Area sq meter/g</u> |
|-----------------------------------|------------------------------------|
|-----------------------------------|------------------------------------|

* Alcoa, Alumina F-1 (8-14 mesh)

52

210

** American Norit, Sorbonorit B-4 (6-14 mesh)

24-26

1050-1200

*** Davison, Type 13X (4-8 mesh)

38

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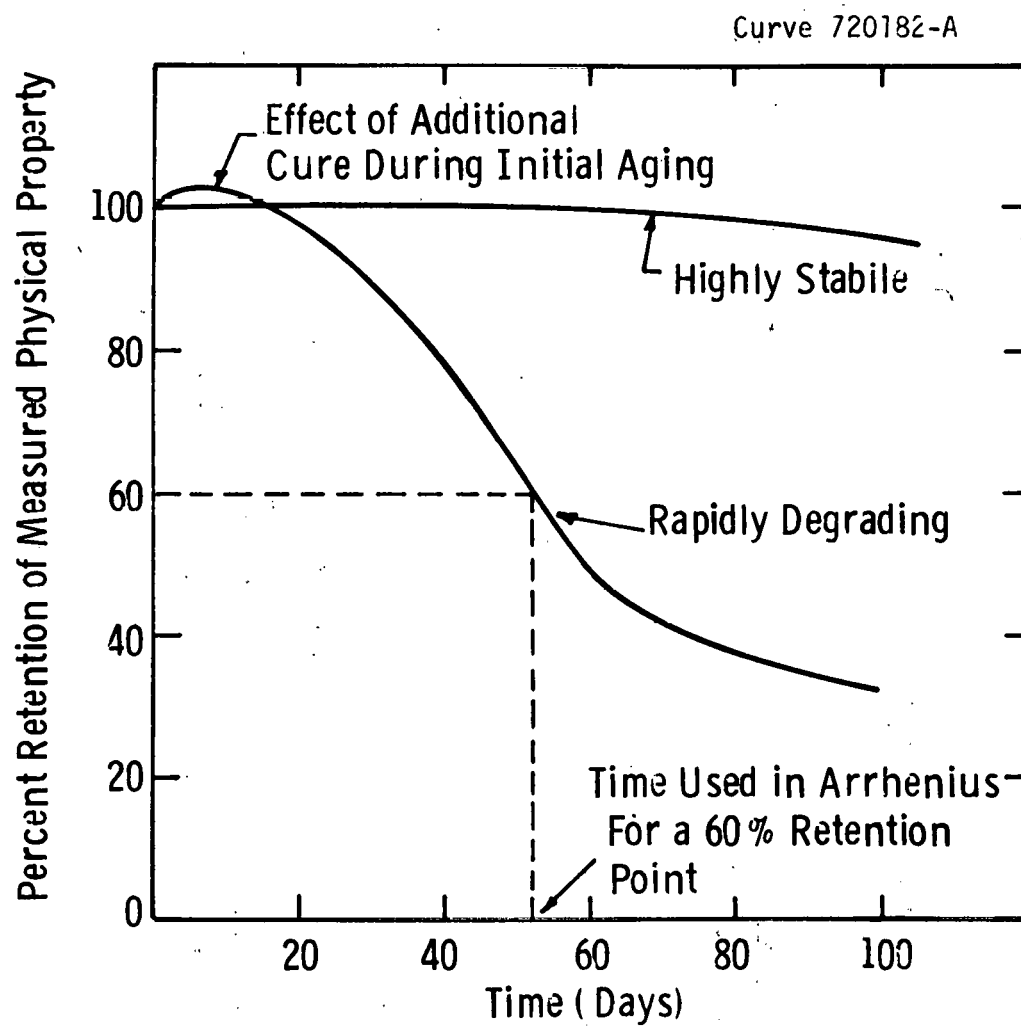


Fig. 1 — Typical effects of thermal aging on specimens

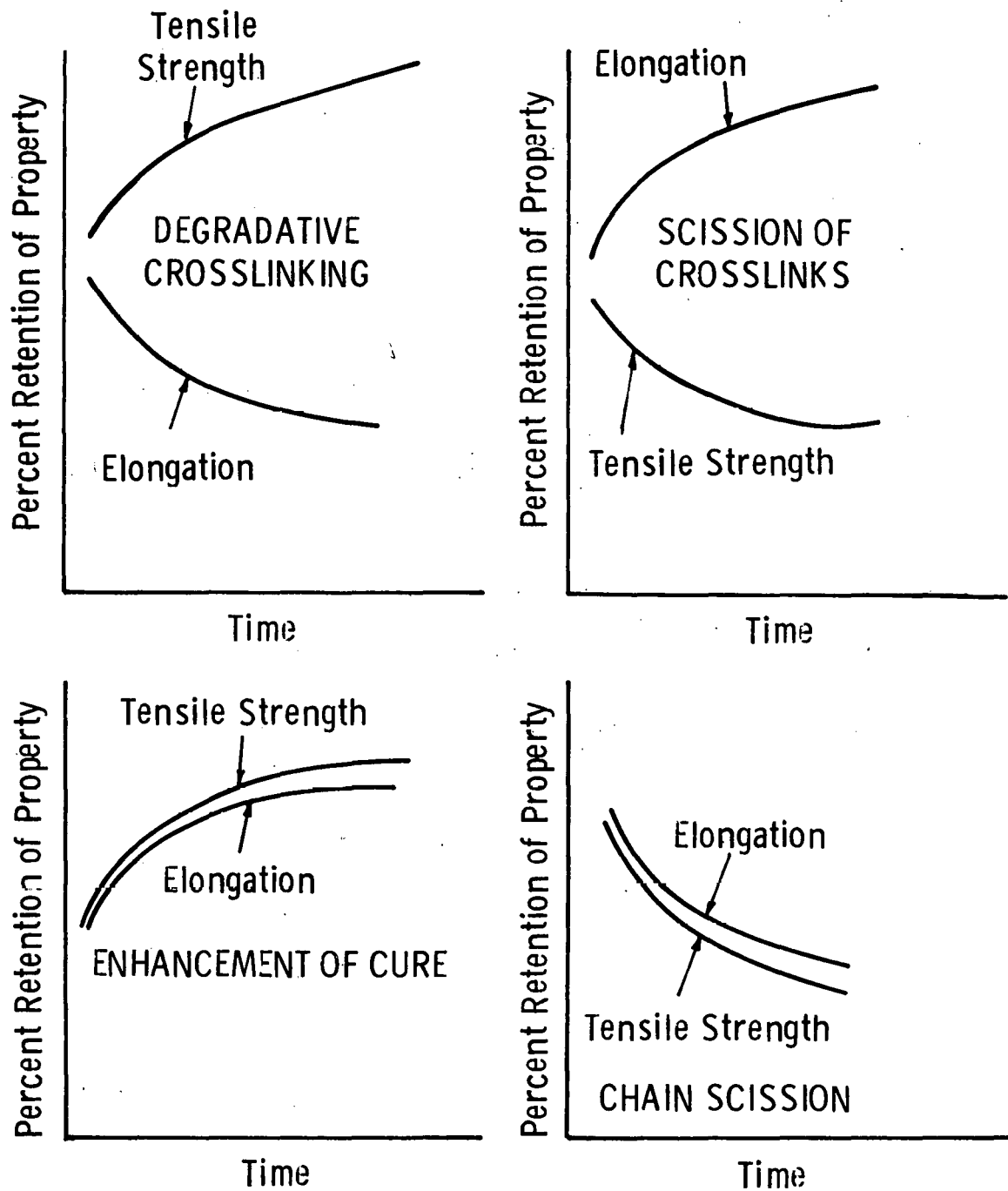


Fig. 2 — Typical effects of various types of degradation processes on physical properties

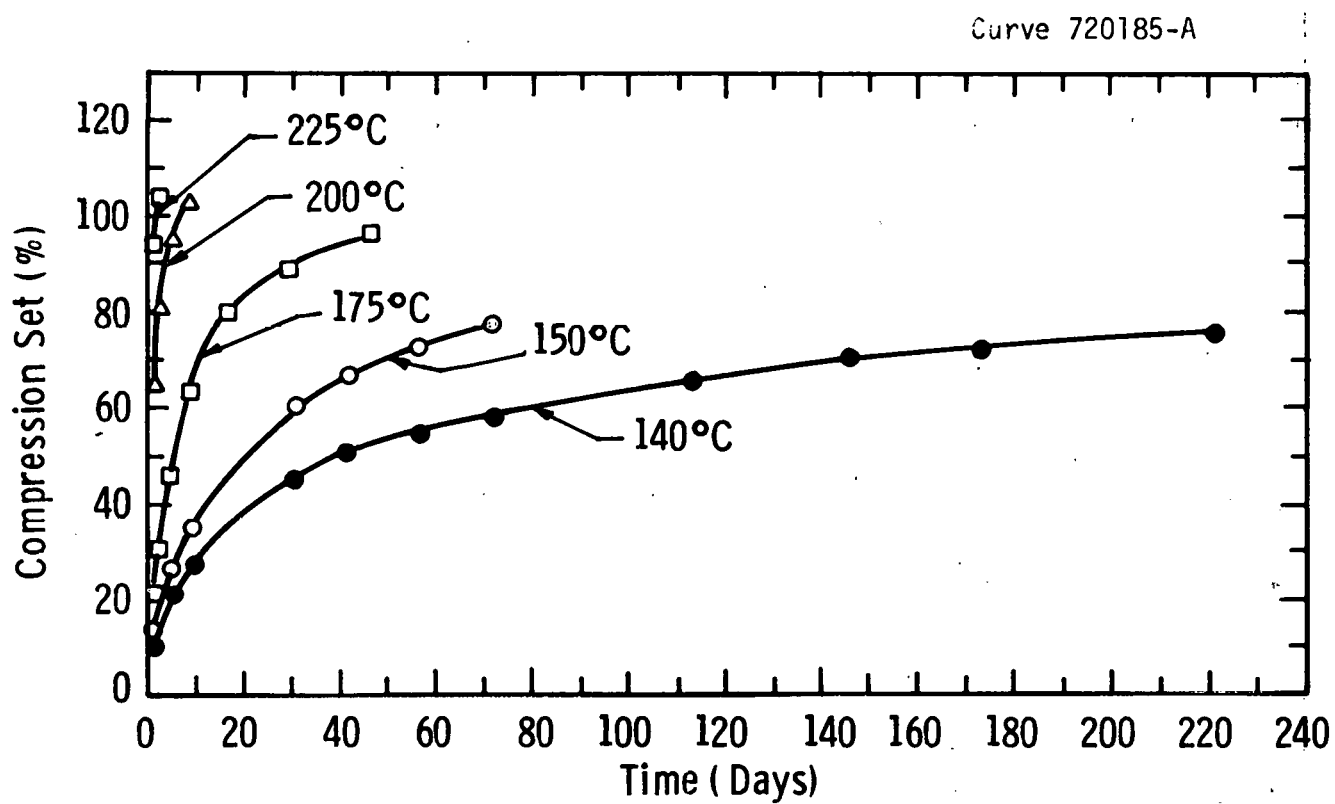


Fig. 3 — Aging of silicone rubber NPC 80/40 under compression

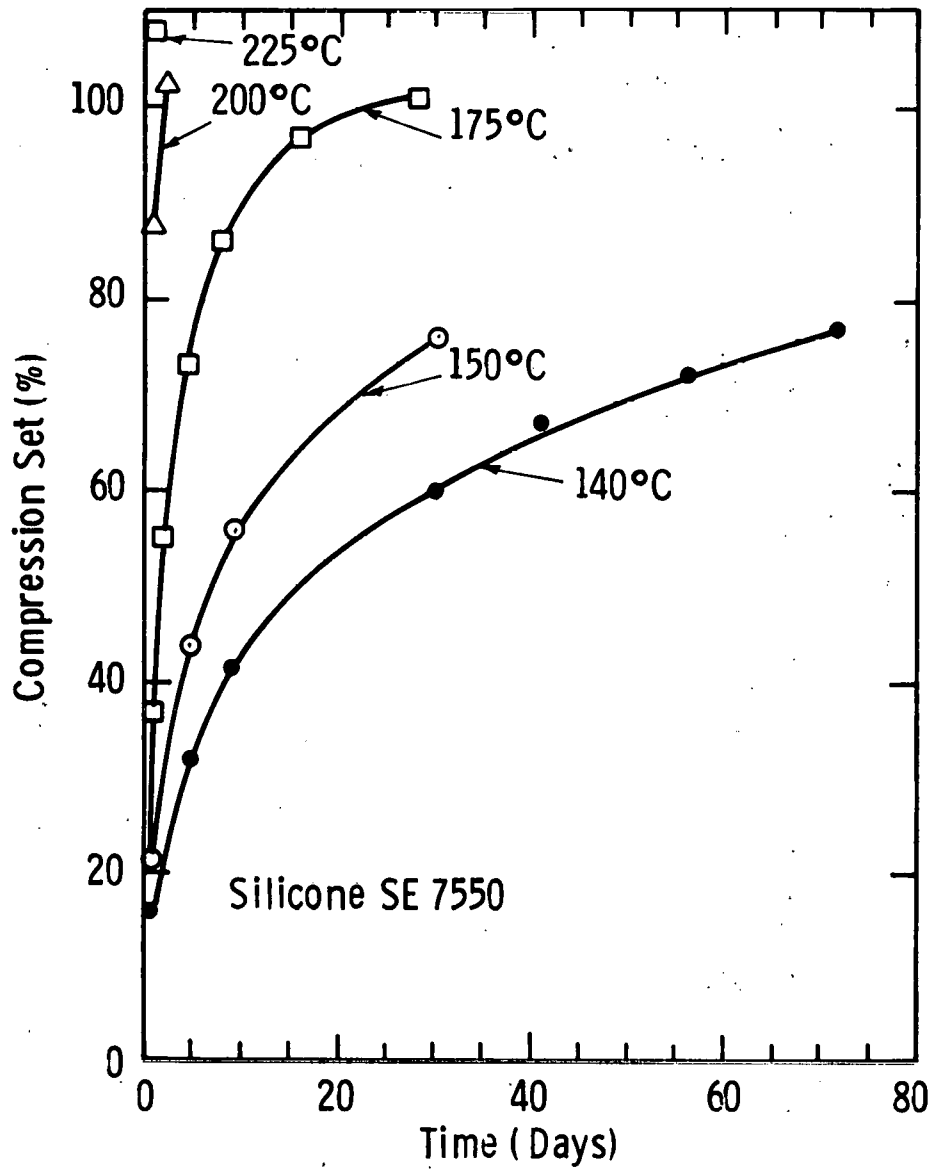


Fig. 4 — Aging of silicone rubber SE 7550 under compression

Curve 720176-A

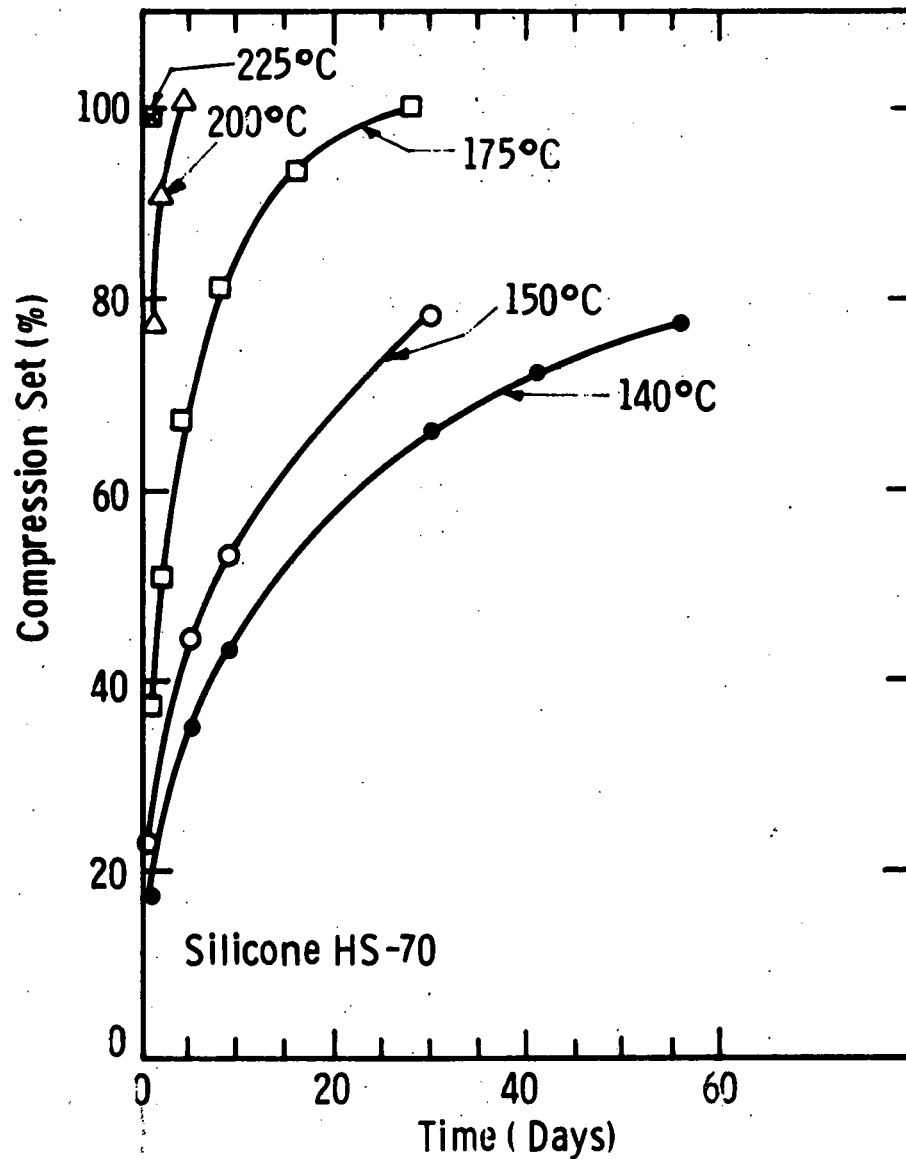


Fig. 5 - Aging of silicone rubber HS-70 under compression

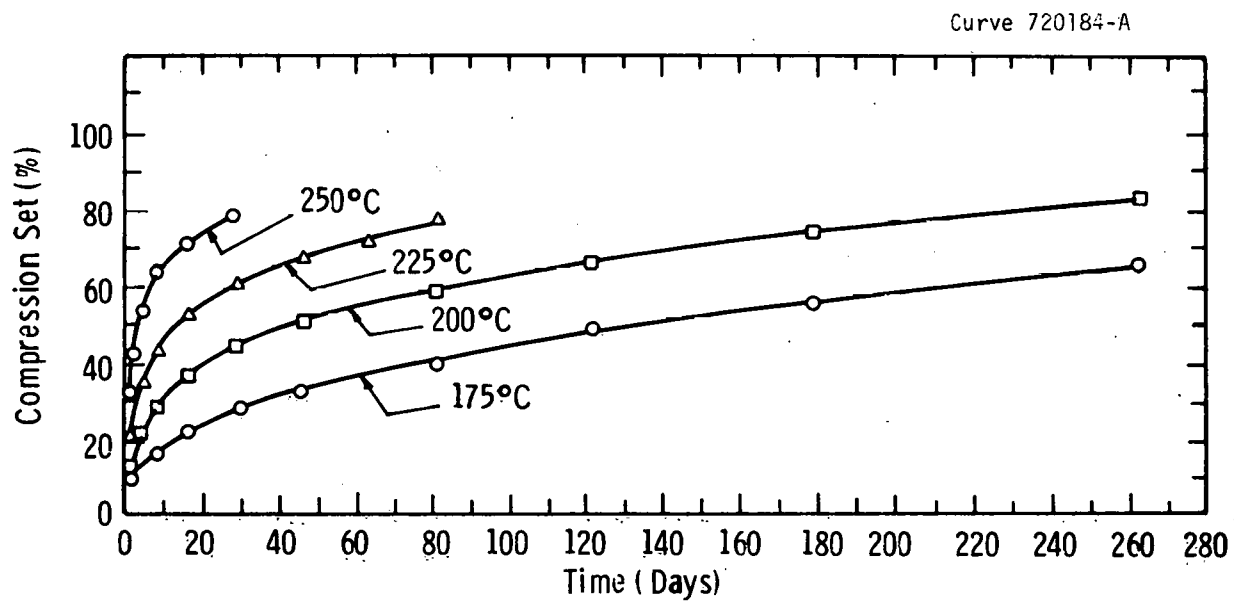


Fig. 6 — Aging of fluorocarbon elastomer Viton 31323-0731A under compression

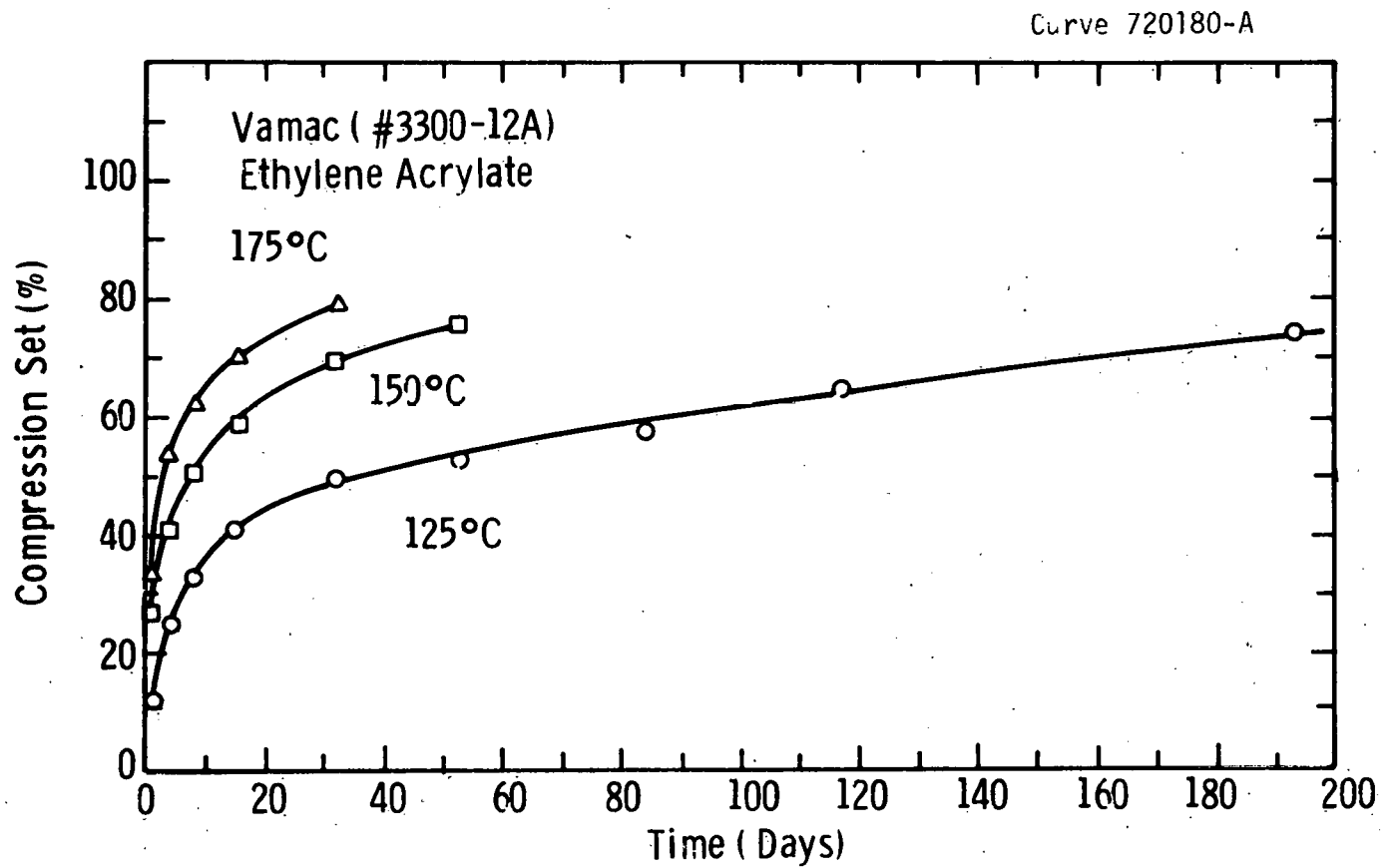


Fig. 7 - Aging of ethylene-acrylic copolymer Vamac 3300-12A under compression

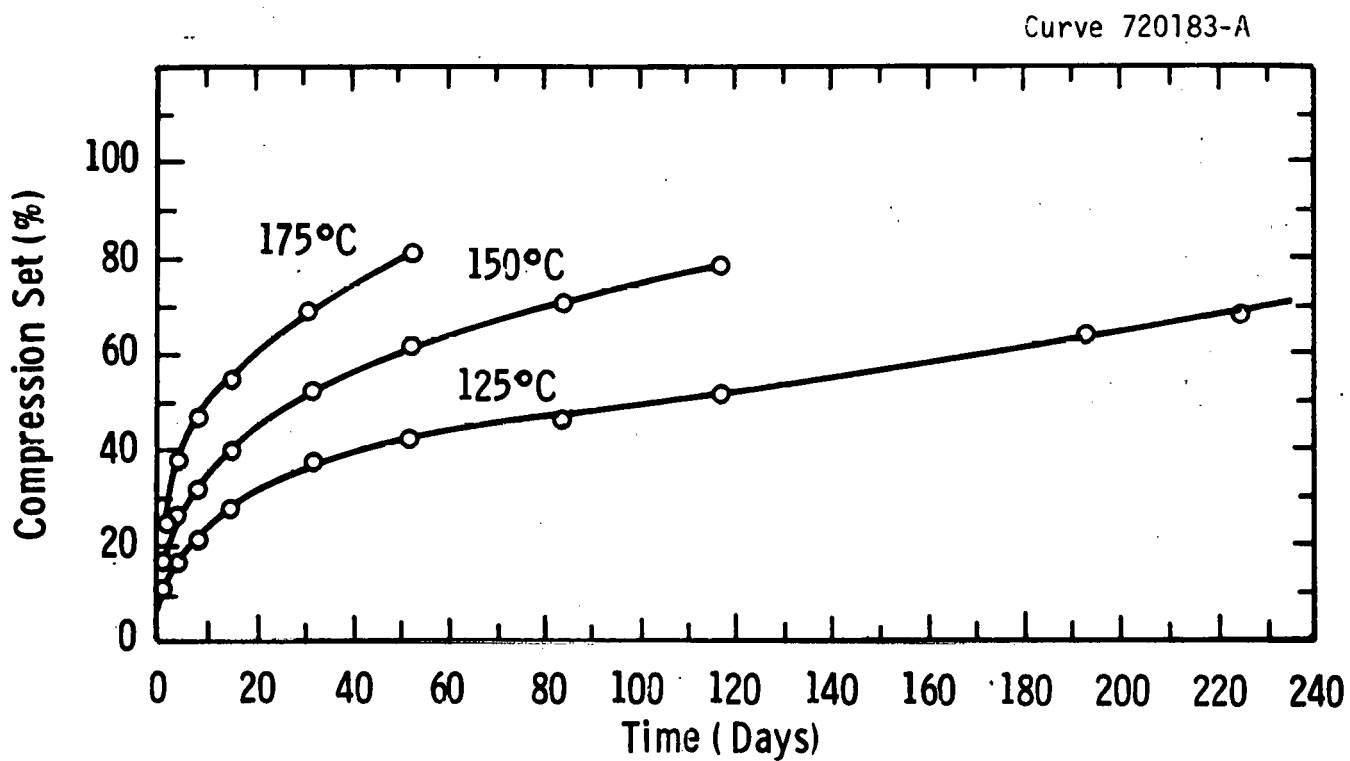


Fig. 8 — Aging of acrylic elastomer 210-108-35-1 under compression

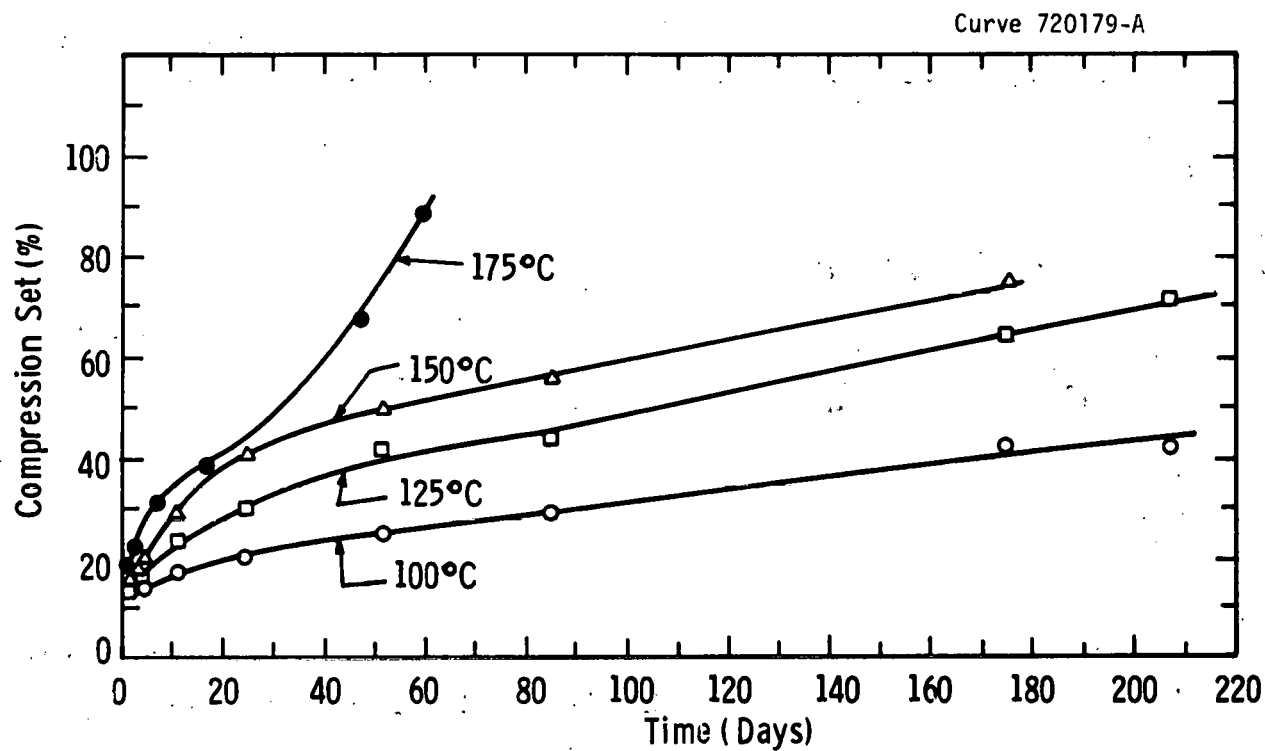


Fig. 9 — Aging of ethylene propylene terpolymer Nordel 3300-11 under compression

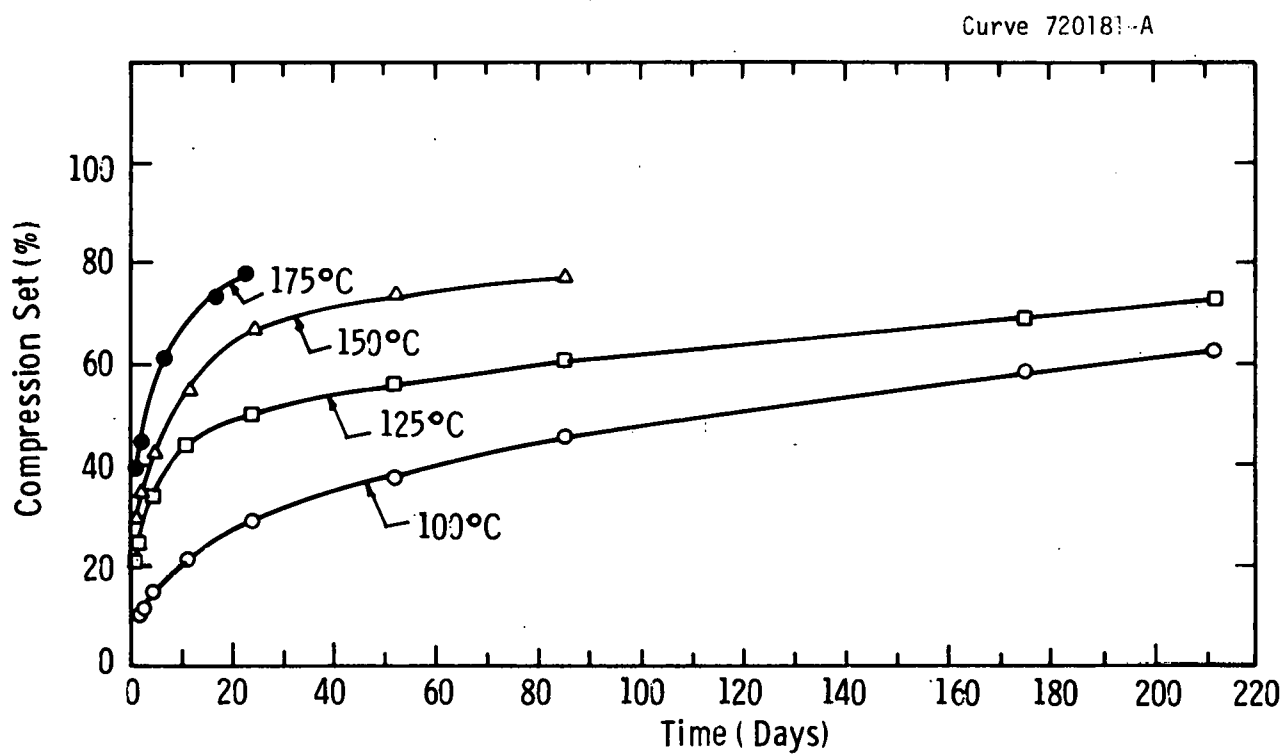


Fig. 10 — Aging of butyl rubber 8EX-123 under compression

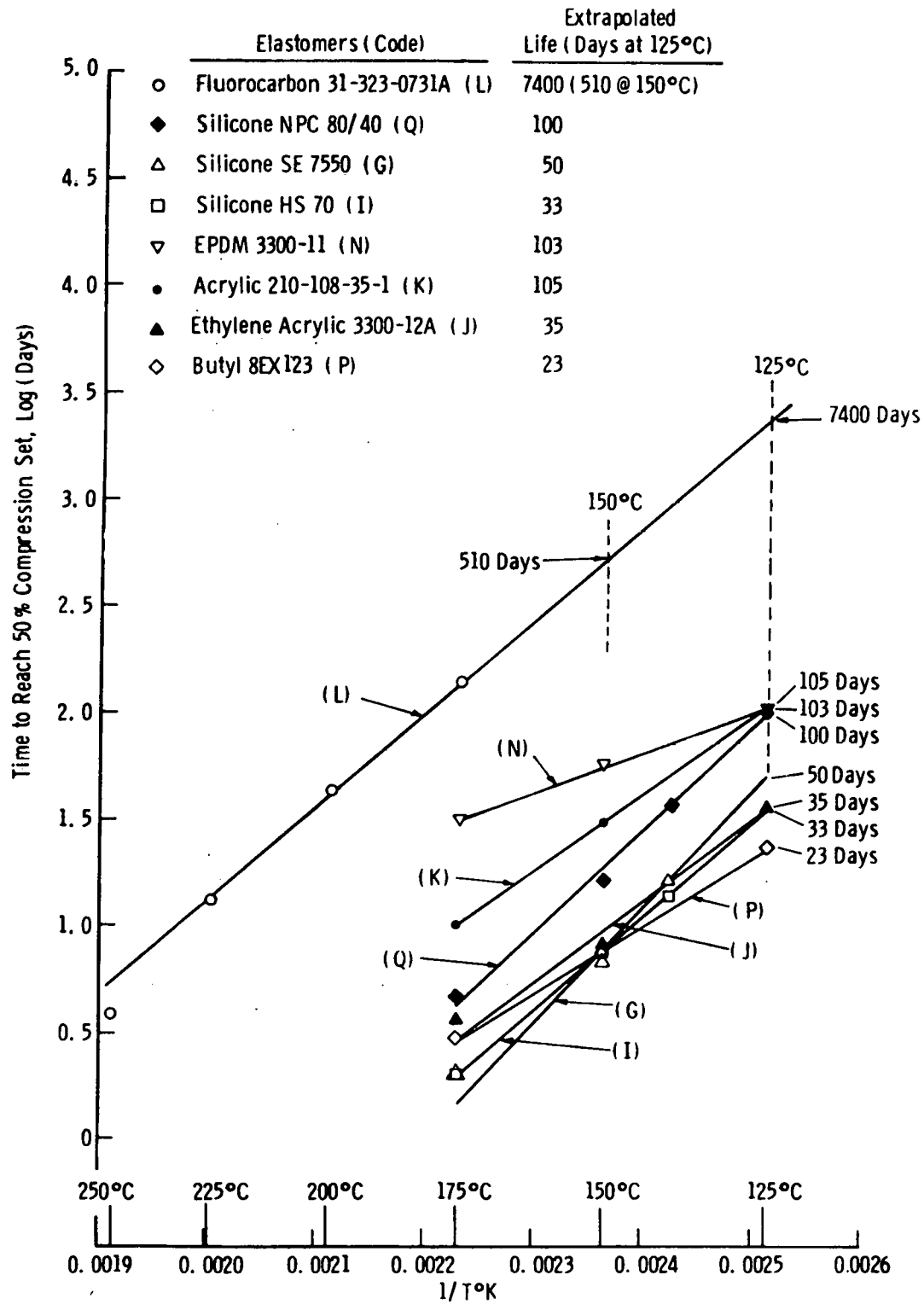


Fig. 11 — Arrhenius plot of compression set aging data

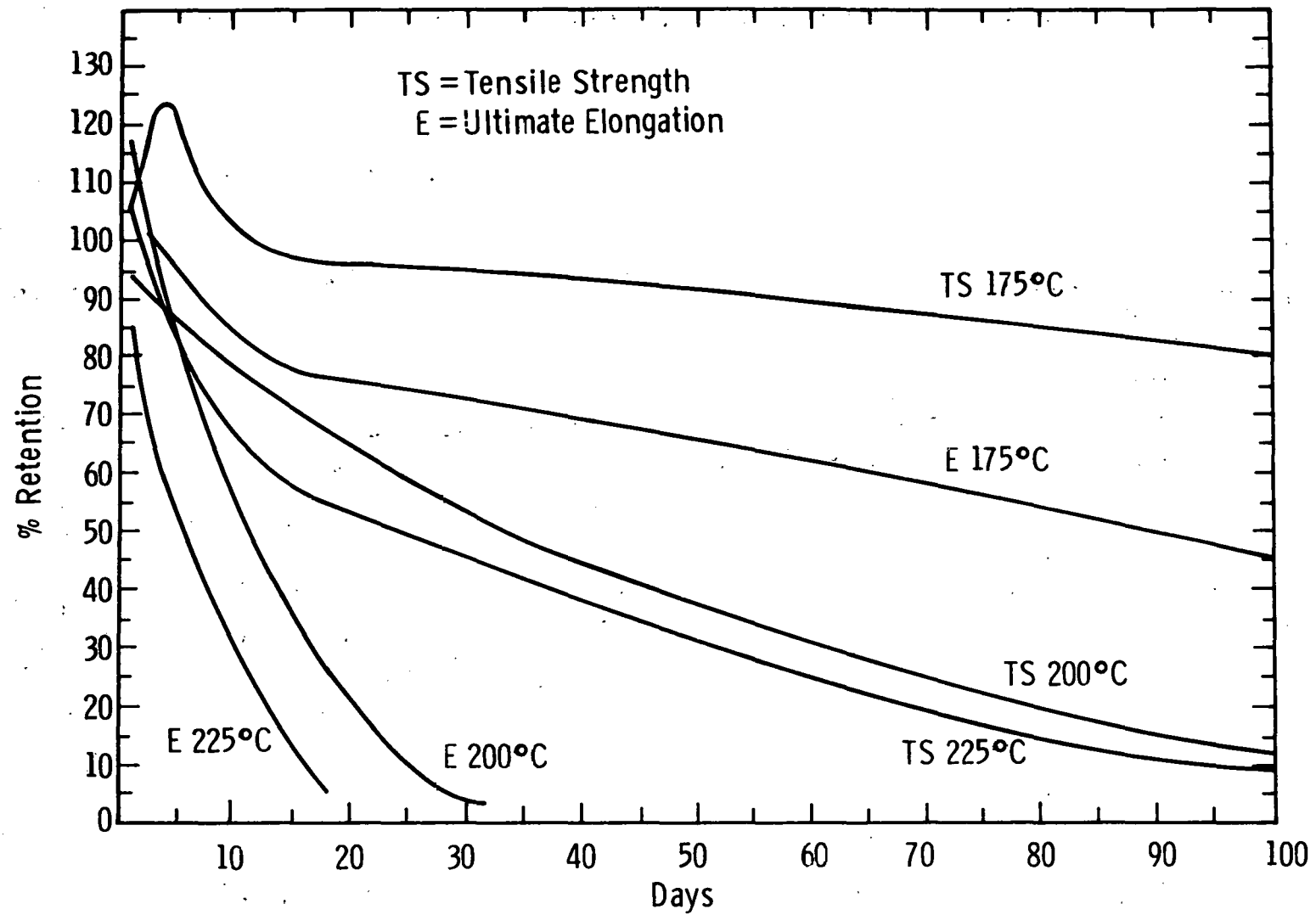


Fig. 12 — Thermal aging in air of silicone rubber NPC 80/40

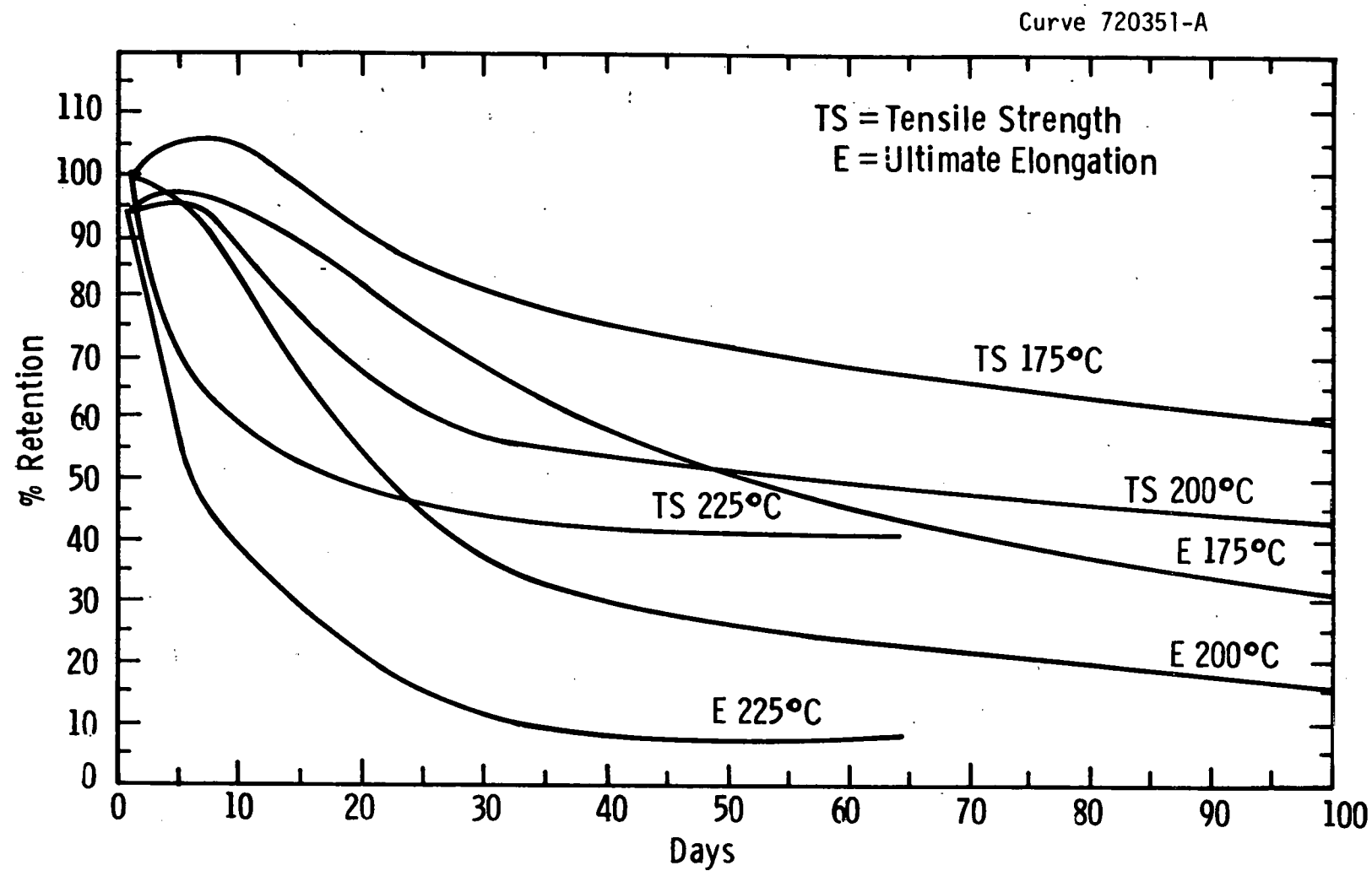


Fig. 13 — Thermal aging of silicone rubber SE-7550 in air

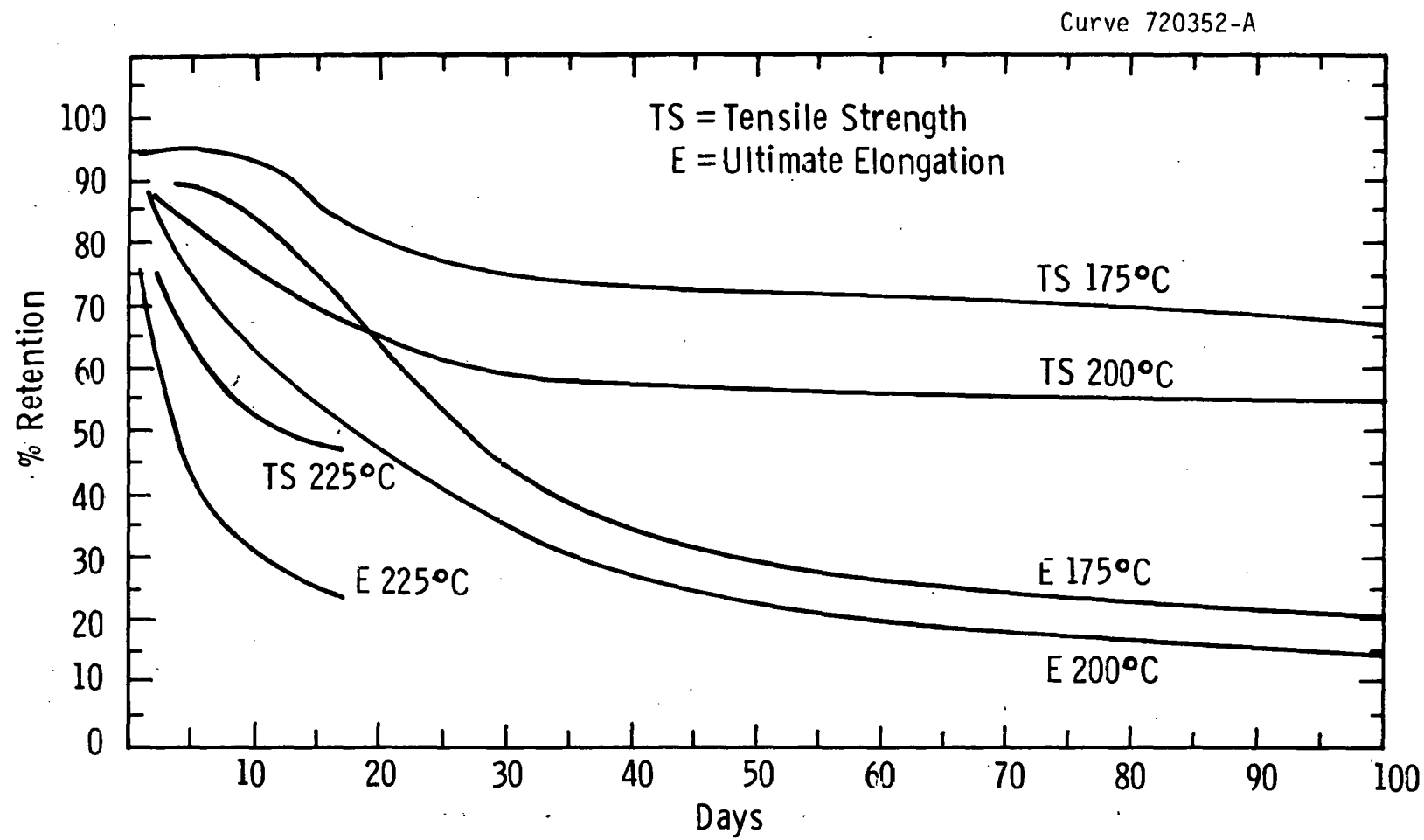


Fig. 14 — Thermal aging of silicone rubber HS-70 in air

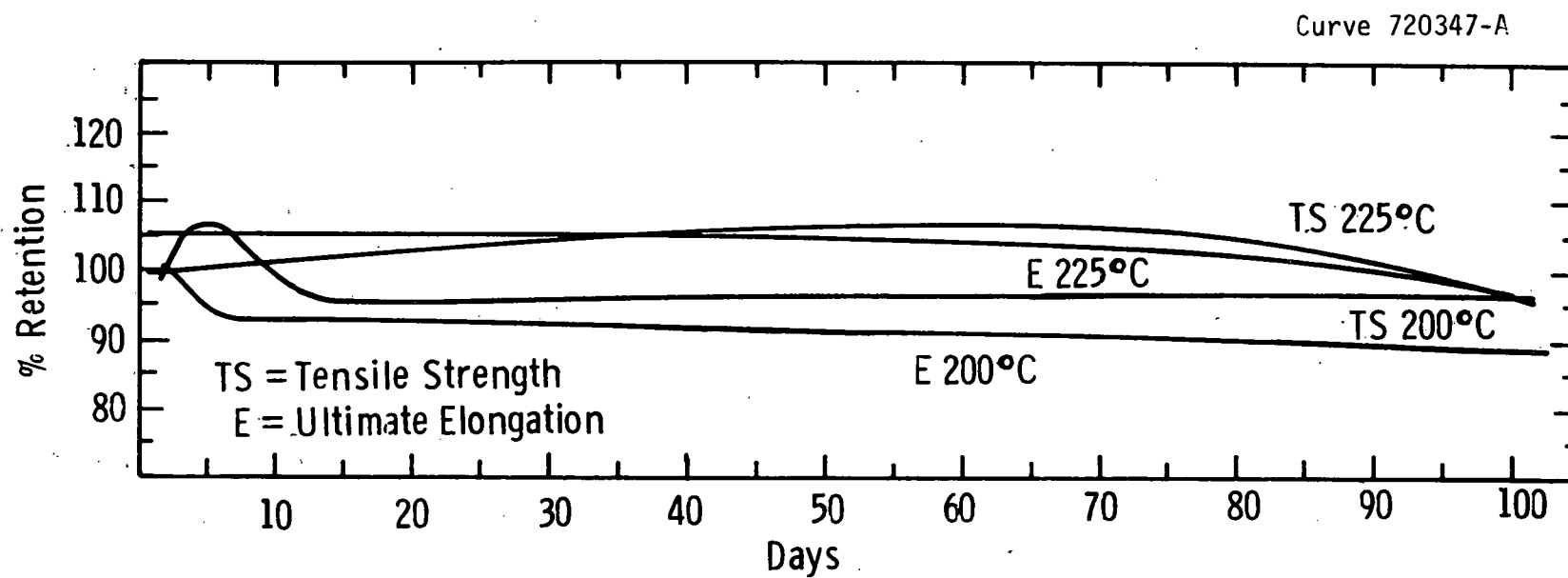


Fig. 15 — Thermal aging in air of fluorocarbon elastomer Viton 31-323-0731A

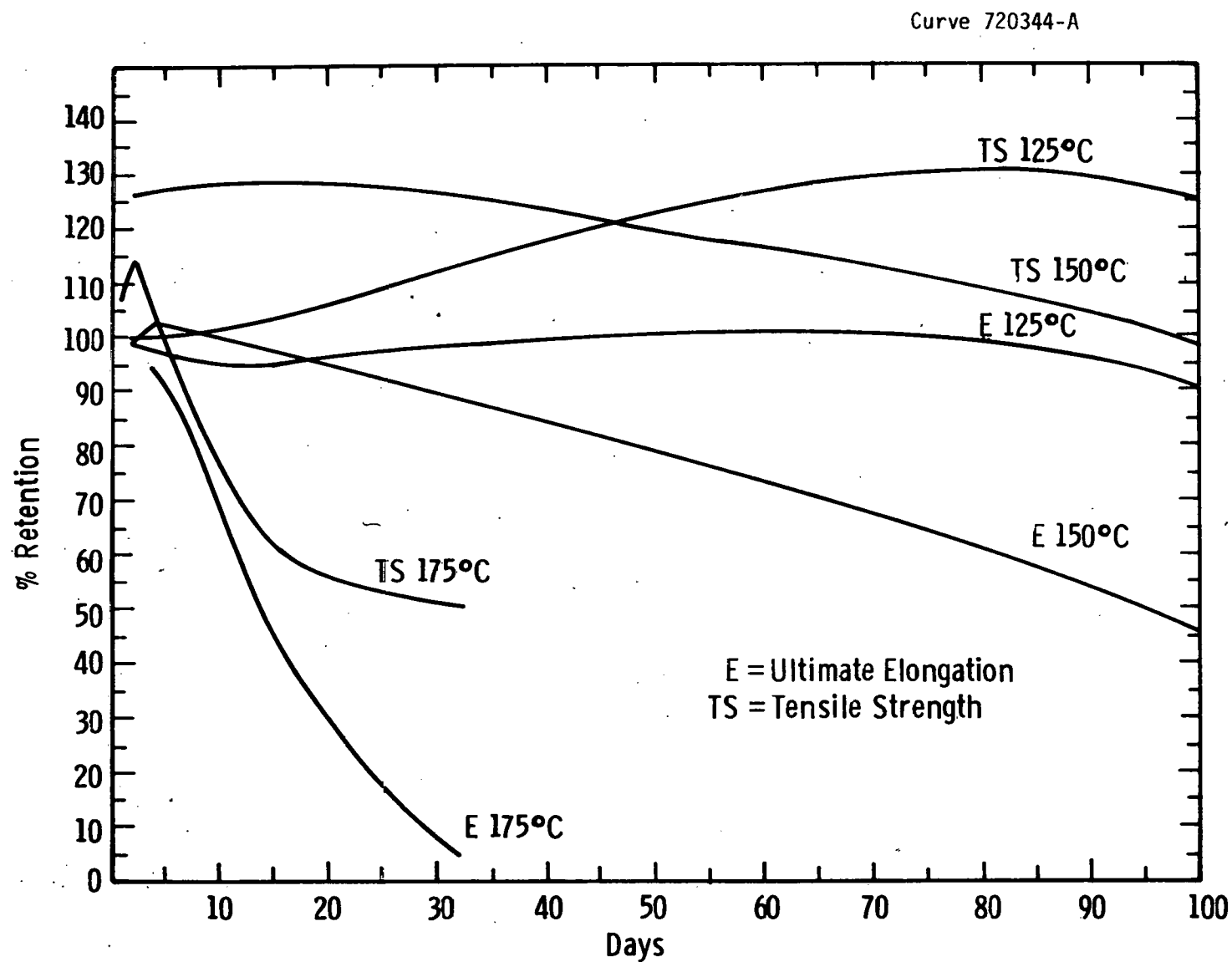


Fig. 16 — Thermal aging of ethylene-acrylic copolymer Vamac 3300-12A in air

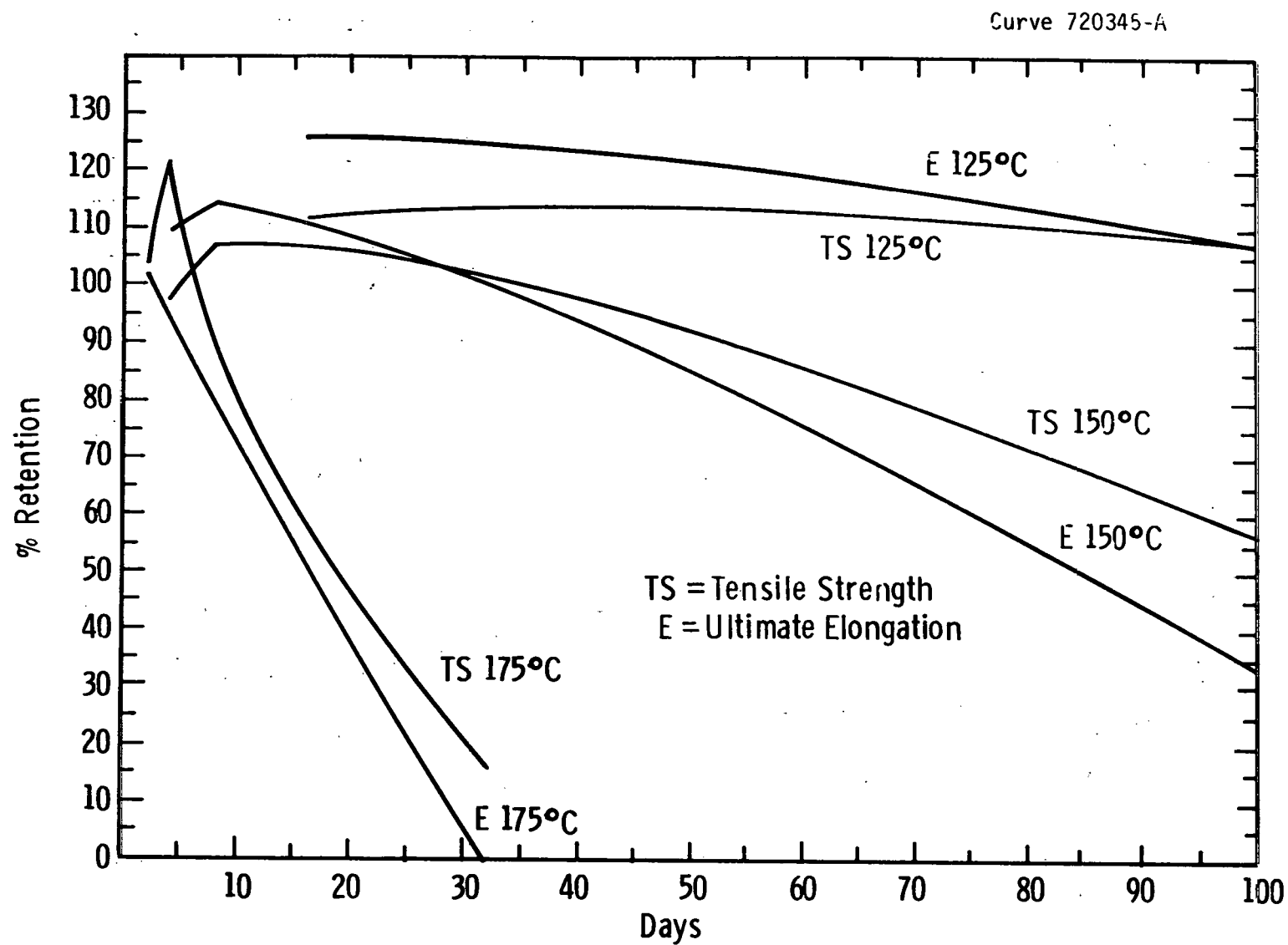


Fig. 17 — Thermal aging in air of acrylic elastomer Hycar 4054 210-108-35-1

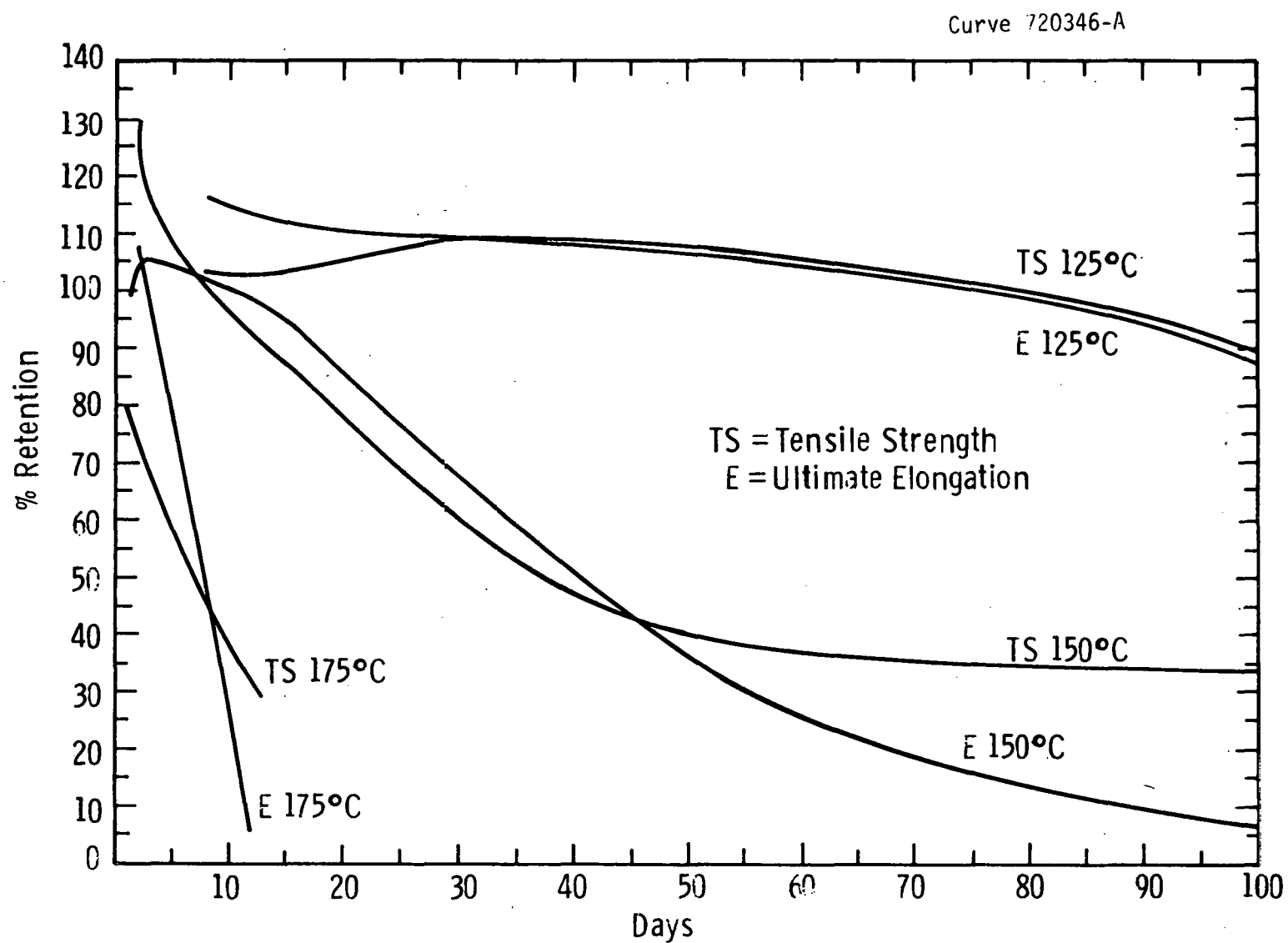


Fig. 18 — Thermal aging in air of ethylene-propylene terpolymer (EPDM) Nordel 3300-11

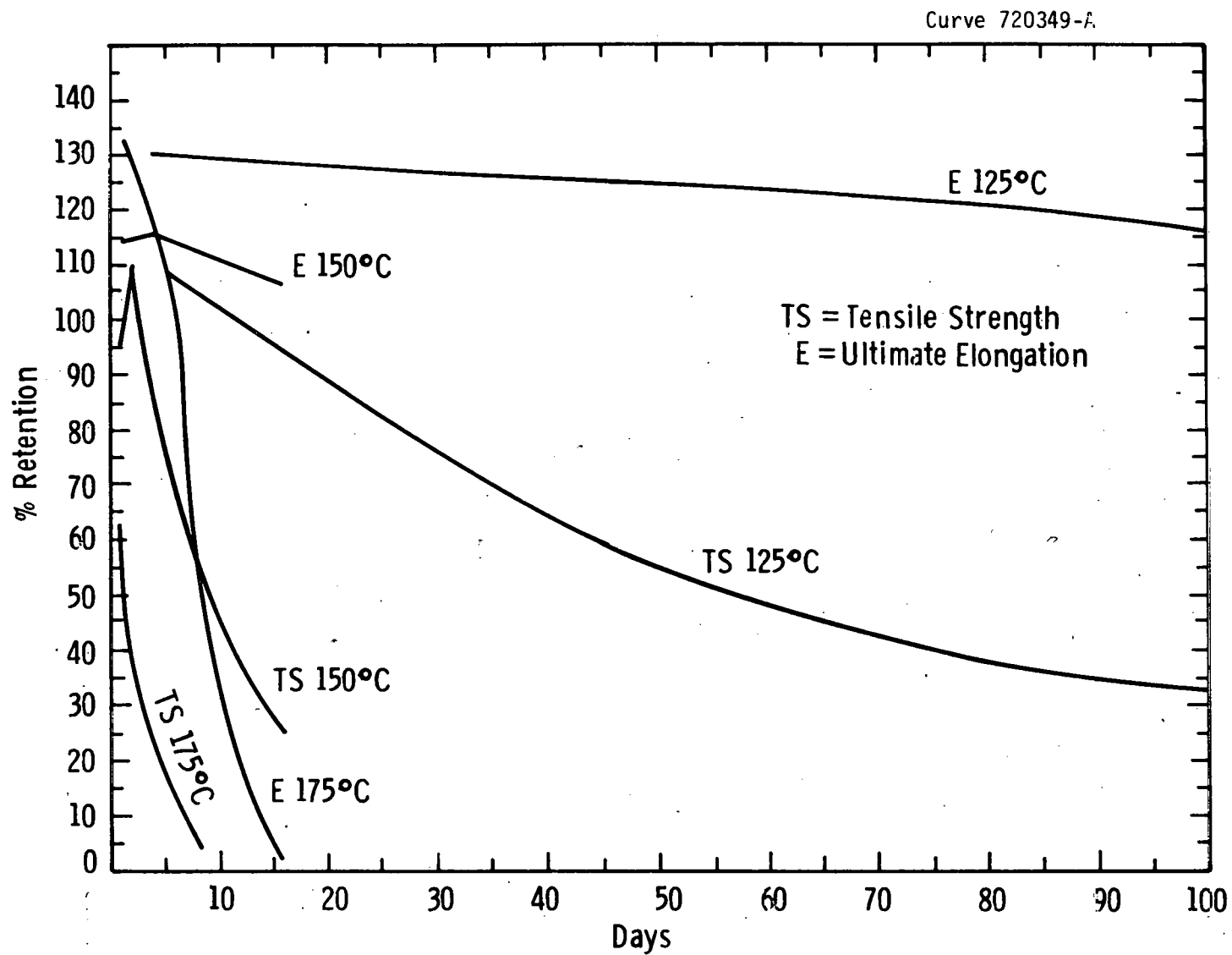


Fig. 19 — Thermal aging in air of butyl rubber 8 Ex-123

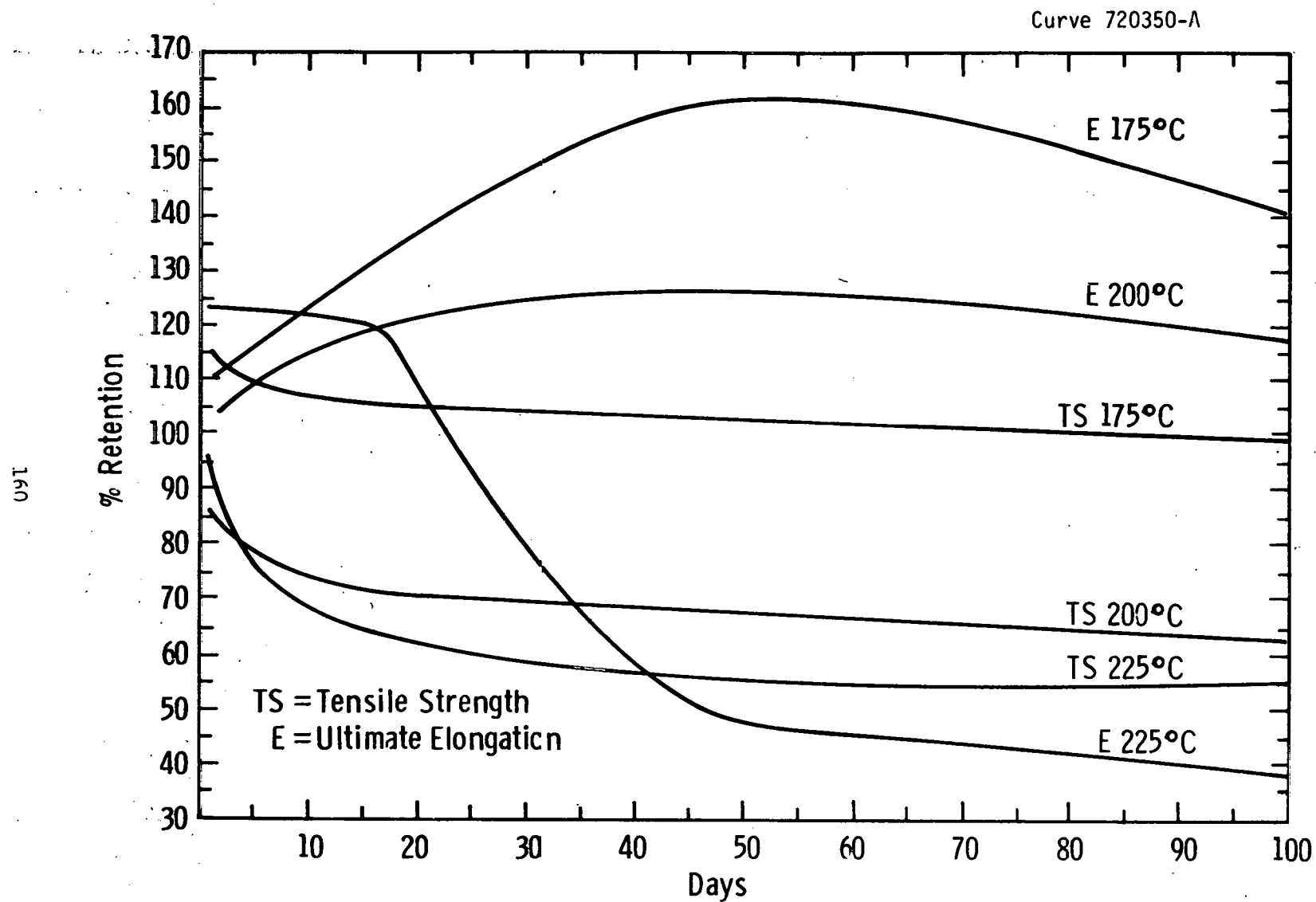


Fig. 20 — Thermal aging in air of silicone caulking compound DC-732

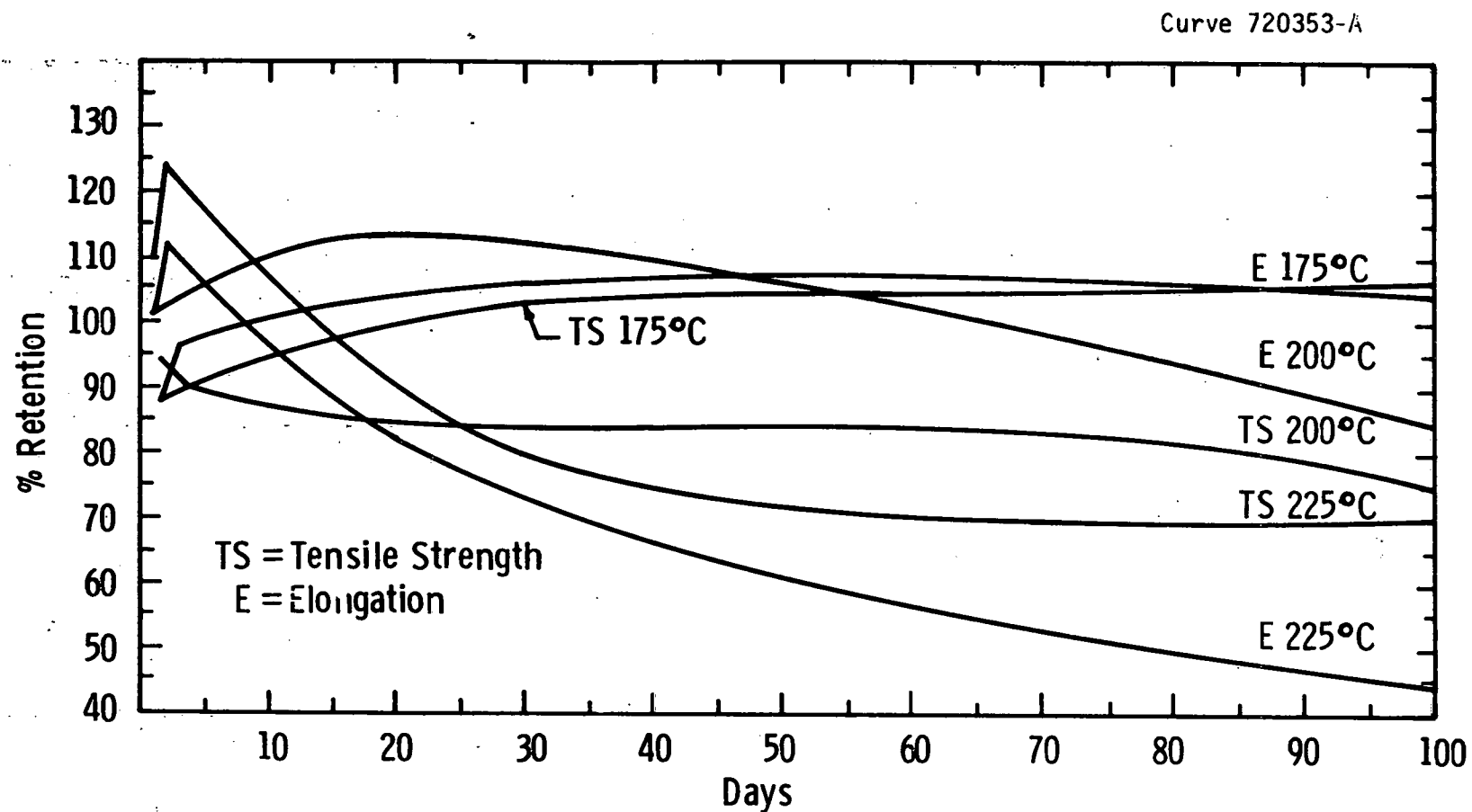


Fig. 21 — Thermal aging in air of silicone caulking compound RTV 103

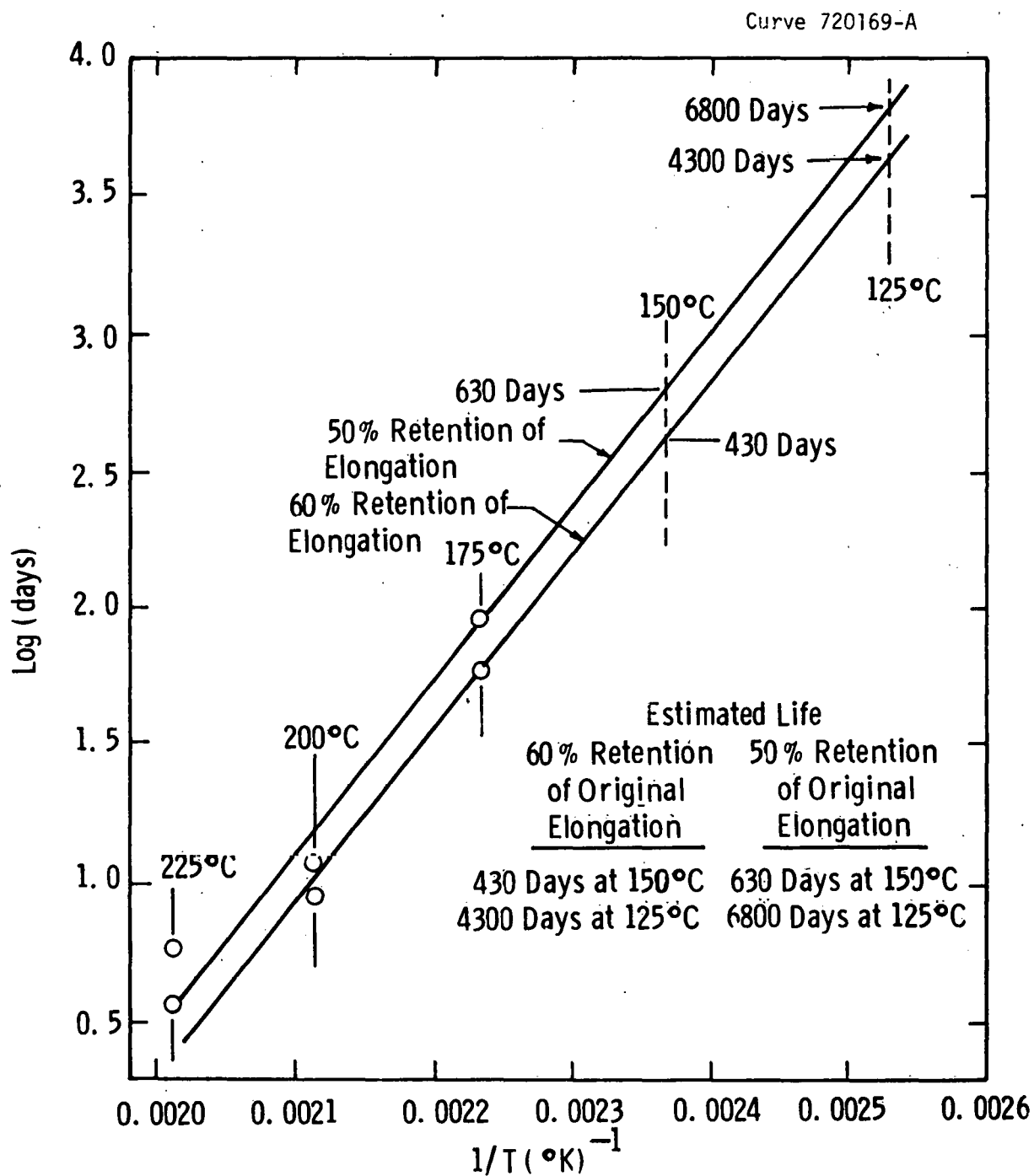


Fig. 22 — Arrhenius plot of thermal aging in air of silicon elastomer NPC 80/40

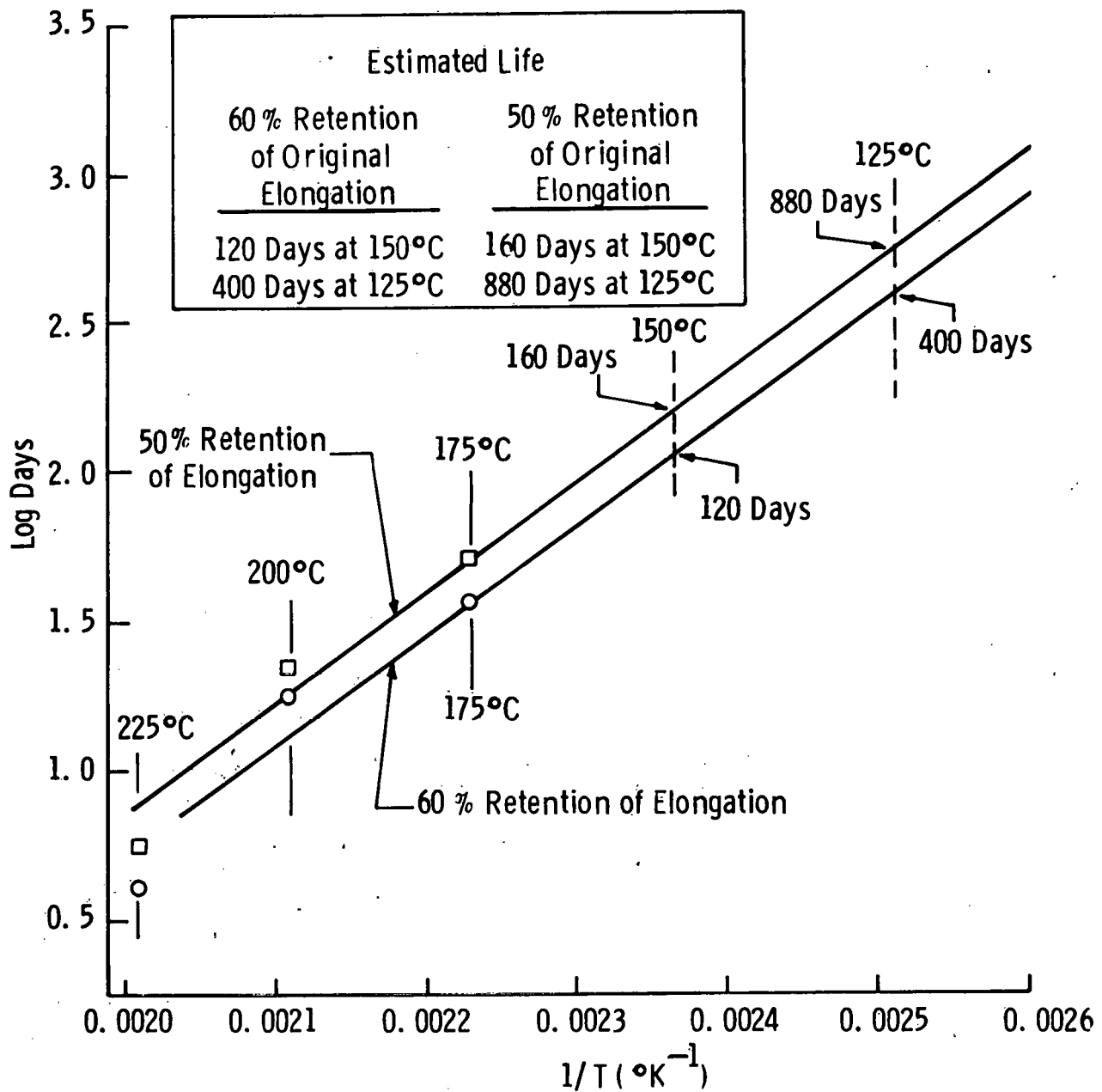


Fig. 22a — Arrhenius plot of thermal aging in air of silicone elastomer SE-7550

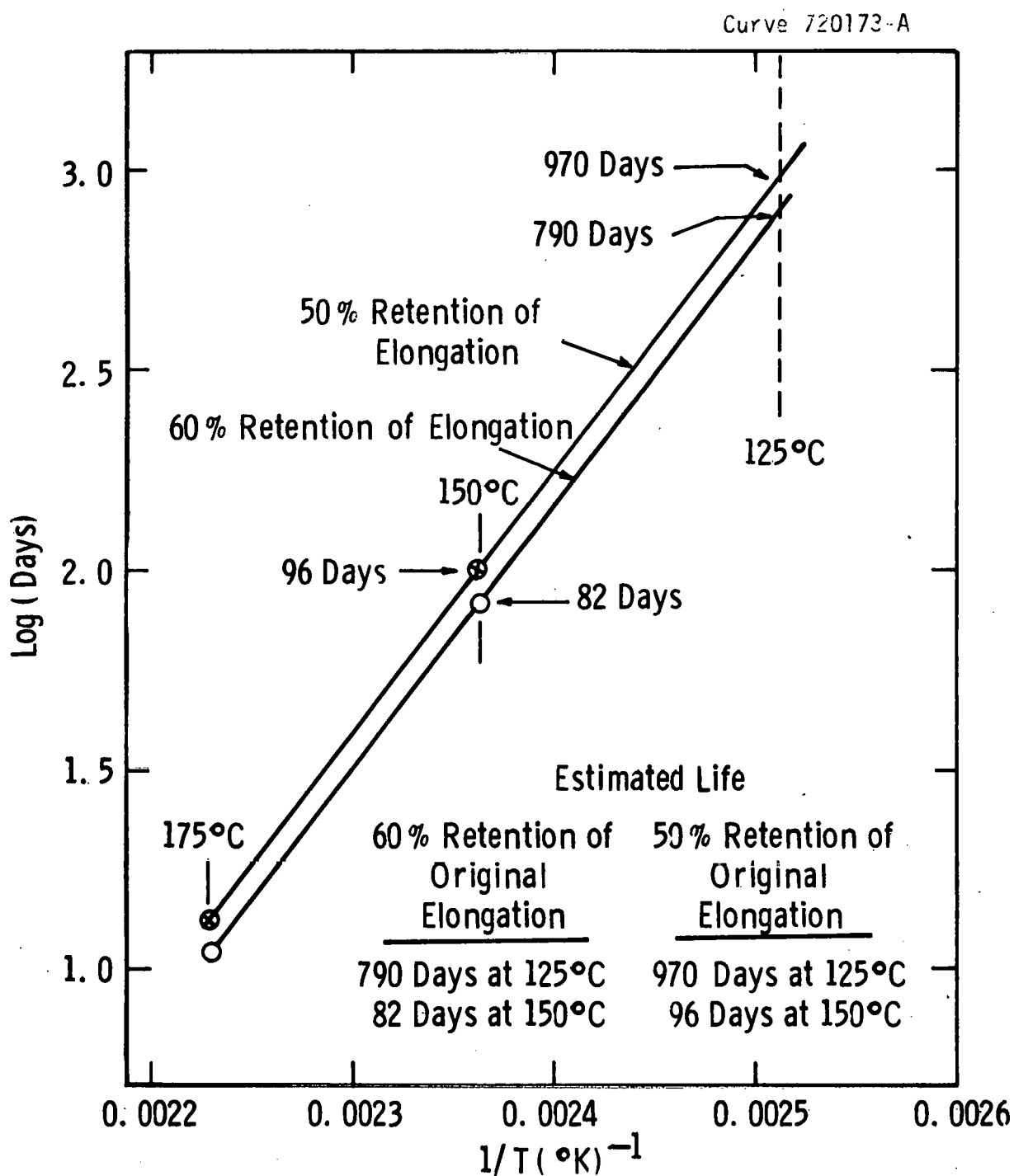


Fig. 23 - Arrhenius plot of thermal aging in air of ethylene/acrylic elastomer 3300-12A

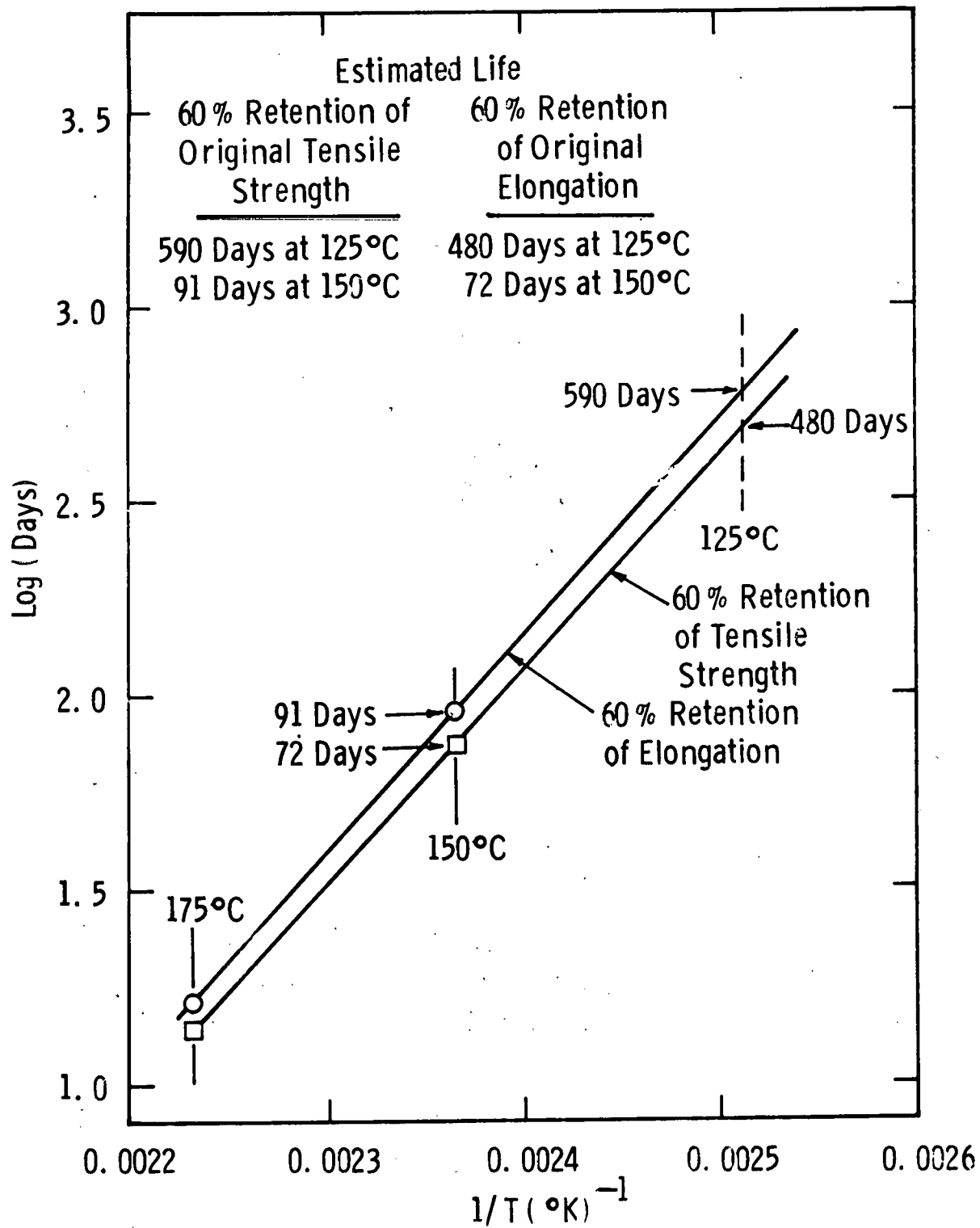


Fig. 24 — Arrhenius plot of thermal aging in air of acrylic elastomer 210-108-35-1

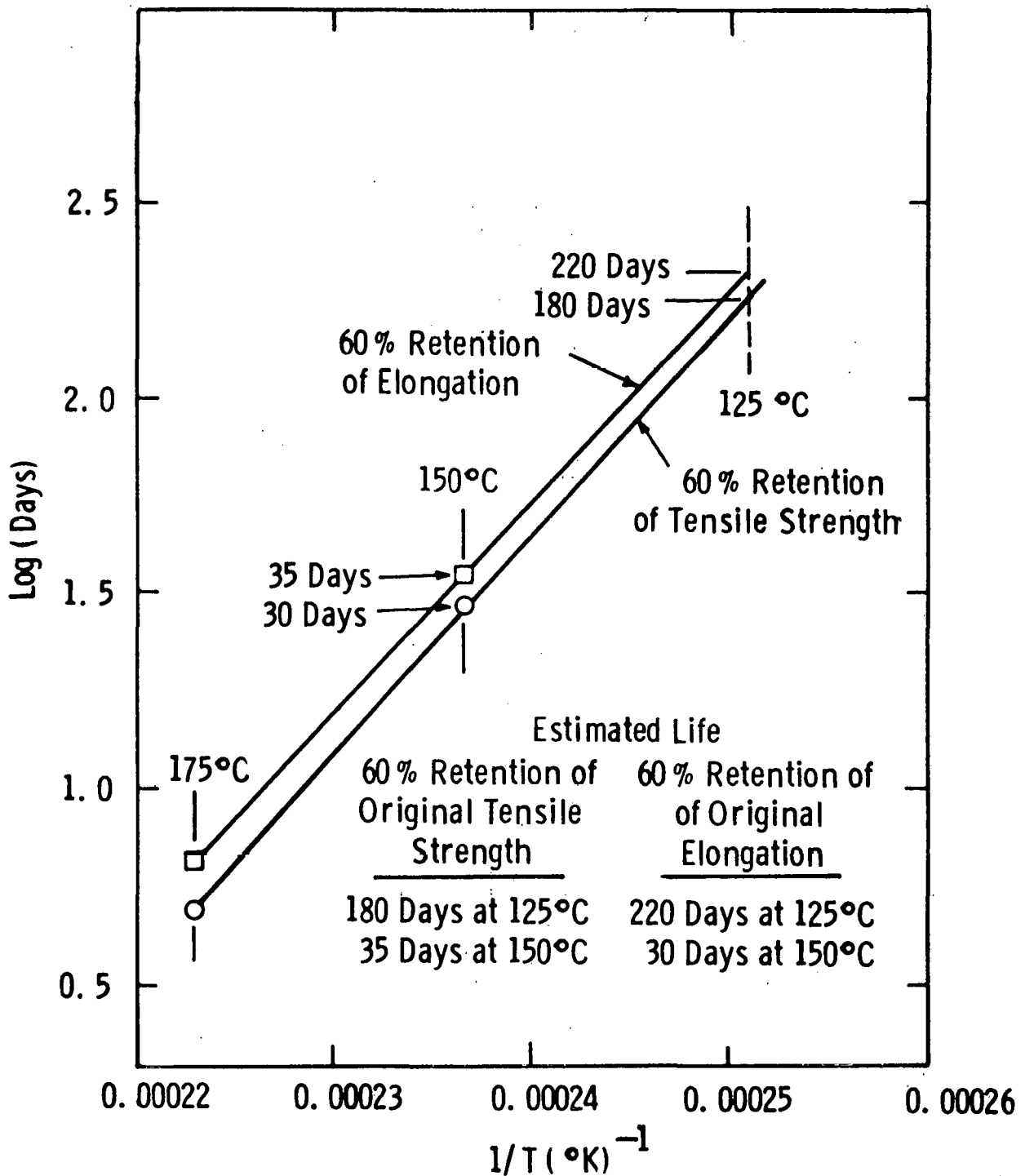


Fig. 25 — Arrhenius plot of thermal aging in air of EPDM 3300-11

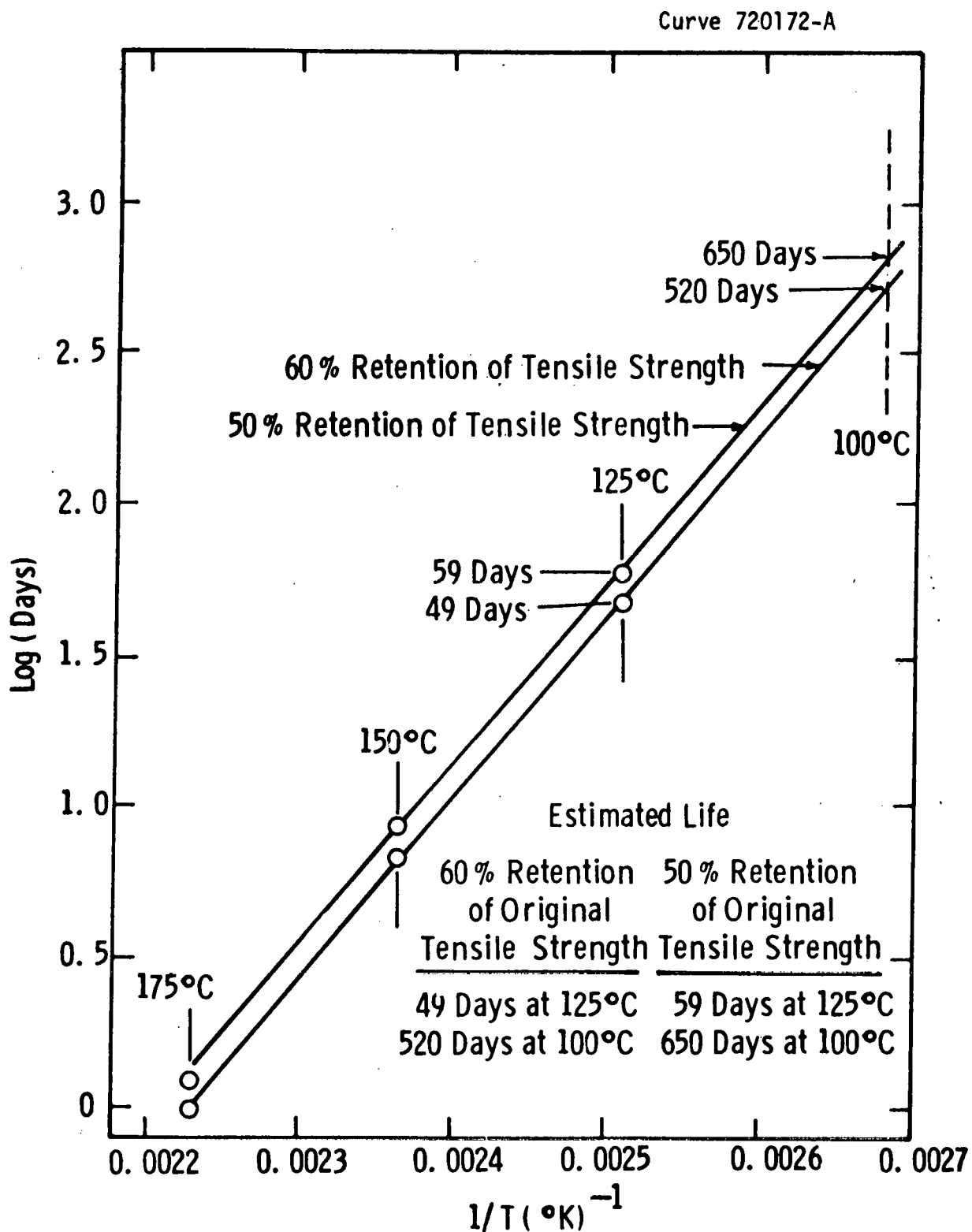


Fig. 26 — Arrhenius plot of thermal aging in air of butyl rubber 8EX123

Curve 720367-A

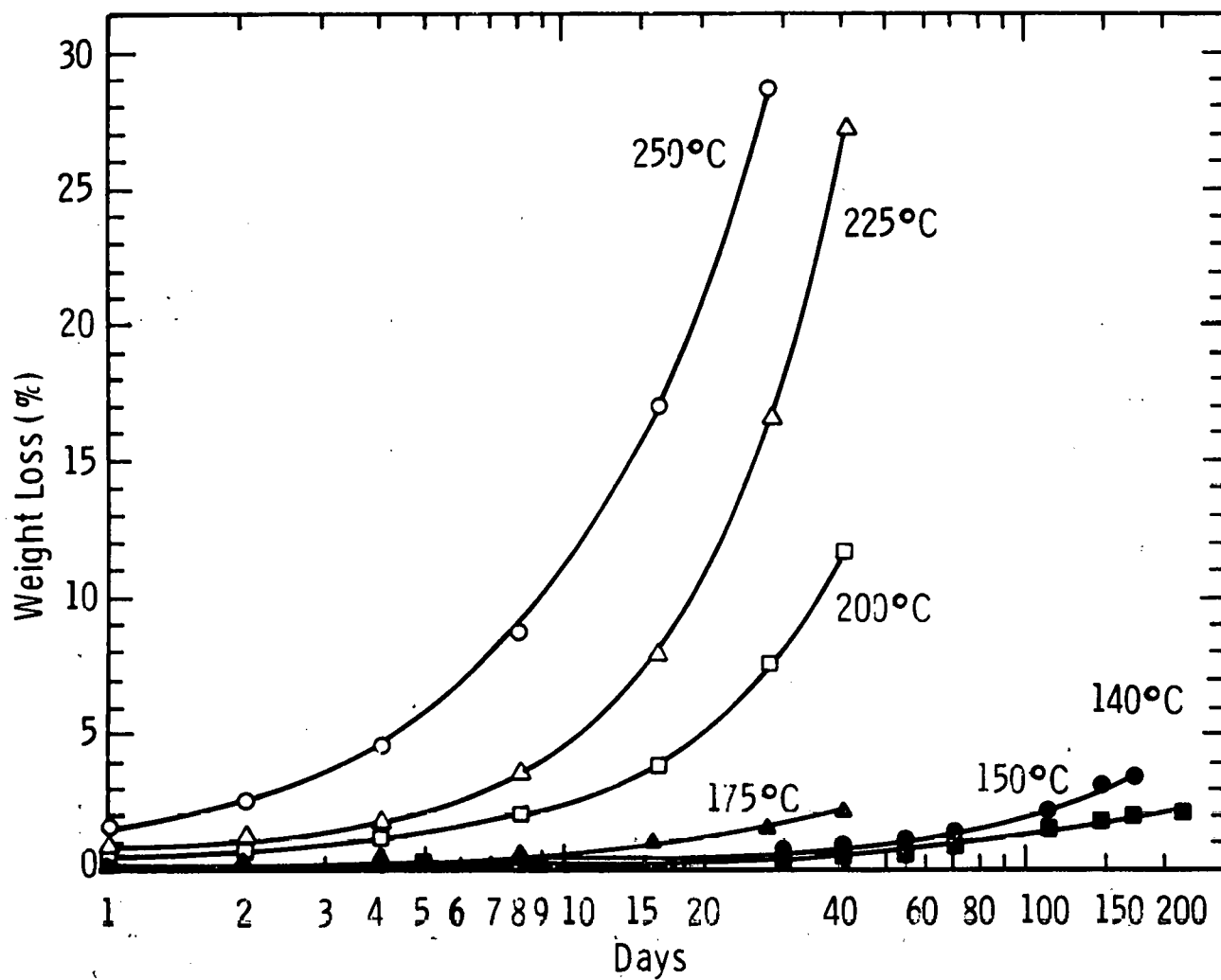


Fig. 27 — Weight loss resulting from aging silicone rubber NPC 80/40 in air

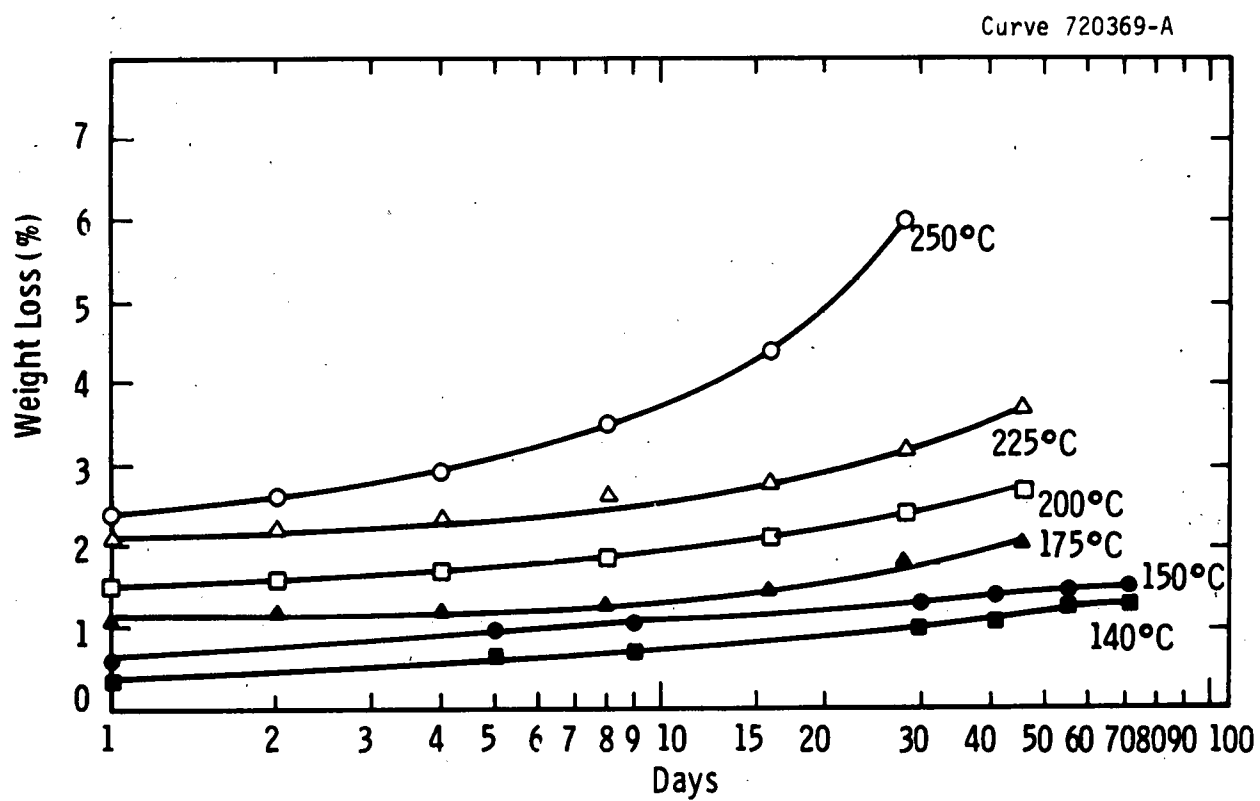


Fig. 28 — Weight loss resulting from aging silicone rubber SE-7550 in air.

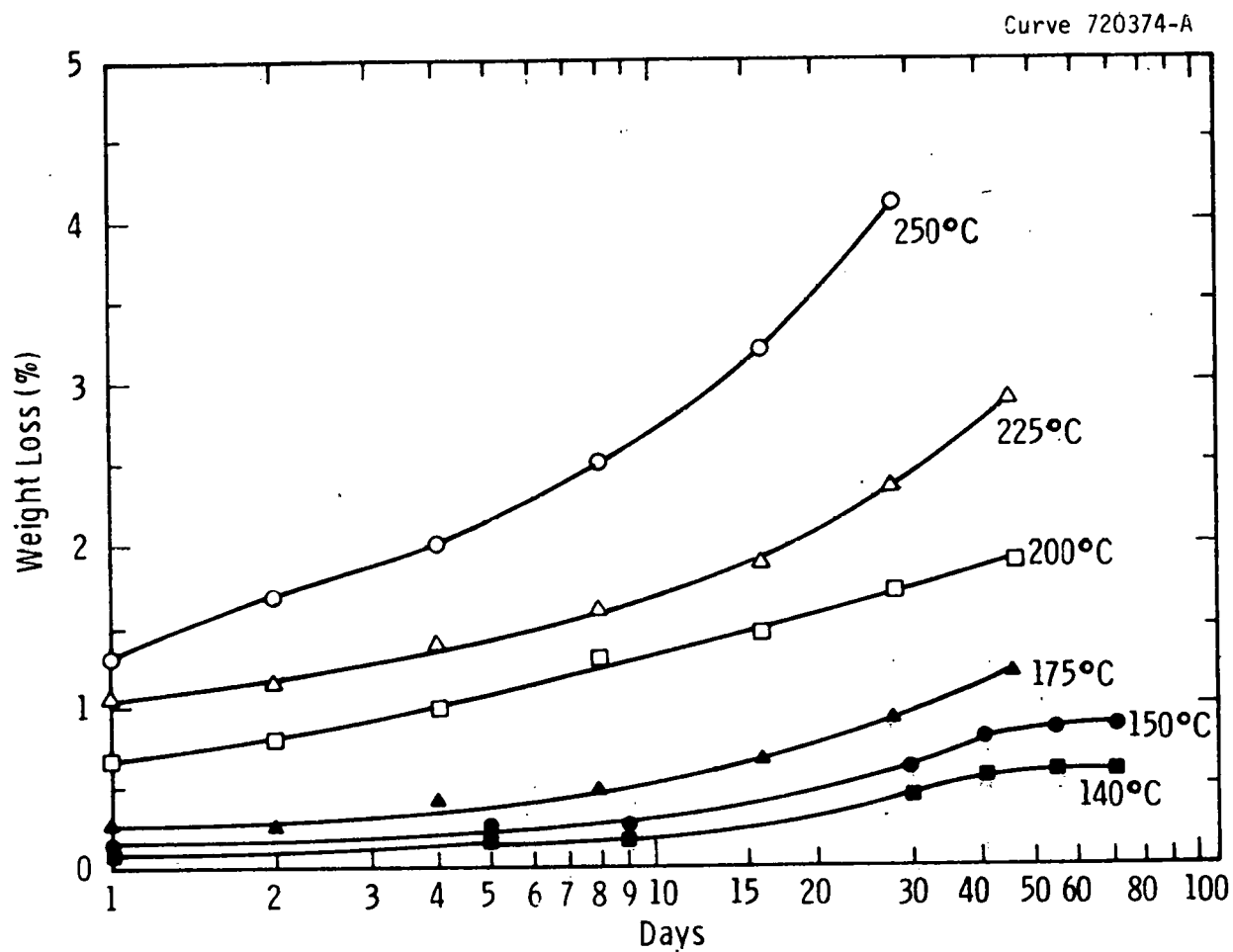


Fig. 29 — Weight loss resulting from aging silicone rubber HS-70 in air.

Curve 720368-A

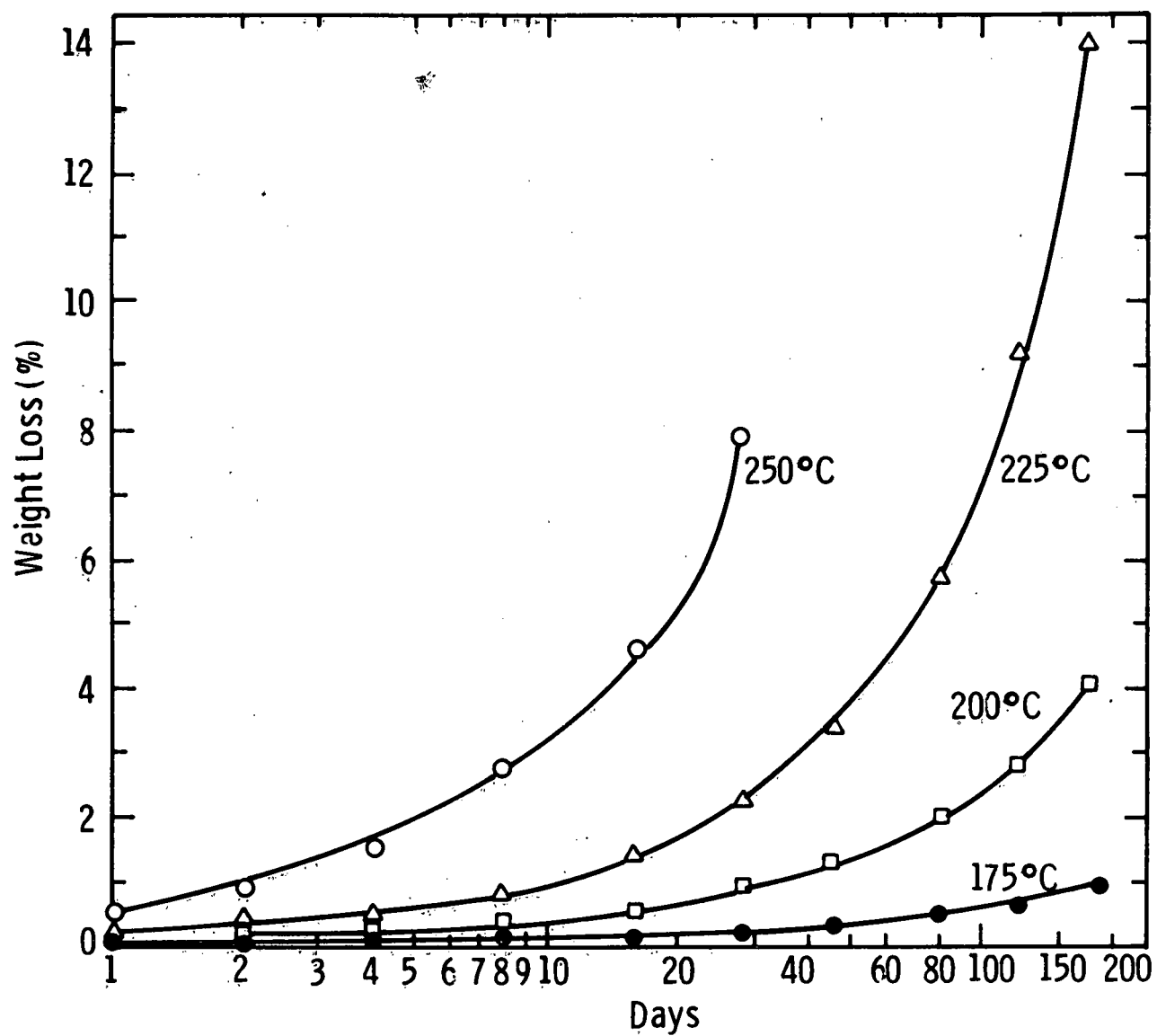


Fig. 30 — Weight loss resulting from aging fluorocarbon elastomer 31-323-0731A in air

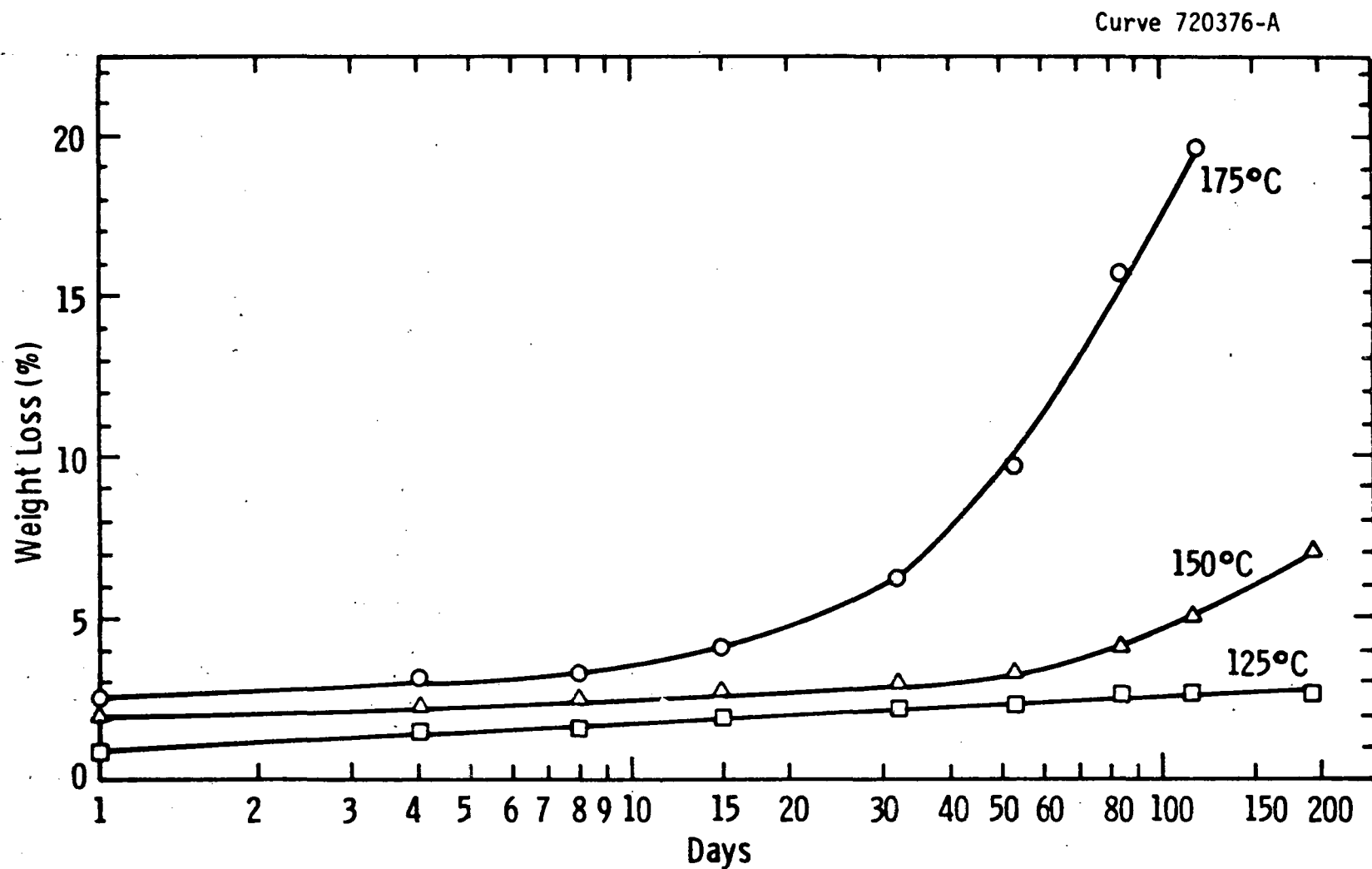


Fig. 31 — Weight loss resulting from aging ethylene-acrylic copolymer Vamac 3300-12A in air

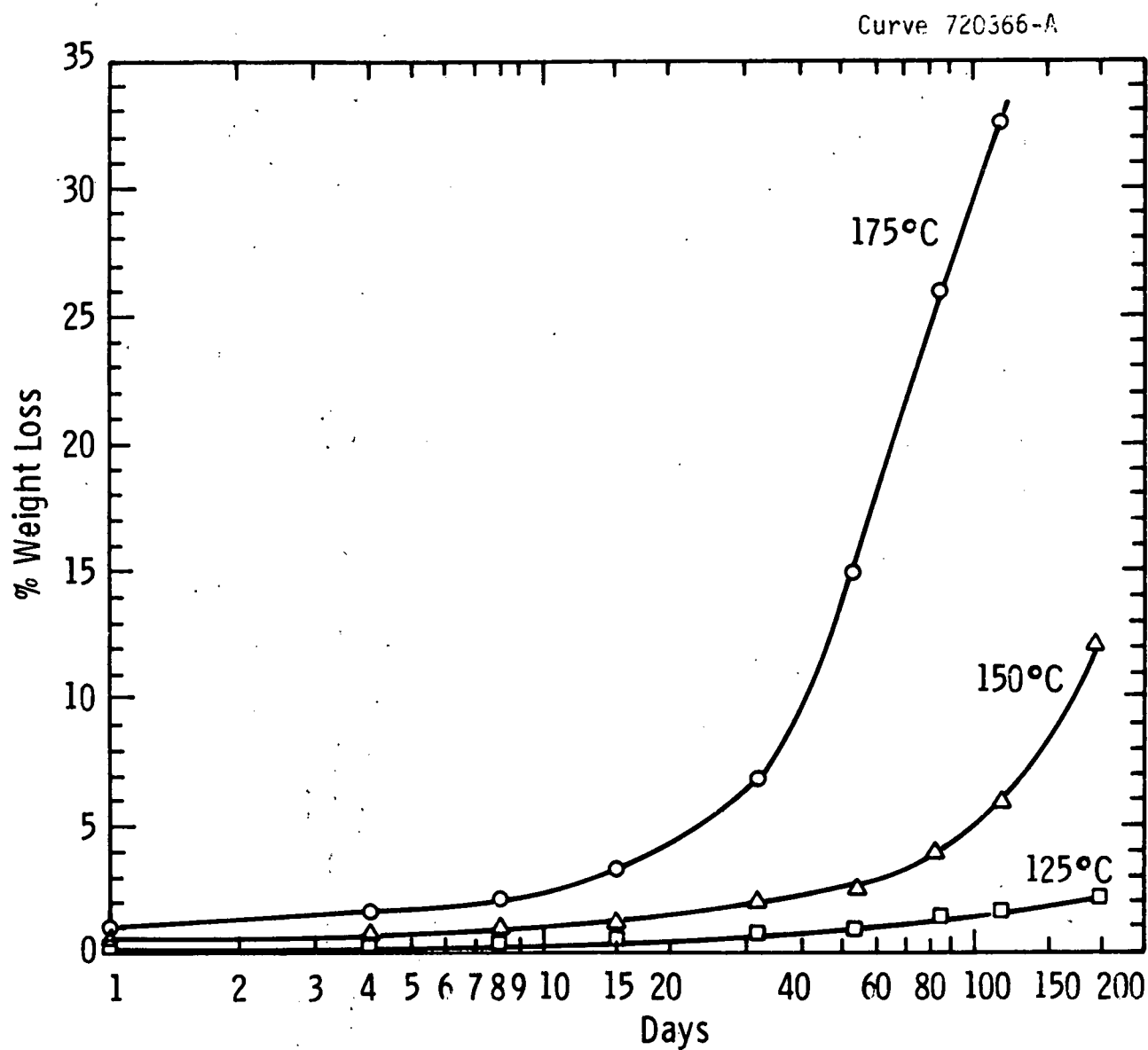


Fig. 32 — Weight loss resulting from aging acrylic elastomer Hycar 4054 210-108-35-1 in air

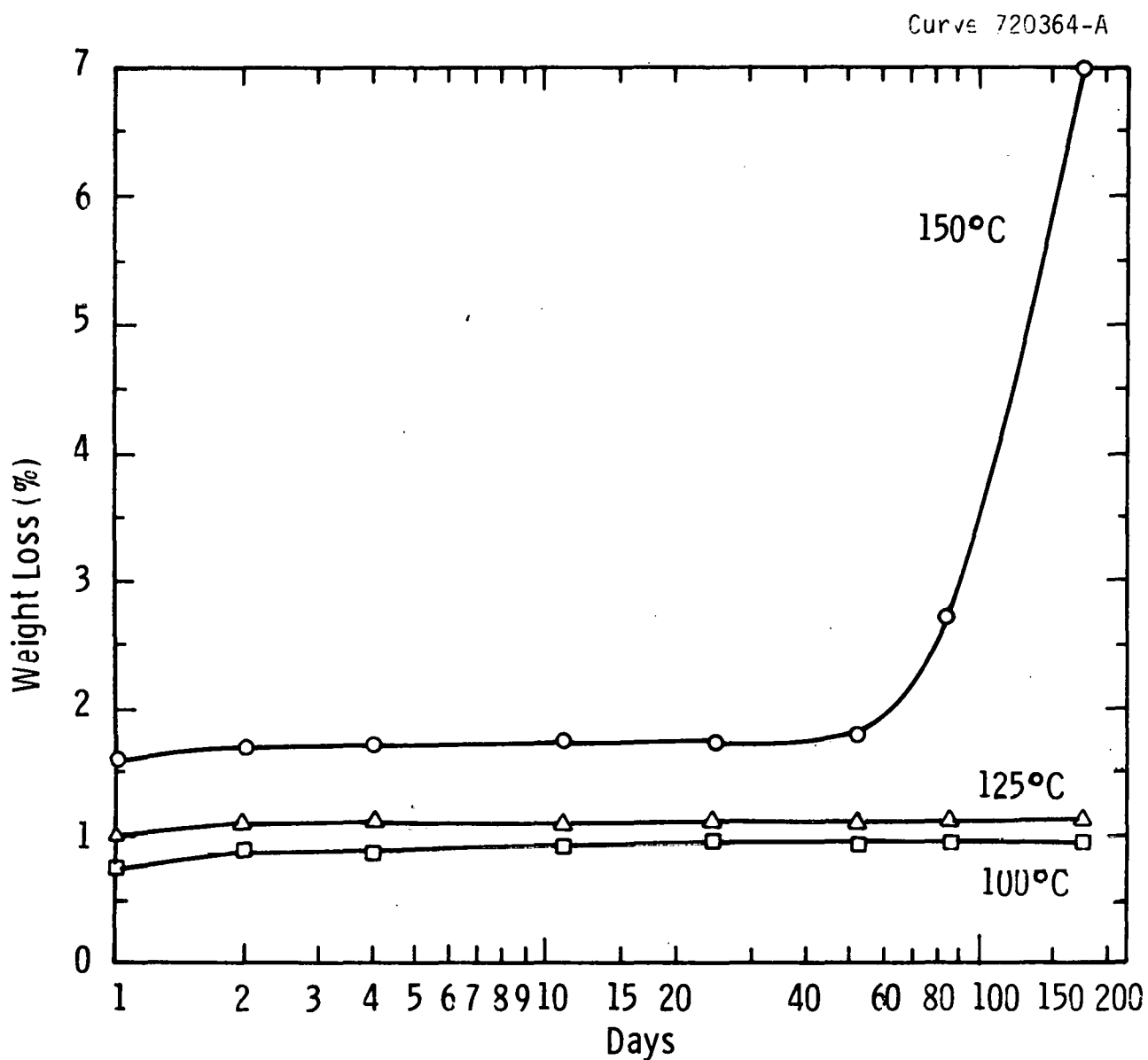


Fig. 33 — Weight loss resulting from aging ethylene-propylene terpolymer (EPDM) Nordel 3300-11 in air.

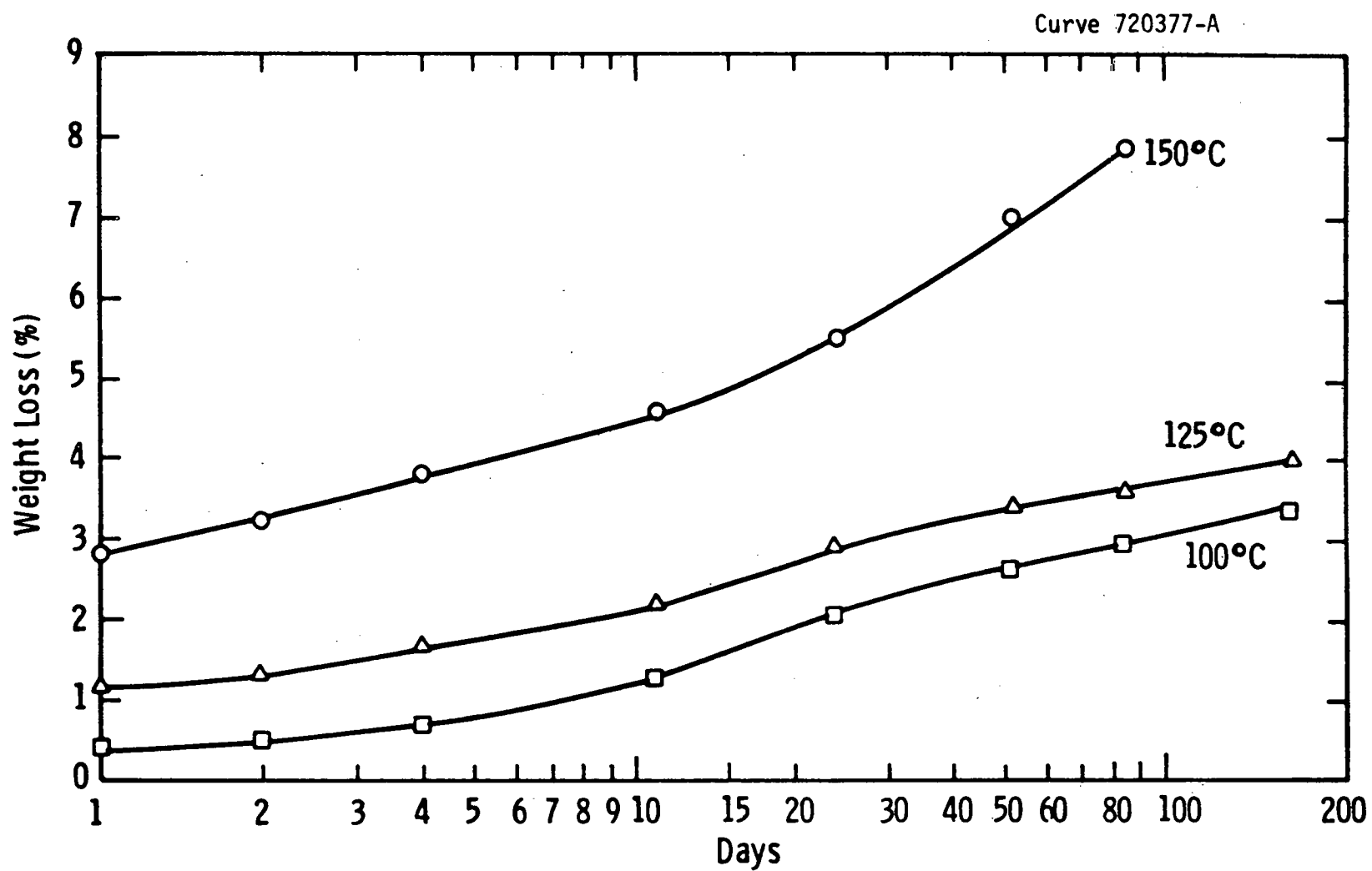


Fig. 34 — Weight loss resulting from aging butyl rubber 8EX 123 in air

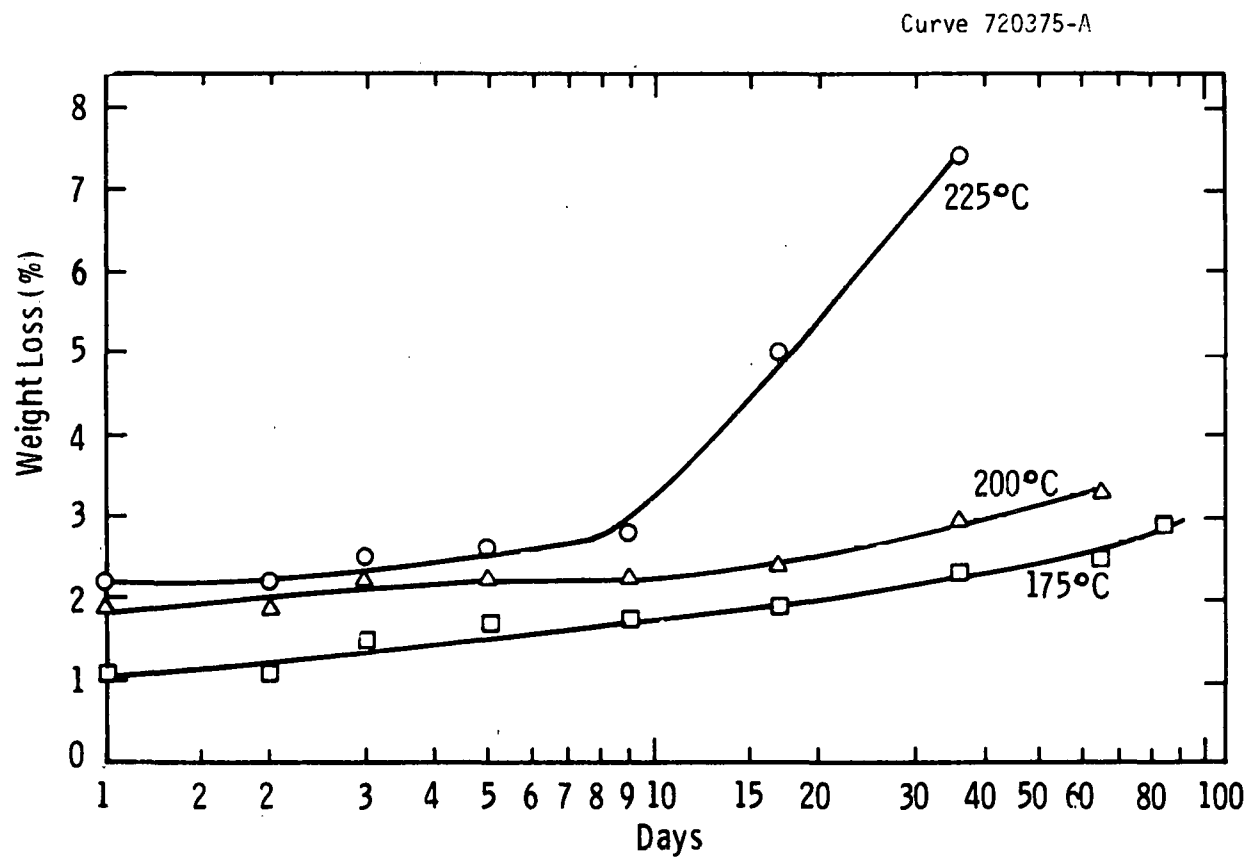


Fig. 35 — Weight loss resulting from aging silicone DC-732 (white) caulk compound in air

Curve 720370-A

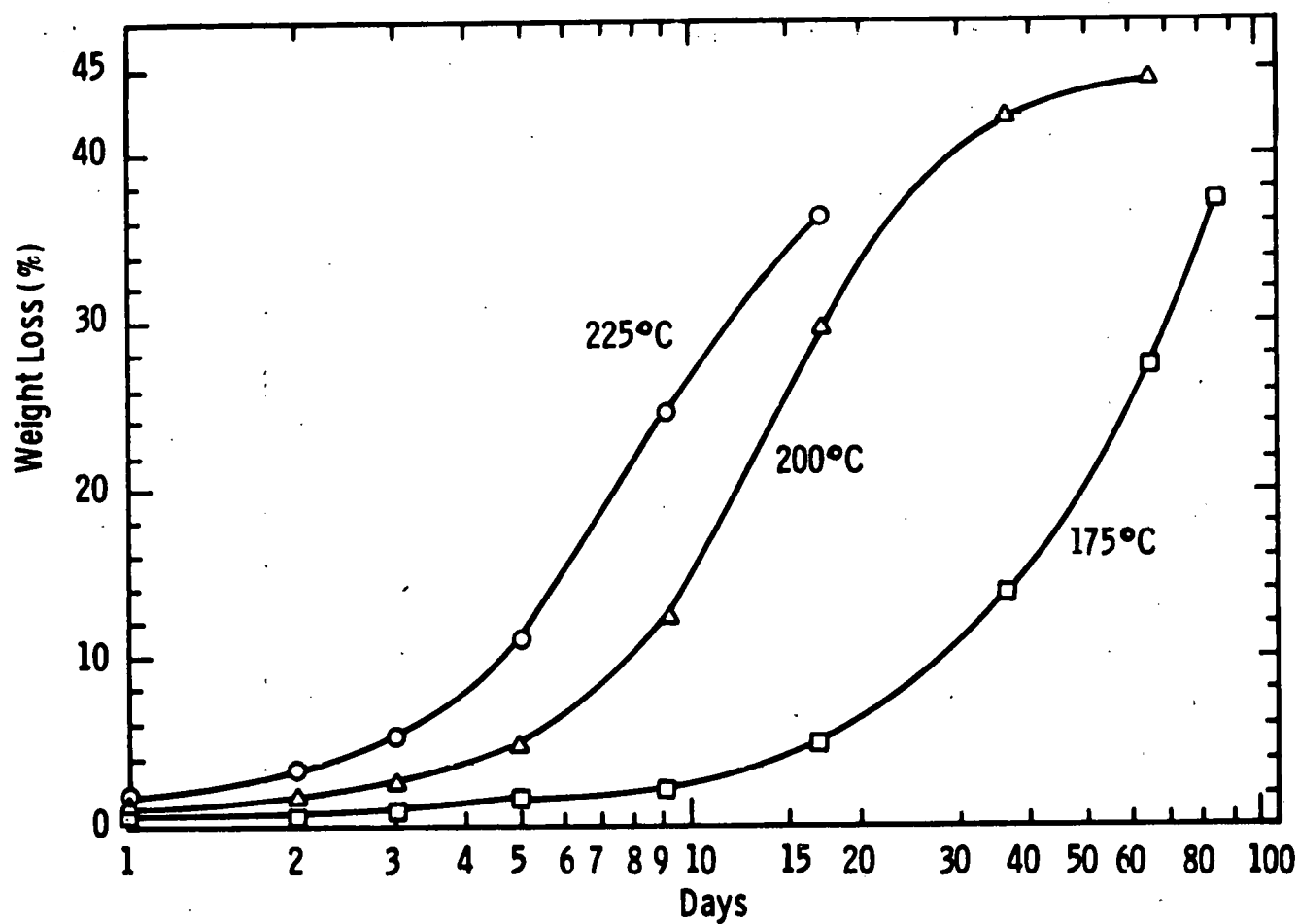


Fig. 36 — Weight loss resulting from aging silicone DC790 caulk compound in air.

Curve 720378-A

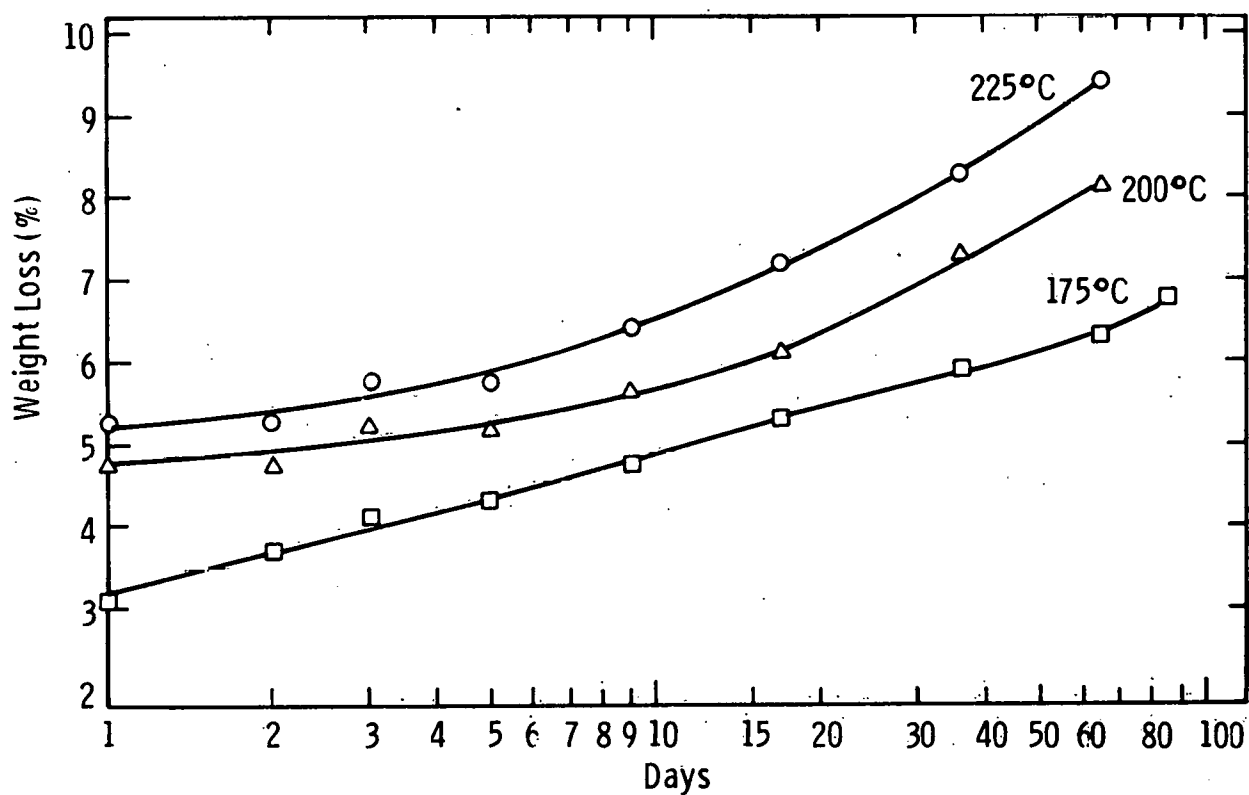


Fig. 37 — Weight loss resulting from aging silicone RTV-103 caulk compound in air.

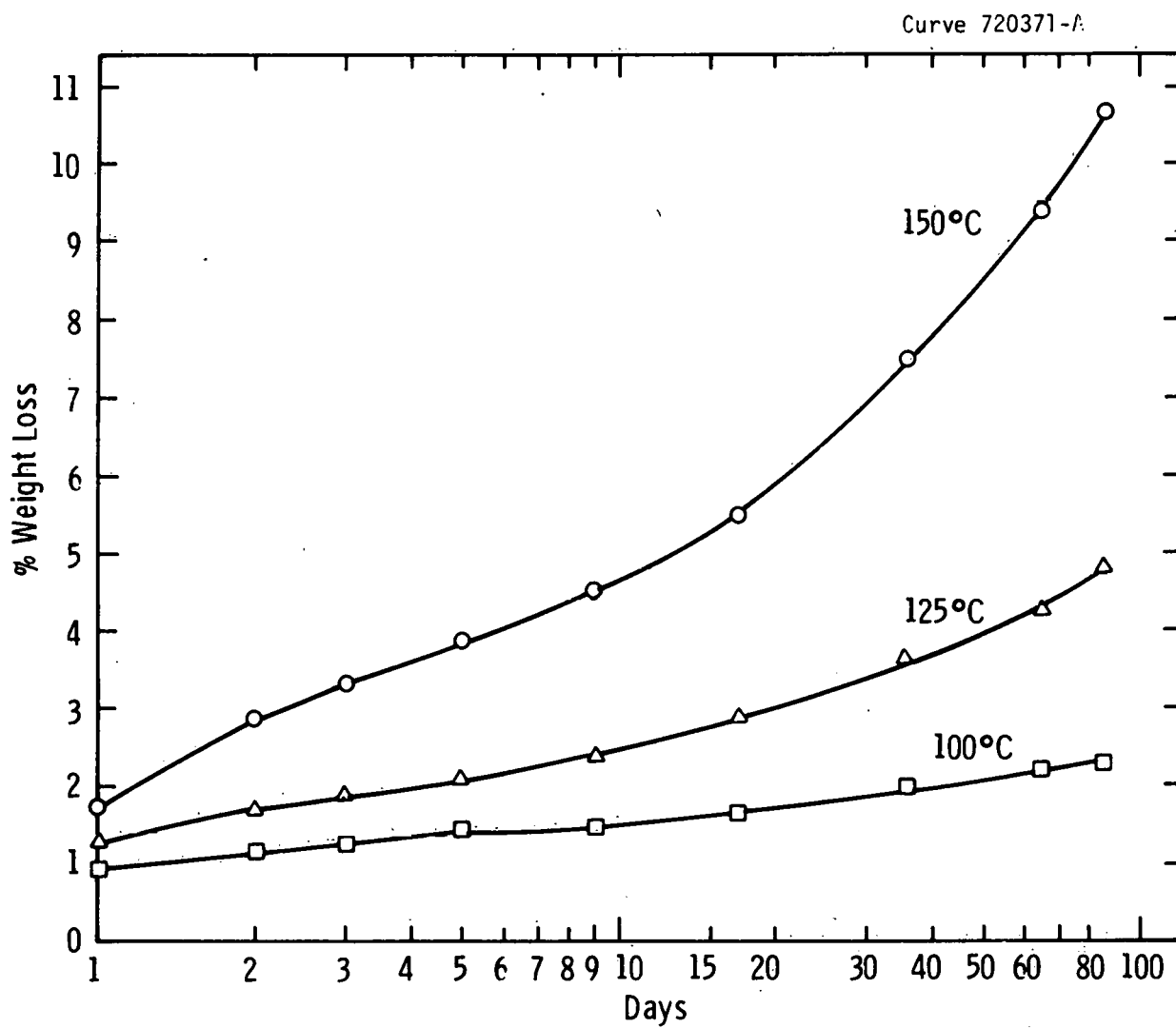


Fig. 38 — Weight loss resulting from aging acrylic terpolymer (Mono-Tremco) caulk compound in air

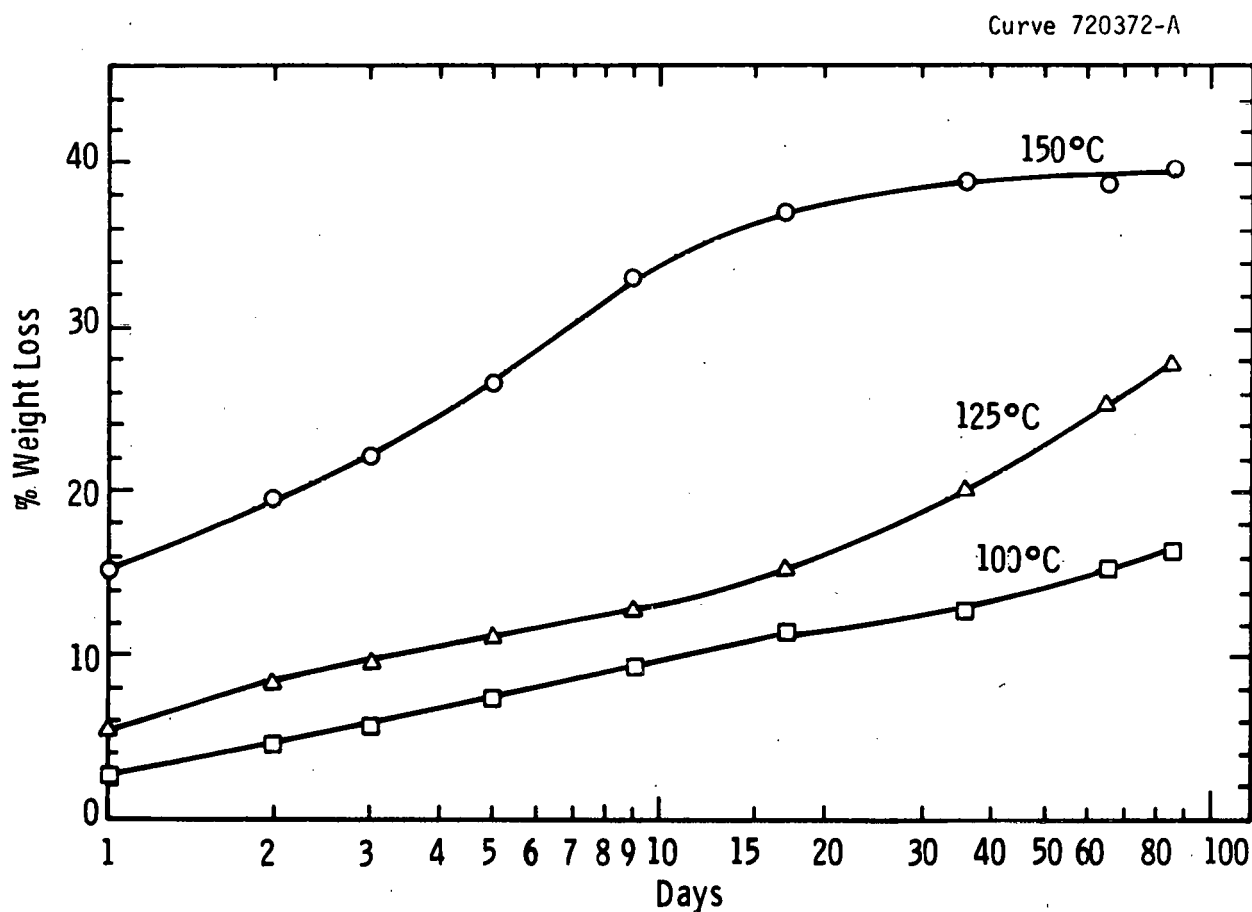


Fig. 39 — Weight loss resulting from aging Hypalon (G & H) caulk compound in air

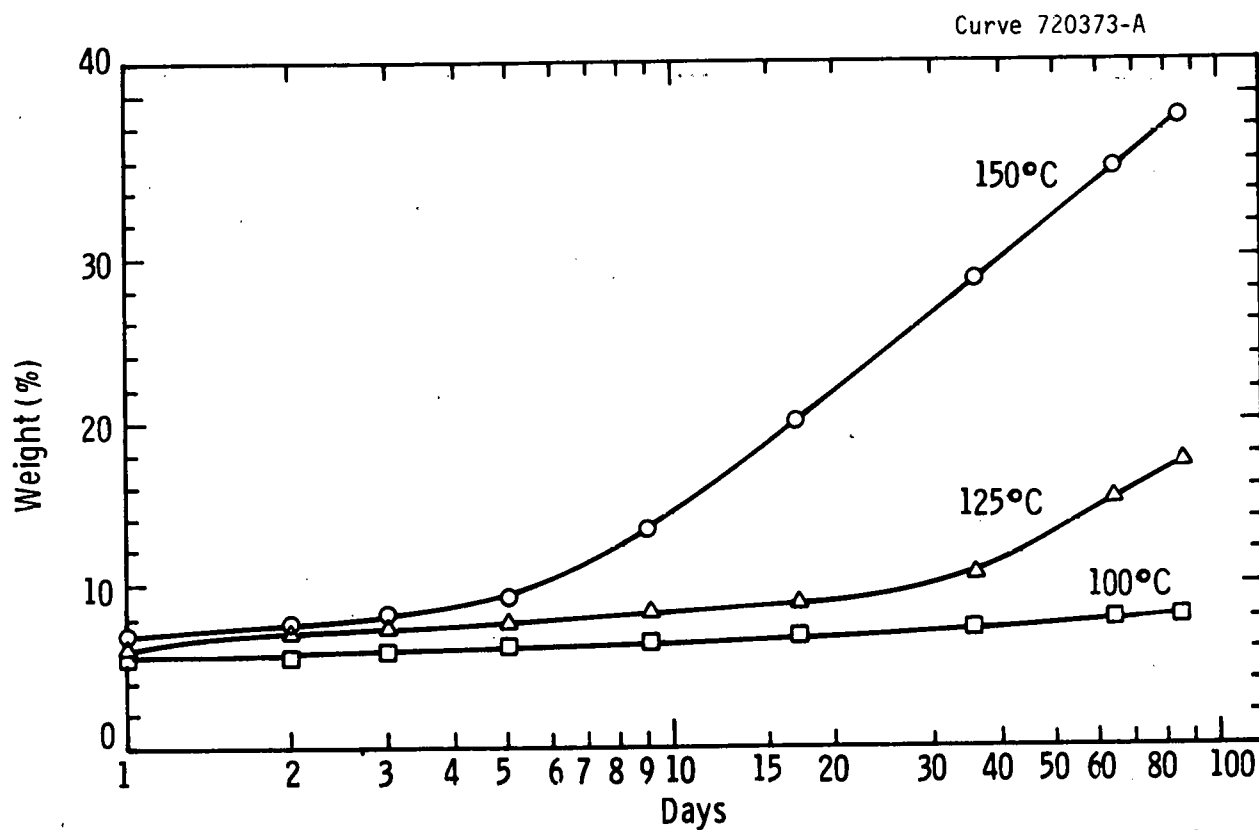


Fig. 40 — Weight loss resulting from aging butyl (Tremco) caulk compound in air

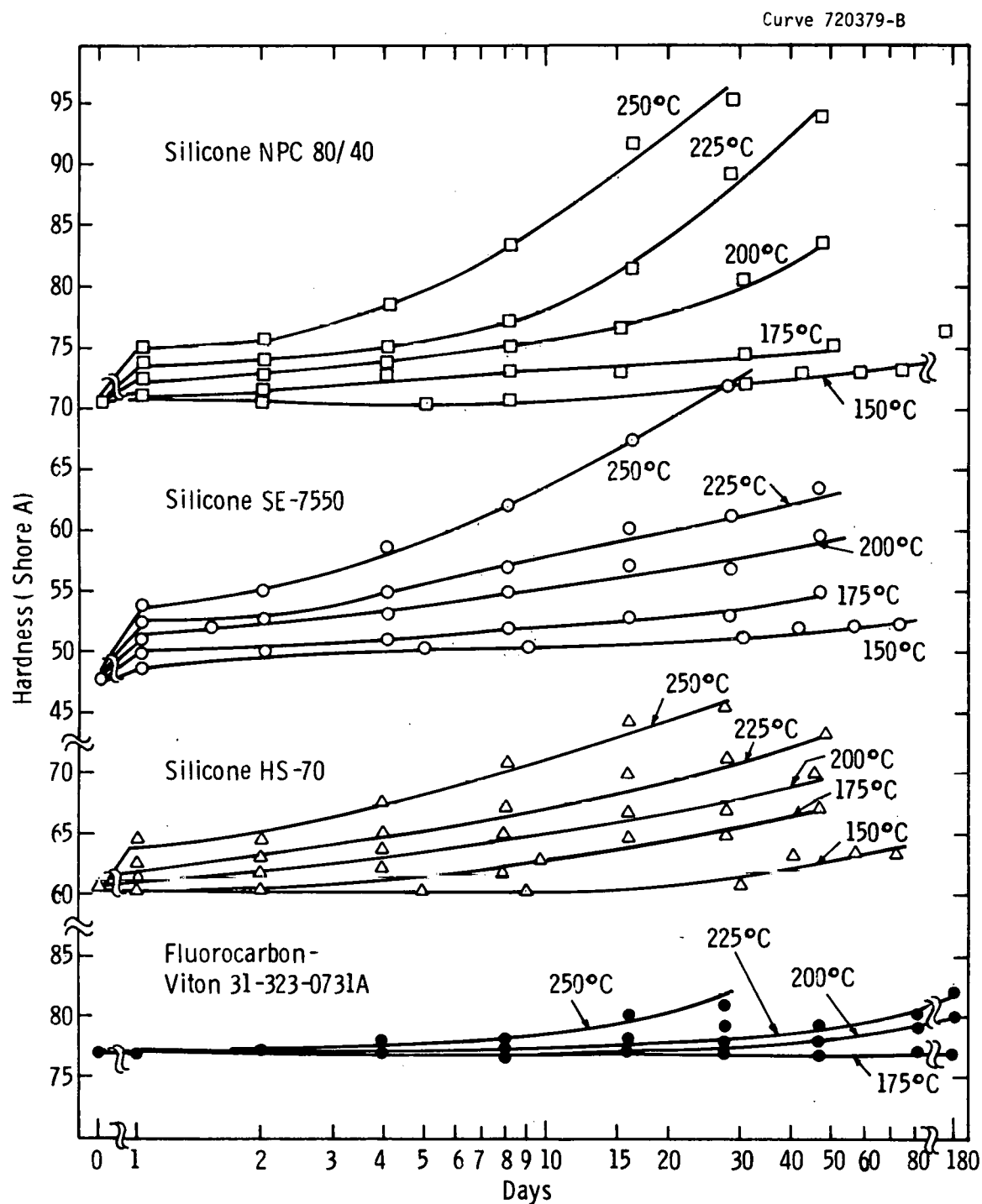


Fig. 41 — Effect of thermal aging in air on hardness of high temperature preformed sheet elastomers.

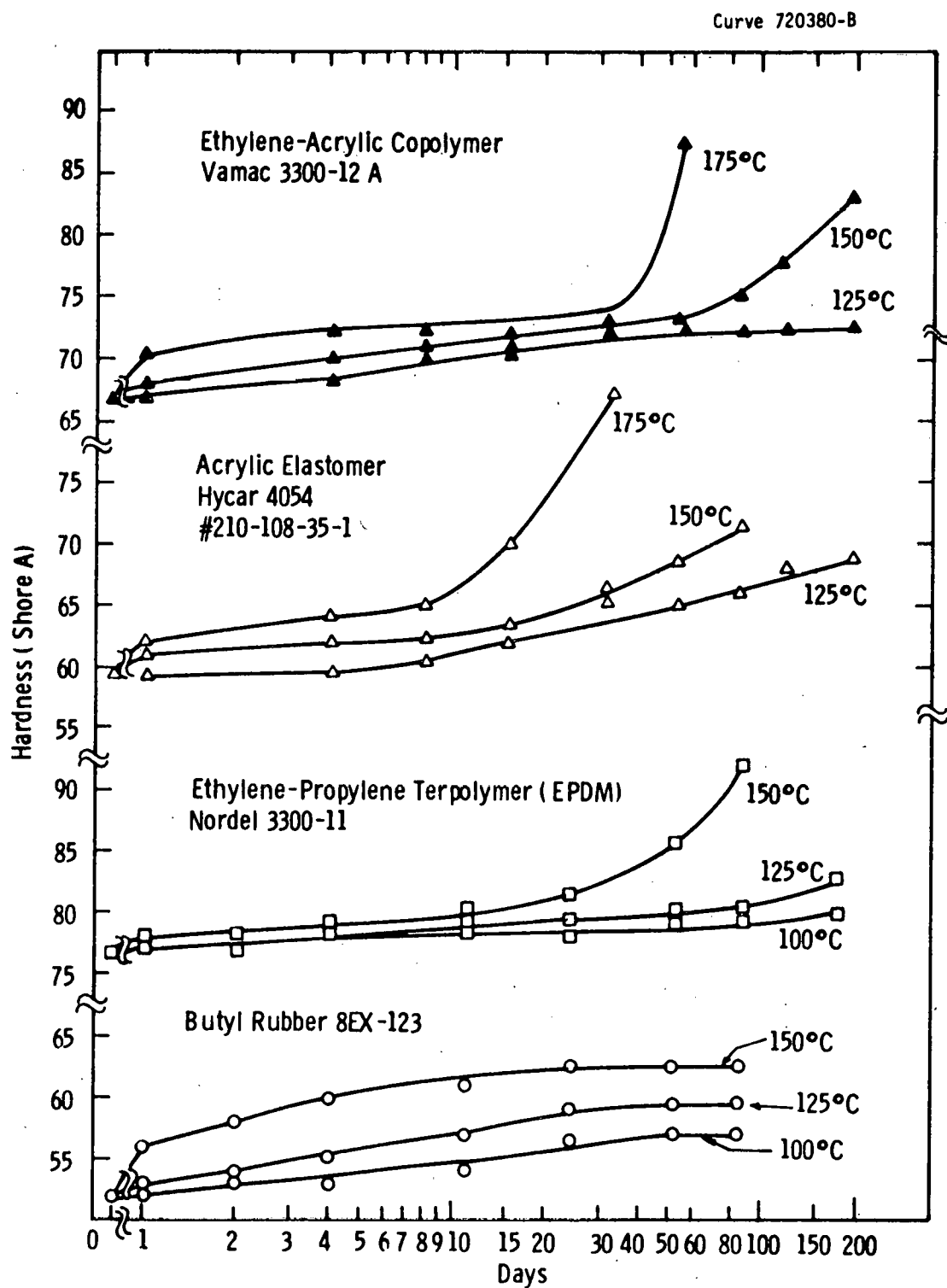


Fig. 42 — Effect of thermal aging in air on hardness of intermediate temperature preformed sheet elastomers

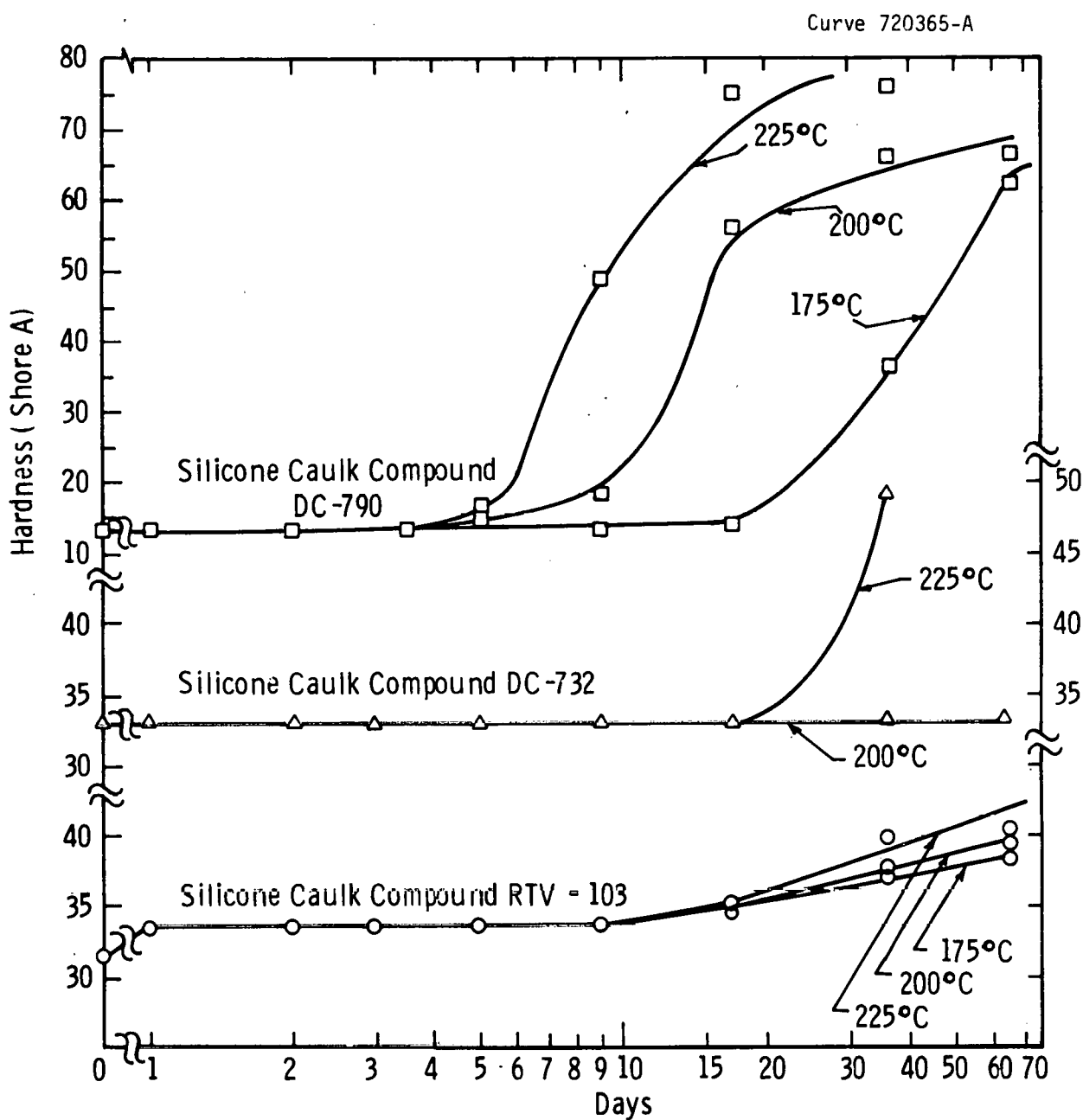


Fig. 43 — Effect of thermal aging in air on hardness of silicone caulk compounds

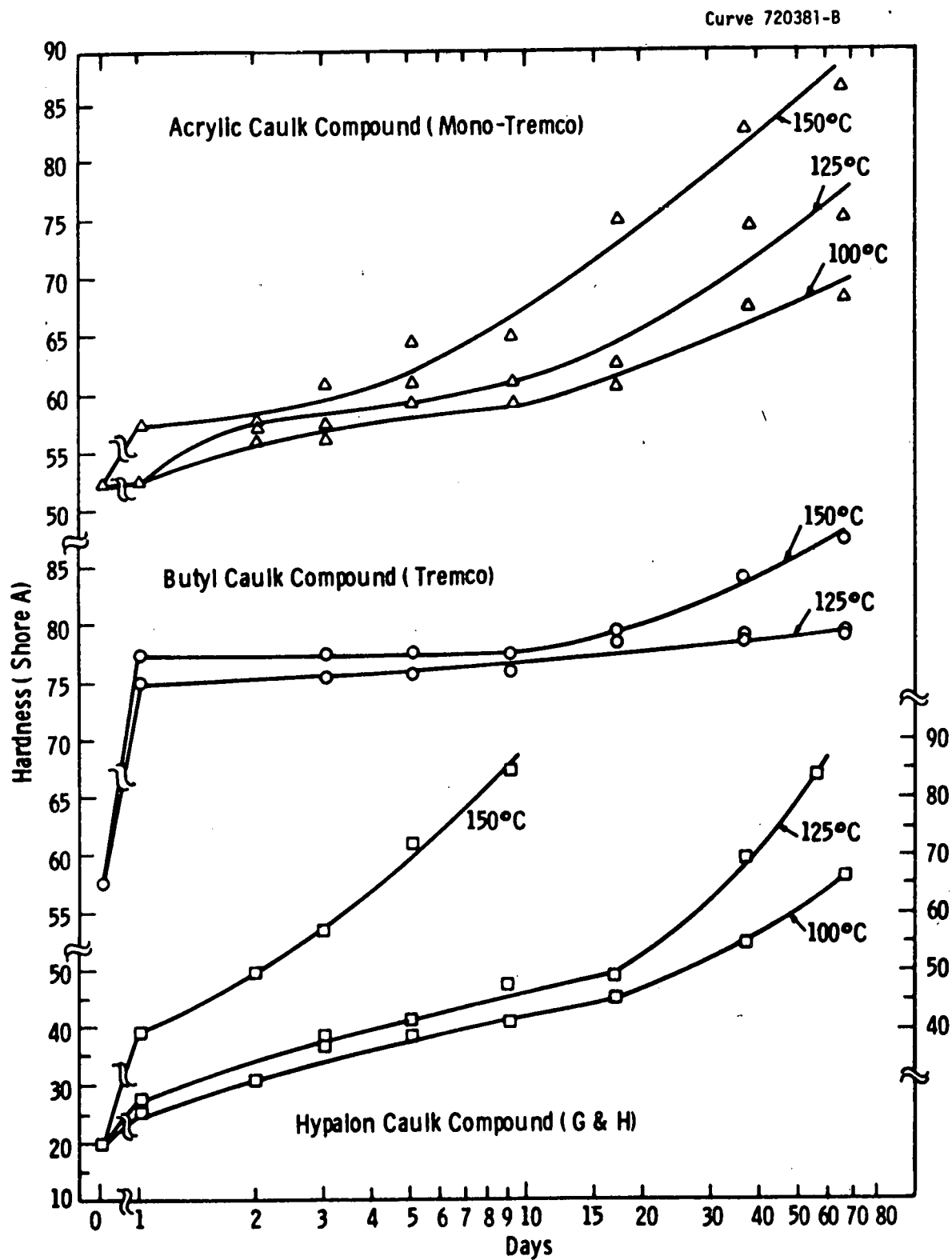


Fig. 44 — Effect of thermal aging in air on hardness of caulk compounds.

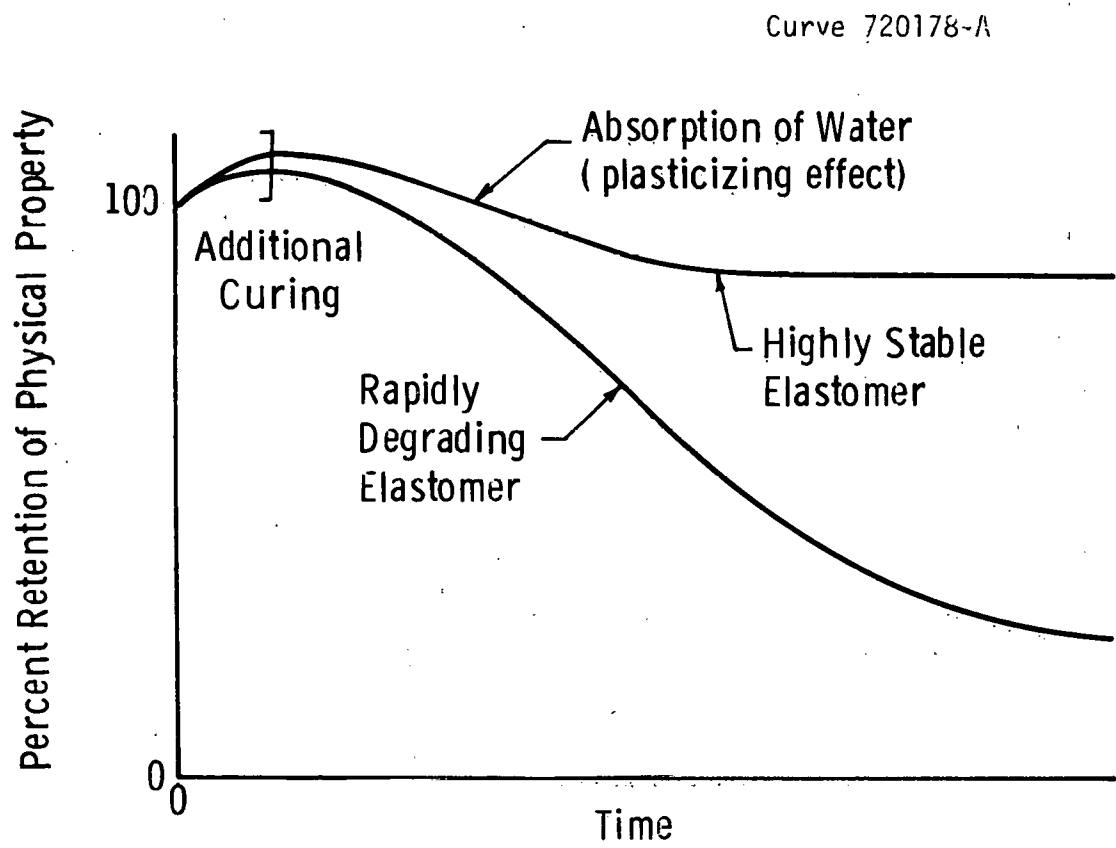


Fig.45 — Typical effects of combination of high humidity and high temperature on elastomers

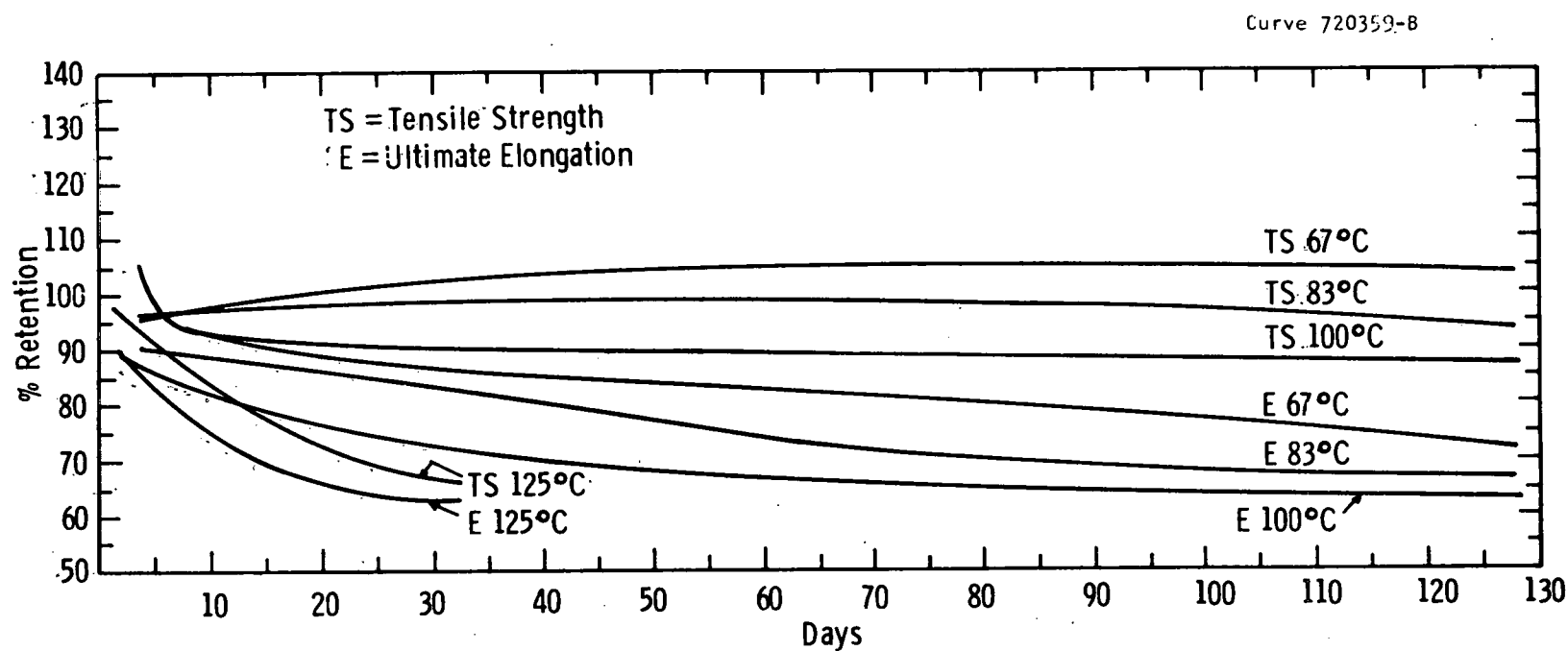


Fig. 46 — Hydrolytic aging of silicone rubber NPC 80/40

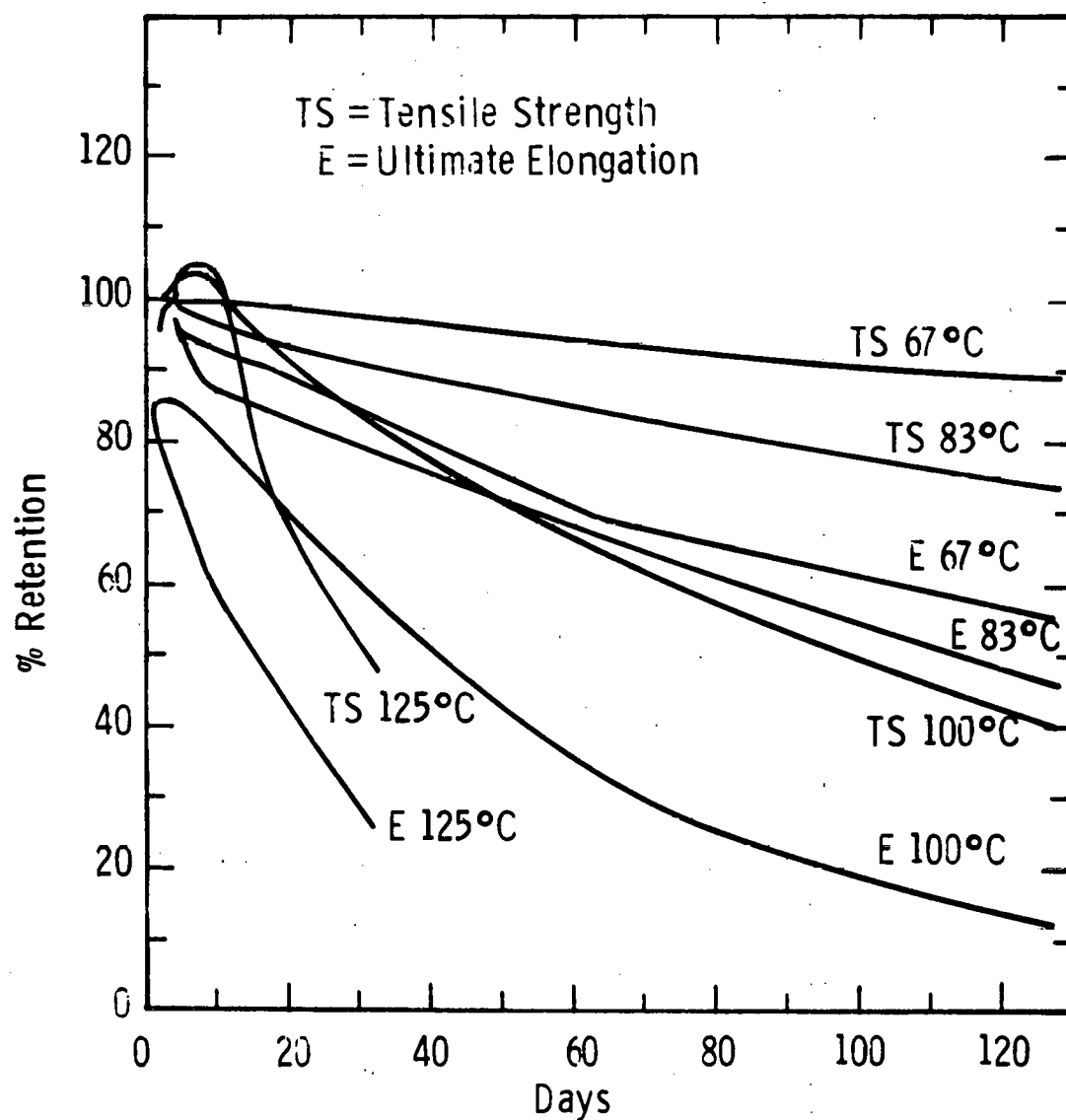


Fig. 47 — Hydrolytic aging of silicone rubber HS-70

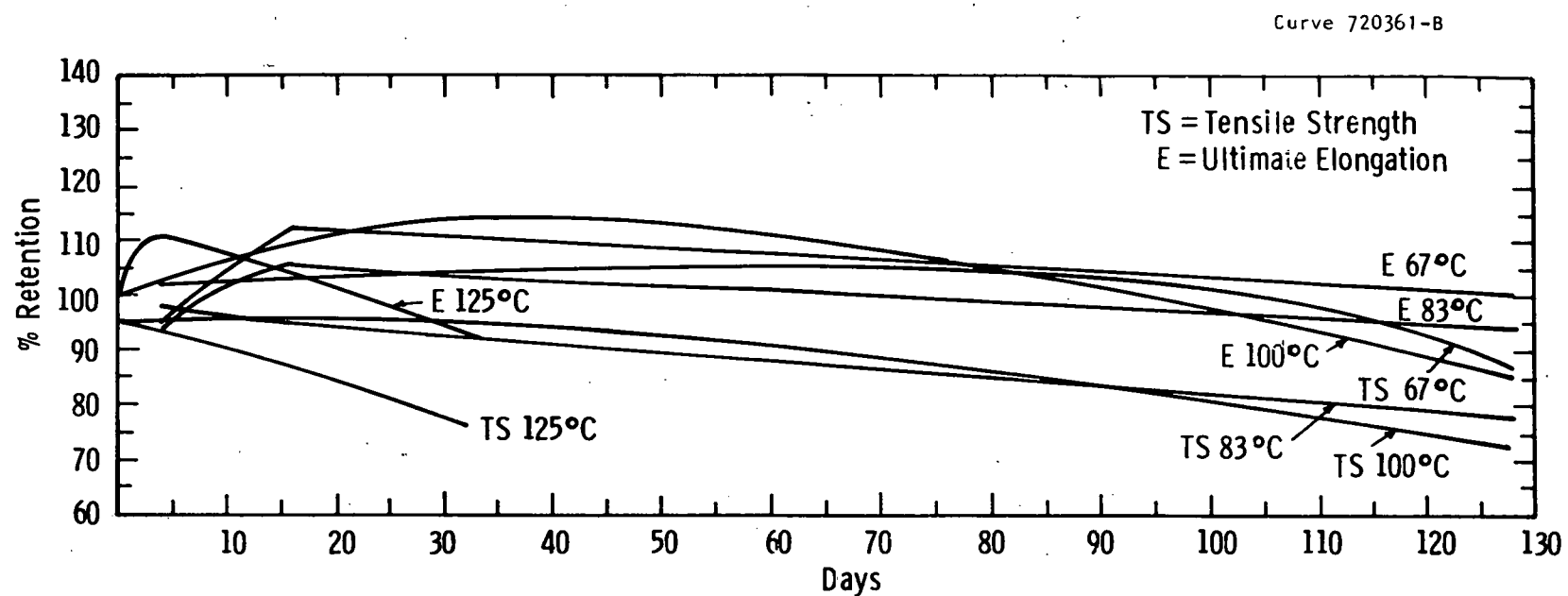


Fig. 48 — Hydrolytic aging of fluorocarbon elastomer Viton 31-323-0731A

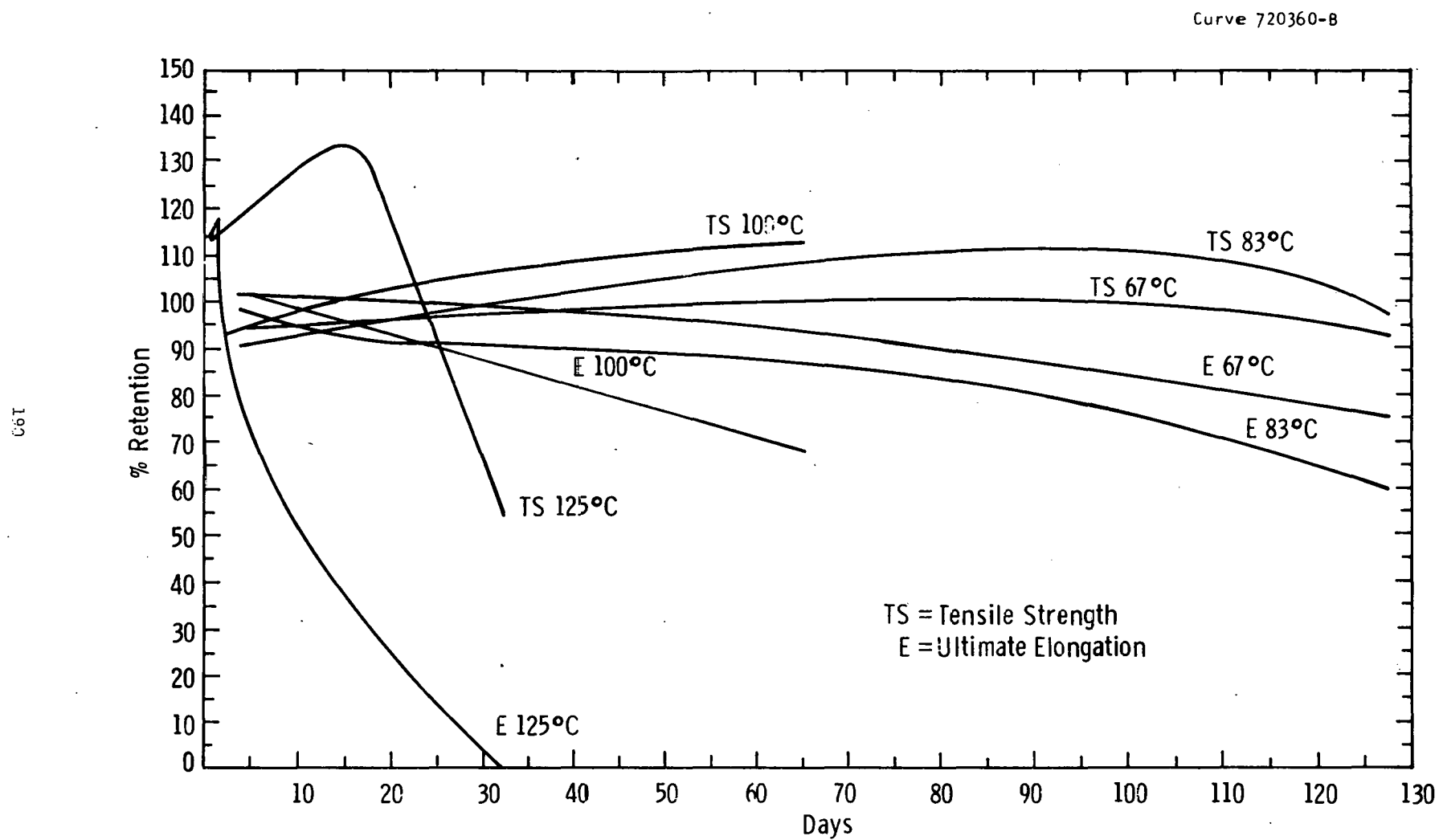


Fig. 49 - Hydrolytic aging of ethylene-acrylic copolymer Vamac 3300-12A

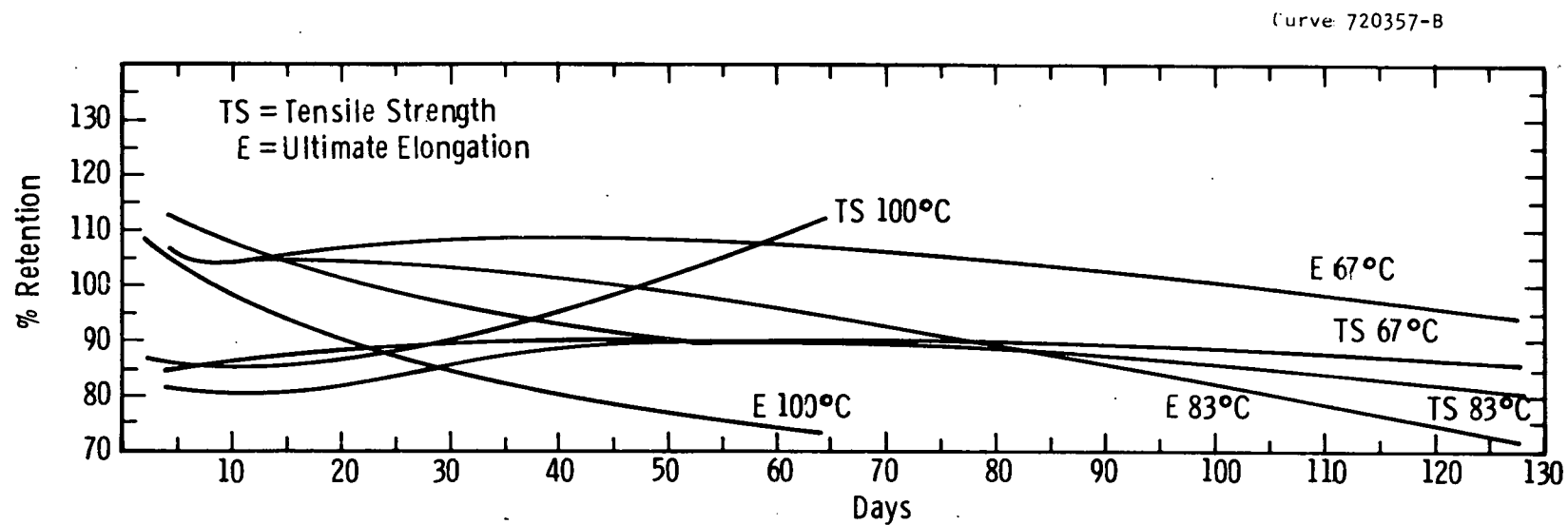


Fig. 50 — Hydrolytic aging of acrylic elastomer Hycar 4054 210-108-35-1

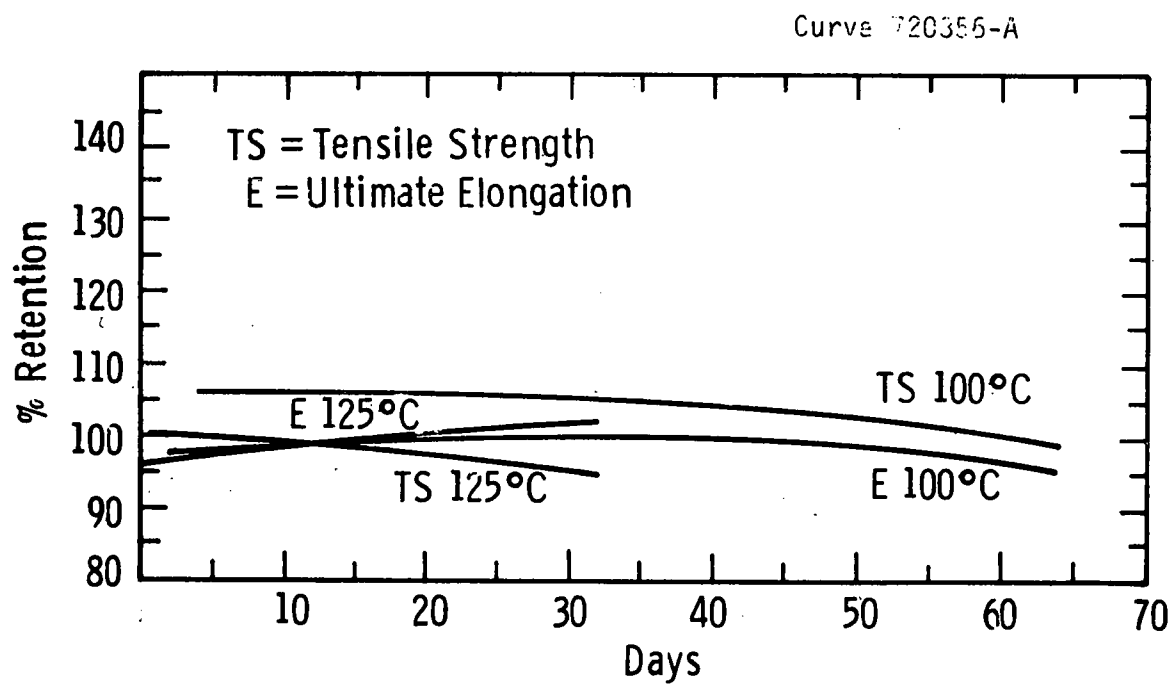


Fig. 51 — Hydrolytic aging of ethylene-propylene terpolymer (EPDM) Nordel 3300-11

Curve 720358-B

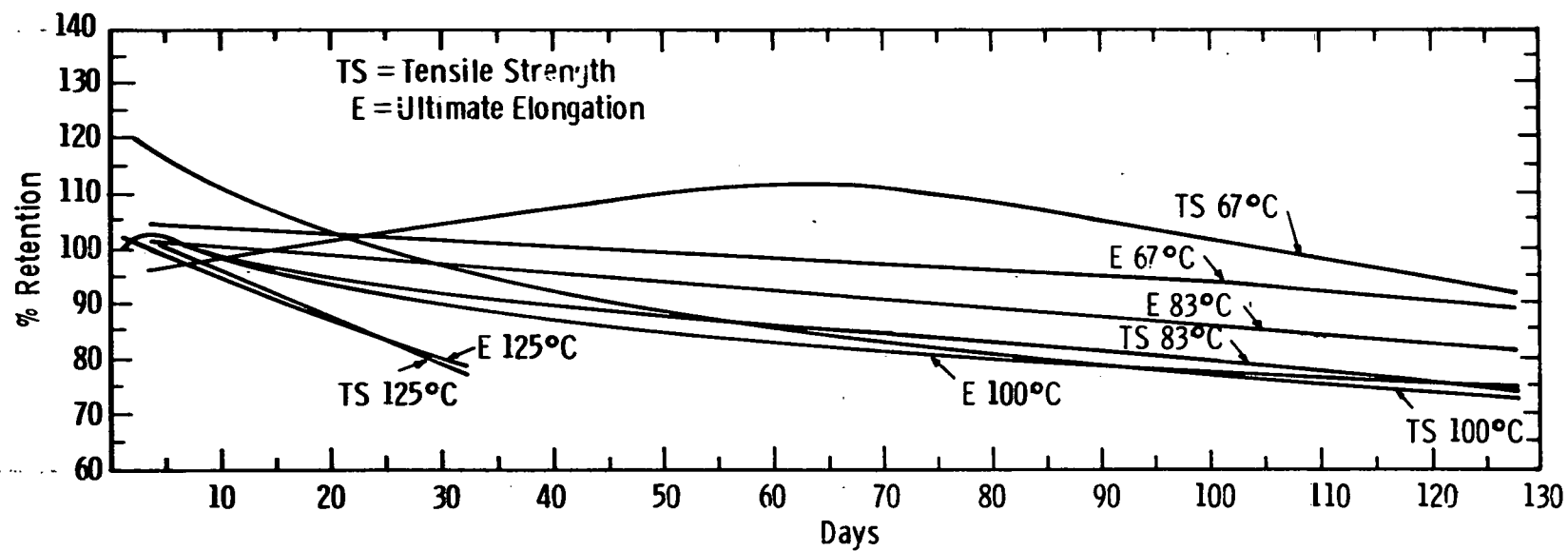


Fig. 52 — Hydrolytic aging of butyl rubber 8EX-123

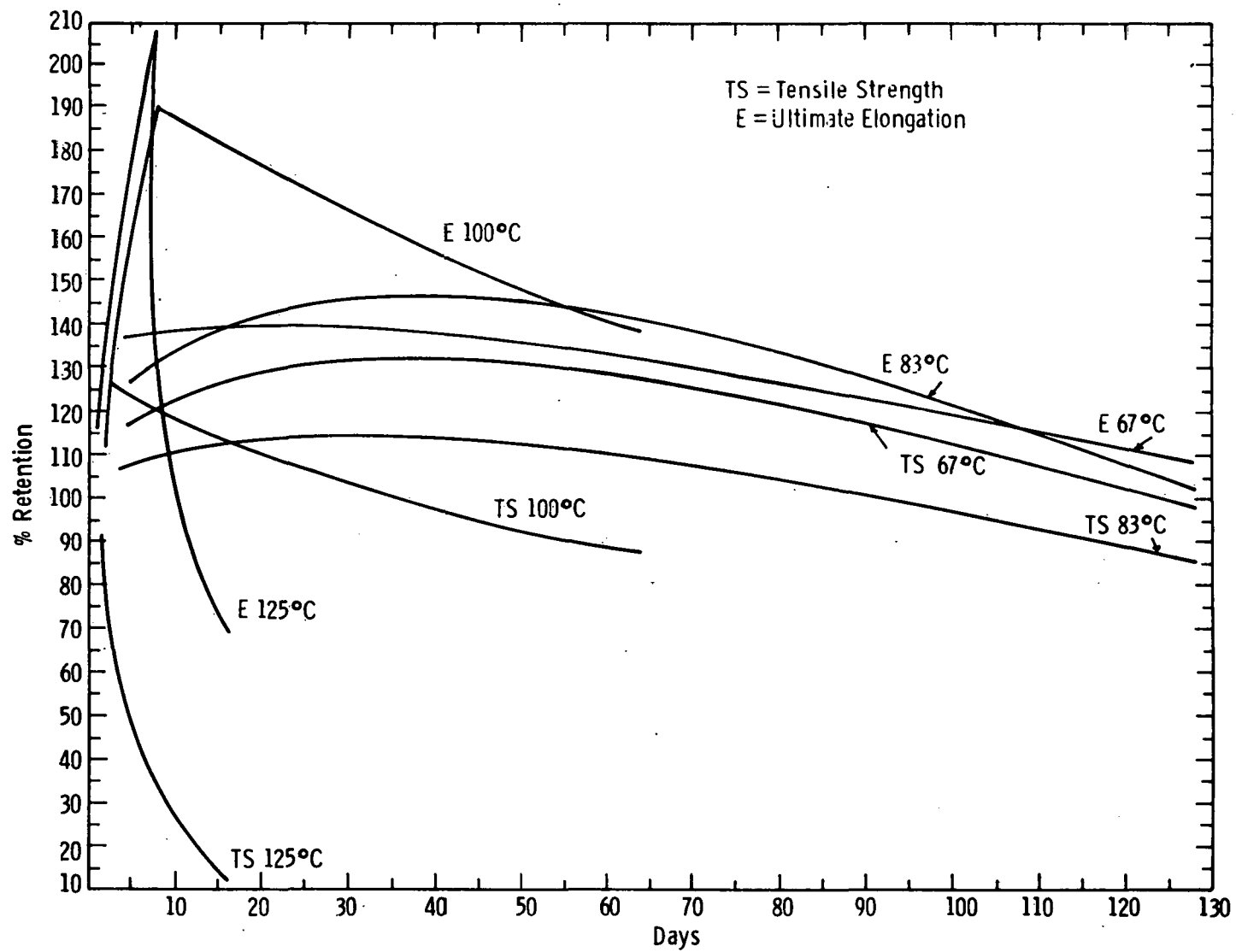


Fig. 53 — Hydrolytic aging of silicone caulking compound DC-732

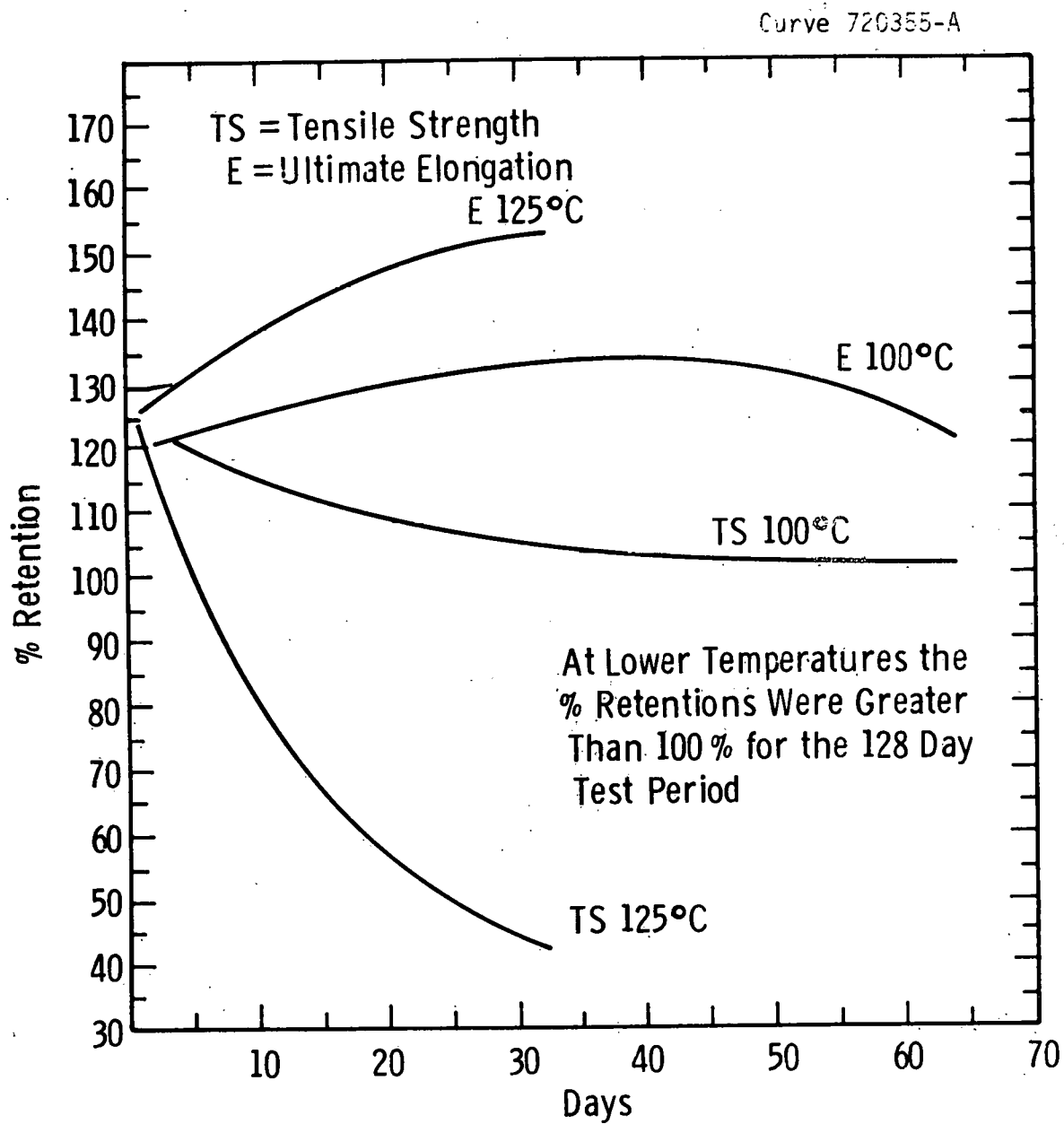


Fig. 54 — Hydrolytic aging of silicone caulking compound RTV-103

The Sunshine Carbon Arc Lamp

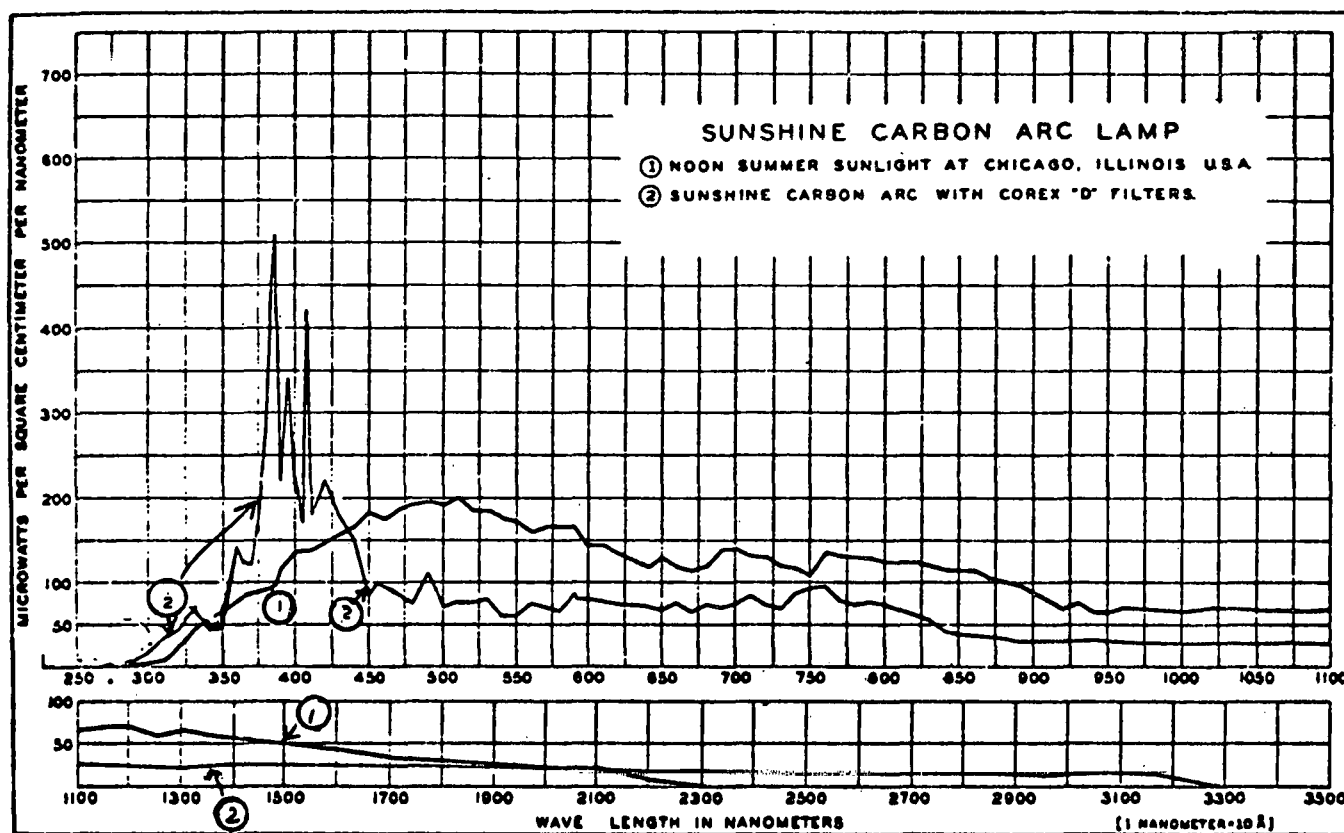
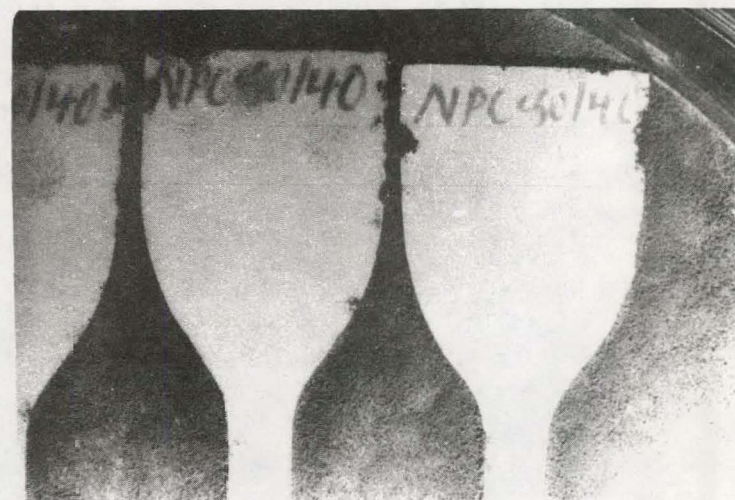


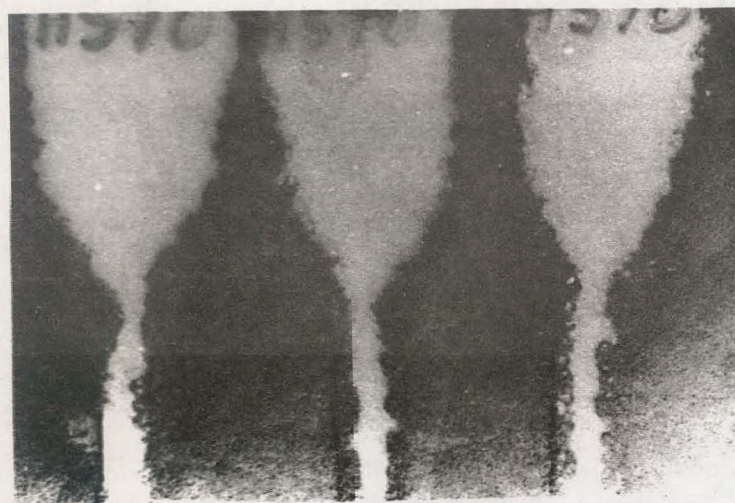
Fig. 55—Light sources — energy distribution curves



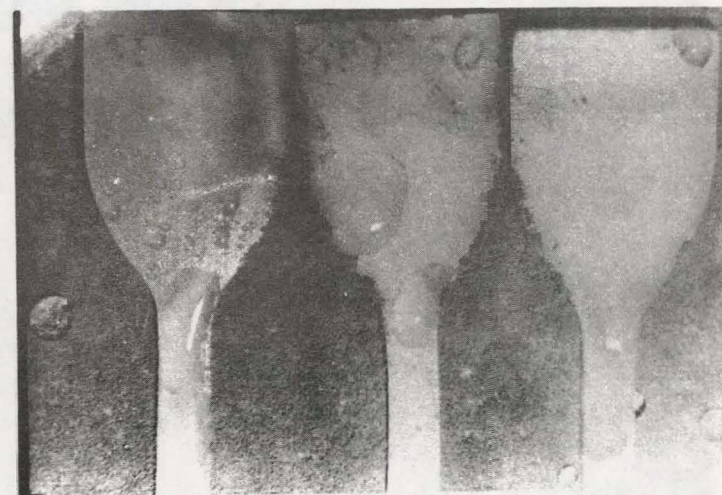
FLUOROCARBON ELASTOMER VITON 31-323-0731A



SILICONE RUBBER NPC 80/40



SILICONE RUBBER HS-70

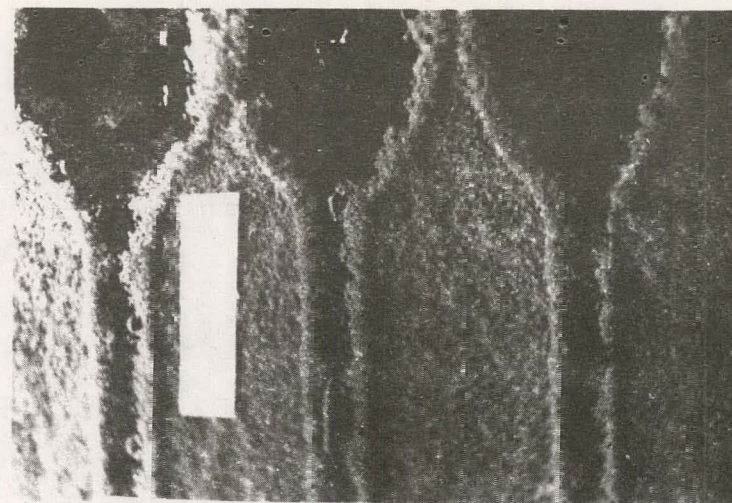


SILICONE RUBBER SE7550

FIG. 56A -- FUNGAL GROWTH ON SEALANTS.



ETHYLENE-PROPYLENE TERPOLYMER (EPDM)
NORDEL 3300-11



ETHYLENE-ACRYLIC COPOLYMER VAMAC 3300-12A

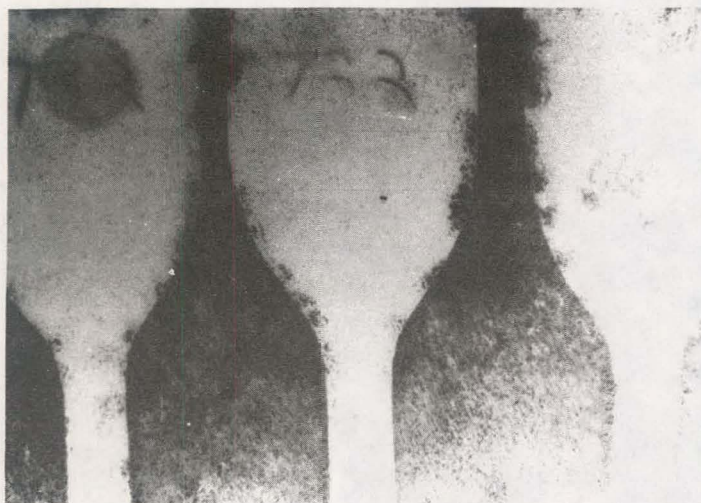


ACRYLIC ELASTOMER Hycar 4054 210-108-35-1

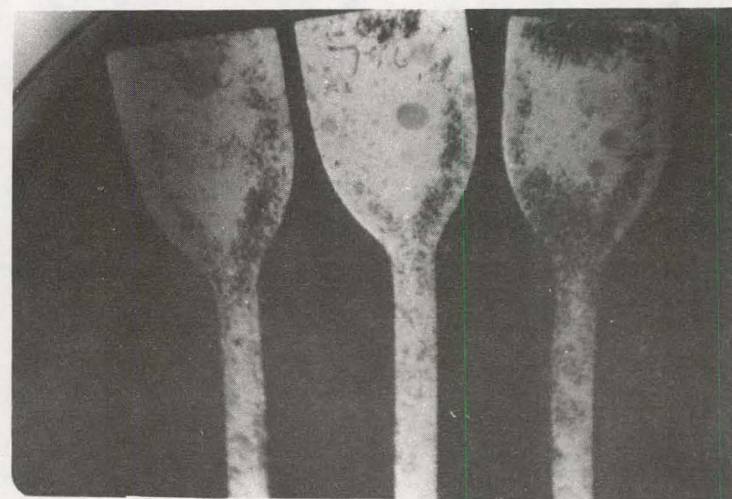


BUTYL RUBBER 8EX123

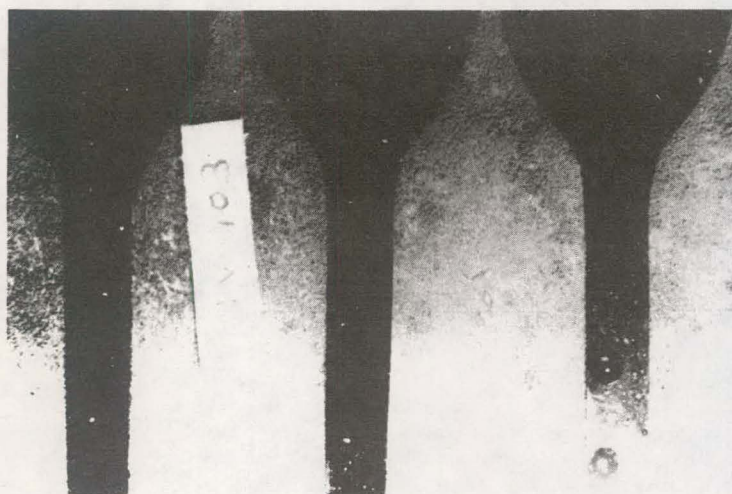
FIG. 56B -- FUNGAL GROWTH ON SEALANTS.



SILICONE CAULKING COMPOUND DC-732



SILICONE CAULKING COMPOUND DC-790

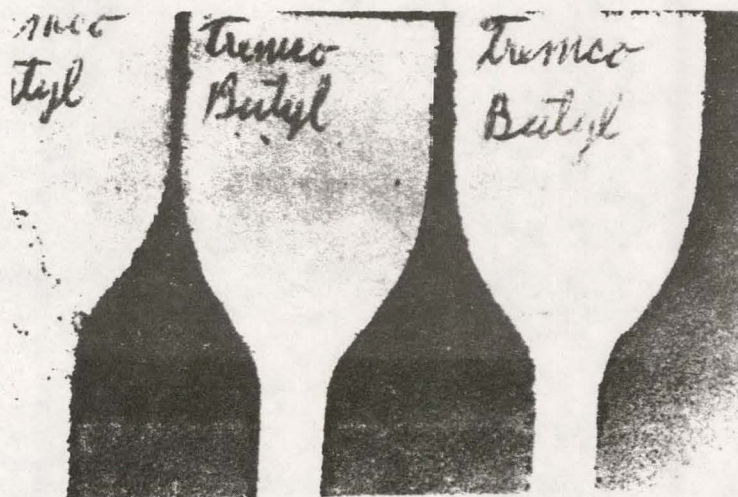


SILICONE CAULKING COMPOUND RTV-103

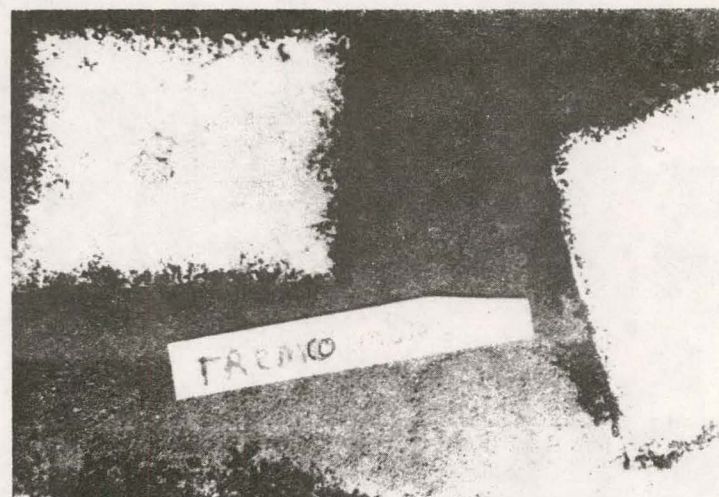


HYPALON CAULK COMPOUND (G+H)

FIG. 56C -- FUNGAL GROWTH ON SEALANTS.



BUTYL CAULK COMPOUND (TREMCO)



ACRYLIC CAULK COMPOUND (MONG-TREMCO)

FIG. 56D -- FUNGAL GROWTH ON SEALANTS.

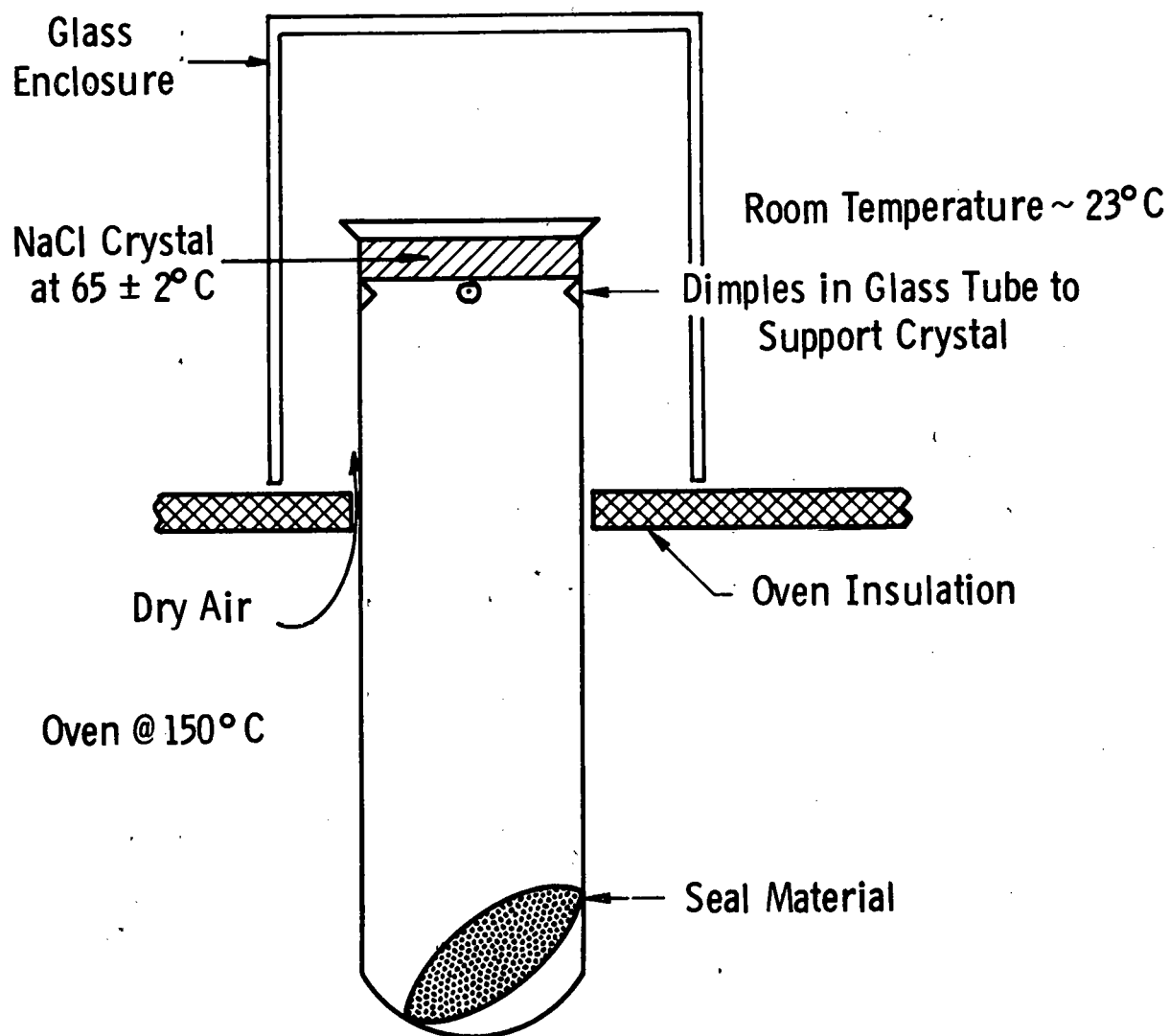


Fig.57 - Outgassing measurement apparatus

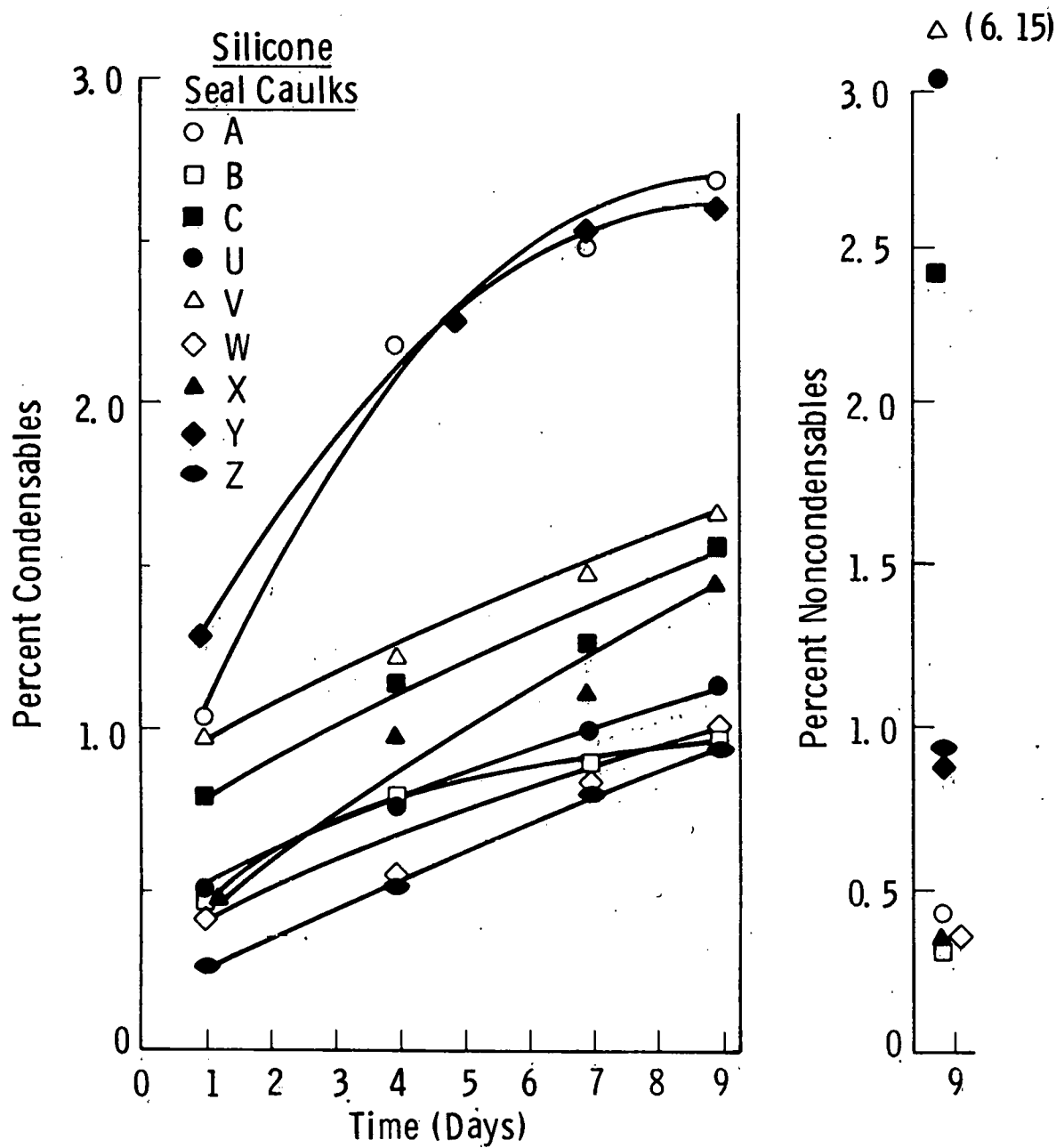


Fig.58 — Sealant aging at 150°C - percent condensables and noncondensables (percents are based on original weight of specimen)

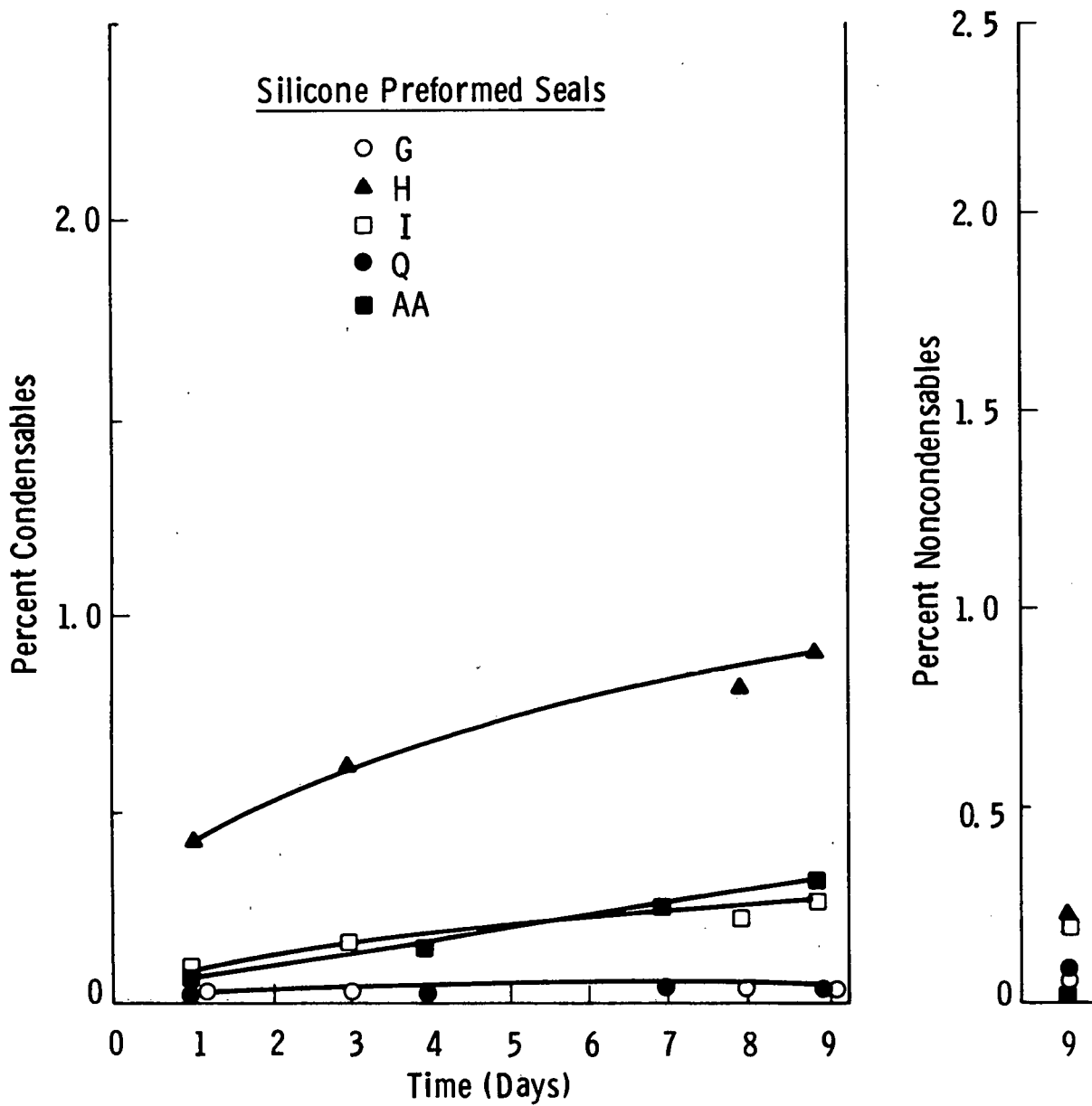


Fig. 59 — Sealant aging at 150°C - percent condensables and noncondensables (percents are based on original weight of specimen)

Curve 720343-B

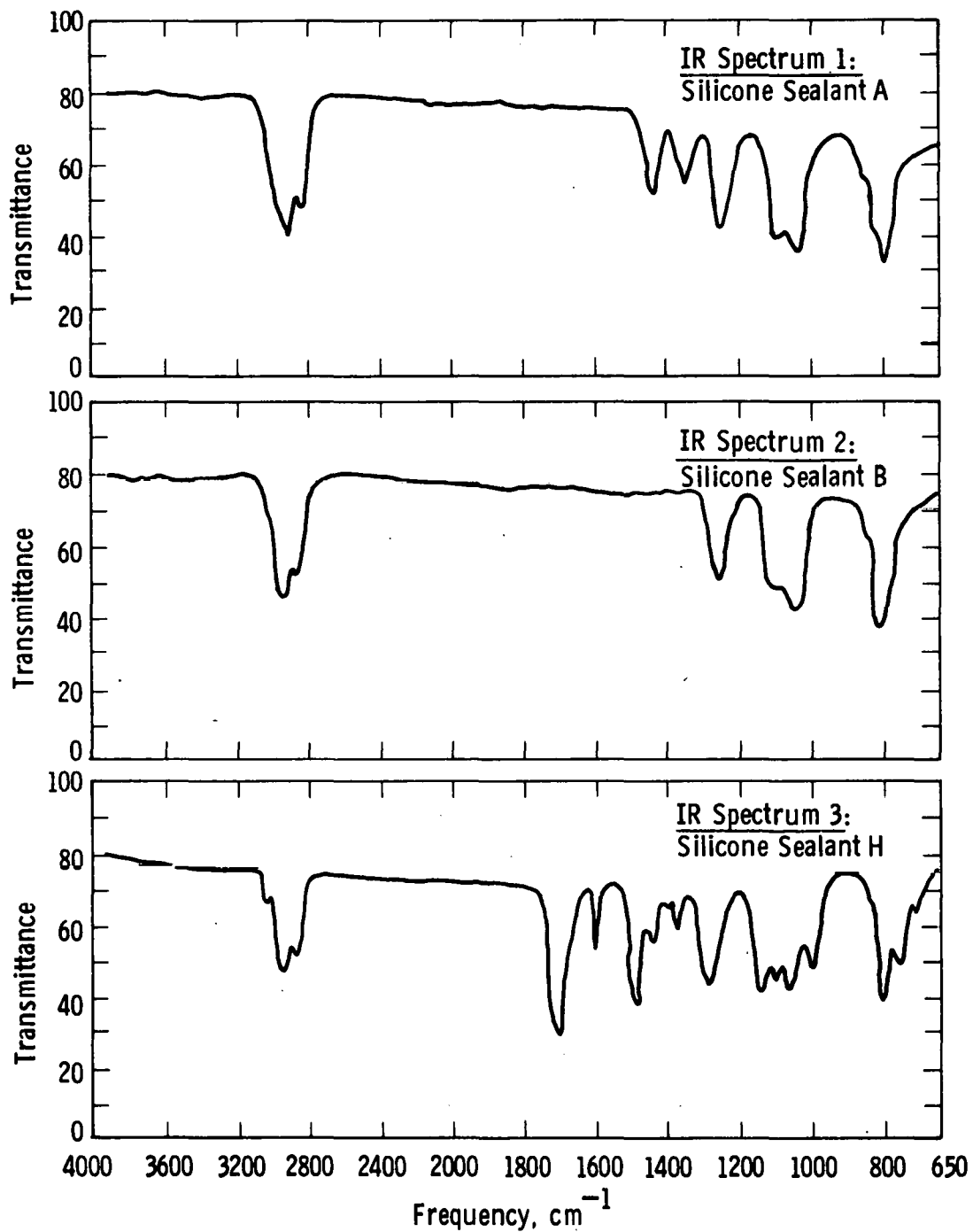


Fig. 60— Infrared spectra of condensable volatiles

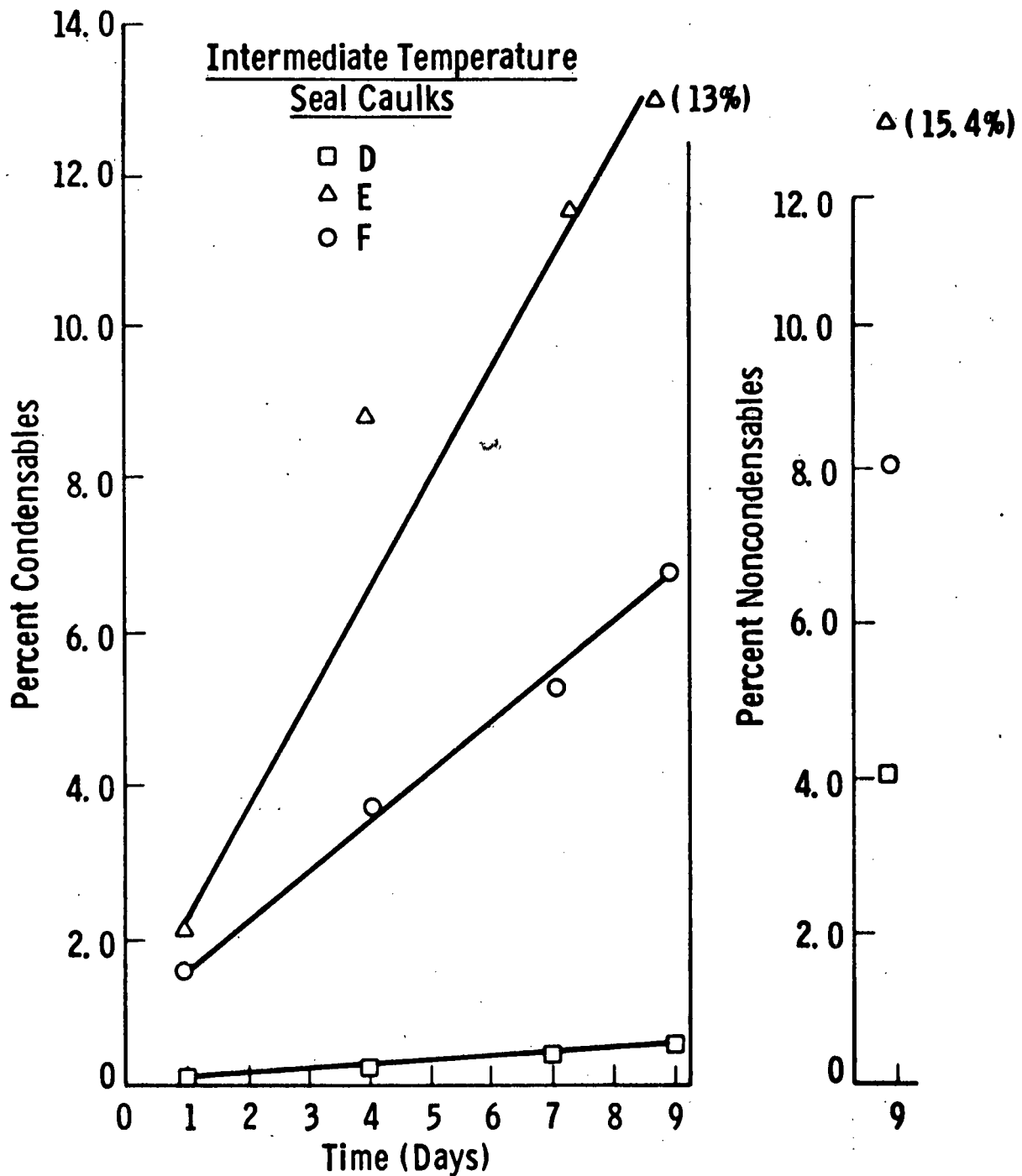


Fig. 61— Sealant aging at 150°C - percent condensables and noncondensables (percents are based on original weight of specimen)

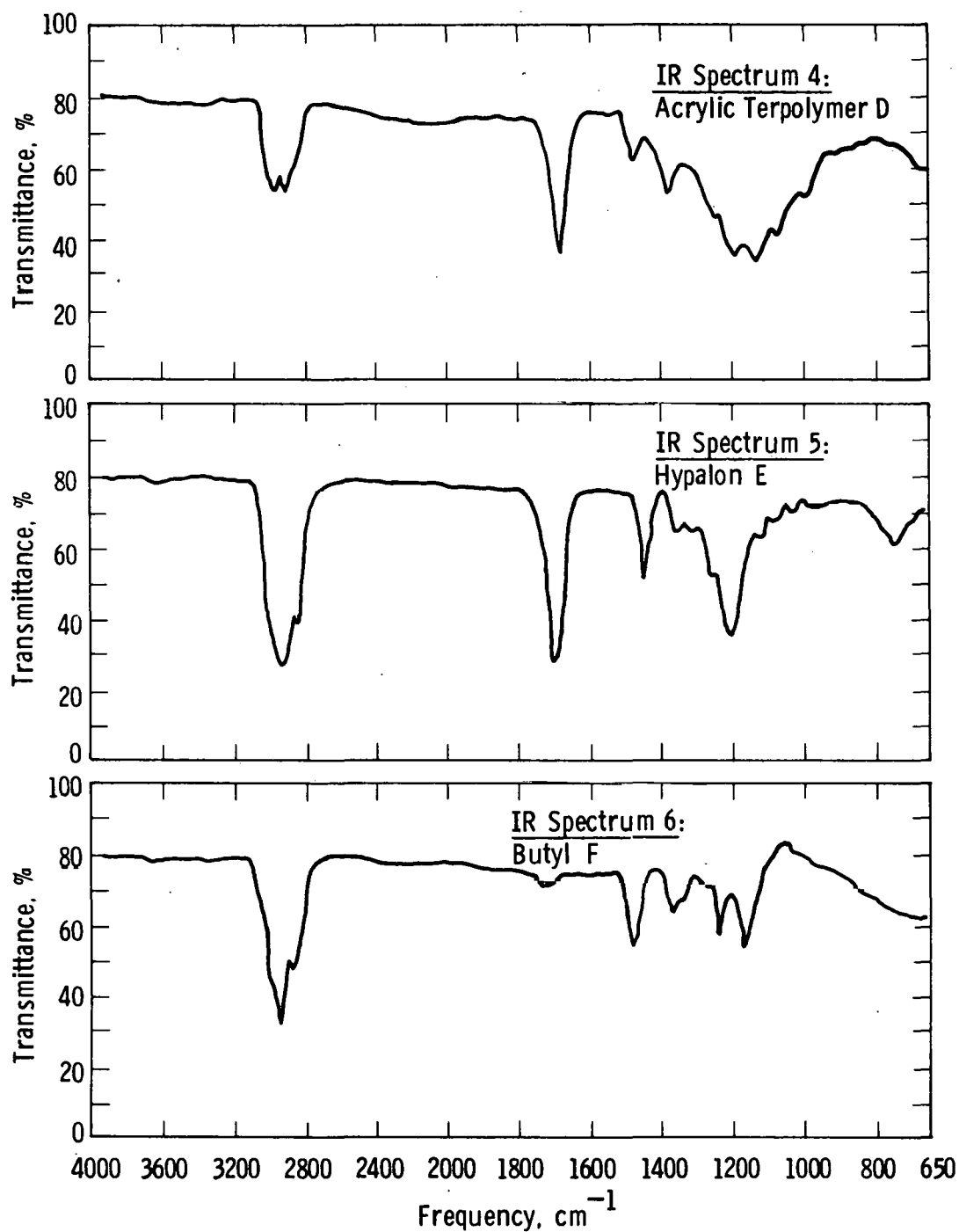


Fig.62— Infrared spectra of condensable volatiles

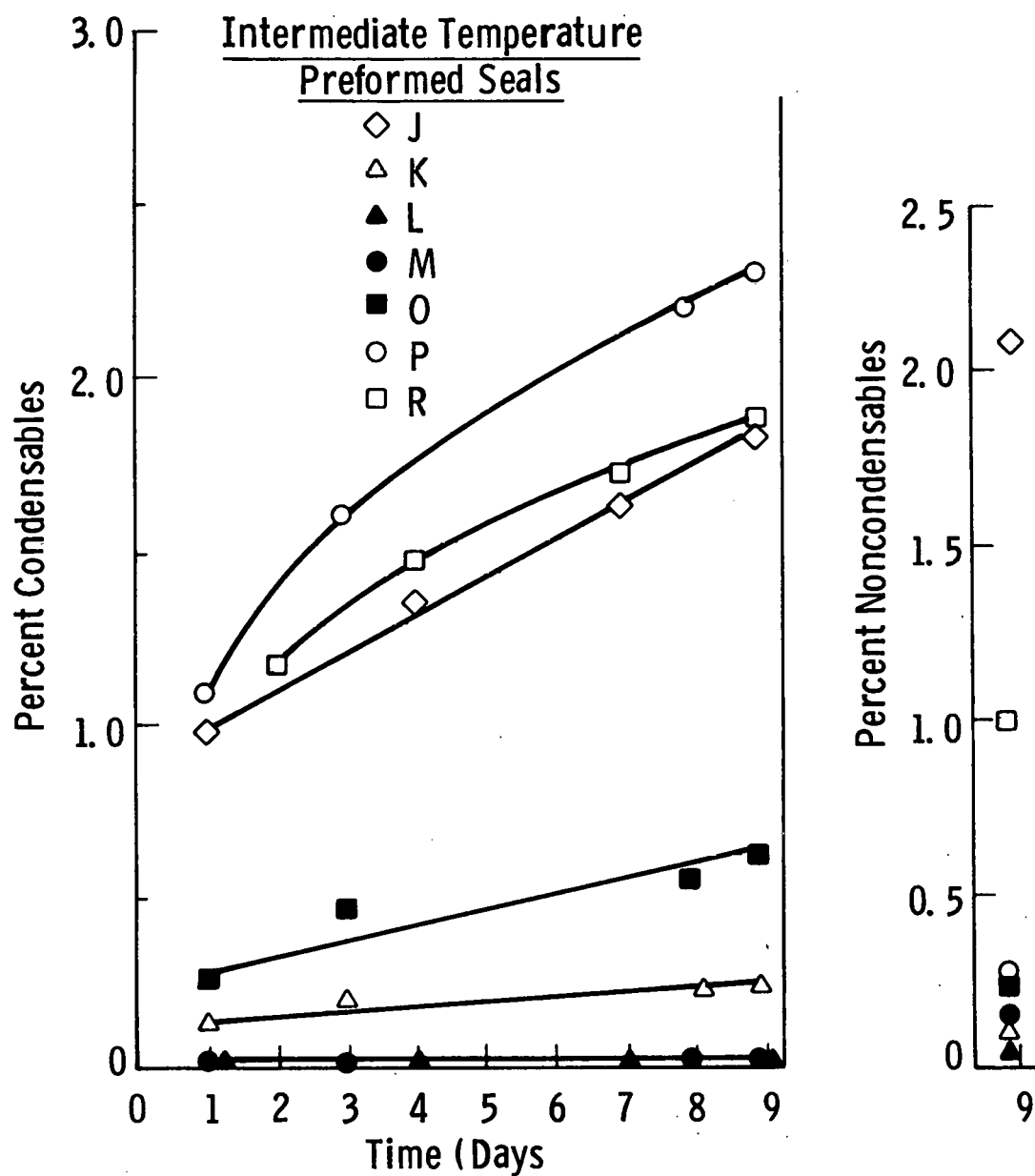


Fig. 63 — Sealant aging at 150°C - percent condensables and noncondensables (percents are based on original weight of specimen)

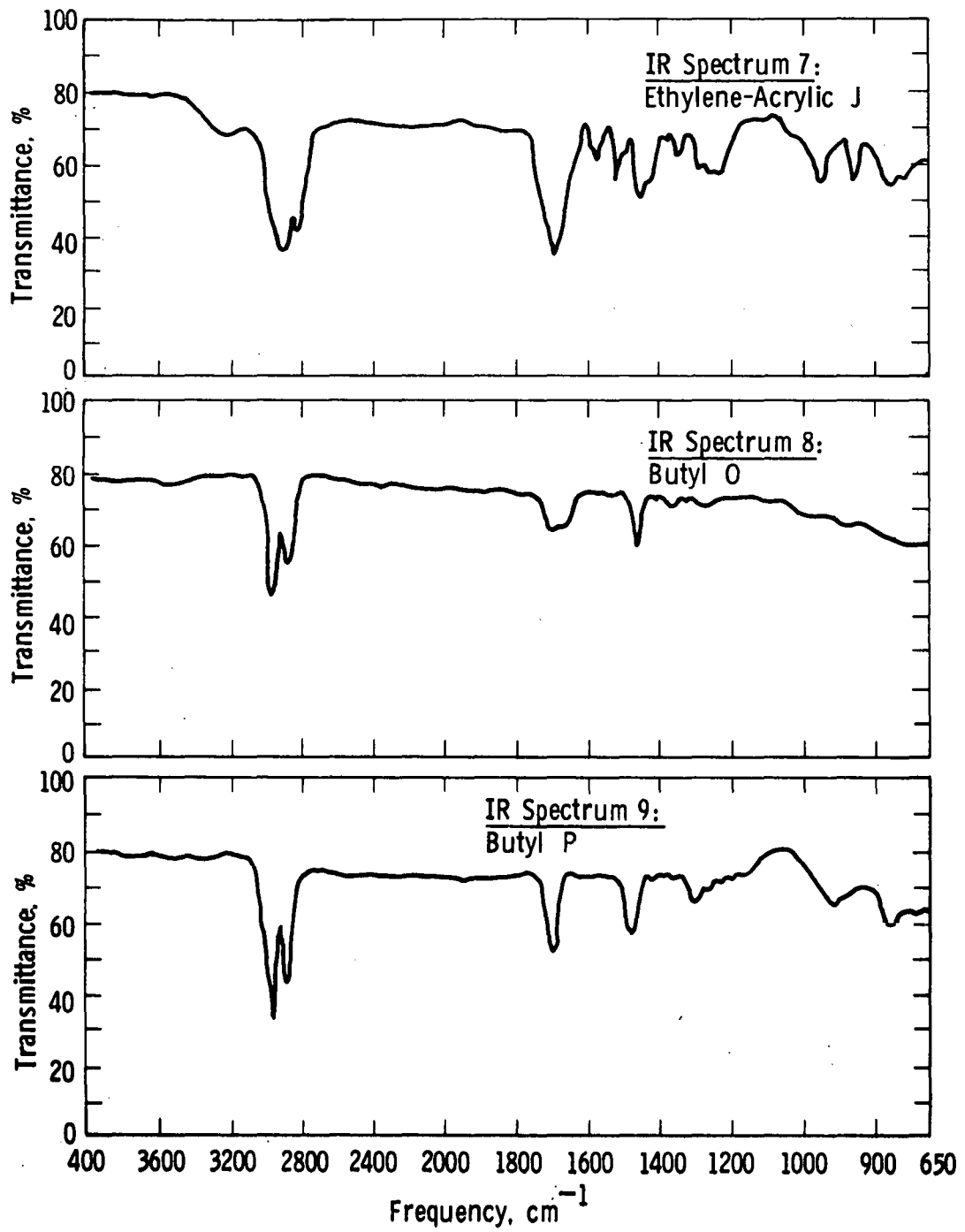


Fig.64— Infrared spectra of condensable volatiles

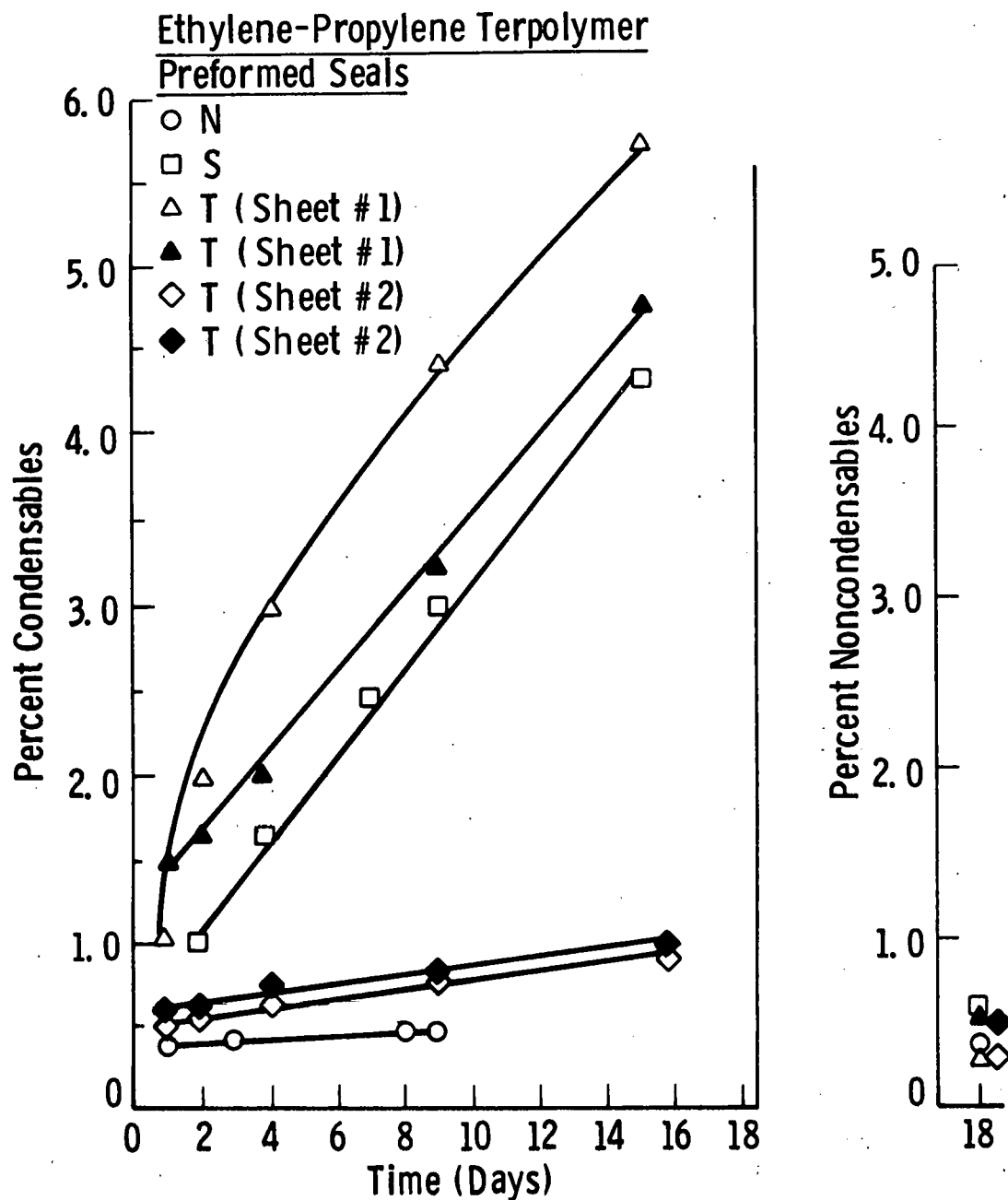


Fig.65 — Sealant aging at 150°C - percent condensables and noncondensables (percents are based on original weight of specimen)

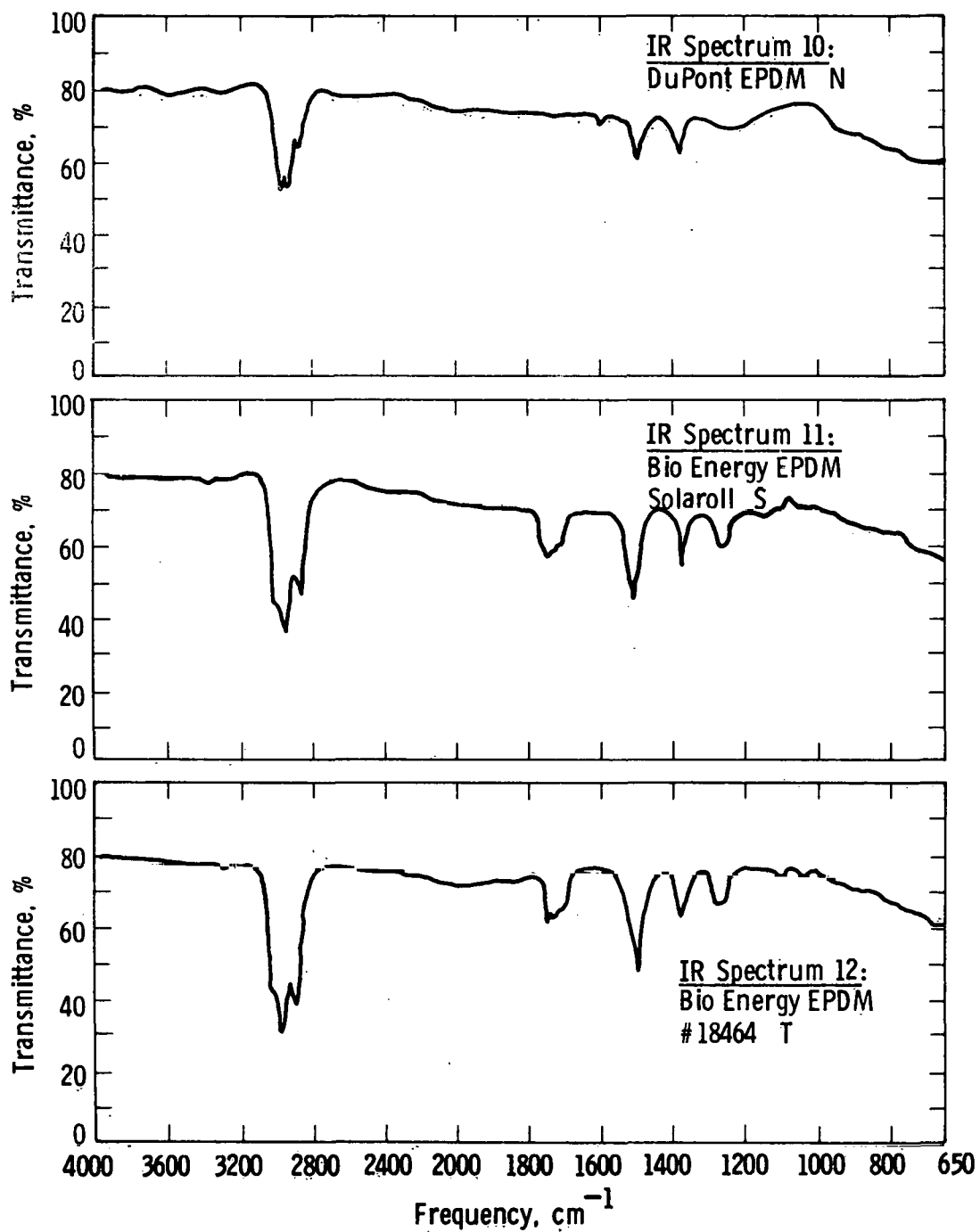


Fig. 66 — Infrared spectra of condensable volatiles

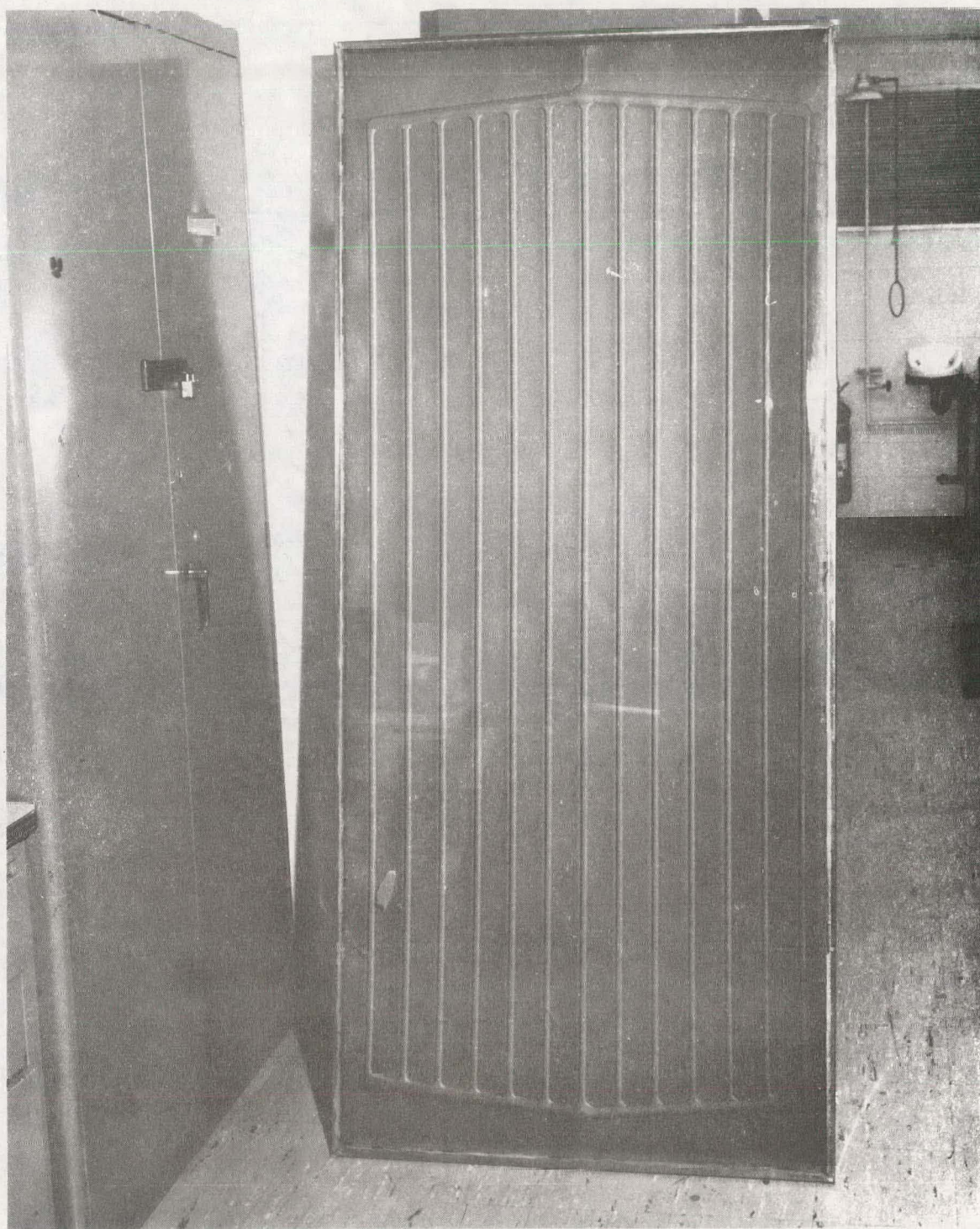


FIG.67 -- PITTSBURGH PLATE GLASS SOLAR COLLECTOR
FROM TOWNS ELEMENTARY SCHOOL,
ATLANTA, GEORGIA.

Dwg. 7694A89

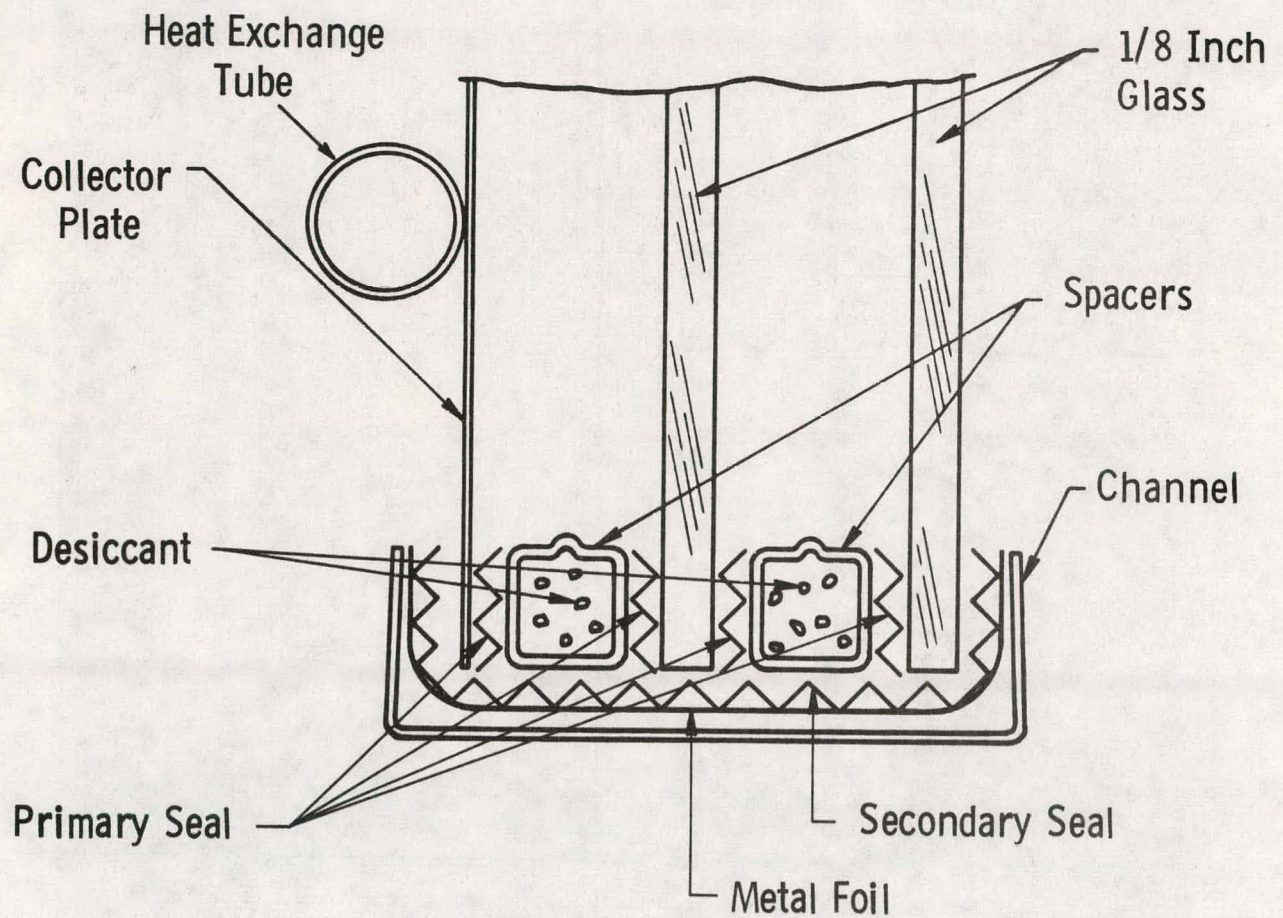


Fig. 68 — Pittsburgh Plate Glass solar collector from Towns elementary school

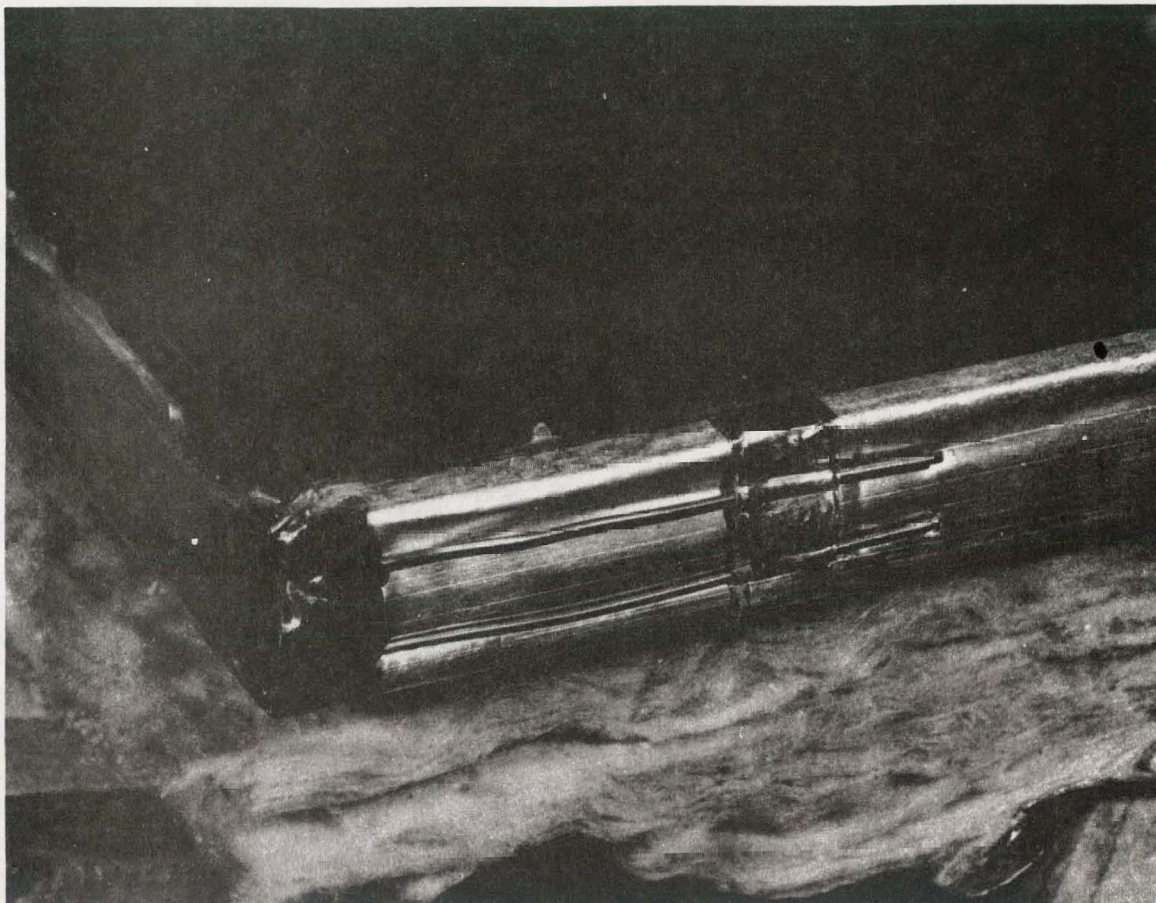


FIG. 69 -- BREATHING TUBES OF PITTSBURGH
PLATE GLASS SOLAR COLLECTOR.

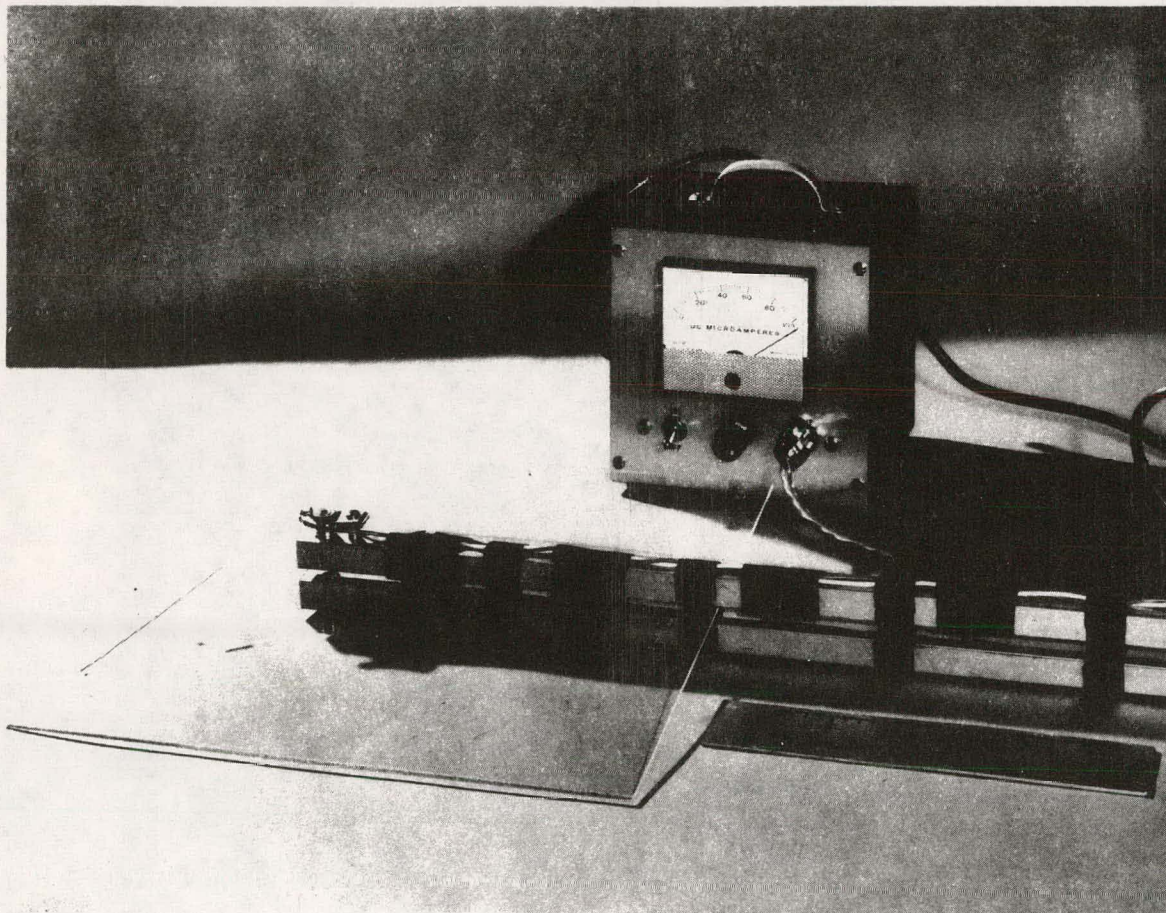


FIG. 70 -- INCANDESCENT/PHOTOCELL APPARATUS FOR
MEASURING COMPARATIVE LIGHT TRANSMITTANCE.

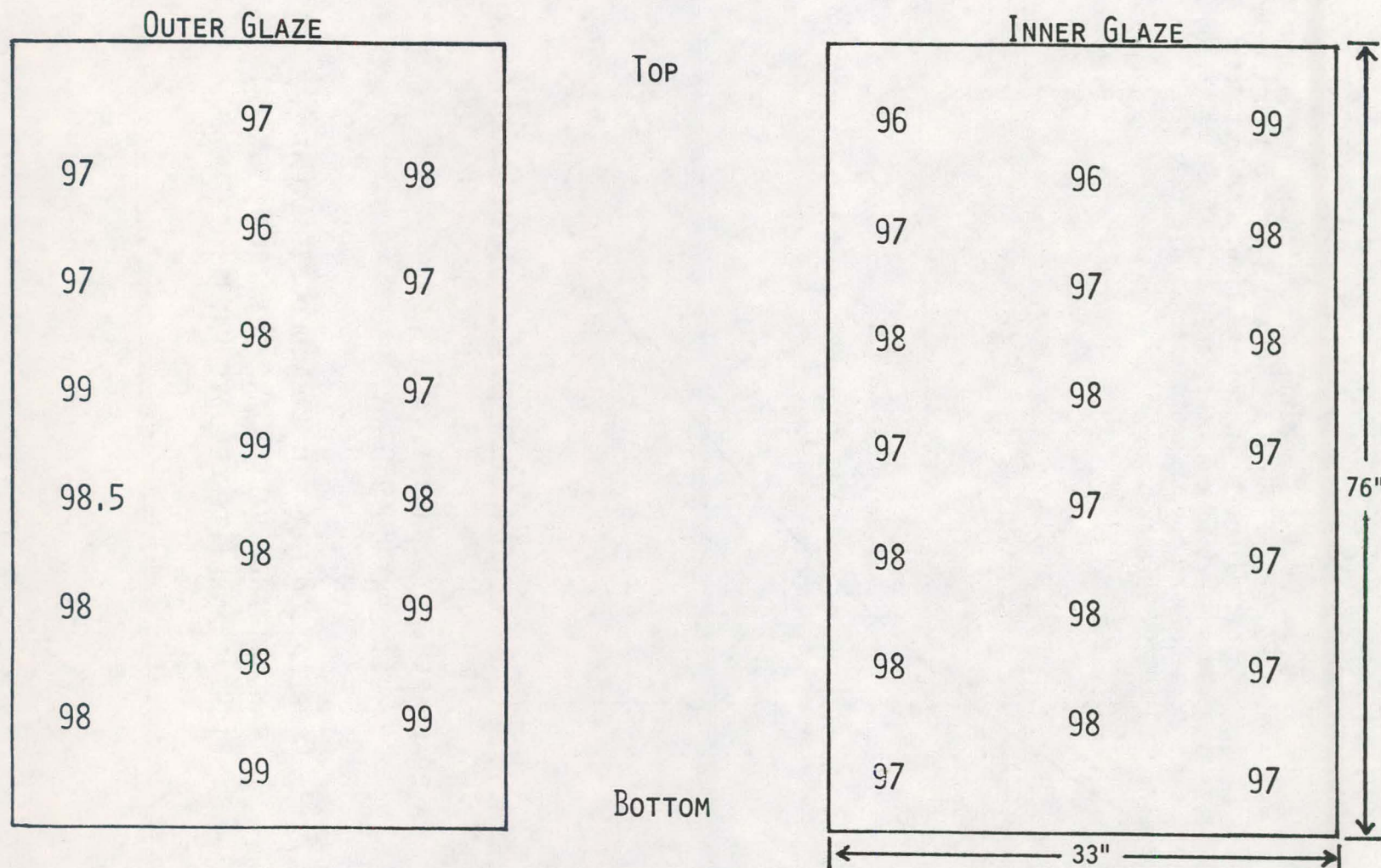


FIG. 71 -- RELATIVE LIGHT TRANSMITTANCE VALUES ON THE PITTSBURGH PLATE GLASS SOLAR COLLECTOR FROM TOWNS ELEMENTARY SCHOOL AS MEASURED USING THE INCANDESCENT LIGHT SOURCE/PHOTOCELL (100 = NO LOSS).

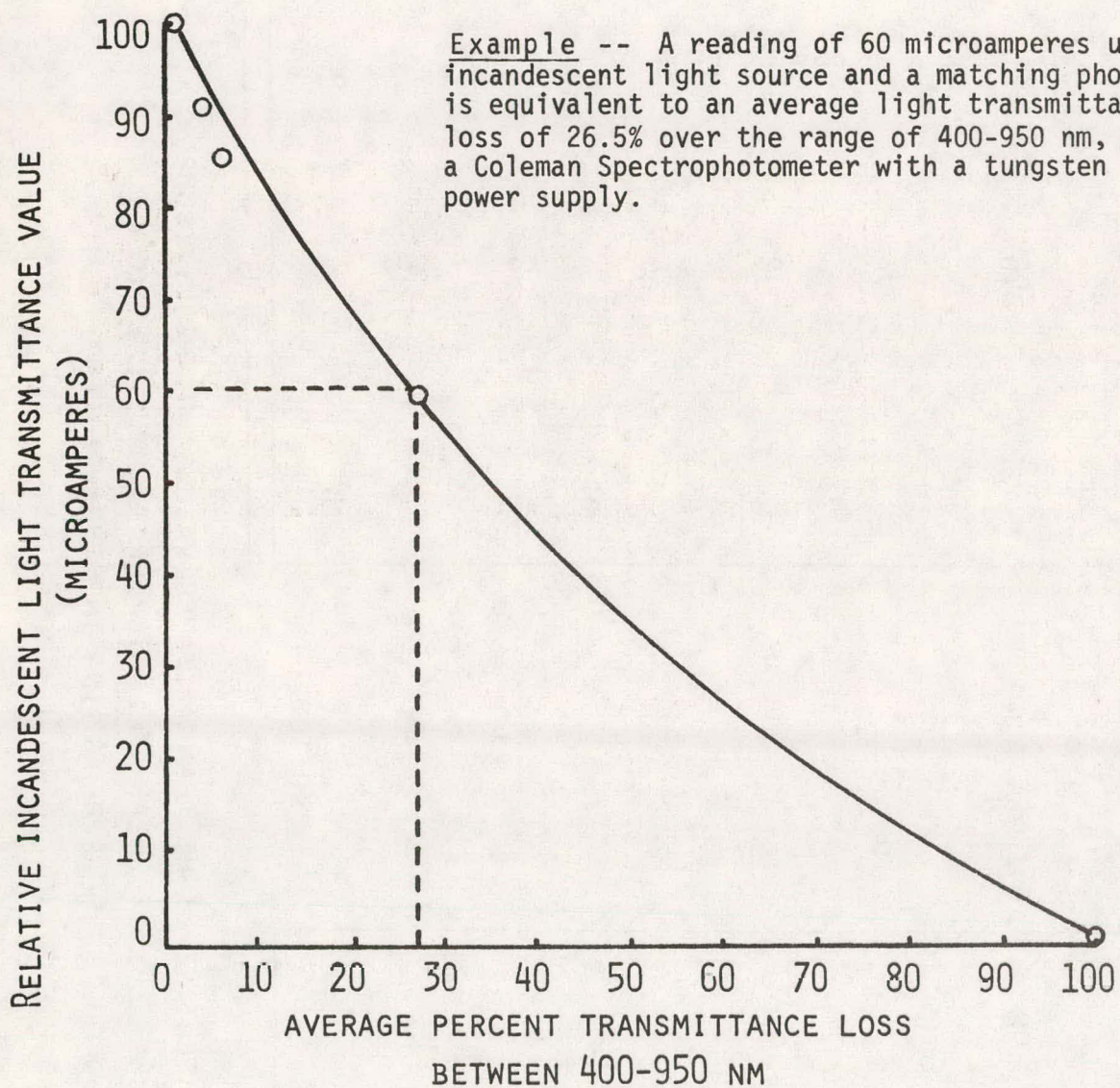


FIG.72 -- CORRELATION BETWEEN INCANDESCENT LIGHT TRANSMITTANCE VALUES AND SPECTROPHOTOMETRIC LIGHT TRANSMITTANCE LOSS VALUES.



FIG. 73 -- CORROSION OF SPACER.



FIG. 74 -- DEPOSIT ON BOTTOM EDGE OF ABSORBER PLATE
OF PITTSBURGH PLATE GLASS SOLAR COLLECTOR
FROM TOWNS ELEMENTARY SCHOOL.

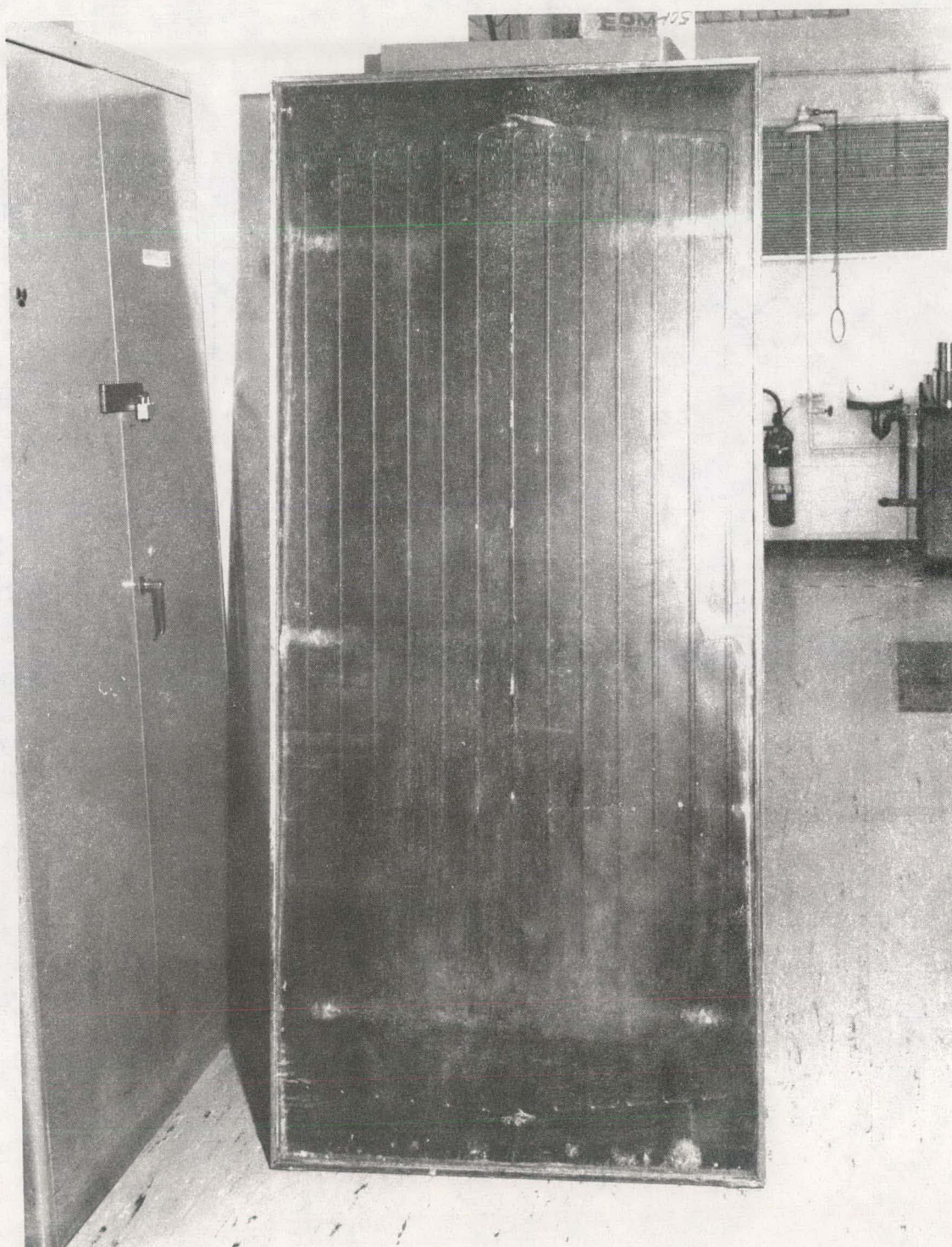


FIG. 75 -- PITTSBURGH PLATE GLASS SOLAR COLLECTOR
FROM THE NATIONAL BUREAU OF STANDARDS,
GAITHERSBURG, MARYLAND.

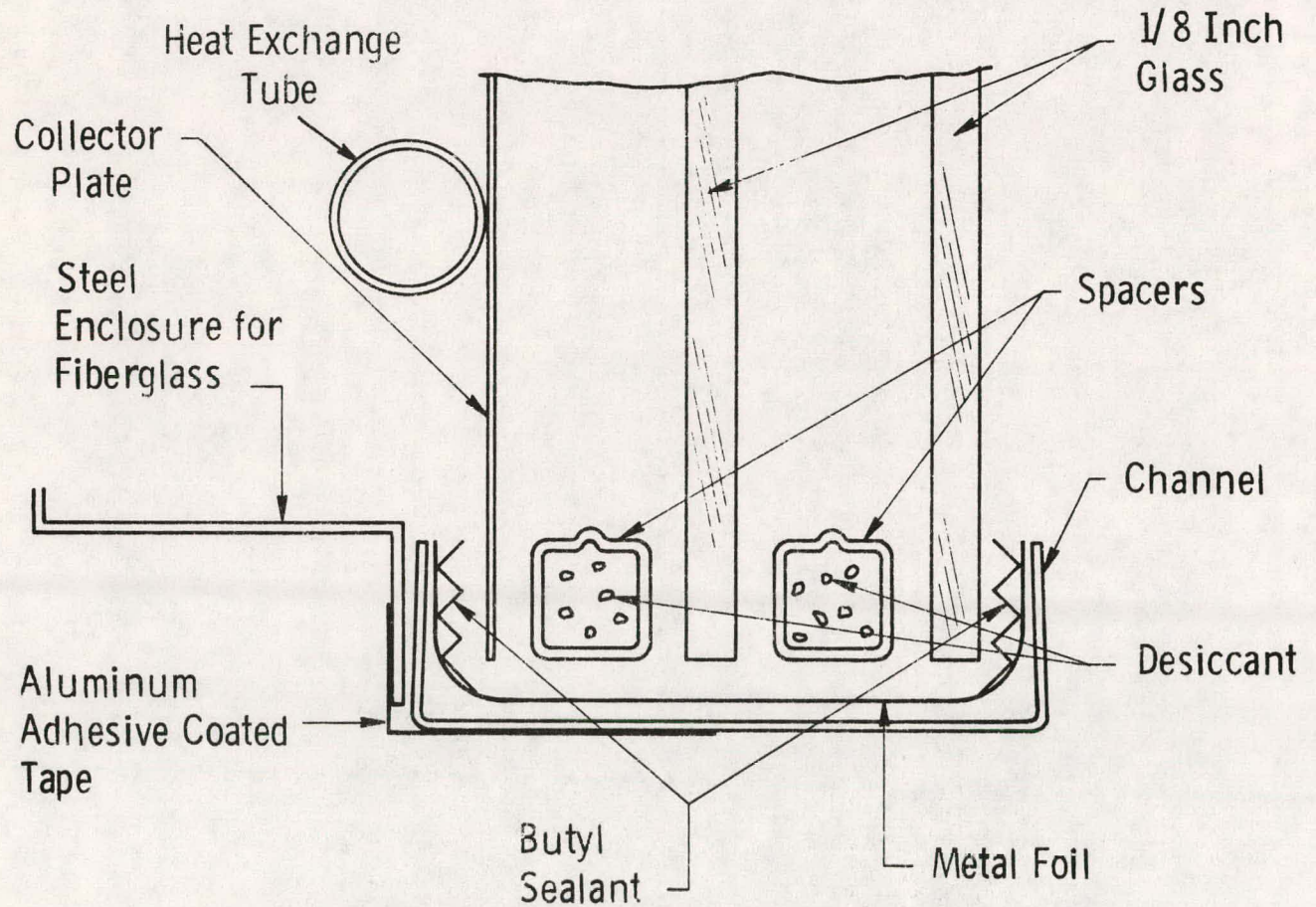


Fig.76—Pittsburgh Plate Glass solar collector from National Bureau of Standards

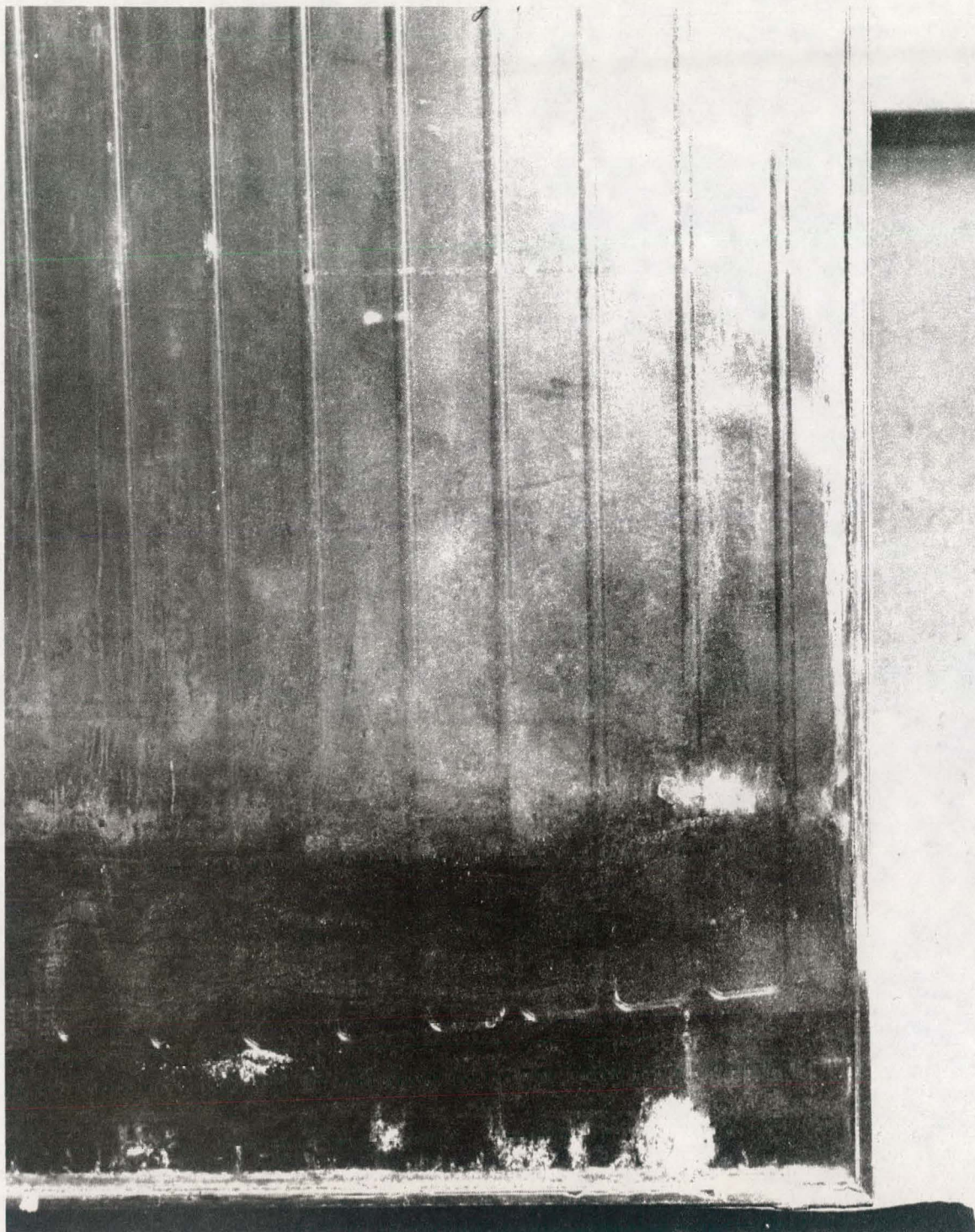


FIG. 77 -- CORROSION SITES ON THE PITTSBURGH
PLATE GLASS ABSORBER PLATE FROM
THE NATIONAL BUREAU OF STANDARDS.

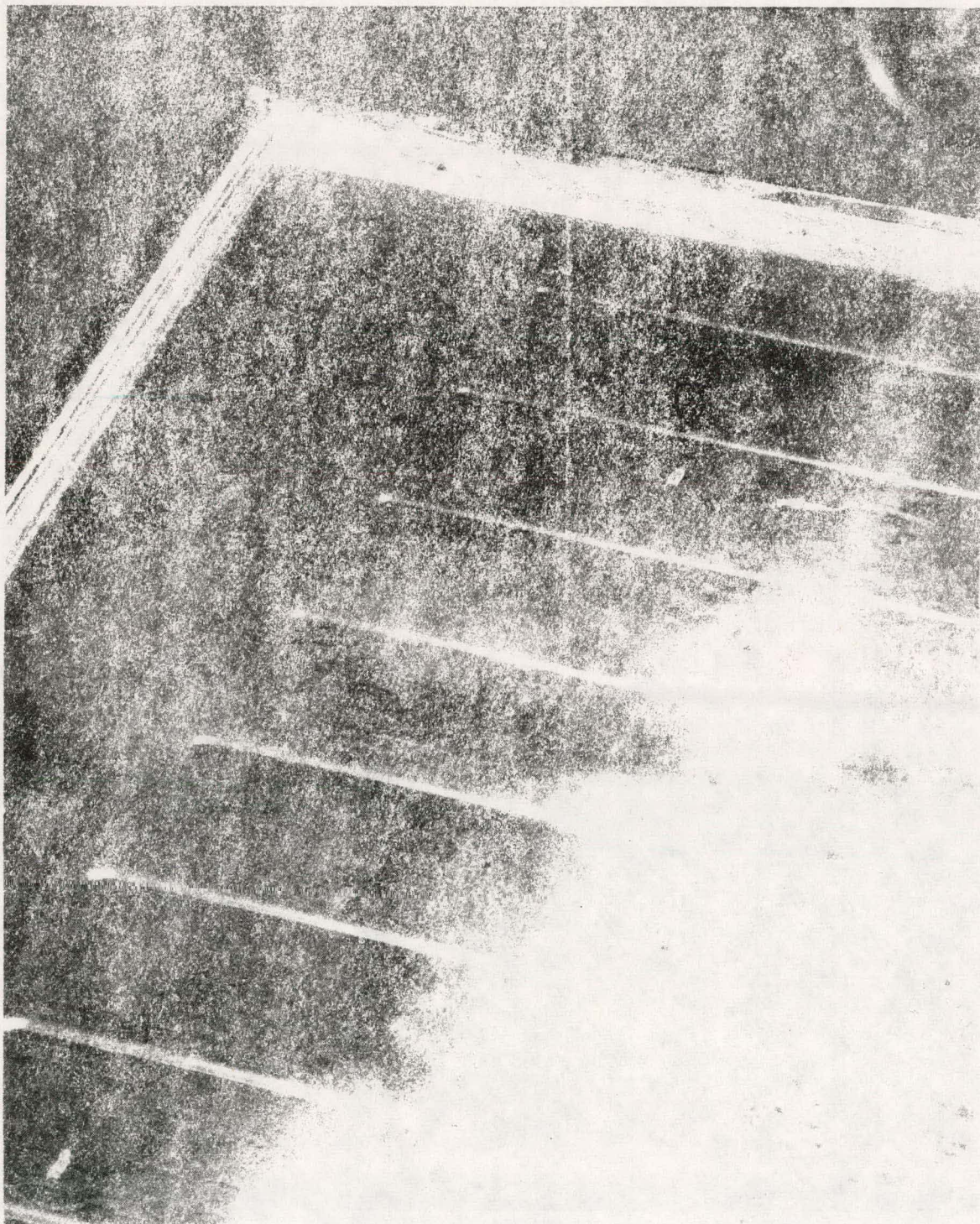


FIG. 78 -- WHITE SALT AND CORROSION DEPOSIT ON THE PITTSBURGH PLATE GLASS SOLAR COLLECTOR GLAZE SUPPLIED BY THE NATIONAL BUREAU OF STANDARDS.

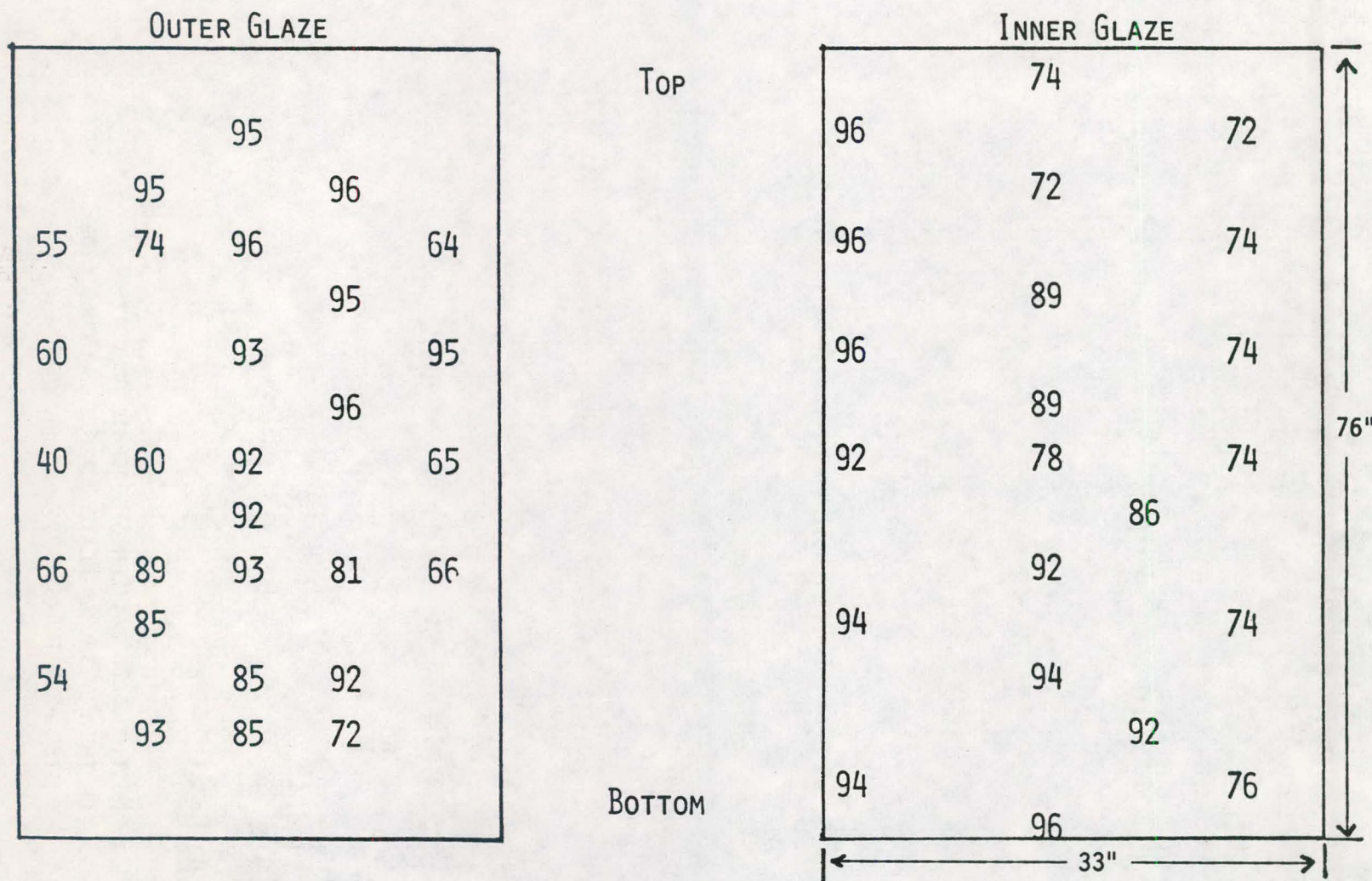


FIG. 79 -- RELATIVE LIGHT TRANSMITTANCE VALUES ON THE PITTSBURGH PLATE GLASS SOLAR COLLECTOR FROM THE NATIONAL BUREAU OF STANDARDS AS MEASURED BY THE INCANDESCENT LIGHT SOURCE/PHOTOCELL (100 = NO LOSS).



FIG. 80 -- MULTICOLORED PATTERN OF ORGANIC DEPOSITS
ON THE PITTSBURGH PLATE GLASS SOLAR COLLECTOR
GLAZED FROM THE NATIONAL BUREAU OF STANDARDS.

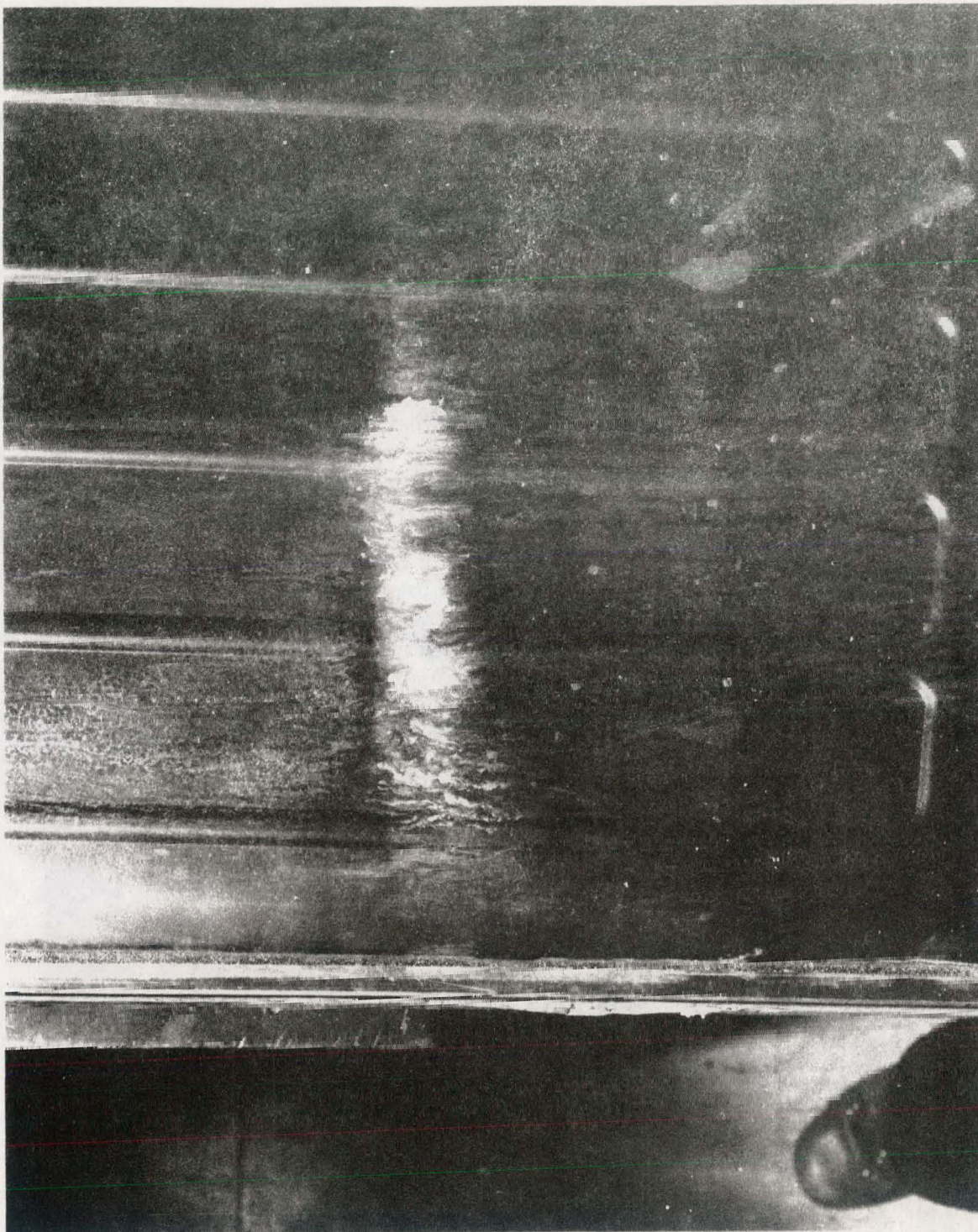


FIG. 81 -- ONE OF THREE WHITE SALT DEPOSIT LINES ON THE OUTER GLAZE OF THE PITTSBURGH PLATE GLASS SOLAR COLLECTOR FROM THE NATIONAL BUREAU OF STANDARDS.

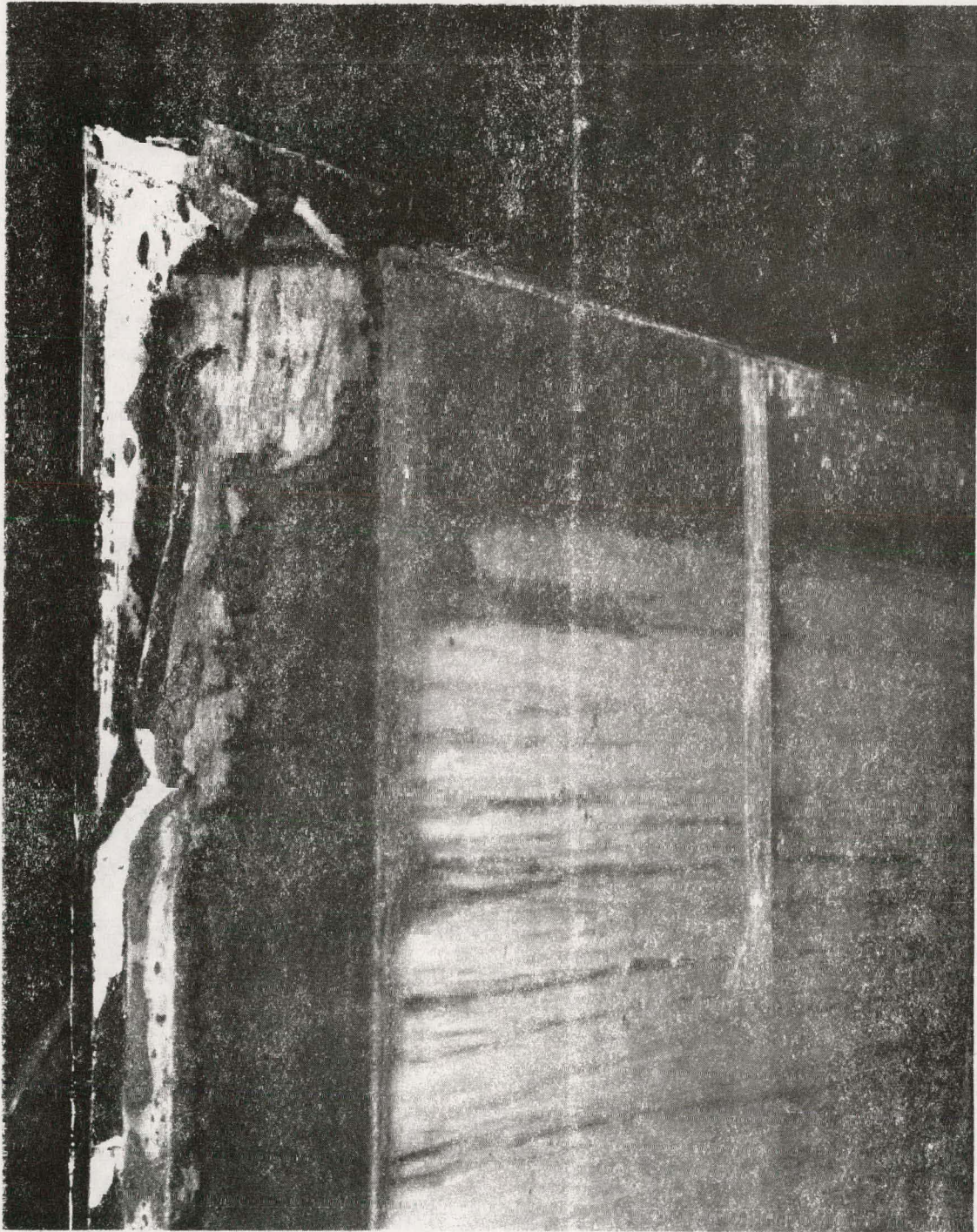


FIG. 82 -- SEVERE CORROSION OF THE STEEL ENCLOSURE AROUND
THE FIBERGLASS OF THE PITTSBURGH PLATE GLASS
SOLAR COLLECTOR FROM THE NATIONAL BUREAU
OF STANDARDS.

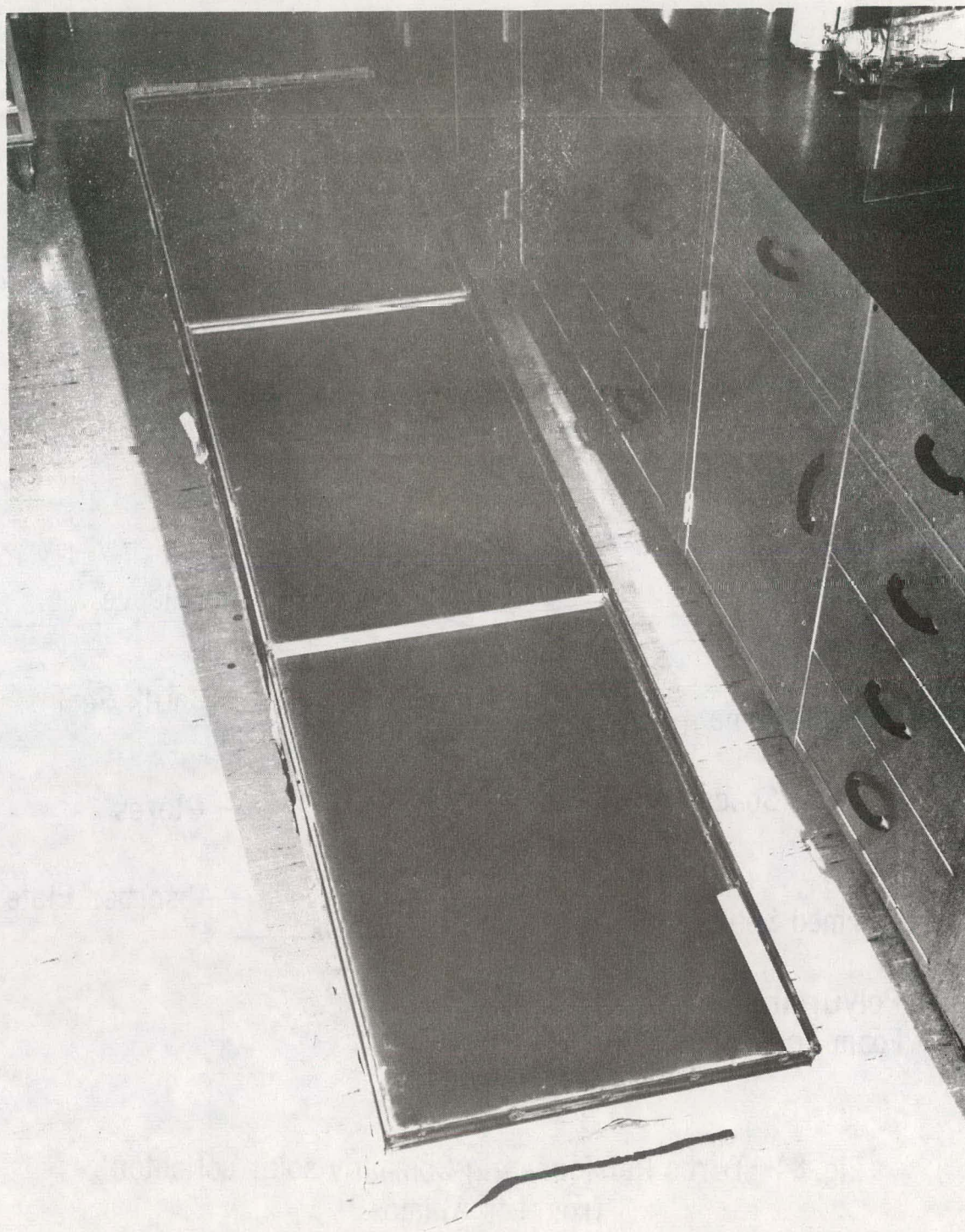
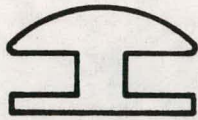


FIG. 83 -- TURBO REFRIGERATING COMPANY'S SOLAR
COLLECTOR SUPPLIED BY THE UNIVERSITY OF
CALIFORNIA LABORATORY AT LOS ALAMOS,
NEW MEXICO.



Seal Shape used
Between Glaze Panels



Seal Shape used
Between Glazes and Spacer

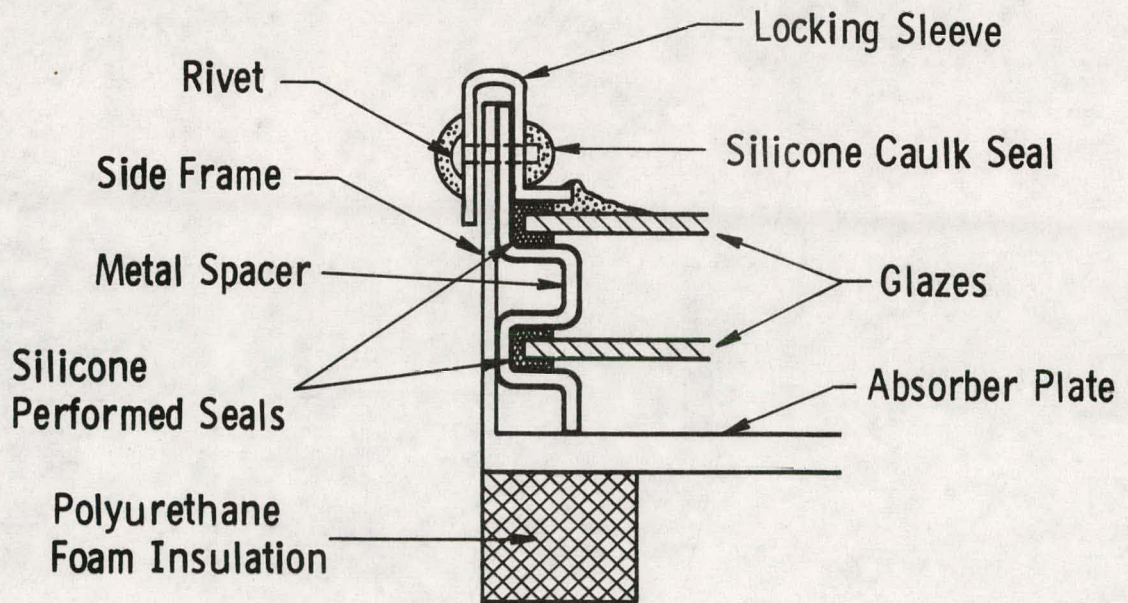


Fig. 84—Turbo Refrigerating Company solar collector
from Los Alamos

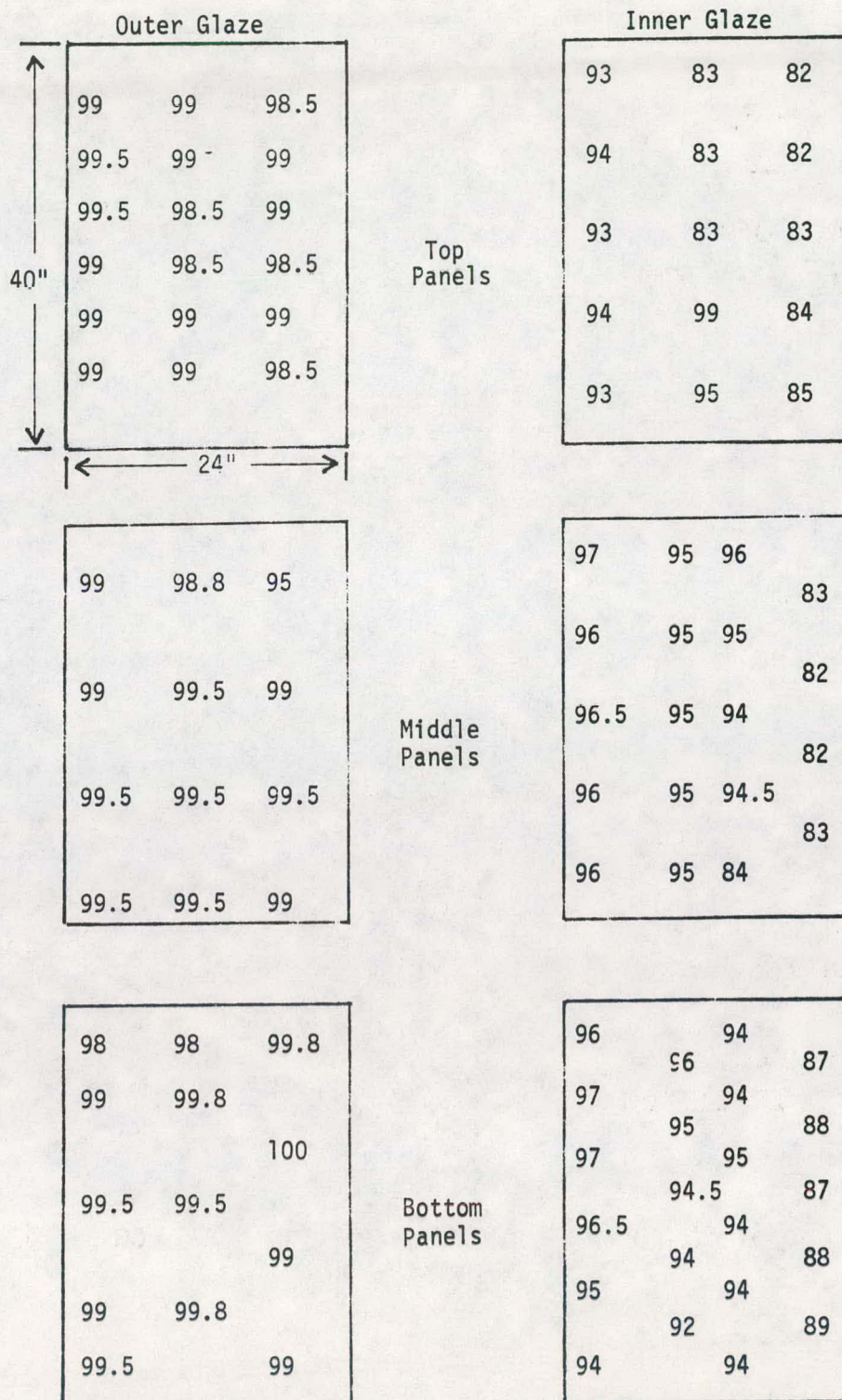


Fig. 85 -- Relative light transmittance values on the Turbo Refrigerating Company's solar collector from Los Alamos using the incandescent light source/photocell (100 = no loss).

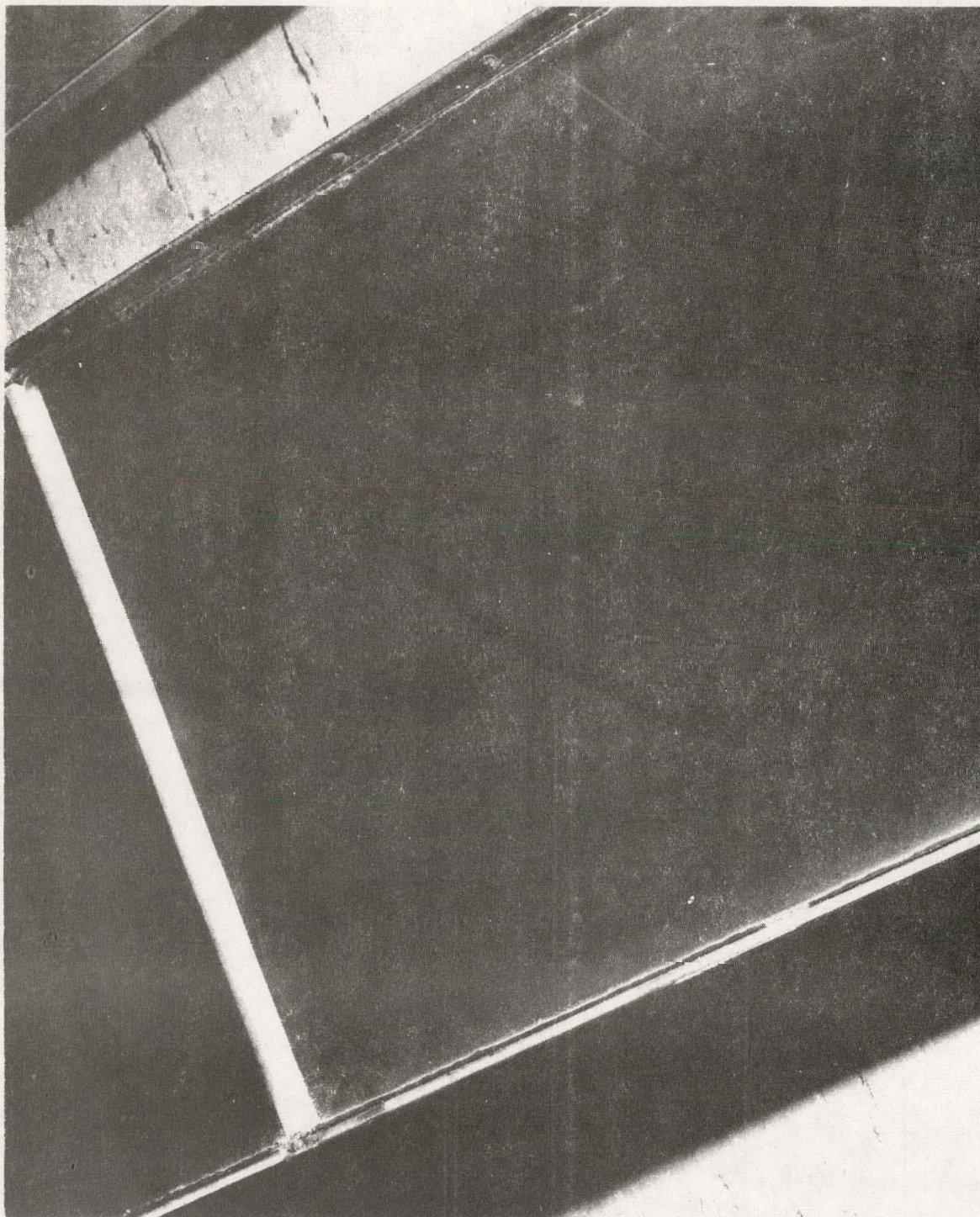


FIG. 86 -- WHITE BLOTCHY SILICA DEPOSIT ON THE INNER
GLAZE OF THE TURBO REFRIGERATING COMPANY'S
SOLAR COLLECTOR FROM LOS ALAMOS, NEW MEXICO.

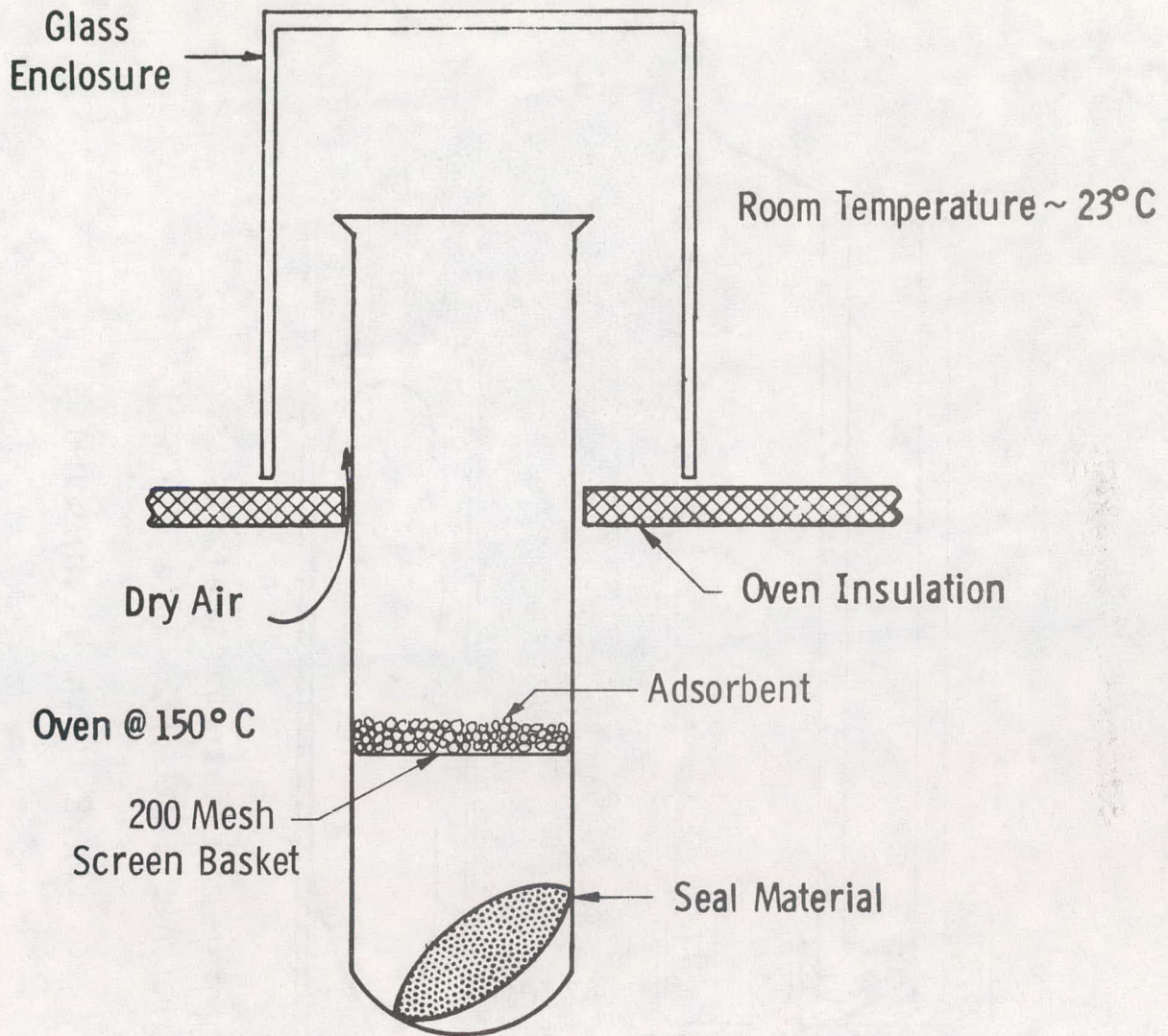
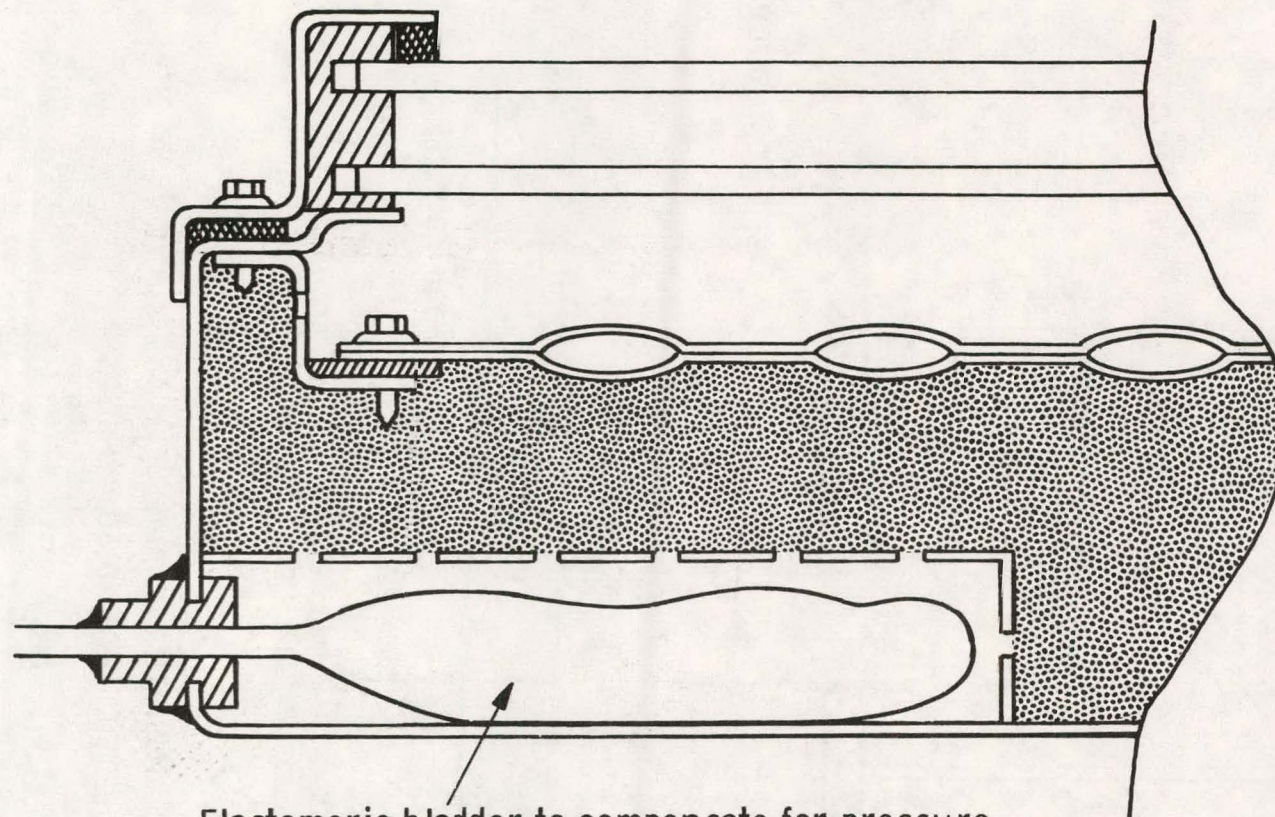


Fig. 87 — Apparatus used to determine the effectiveness of adsorbents toward organic vapors



Elastomeric bladder to compensate for pressure
due to barometric and temperature changes

Fig. 88 — Hermetically sealed design

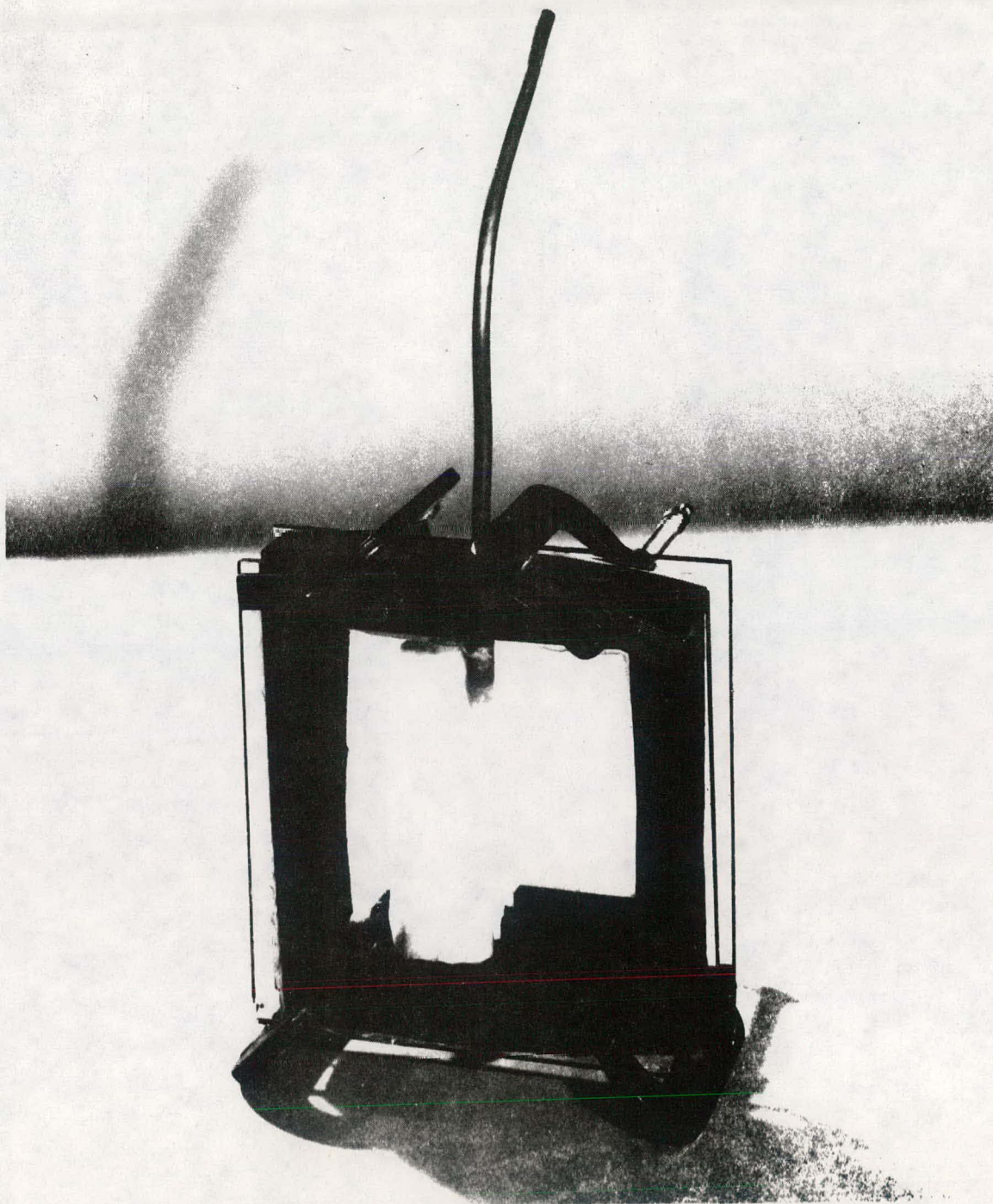


FIG. 89 -- INTERNALLY CONTAINED BREATHING BLADDER.

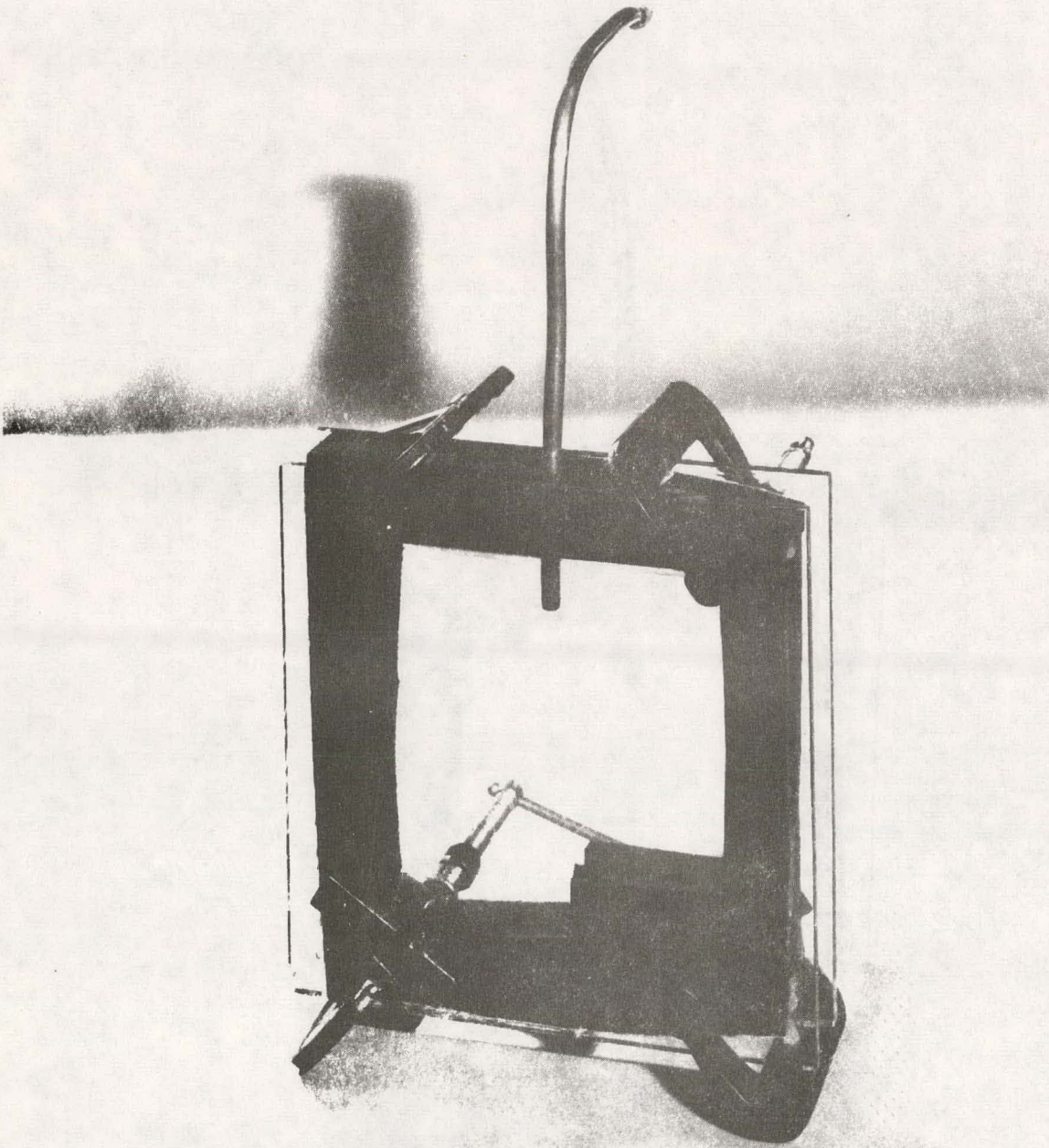


FIG. 90 -- EXTERNALLY MOUNTED BREATHING BLADDER.

A P P E N D I X I.

REFERENCED TEST METHODS



AMERICAN NATIONAL
STANDARD

ANSI/ASTM D 3867 - 78

AMERICAN SOCIETY FOR TESTING AND MATERIALS

1916 Race St., Philadelphia, Pa. 19103

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If not listed in the current combined index, will appear in the next edition.

Standard Specification for RUBBER SEALS USED IN FLAT-PLATE SOLAR COLLECTORS¹

This Standard is issued under the fixed designation D 3867, the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This specification covers the general requirements for materials used in rubber seals of flat-plate solar collectors. Particular applications may necessitate other requirements that would take precedence over these requirements when specified.

1.2 The design requirement pertains only to permissible deflections of the rubber during thermal expansion or contraction of the seal in use and the tolerances in dimensions of molded and extruded seals.

1.3 This specification does not include requirements pertaining to the fabrication or installation of the seals.

2. Applicable Documents

2.1 ASTM Standards:

- C 661 Test for Indentation Hardness of Elastomeric-Type Sealants by Means of a Durometer²
- C 717 Definitions of Terms Relating to Building Seals²
- C 719 Test for Adhesion and Cohesion of Elastomeric Joint Sealants Under Cyclic Movement²
- D 395 Tests for Rubber Property—Compression Set³
- D 412 Test for Rubber Properties in Tension³
- D 455 Test for Rubber Deterioration by Heating in a Test Tube³
- D 1149 Test for Rubber Deterioration—Surface Ozone Cracking in a Chamber (Flat Specimens)³
- D 1226 Test for Rubber Property—Compression Set at Low Temperatures³
- D 1349 Recommended Practice for Rubber—Standard Temperatures and Atmospheres for Testing and Conditioning⁴

D 1415 Test for Rubber Property—International Hardness³

D 1566 Definitions of Terms Relating to Rubber³

D 2137 Test for Rubber Property—Brittleness Point of Flexible Polymers and Coated Fabrics³

D 2240 Test for Rubber Property—Durometer Hardness⁴

D 3182 Recommended Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets³

D 3183 Recommended Practice for Rubber—Preparation of Pieces for Test from Other Than Standard Vulcanized Sheets³

2.2 Other Standards:

RMA Handbook—Rubber Products: Molded, Extruded, Lathe Cut, and Cellular⁵

3. Classification

3.1 Types:

3.1.1 Type C, intended for use in cold climates (below -10°C in winter).

3.1.2 Type W, intended for use in warm

This specification is under the jurisdiction of ASTM Committee D-11 on Rubber and Rubber-Like Materials, and is the direct responsibility of Subcommittee D11.36 on Seals.

Current edition approved May 11, 1978. Published July 1978.

¹ Annual Book of ASTM Standards, Part 18.

² Annual Book of ASTM Standards, Part 37.

³ Annual Book of ASTM Standards, Parts 35, 36, and 38.

⁴ Annual Book of ASTM Standards, Parts 37 and 38.

⁵ Annual Book of ASTM Standards, Parts 35 and 37.

⁶ Available from the Rubber Manufacturers Association (RMA), 444 Madison Ave., New York, N.Y. 10022.

climates (above -10°C in winter).

3.2 Grades:

3.2.1 Grade designations represent differing degrees of hardness as follows:

3.2.1.1 Grade 2, hardness of 20 ± 5 .

3.2.1.2 Grade 3, hardness of 30 ± 5 .

3.2.1.3 Grade 4, hardness of 40 ± 5 .

3.2.1.4 Grade 5, hardness of 50 ± 5 .

3.2.1.5 Grade 6, hardness of 60 ± 5 .

3.2.1.6 Grade 7, hardness of 70 ± 5 .

3.2.1.7 Grade 8, hardness of 80 ± 5 .

NOTE 1—The grade to be used in a particular application depends on the design of the seal and must be specified by the designer.

3.3 Classes:

3.3.1 Seals shall be classified as follows:

3.3.1.1 Class PS, preformed rubber seal.

3.3.1.2 Class SC, sealing compound.

NOTE 2—Class SC material should not be used in designs where the seal is under mechanical stress.

4. Definitions

4.1 Refer to the definitions of terms in Definitions C 717 and D 1566.

5. Materials

5.1 Seals shall be made from rubber compounds that are impermeable to ultraviolet light and when vulcanized, conform to the requirements in Section 6.

6. Requirements

6.1 Class PS material shall conform to the requirements given in Table 1.

6.2 Class SC material shall conform to the requirements given in Table 2.

7. Dimensions

7.1 The design of the seal shall not permit the rubber to deflect more than 25 % in any direction during thermal expansion and contraction of the solar collector.

NOTE 3—If the thermal coefficient of linear expansion for the rubber is not known, a value of $0.0003/\text{K}$ may be assumed for design purposes.

7.2 The tolerances in dimensions shall conform to the following designations in the RMA Handbook:

Molded Seals:

Commercial Dimensions—RMA A2-F-1032

Critical Dimensions—RMA A2-F-1032

Extruded Seals:

U 3867

7.2.2.1 Commercial Dimensions—RMA A2-F3.

8. Workmanship

8.1 Class PS seals shall be free of blisters, checks, cracks, and other imperfections that can affect their ability to make or maintain a water-tight seal.

8.2 Class SC material shall be uniform in composition and be free of defects that may affect serviceability.

9. Test Methods

9.1 Class PS Material—Prepare the specimens as prescribed in Recommended Practice D 3183 and test the material in accordance with the test methods given in Table 1. For control of production, specimens may be taken from standard test sheets prepared in accordance with Recommended Practice D 3182, using the same unvulcanized material used to prepare the seals and vulcanizing the material at the same temperature used for the seals to an equivalent state of vulcanization.

9.2 Class SC Material—Prepare five sheets approximately 150 by 150 by 2 mm in accordance with instructions supplied with the sealing material. Also, prepare five adhesion specimens in accordance with Method C 719. Preferably, prepare each sheet and adhesion specimen from material in a different container. Condition the sheets and adhesion specimens for 14 days at a temperature of 23°C and relative humidity of 50 %. Test the material in accordance with the test methods given in Table 2.

9.3 Determine volatiles lost from the difference in mass of the specimens before and after heating for 166 h at the temperature given in Table 1 or 2 and as prescribed in Method D 865.

9.4 Determine volatiles condensable at 23°C from the difference in mass of the outlet tubes before and after heating the specimens for 166 h at the temperature given in Table 1 or 2 in accordance with Method D 865. If necessary, cool the exposed portion of the outlet tube with a stream of air to maintain a temperature of $25 \pm 2^{\circ}\text{C}$. If any volatiles condense on the inlet tube or other parts of

the apparatus, add the mass of this condensed material to the mass of the material on the outlet tube.

10. Inspection and Rejection

10.1 *Class PS Material*—Manufacturers of preformed seals may use their quality control systems for production inspection to assure the seals conform with this specification, provided appropriate records are kept. In case of dispute regarding the quality of a delivered product, a sample of five seals shall be taken from the lot and tested for compliance with this specification. If one of the five seals does not conform, a second sample of five seals may be taken and tested. If two or more of the ten seals do not conform, the lot may be rejected.

10.2 *Class SC Material*—Manufacturers may use their quality control systems to assure production conforms with this specification. In case of dispute regarding the quality of a delivered product, five test sheets and five adhesion specimens shall be prepared, preferably from five different packages, in accord-

ance with the instructions supplied with the sealing material. If one of the five sheets or adhesion specimens does not conform, an additional five sheets or adhesion specimens may be prepared and tested. If two or more of the ten sheets or adhesion specimens do not conform, the lot may be rejected.

11. Marking

11.1 The following information shall be marked either on the seal, packaging, label, or tag:

11.1.1 Name, brand, or trademark of the manufacturer.

11.1.2 Type and grade.

11.1.3 Compliance with Specification D 3667, and

11.1.4 Other information required by the manufacturer or purchaser.

12. Packaging

12.1 Material shall be protected by suitable packaging to prevent damage during shipment or storage prior to installation in the solar collector.

TABLE 1 Requirements for Class PS Material Used to Seal Flat-Plate Solar Collectors

| Property | Grade | | | | | | ASTM Method |
|---|-------|-----|-----|-----|-----|-----|---------------------|
| | 3 | 4 | 5 | 6 | 7 | 8 | |
| Ultimate elongation, min, % | 350 | 300 | 250 | 200 | 150 | 100 | D 412 |
| Compression set, max, %: | | | | | | | |
| after 70 h at 150°C | 30 | 30 | 30 | 30 | 30 | 30 | D 395 ^a |
| after 166 h at -10°C | 60 | 60 | 60 | 60 | 60 | 60 | D 1220 ^b |
| Resistance to heating (for 166 h at 150°C) ^c | | | | | | | D 865 |
| Hardness change, max | 10 | 10 | 10 | 10 | 10 | 10 | D 1415 or D 2240 |
| Ultimate elongation change, max, % | 30 | 30 | 30 | 30 | 30 | 30 | D 412 |
| Tensile strength change, max, % | 20 | 20 | 20 | 20 | 20 | 20 | D 412 |
| Volatiles lost, max, % | 1 | 1 | 1 | 1 | 1 | 1 | See 10.3 |
| Volatiles condensable, max, % | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | See 10.4 |
| Resistance to ozone: | | | | | | | D 1149 |
| 100 mPa ^d for 166 h at 40°C | | | | | | | |
| Resistance to low temperature | | | | | | | D 2137 ^e |
| Type C only, max, °C | -40 | -40 | -40 | -40 | -40 | -40 | |

^a Method B

^b Set to be measured at 10 s after release. Lubricated plates or polytetrafluoroethylene film is recommended if the rubber adheres to the metal compression plates during test.

^c The test temperature of 150°C is used to test seals for cover plates. A seal in contact with an absorber plate should be tested at a standard test temperature listed in Recommended Practice D 1349 next above the maximum temperature of the absorber plate in service (which generally occurs under stagnation conditions and maximum radiation flux) but not less than 150°C.

^d 100 mPa of ozone partial pressure is equivalent to 100 ppm at standard atmospheric pressure (100 kPa). The higher test temperatures are: 175, 200, 225, and 250. See new terminology on ozone content expressions described in Method D 1149.

TABLE 2 Requirements for Class SC Material Used to Seal Flat-Plate Solar Collectors

| Property | Grade | | | ASTM Method |
|--|-------|-----|-----|-----------------------|
| | 2 | 3 | 4 | |
| Ultimate elongation, min, % | 200 | 150 | 100 | D 412 |
| Resistance to heating (for 166 h at 125°C) | | | | D 865 |
| Hardness change, max | 10 | 10 | 10 | C 661 |
| Ultimate elongation change, max, % | 30 | 30 | 30 | D 412 |
| Tensile strength change, max, % | 20 | 20 | 20 | D 412 |
| Volatiles lost, max, % | 1 | 1 | 1 | See 10.3 ^a |
| Volatiles condensable, max, % | 0.1 | 0.1 | 0.1 | See 10.4 ^a |
| Resistance to ozone: | | | | D 1149 |
| 100 mPa, for 166 h at 40°C | | | | |
| Resistance to low temperature: | | | | D 2137 |
| Type C only, max, °C | -40 | -40 | -40 | |
| Adhesion loss, max, cm ^b | 9 | 9 | 9 | C 719 |

^a This test is not required if the design precludes condensing of the volatiles on the cover plate(s) of the solar collector.

^b The combined loss in bond and cohesion areas for the three specimens tested shall not exceed 9 cm².

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19101, which will schedule a further hearing regarding your comments. Failing satisfaction there, you may appeal to the ASTM Board of Directors.



Standard Test Methods for RUBBER PROPERTY—COMPRESSION SET¹

This Standard is issued under the fixed designation D 395; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

¹ NOTE—Editorial changes were made throughout in October 1975.

1. Scope

1.1 These methods cover the testing of rubber intended for use in applications in which the rubber will be subjected to compressive stresses or shear. They are applicable particularly to the rubber used in machinery mountings and vibration dampers. Two methods are covered as follows:

Method A—Compression set under constant load.

Method B—Compression set under constant deflection.

1.2 The choice of method is optional, but consideration should be given to the nature of the service for which correlation of test results may be sought. In case of conflict between the provisions of these methods and those of detailed specifications or methods of test for a particular material, Method B shall take precedence.

1.3 This method is not suitable for vulcanizates in excess of 90 IRHD.

2. Definition

2.1 *Compression Set of Rubber*—For the purpose of these tests, the residual deformation of a test specimen measured 30 min after removal from a suitable compression device in which the specimen had been subjected for a definite time to compressive deformation under specified conditions.

3. Significance

3.1 Compression set tests are intended to measure the ability of rubber compounds to retain elastic properties after prolonged action of compressive stresses. The actual stress-

ing service may involve the maintenance of a definite deflection, the constant application of a known load, or the rapidly repeated deformation and recovery resulting from intermittent compressive forces. Though the latter dynamic stressing, like the others, produces compression set, its effects as a whole are more closely simulated by compression flexing or hysteresis tests. Therefore, compression set tests are considered to be only those involving static loading.

METHOD A. COMPRESSION SET UNDER CONSTANT LOAD

4. Apparatus

4.1 *Cutting Die*—The circular die used to prepare standard test specimens shall have an inside diameter measurement of 23.68 ± 0.03 mm (1.129 ± 0.001 in.). This die shall be carefully maintained so that the cutting edges are sharp and free from nicks.

4.2 *Dial Micrometer*—to measure the thickness of the specimen. The micrometer shall have an anvil of 9.5 ± 0.5 mm (0.38 ± 0.02 in.) in diameter and a hemispherical presser foot 6 ± 1 mm (0.24 ± 0.04 in.) in diameter. The force on the presser foot shall be 0.8 ± 0.1 N (0.18 ± 0.02 lbf).

NOTE 1—Other ways of measuring specimen thickness before and after testing may be used as agreed upon by purchaser and seller.

¹ These methods are under the jurisdiction of ASTM Committee D-11 on Rubber and Rubber-Like Materials and is the direct responsibility of Subcommittee D11.10 on Physical Testing.
Current edition effective Oct. 3, 1969. Originally issued 1934. Replaces D 395 - 67.

4.3 *Compression Device*—The compression device shall consist of a loading spring and two parallel compression plates assembled by means of a frame or threaded bolts in such a manner that the device shall be portable and self-contained after the load has been applied and that the parallelism of the plates shall be maintained. The load may be applied as described in either 4.3.1 or 4.3.2.

4.3.1 *Calibrated Spring Loading*—In the case of calibrated spring loading, the required load shall be applied by a screw mechanism for compressing a calibrated spring the proper amount. The spring shall be of properly heat-treated spring steel with ends ground square and perpendicular to the longitudinal axis of the spring. A suitable compression device is shown in Fig. 1. The spring shall conform to the following requirements:

4.3.1.1 The spring shall be calibrated at room temperature ($23 \pm 5^\circ\text{C}$ ($73.4 \pm 9^\circ\text{F}$)) by applying successive increments of force not exceeding 250 N or 50 lbf and measuring the corresponding deflection to the nearest 0.2 mm or 0.01 in. The curve obtained by plotting the forces against the corresponding deflections shall have a slope of 70 ± 3.5 kN/m (400 ± 20 lbf/in.) at 1.8 kN or 400 lbf force. The slope is obtained by dividing the difference between two loads above and below 1.8 kN (400 lbf) by the difference between the corresponding deflections.

4.3.1.2 The original dimensions of the spring shall not change due to fatigue by more than 0.3 mm (0.01 in.) after it has been mounted in the compression device, compressed under a force of 1.8 kN (400 lbf), and heated in the oven for 1 week at 70°C (158°F). In ordinary use, a weekly check of the dimensions shall show no greater change than this over a period of 1 year.

4.3.1.3 The minimum load required to close the spring solid shall be 2.4 kN (530 lbf).

4.3.2 *External Loading*—In the case of external loading, the required load shall be applied to the compression plates and spring by external means after the test specimen is mounted in the apparatus. Either a calibrated compression machine or dead weights may be used for load application. Provision shall be made by the use of bolts and nuts or other devices to prevent the specimen and spring from losing their initial deflections when the

external load is removed. The spring shall have essentially the same characteristics as described in 4.3.1, but calibration is not required. A suitable compression device is shown in Fig. 2.

4.4 *Plates*—The plates between which the test specimen is compressed shall be made of steel of sufficient thickness to withstand the compressive stresses without bending. The surfaces against which the specimen is held shall have a highly polished chromium-plated finish and shall be thoroughly cleaned and wiped dry before each test.

4.5 *Oven*—The oven used shall conform to the specifications for a Type IIB laboratory oven given in ASTM Specification E 145, for Gravity-Convection and Forced-Ventilation Ovens.²

5. Test Specimen

5.1 The standard test specimen shall be a cylindrical disk cut from a laboratory-prepared slab or between 12.5 and 13.0 mm (0.49 and 0.51 in.) in thickness using the cutting die specified in 4.1. Aging of samples shall be in accordance with ASTM Recommended Practice D 3183, Preparation of Pieces for Test from Other Than Standard Vulcanized Sheets.³

5.2 When cutting a specimen, the specified circular die shall be suitably rotated in a drill press or similar device and lubricated by means of a soap solution. A minimum distance of 13 mm (0.5 in.) shall be maintained between the cutting edge of the die and the edge of the slab. The cutting pressure shall be as light as possible to minimize cupping of the cut edges.

5.3 An optional method of preparing the standard specimen may be the direct molding of a circular disk having the dimensions specified in 4.1 and 5.1.

NOTE 2—It should be recognized that an equal time and temperature if used for both the slab and molded specimen will not produce an equivalent state of cure in the two types of specimen. A "tighter" cure will be obtained in the molded specimen. Adjustments, preferably in the time of cure,

² Annual Book of ASTM Standards, Part 41.

³ Annual Book of ASTM Standards, Part 37.

⁴ Conant, F. S., Svetlik, J. F., and Juve, A. E., "Equivalent Cures in Specimens of Various Shapes," *Rubber World*, RUBWA, Vol. 137, No. 6, March, 1958, p. 856; *Rubber Age*, RUAGA, Vol. 82, No. 6, March, 1958, p. 1031; *Rubber Chemistry and Technology*, RCTEA, Vol. XXXI, No. 3, July-September, 1958.

must be taken into consideration if comparisons between the two types of specimen are to be considered valid.

NOTE 3—It is suggested, for purposes of uniformity and closer tolerances in the molded specimen, that the dimensions of the mold be specified and shrinkage compensated for therein. A plate cavity 13.0 ± 0.1 mm (0.510 ± 0.004 in.) in thickness and 29.20 ± 0.05 mm (1.148 ± 0.002 in.) in diameter, with overflow cavities both top and bottom when combined with two end plates, will provide one type of a suitable mold.

5.4 When the standard test specimen is to be replaced by a specimen taken from a vulcanized rubber part, the circular die as used in 5.1 shall be used. The sample thickness may be reduced where necessary by first cutting transversely with a sharp knife and then followed by buffing to the required thickness. The buffing shall be done in accordance with Recommended Practice D 3183.

5.5 For routine testing or product specifications, it is sometimes more convenient to prepare specimens of a different size and shape or both than specified above. When such specimens are used, the results should be compared only with those obtained from specimens of similar size and shape and not with those obtained with the standard specimen. The product specification should in such cases define the specimen as to size and shape. If suitable specimens cannot be prepared from the product, the method of test and allowable limits must be agreed upon by consumer and producer.

6. Procedure

6.1 Original Thickness Measurement—Measure the original thickness of the specimen to the nearest 0.02 mm or 0.001 in. Place the specimen on the anvil of the dial micrometer so that the presser foot will indicate the thickness at the central portion of the top and bottom faces.

6.2 Application of Load—Assemble the specimen in the compression device, using extreme care to place it exactly in the center between the plates to avoid tilting. If the calibrated spring device (Fig. 1) is used, apply the load by tightening the screw until the deflection as read from the scale is equivalent to that shown on the calibration curve for the spring corresponding to a force of 1.0 kN or 400 lbf. With the external loading device (Fig. 2), apply this force to the assembly in

the compression machine or by dead weight, but in the latter case take care to apply the weight gradually without shock. Tighten the nuts and bolts just sufficiently to hold the initial deflections of the specimen and spring. It is imperative that no additional load be applied in tightening the bolts.

6.3 Heat Treatment—Choose a suitable temperature and time for the heat treatment, depending upon the conditions of the expected service. In comparative tests, use identical temperature and heating periods. It is suggested that the test temperature be chosen from those listed in ASTM Recommended Practice D 1349, Rubber—Standard Temperatures and Atmospheres for Testing and Conditioning.¹ Suggested heating periods are 22 h and 70 h. The specimen shall be at room temperature when inserted in the compression device. Place the assembled compression device in the oven within 2 h after completion of the assembly and allow it to remain there for the required heating period in dry air at the test temperature selected. At the end of the heating period, take the device from the oven and remove the specimen immediately and allow it to cool.

6.4 Cooling Period—While cooling, allow the specimen to rest on a thermally nonconducting surface, such as wood, for 30 min before making the measurement of the final thickness.

6.5 Final Thickness Measurement—After the rest period, measure the final thickness at the center of the specimen using the procedure in 6.1.

7. Calculation

7.1 Calculate the compression set as a percentage of the original thickness, as follows:

Compression set, percent = $[(t_o - t_f)/t_o] \times 100$ where:

t_o = original thickness (6.1), and

t_f = final thickness (6.5).

8. Report

8.1 The report shall include the following:

8.1.1 Original dimensions of the test specimen, including the original thickness, t_o .

8.1.2 Actual compressive load on the specimen as determined from the calibration curve of the spring and the spring deflection

reading (4.3.1) or as applied by external loading (4.3.2).

8.1.3 Thickness of the test specimen 30 min after removal from the clamp, t_f .

8.1.4 Type of test specimen used, together with the time and temperature of test.

8.1.5 Compression set, expressed as a percentage of the original thickness, and

8.1.6 Method used (Method A).

METHOD B. COMPRESSION SET UNDER CONSTANT DEFLECTION

9. Apparatus

9.1 Cutting Dies The circular dies used for cutting the test specimens shall be carefully maintained so that the cutting edges are sharp and free of nicks.

9.1.1 Type 1—The circular die used to prepare Type 1 standard test specimens shall have an inside diameter measurement of 29.0 ± 0.5 mm (1.14 ± 0.02 in.).

NOTE 4 The cutting die described in 9.1 or in Fig. 1 of ASTM Methods D 575, Tests for Rubber Properties in Compression² may be used.

9.1.2 Type 2—The circular die used to prepare Type 2 standard test specimens shall have an inside diameter measurement of 13.0 ± 0.2 mm (0.51 ± 0.01 in.).

9.2 Dial Micrometer—A dial micrometer shall be provided to measure the thickness of the specimen. The micrometer shall have an anvil of 9.5 ± 0.5 mm (0.38 ± 0.02 in.) in diameter and a hemispherical presser foot 6 ± 1 mm (0.24 ± 0.04 in.) in diameter. The force of the presser foot shall be 0.8 ± 0.1 N (0.18 ± 0.02 lbf).

NOTE 5 Other ways of measuring specimen thickness before and after testing may be used as agreed upon by producer and consumer. For vulcanizates having a hardness below 35 IRHD the force of the presser foot should be reduced to 0.2 ± 0.05 N (0.04 ± 0.01 lbf).

9.3 Spacer Bars—To maintain the constant deflection required under Method B, spacer bars shall be used.

9.3.1 Spacer bars for Type 1 samples shall have a thickness of 9.5 ± 0.02 mm (0.375 ± 0.001 in.).

9.3.2 Spacer bars for Type 2 samples shall have a thickness of 4.50 ± 0.01 mm (0.177 ± 0.0005 in.).

9.4 Compression Device—The compression device shall consist of two or more flat steel plates between the parallel faces of which the specimens may be compressed as shown in Fig. 3. Steel spacers for the required percentage of compression given in 11.2 shall be placed on each side of the rubber specimens to control their thickness while compressed. The steel surfaces contacting the rubber specimens shall be ground to a maximum roughness of 10 μ m, and then chromium plated and polished. They shall be thoroughly cleaned before each use.

9.5 Oven—The oven used shall conform to the specifications for a Type IIB laboratory oven given in ASTM Specification E 145.

9.6 Plates—The plates between which the test specimen is compressed shall be made of steel of sufficient thickness to withstand the compressive stresses without bending. The surfaces against which the specimen is held shall have a highly polished chromium-plated finish and shall be thoroughly cleaned and wiped dry before each test.

10. Test Specimen

10.1 Type 1 specimen shall be a cylindrical disk cut from a laboratory-prepared slab of thickness 12.5 ± 0.5 mm (0.50 ± 0.02 in.) using the cutting die specified in 9.1.1.

10.2 Type 2 specimen shall be a cylindrical disk cut from a laboratory-prepared slab of thickness 6.0 ± 0.2 mm (0.24 ± 0.01 in.) using the cutting die specified in 9.1.2.

10.3 When cutting a specimen, the specified circular die shall be suitably rotated in a drill press or similar device and lubricated by means of a soap solution. A minimum distance of 13 mm (0.5 in.) shall be maintained between the cutting edge of the die and the edge of the slab. The cutting pressure shall be as light as possible to minimize the cupping of the cut edges.

10.4 Alternative samples of the above test specimens, Types 1 and 2, may be prepared by direct molding or by plying up cylindrical disks cut from a standard sheet prepared in accordance with Recommended Practice D 3182, Rubber—Materials, Equipment, and Procedures for Mixing Vulcanized Compounds and Preparing Standard Vulcanized Sheets.³

NOTE 6 It should be recognized that an equal

time and temperature if used for both the slab and molded specimen will not produce an equivalent state of cure in two types of specimen. A "tighter" cure will be obtained in the molded specimen. Adjustments, preferably in the time of cure, must be taken into consideration if comparisons between the two types of specimen are to be considered valid.

NOTE 7—The mold described in Note 4 may be used for Type 1 specimen. A similar mold for Type 2 specimen would have a plate cavity of 6.3 ± 0.3 mm (0.25 ± 0.01 in.) in thickness and 13.2 ± 0.1 mm (0.53 ± 0.004 in.) in diameter.

10.5 Plyed disks may be obtained from the part in the same manner and, in cases where the thickness must be reduced, it can be cut transversely with a sharp knife and then followed by buffing flat. See Recommended Practice D 3183 for buffing procedures.

10.6 The disks shall be plyed, without cementing, to the thickness required. Such plies shall be smooth, flat, of uniform thickness and shall not exceed seven in number for Type 1 specimens and four in number for Type 2 specimens. Care shall be taken during handling and placing of the test specimen in the test fixture by keeping the circular faces parallel and at right angles to the axis of the cylinder. The results obtained on specimens so prepared may be different from those obtained using a solid specimen and the results may be more variable, particularly if air is trapped between disks.

10.7 For routine testing or product specifications, it is sometimes more convenient to prepare specimens of a different size and shape, or both, than specified above. When such specimens are used, the results should be compared only with those obtained from specimens of similar size and shape and not with those obtained with the standard specimen. The product specification should in such cases define the specimen as to size and shape. If suitable specimens cannot be prepared from the product, the method of test and allowable limits must be agreed upon by consumer and producer.

11. Procedure

11.1 Original Thickness Measurement

Measure the original thickness of the specimen to the nearest 0.02 mm (0.001 in.). Place the specimen on the anvil of the dial micrometer so that the presser foot will indicate the thickness at the center portion of the top and

bottom faces.

11.2 Application of Load—Place the test specimen between the plates of the compression device with the spacers on each side. If only one specimen is being tested, place it in the center. If two specimens are being tested, provide three spacers, one in the center and one outside of each specimen, allowing sufficient clearance for the bulging of the rubber when compressed (Fig. 3). Tighten the bolts so that the plates are drawn together uniformly until they are in contact with the spacers. The percentage of compression employed shall be approximately 25 % for all hardnesses. A suitable mechanical or hydraulic device may be used to facilitate assembling and disassembling the test fixture.

11.3 Heat Treatment—Choose a suitable temperature and time for the heat treatment, depending upon the conditions of the expected service. In comparative tests, use identical temperatures and heating periods. It is suggested that the test temperature be chosen from those listed in Recommended Practice D 1349. Suggested heating periods are 22 h and 70 h. The test specimen shall be at room temperature when inserted in the compression device. Place the assembled compression device in the oven within 2 h after completion of the assembly and allow it to remain there for the required heating period in dry air at the test temperature selected. At the end of the heating period, take the device from the oven and remove the test specimen immediately and allow it to cool.

11.4 Cooling Period—While cooling, allow the test specimen to rest on a thermally non-conducting surface, such as wood, for 30 min before making the measurement of the final thickness.

11.5 Final Thickness Measurement—After the rest period, measure the final thickness at the center of the test specimen using the procedure in 11.1.

12. Calculation

12.1 Calculate the compression set expressed as a percentage of the original deflection as follows:

$$C = [(t_o - t_f)/(t_o - t_s)] \times 100$$

where:

C = compression set expressed as percent

age of the original deflection.

t_o = original thickness of specimen (11.1),
 t_f = final thickness of specimen (11.5), and
 t_s = thickness of the spacer bar used.

13. Report

13.1 The report shall include the following:

13.1.1 Original dimensions of the test specimen including the original thickness, t_o .

13.1.2 Percentage compression of the specimen actually employed.

13.1.3 Thickness of the test specimen 30 min after removal from the clamp, t_f .

13.1.4 Type of test specimen used, together with the time and temperature of test.

13.1.5 Compression set, expressed as a percentage of the original deflection, and

13.1.6 Method used (Method B).

14. Precision

14.1 In column 3 are the coefficients of variation estimated for the ranges of property levels in column 2 for both Methods A and B. In column 4 are the maximum differences (ranges) that should be considered acceptable for two results. These precision data are based

on tests conducted exclusively with pellets prepared by a single, participating laboratory.

| Method | Applicable Range, °F | Coefficient of Variation, % of mean | Acceptable Range of Two Results, DTS, % of mean |
|--------|------------------------------------|-------------------------------------|---|
| A | Single-Operator (repeatability): | | |
| | from 2 to 4 | 14 | 44 |
| | from 6 to 16 | 8 | 23 |
| | Multilaboratory (reproducibility): | | |
| B | from 2 to 4 | 21 | 72 |
| | from 6 to 16 | 12 | 35 |
| | Single-operator (repeatability): | | |
| | from 24 to 74 | 2.3 | 6.5 |
| | Multilaboratory (reproducibility): | | |
| | from 24 to 74 | 9 | 26 |

NOTE 8—These precision data are approximations based on limited results, namely triplicate tests on each of four materials (4 levels) at eight (9 for Method B) laboratories. The degrees of freedom (DF) estimated for the coefficients of variation given in order in column 3 are 16, 48, 7, 21, 72, and 32 respectively.

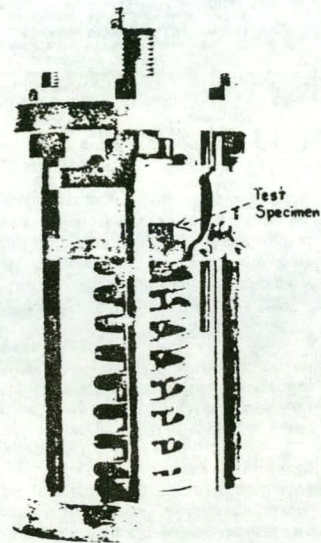


FIG. 1 Device for Compression Set Test, Using Calibrated Spring Loading, Method A

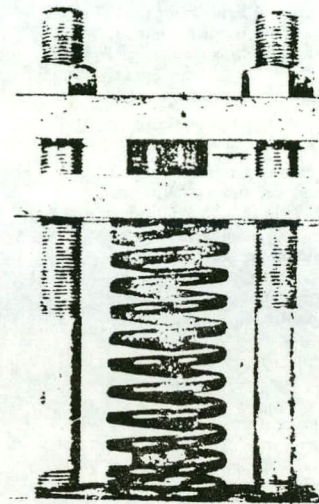


FIG. 2 Device for Compression Set Test, Using External Loading, Method A

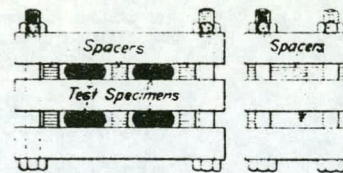


FIG. 3 Device for Compression Set Test Under Constant Deflection, Method B

By publication of this standard no position is taken with respect to the validity of any patent rights in connection therewith, and the American Society for Testing and Materials does not undertake to insure anyone utilizing the standard against liability for infringement of any Letters Patent nor assume any such liability.



Standard Test Methods for RUBBER PROPERTIES IN TENSION¹

This Standard is issued under the fixed designation D 412; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

These methods have been approved for use by agencies of the Department of Defense to replace methods 4001, 4116, -1121, and 4411 of Federal Test Method Standard No. 601 and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 These methods cover the tension testing of rubber at various temperatures. Measurements of tensile stress at given elongations, tensile strength, ultimate elongation, and tensile set are included. The methods are not applicable to the testing of ebonite and similar hard, low-elongation materials.

1.2 The methods appear as follows:

| | Sections |
|--|----------|
| Method A — Dumbbell and Straight Specimens | 11 to 14 |
| Method B — Cut Ring Specimens | 15 to 18 |

1.3 The agreement between data from Method A and Method B specimens is generally very good. However, when such specimens are used, the results should be compared only with those obtained from specimens of similar size and shape.

2. Applicable Documents

2.1 ASTM Standards:

D1349 Recommended Practice for Rubber—Standard Temperatures and Atmospheres for Testing and Conditioning²

D3182 Recommended Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets³

D3183 Recommended Practice for Rubber—Preparation of Pieces for Test from Other Than Standard Vulcanized Sheets⁴

E 4 Verification of Testing Machines⁵

3. Summary of Methods

3.1 The methods start with a piece taken from the sample and includes: (1) the preparation of the specimen and (2) testing of the specimen. Specimens may be in the shape of a dumbbell, ring, or straight piece of uniform section and may be of various sizes.

3.2 Measurements of tensile stress, tensile strength, and ultimate elongation are made on specimens that have not been prestressed. Tensile stress and tensile strength are based on the original cross sectional area of a uniform section of the specimen.

3.3 Measurement of tensile set is made after a specimen has been extended and allowed to retract by a prescribed procedure without prestressing. Measurement of set after break is also described.

4. Significance

4.1 The tensile properties of rubber are not intrinsic characteristics, but depend on both the material and the conditions of test such as rate of extension, temperature, humidity, geometry of specimen, inertia of dynamometer

¹ These methods are under the jurisdiction of ASTM Committee D-11 on Rubber and Rubber-Like Products, and are the direct responsibility of Subcommittee D11.10 on Physical Testing.

² Current edition approved Nov. 28, 1975. Published February 1976. Originally published as D 412-35 T. Last previous edition D 412-68.

³ Annual Book of ASTM Standards, Parts 37 and 38.

⁴ Annual Book of ASTM Standards, Part 37.

⁵ Annual Book of ASTM Standards, Part 41.

Detailed drawings available from ASTM Headquarters, 1916 Race St., Philadelphia, Pa. 19103. Request Adj. No. 12-404120-00.

in tester, and environmental or mechanical preconditioning. For this reason, the tensile properties are only relative indicators of quality and various rubbers should only be compared when tested under the same conditions. The report should include a statement of the test conditions.

4.2 Modest changes in rate of extension caused by type of tester (pendulum versus inertialess) have little or no effect on the tensile properties of most rubbers.

4.3 Temperature may have a significant effect on tensile properties and therefore should be controlled.

4.4 For most rubbers, humidity has a small effect that can be neglected. There are sensitive rubbers that necessitate humidity conditioning.

4.5 Tensile strength and ultimate elongation depend on the volume of the specimen (or the volume of the restricted portion in the case of dumbbells) and stress concentration due to shape of specimens. For ring specimens, ultimate elongation is based on inside circumference where the stress is highest. Elongation of ring specimens theoretically should be based on mean circumference to give values for tensile stress at a given elongation in accord with that determined with dumbbell or straight specimens.

4.6 Inertia-type dynamometers may give erroneous results if the load capacity of the tester is too high or too low for the material being tested, or if there is friction. Inertialess dynamometers have greater versatility, but calibration may be less stable.

4.7 Tensile set represents deformation, after stretching and retraction, that is partly permanent and partly recoverable. For this reason, the periods of extension and recovery and other conditions of test must be controlled to obtain comparable results.

5. Definitions

5.1 *elongation*—extension produced by a tensile stress.

5.2 *elongation, ultimate*—the elongation at the time of rupture.

5.3 *piece*—the portion of the sample that is prepared for testing.

5.4 *sample*—the portion or unit(s) selected to represent the lot.

5.5 *set after break*—the tensile set of a specimen stretched to rupture.

5.6 *specimen*—a piece of material appropriately shaped and prepared so that it is ready to use for a test.

5.7 *tensile set*—the extension remaining after a specimen has been stretched and allowed to retract in a specified manner expressed as a percentage of the original length.

5.8 *tensile strength*—the maximum tensile stress applied during stretching a specimen to rupture.

5.9 *tensile stress*—a stress applied to stretch a test piece (specimen).

5.10 *tensile stress at given elongation*—the stress required to stretch the uniform cross section of a test specimen to a given elongation.

6. Apparatus

6.1 *Testing Machine*—Tension tests shall be made on a power-driven machine so equipped that a uniform rate of grip separation at 8.5 ± 0.8 mm/s (20 ± 2 in./min) (Note 1) for at least 750 mm (30 in.) is maintained, and having a suitable dynamometer and an indicating or recording device for measuring the applied force within $\pm 2\%$. If the capacity range cannot be changed during a test as in the case of pendulum dynamometers, the applied force at break shall be measured within $\pm 2\%$, and the smallest tensile force measured shall be accurate to within $\pm 10\%$. If the dynamometer is of the compensating type for measuring tensile stress directly, means shall be provided to adjust for the cross-sectional area of the specimen. The response of the recorder shall be sufficiently rapid that the applied force is measured with the requisite accuracy during the extension of the specimen to rupture. If the tester is not equipped with a recorder, a device shall be provided that indicates after rupture the maximum force applied during extension. Testers shall be capable of measuring elongation in increments of 10%.

NOTE 1—A rate of separation of 17.0 ± 1.6 mm/s (40 ± 4 in./min) may be used in routine work (with notation of the speed used made on the report), but in case of dispute, the rate shall be 8.5 ± 0.8 mm/s (20 ± 2 in./min).

6.2 *Test Chamber for Elevated and Low Temperature*—The test chamber shall con-

form to the following requirements:

6.2.1 Air shall be circulated through the chamber with a speed of 1 to 2 m/s (200 to 400 f/min) at the location of the grips and specimens, and maintained within 2°C of the specified temperature.

6.2.2 A calibrated sensing device shall be located near the grips for measuring the actual temperature.

6.2.3 The chamber shall be vented to an exhaust system or the outside atmosphere to remove any toxic fumes liberated at high temperatures.

6.2.4 Provision shall be made for suspending specimens vertically near the grips for conditioning prior to test. The specimens should not touch each other or the sides of the chamber except for momentary contact when agitated by the circulating air.

6.2.5 Suitable fast-acting grips for manipulation at high or low temperature shall be provided to permit placing test specimens in the grips or around the spindles, whichever is applicable, in the shortest time possible to minimize any change in the temperature of the chamber.

6.2.6 The dynamometer shall be suitable for use at the temperature of test or it shall be thermally insulated from the chamber.

6.2.7 Provision shall be made for measuring elongation of specimens in the chamber. If a scale is used to measure the extension between bench marks, the scale shall be located parallel and close to the grip path during extension and shall be controlled from outside the chamber.

6.3 *Micrometers*—The dial micrometer used to measure the thickness of flat specimens shall be capable of exerting a pressure of 25 ± 5 kPa (3.6 ± 0.7 psi) on the specimens (Note 3) and measuring the thickness to within ± 0.025 mm (0.001 in.). For flat specimens the foot in contact with Die C specimens shall be at least 6 mm (0.236 in.) in diameter. The anvil of the micrometer shall be at least 35 mm (1.4 in.) in diameter and shall be parallel to the face of the contact foot. For ring specimens, the base used to measure the radial width shall be at least 12 mm (0.5 in.) long and 15.5 ± 0.3 mm (0.61 ± 0.01 in.) in diameter. Curved feet to fit the curvature of the ring may also be used.

NOTE 2—Dial micrometers exerting a force of 0.80 ± 0.15 N (85 gf) on a circular foot 6.35 mm (0.25 in.) in diameter, or 0.20 ± 0.04 N (20 gf) on a circular foot 3.2 mm (0.125 in.) in diameter conform to this pressure requirement. A micrometer should not be used to measure the thickness of specimens narrower in width than the diameter of the foot unless the contact pressure is properly adjusted.

6.4 *Apparatus for Tensile Set Test*—The testing machine described in 6.1 or an apparatus similar to that shown in Fig. 1 may be used. A stop watch or other suitable timing device that will register the time in minutes for at least 30 min shall be provided. A scale or other device shall be provided for measuring tensile set to within 1%.

7. Selection of Test Specimens

7.1 The following information should be considered in making a selection:

7.1.1 Since "grain" will have a bearing on stress-strain results, dumbbell or straight specimens should be cut in the lengthwise portion of the specimen will be parallel with the mill direction when known. Ring specimens enable the tensile stress with and across the grain to be averaged.

7.1.2 Ring specimens enable elongation to be measured by grip separation, but the elongation across the radial width of ring specimens is not uniform. To minimize this effect, the width of ring specimens (as is the case with the Type I ring) must be small compared to the diameter.

7.1.3 Straight specimens tend to break in the grips and are used only when it is not feasible to prepare another type of specimen.

7.1.4 The size of specimen will be influenced by the material, test equipment, and the piece available for test. A longer specimen may be used for rubbers having low ultimate elongations to improve the precision of measurement.

8. Calibration of Testing Machine

8.1 Calibrate the testing machine in accordance with Procedure A of Methods E 4. If the dynamometer is of the strain-gage type, calibrate the tester at one or more forces daily, in addition to the requirements in Sections 7 and 18 of Method E 4. Testers having pendulum dynamometers may be calibrated as follows:

place one end of a dumbbell specimen in the upper grip of the testing machine. Remove the lower grip from the machine and attach to the specimen. To this lower grip, attach a hook suitable for holding known masses. Suspend a mass from the hook on the specimen in such a way (Note 3) as to permit the mass assembly to rest on the machine grip holder. If the machine has a dynamometer head of the compensating type, calibrate it at two or more settings of the compensator. Start the motor and run as in normal testing until the mass assembly is freely suspended by the specimen. If the dial or scale (whichever is normally used in testing) does not indicate the force applied (or its equivalent in stress for compensating tester) within the specified tolerance, thoroughly check the machine for excess friction in the bearings and all other moving parts. After eliminating as nearly as possible all the excess friction, recalibrate the machine at a maximum of three points, using known masses to produce forces of approximately 10, 20, and 50 % of capacity. The mass of the lower grip and hook shall be included as part of the calibration mass. If pawls and ratchet are used during test, they should also be used during the calibration. Friction in the head can be checked by calibrating with the pawls up.

NOTE 3—It is advisable to provide a means for preventing the masses from falling to the floor in case the dumbbell should break.

8.2 A rapid and frequent approximate check on accuracy of the tensile tester calibration may be obtained by using a spring calibration device.

9. Test Temperature

9.1 Unless otherwise specified, the standard temperature for testing shall be $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$). If the material is affected by moisture, the relative humidity shall be maintained at $50 \pm 5\%$ and the specimen shall be conditioned for at least 24 h, prior to testing. When testing at some other temperature is required, the temperature specified shall be one of those listed in Recommended Practice D 1349, and the report shall include a statement of the temperature at which the test was made. Specimens shall be conditioned for at least 3 h when the test temperature is $23 \pm 2^\circ\text{C}$.

9.2 For exposure at any temperature above standard (see 9.1), condition Method A specimens for 10 ± 2 min and Method B specimens for 6 ± 2 min (Note 4). Place each specimen in the test chamber at intervals ahead of testing so that all specimens of a series will be in the test chamber the same length of time, that is, if 30 s is required to run the test for Method A specimens, the first specimen placed in the test chamber 10 min prior to testing, would be followed by other specimens at 30 s intervals. The conditioning time at elevated temperature must be limited to avoid additional curing or heat aging.

NOTE 4: Caution—Suitable heat- or cold-resistant gloves should be worn for hand and arm protection when testing at high or low temperatures. At high temperatures, an air mask is very desirable when the door of the chamber is opened to insert specimens; toxic fumes may be present and should not be inhaled.

9.3 For testing at sub-ambient temperature, condition the specimen at least 10 min prior to testing.

10. Characteristics of Piece Tested

10.1 The median of the values for three specimens shall be taken as the characteristics of the piece of rubber tested, except that under the following conditions, the median of the values for five specimens shall be used:

10.1.1 If one or two values do not meet the specified requirements when testing for compliance with specifications.

10.1.2 If referee tests are being made.

METHOD A—DUMBBELL AND STRAIGHT SPECIMENS

11. Apparatus

11.1 *Dies*—The shapes and dimensions of dies for preparing dumbbell specimens shall conform with those shown in Fig. 2. The inside faces in the reduced section shall be polished and perpendicular to the plane formed by the cutting edges for a depth of at least 5 mm (0.2 in.). The dies shall be sharp and free of nicks in order to prevent ragged edges on the specimen.

NOTE 5—Careful maintenance of die cutting edges is of extreme importance and can be obtained by light daily honing and touching up the cutting edges with jewelers' hard honing stones. The condition of the die may be judged by investigating the rupture point on any series of broken specimens. When broken specimens are removed from the clamps of the testing machine, it is advan-

tageous to pile these specimens and note if there is any tendency to break at or near the same portion of each specimen. Rupture points consistently occurring at the same place may be an indication that the die is dull, nicked, or bent at that particular position.

11.2 Bench Marker. having two parallel straight marking surfaces ground smooth in the same plane. The surfaces shall be between 0.05 and 0.08 mm (0.002 and 0.003 in.) in width and at least 15 mm (0.6 in.) in length. The angles between the marking surfaces and the sides shall be at least 75 deg. The distance between the centers of the marking surfaces shall be within 1 % of the required distance.

11.3 Ink Applicator. having plane unyielding surface (for example, hardwood, plate glass, or plastic). The ink shall have no deteriorating effect on the specimen and shall be of contrasting color to that of the specimen.

11.4 Grips—The tester shall have two grips, one of which shall be connected to the dynamometer.

11.4.1 Grips for testing dumbbell specimens shall tighten automatically and exert a uniform pressure across the gripping surfaces, increasing as the tension increases in order to prevent uneven slipping and to favor failure of the specimen in its constricted section. Constant-pressure pneumatic-type grips are also satisfactory. If possible, at the end of each grip, a positioning device is recommended for positioning specimens to the same depth in the grip and aligning them with the direction of pull.

11.4.2 Grips for testing straight specimens shall be either constant-pressure pneumatic, wedged, or toggle type designed to transmit the applied force over a large surface area of the specimen.

12. Preparation of Test Specimens

12.1 Dumbbell Specimens—The piece of rubber to be tested shall, whenever possible, be flat, not less than 1.5 mm (0.060 in.) nor more than 3 mm (0.120 in.) in thickness and of a size that will permit cutting a specimen by one of the standard methods. If obtained from a manufactured article, the piece of rubber shall be free of surface roughness, fabric layers, etc., in accordance with the procedure described in Recommended Practice D 3183. All specimens shall be cut so the

lengthwise portion of the specimen will be parallel with the grain unless otherwise specified with the exception that specimens of belts wider than 300 mm (12 in.) of sheet packing, of hose, or of dredging sleeves more than 100 mm (4 in.) in inside diameter shall be taken in the transverse direction. In the case of specially cured sheets prepared in accordance with Recommended Practice of D 3182, the specimen shall be 2.0 ± 0.2 mm (0.080 ± 0.008 in.) in thickness and died out in the direction of the grain. Dumbbell specimens shall conform in shape to those shown in Fig. 2. Unless otherwise specified, Die C (Fig. 2) shall be used to prepare the specimens. In all cases, the cutting of test specimens shall be done with a single stroke of the cutting tool so as to assure obtaining smoothly cut surfaces.

12.1.1 Marking Dumbbell Specimens—Dumbbell specimens shall be marked with the bench marker described in 11.2. The sample shall be under no tension at the time it is marked. Marks shall be placed on the reduced section of the specimen equidistant from its center and perpendicular to its longitudinal axis. The distance between centers of the marks shall be within 1 % of either 25.00 ± 0.25 mm (or 1.00 ± 0.01 in.) on specimens cut with Dies C and D, and either 50.00 ± 0.50 mm (or 2.00 ± 0.02 in.) on specimens cut with the other dies shown in Fig. 2. Bench marks 20 mm (0.79 in.) apart may be used to measure elongation where scales graduated in millimetres are required.

12.1.2 Measuring Dumbbell Specimens—Three measurements shall be made for thickness, one at the center and one at each end of the reduced section of the specimen. The median of the three measurements shall be used as the thickness in calculating the cross-sectional area, except that specimens for which the differences between the maximum and minimum thickness exceeds 0.08 mm (0.003 in.) shall be discarded. The width of the specimen shall be taken as the distance between the cutting edges of the die in the restricted section.

12.2 Straight Specimens—Straight specimens may be prepared where it is not practical to cut either a dumbbell or a ring specimen, as in the case of a narrow rubber strip, small tubing, or electrical insulation. These

specimens shall be of sufficient length to permit their installation in the grips used in the test. Bench marks shall be placed on the specimens as described for dumbbell specimens in 12.1.1 to determine the cross sectional area of straight specimens in the form of tubes, the weight, length, and density of the specimen may be determined. The cross-sectional area may then be calculated from the measurements as follows:

$$A = M/DL$$

where:

A = cross-sectional area,
M = mass,
D = density, and
L = length.

13. Procedure

13.1 Determination of Tensile Stress, Tensile Strength, and Ultimate Elongation—Place dumbbell or straight specimens in the grips of the testing machine, using care to adjust the specimen symmetrically in order that the tension will be distributed uniformly over the cross section. If tension is greater on one side of the specimen than on the other, the bench marks will not remain parallel and maximum strength of the rubber will not be developed. Unless otherwise specified, the rate of grip separation shall be 8.5 ± 0.8 mm/s (20 ± 2 in./min) (Note 1). Start the machine and note continuously the distance between the two bench marks, taking care to avoid parallax. Record the stress at the elongation specified and at the time of rupture, preferably by means of an autographic or spark recorder. At rupture, measure and record the elongation to the nearest 10 % on the scale.

13.2 Determination of Tensile Set—Place the specimen in the grips of the testing apparatus and adjust symmetrically so as to distribute the tension uniformly over the cross section. Separate the grips at a rate of speed as uniformly as practicable, requiring about 15 s to reach the specified elongation. Then hold the specimen at the specified elongation for 10 min, release quickly without allowing it to snap back, and allow to rest for 10 min. At the end of the 10-min rest period, measure the distance between the bench marks to the nearest 1 % of the original length and calculate the tensile set. In stretching the specimen, it has

been found convenient to use a measured rod of a length equal to the exact distance required between the two bench marks. Holding the rod behind the test specimen while it is being stretched simplifies the operation and reduces the chance of stretching the specimen more than the required amount. Use a stop watch or equivalent timer for recording the time required for the various operations.

13.3 Determination of Set at Break—Set at break is the set determined on the specimen when stretched to rupture. Ten minutes after the specimen is broken, fit the two pieces carefully together so that they are in contact over the full area of the break. Measure the distance between the bench marks and calculate the set.

14. Calculation

14.1 Calculate the tensile stress as follows:

$$\text{Tensile stress} = F/A \quad (1)$$

where:

F = observed force, and
A = cross-sectional area of the unstretched specimen.

14.2 Calculate the tensile strength by letting F in Eq 1 be equal to the force required to break the specimen. Tensile stress and tensile strength are expressed in either megapascals or pounds-force per square inch.

14.3 Calculate the elongation as follows:

$$\text{Elongation, \%} = [(L - L_0)/L_0] \times 100 \quad (2)$$

where:

L = observed distance between the bench marks on the stretched specimen, and
L₀ = original distance between the bench marks.

14.4 Calculate the ultimate elongation by letting L in Eq 2 be equal to the distance between the bench marks at the time of rupture.

14.5 Calculate the tensile set by substituting the distance between the bench marks after the 10-min retraction period for L in Eq 2.

METHOD B—CUT RING SPECIMENS

15. Apparatus

15.1 Specimens Cut from Flat Sheets.
15.1.1 Cutter—The cutter shown in Fig. 3

is recommended. The cutter is rotated by a machine having sufficient torque to maintain a speed of at least 31 rad/s (300 rpm) during cutting and having precision bearings and spindle for holding the cutter with a maximum run-out of 0.010 mm (0.0004 in.). The machine should have a base perpendicular to the spindle, and preferably an arrangement for positioning the rubber piece with respect to the spindle.

15.1.2 Rubber Holding Device—A vacuum holding plate similar to the one shown in Fig. 4 is recommended. The plate is clamped to the base or table of the cutting machine. Other size plates may be used depending on the size of the rubber piece. The holding device has plane, parallel upper and lower surfaces, and is connected to a source of reduced pressure to hold the rubber piece firmly on the plate without distortion.

15.2 Specimens Cut from Tubes:

15.2.1 Cutter—A sharp knife edge or razor blade held firmly in the tool post of a lathe.

15.2.2 Mandrel—A hard rubber or wood mandrel to fit the tube, used to hold and rotate the rubber piece in the lathe.

15.3 Testing Machine Grips—The assembly shown in Fig. 5 is recommended to extend the ring. The shaft is 4.75 mm (0.187 in.) in diameter. The surface of the shaft should be lubricated with a material that does not affect the rubber, particularly for tests at elevated temperatures.

15.4 Circumference Gages—A cone or frustum of a cone may be used to measure the inside diameter of ring specimens and shall have steps having diametric intervals not exceeding 2% of the diameter to be measured. Alternatively, for rings cut with the cutter shown in Fig. 3, go-no-go gages may be used.

16. Preparation of Test Specimens

16.1 The ring specimen shall be prepared from flat sheets not less than 1.0 mm (0.04 in.) nor more than 3.0 mm (0.12 in.) in thickness and of a size that will permit cutting the specimen. Ring specimens shall conform to the dimensions shown in Fig. 3. Unless otherwise specified, the Type 1 cut ring shall be used.

16.1.1 Place the blades in their proper slots in the holder, and using the blade-setting

gage, set the blade depth as indicated in Fig. 3. Be extremely careful that the blade alignment is good. Place the cutter in position in the drill press and adjust the machine so that the bottom of the blade holder is approximately 13 mm (0.5 in.) above the rubber slab on the hold-down plate.

16.1.2 Set the stop on the vertical travel so that the tips of the cutting blades just penetrate the rubber piece. Lower the cutter at a steady uniform rate until it reaches the stop, then bring it back to its initial position. Be sure that when the blade holder is fully lowered the bottom of the holder does not contact the rubber. For specimens that are thicker than 2.25 mm (0.090 in.) the cutting blades will have to be adjusted. A mild soap solution for lubrication may be used on the cutting blades when cutting the ring specimen.

16.2 Preparation from Tubes—The tube is placed on a mandrel, preferably slightly larger than the inner diameter of the tube so it is held firmly. The mandrel and tube are rotated in a lathe and rings of desired width are cut. Thin-walled rubber tubes that can be laid flat may be cut with dies having two parallel blades.

16.3 Rubber Rings—Products in the form of rings are tested without special preparation if their size permits testing in the tensile tester. Such products include ring seals and rubber bands.

16.4 Measuring Ring Specimens—The radial width is measured at three locations distributed around the circumference of the ring using the micrometer as specified in 6.3. The thickness of the disk cut from the inside of the ring is measured with the micrometer described in 6.3. The cross-sectional area is calculated from the medians of three width and three thickness measurements. Alternatively to measuring the width and thickness, the area may be determined from the mass, density, and mean circumference of the ring.

NOTE 6—For rings of known density and mean circumference, the measurement of mass is more rapid and reliable for determining area than dimensional measurements.

16.5 Ring Circumference—The inside circumference of ring specimens can be determined by the stepped cone or go-no-go gages

described in 15.4, employing no stress in excess of that necessary to overcome any ellipticity of the ring. The mean circumference is obtained by adding to the inside circumference the product of the radial width and pi (3.14).

NOTE 7—Types 1, 2, and 3 ring specimens in Fig. 3 are designed for autographic measurement of elongation. Type 1 has an inside circumference of 2 in. so that spindle separation in inches equals the ultimate elongation in percent. Type 2 has a mean circumference of 100 mm so that each 50 mm of grip separation equals 100% elongation. Type 3 has a mean circumference of 4 in. so that each 2 in. of grip separation equals 100% elongation.

17. Procedure

17.1 Determination of Tensile Stress, Tensile Strength, and Ultimate Elongation:

NOTE 8—For materials having a yield point under 20% elongation when tested at 8.50 ± 0.08 mm/s (20 ± 2 in./min), the rate shall be reduced to 0.85 mm/s (2.0 in./min). If the material still has a yield point under 20% elongation, the rate shall be reduced to 0.085 mm/s (0.20 in./min). The rate of separation shall be reported.

17.1.1 Place the ring specimen around the lubricated spindles. The initial setting between the spindles from center to center shall be adjusted so that a minimum of tension is placed on the specimen prior to testing. Unless otherwise specified, spindle separation shall be 8.5 ± 0.8 mm/s (20 ± 2 in./min). Start the test machine and record continuously the distance between the centers of the spindles. If the stress and strain are not autographically recorded, predetermine the distance between the centers of the spindles as prescribed in 17.4 for the elongation specified for the material under test.

17.1.2 Record the stress at the predetermined distance between the centers of the spindles and at the time of rupture, preferably by means of an autographic recorder. At rupture, measure the distance between the centers of spindles to within 2.5 mm (0.1 in.) and record.

18. Calculation

18.1 Calculate the tensile stress as follows:

$$\text{Tensile stress} = F/2A$$

where:

F = observed force, and
 $2A$ = twice the cross-sectional area.

18.2 Calculate the tensile strength by setting F in the above equation for tensile stress be equal to the force required to break the specimen.

18.3 Calculate the ultimate elongation as follows:

$$E = 100(2D + G - C)/C$$

where:

E = ultimate elongation, %
 C = inside circumference of ring specimen,
 D = distance between centers of spindles and
 G = circumference of one spindle

18.4 Determine spindle separation at elongation in determining the tensile stress at a specified elongation as follows:

$$D = 1/2[(EM/100) + C]$$

where:

D = distance between spindle centers for specified elongation, and
 M = mean circumference of ring specimen.

19. Report

19.1 The report shall include the following:

19.1.1 Results calculated in accordance with Section 14 or 18, whichever is applicable.

19.1.2 Type of test specimen.

19.1.3 Rate of extension, if other than 8.50 ± 0.08 mm/s (20 ± 2 in./min).

19.1.4 Temperature and humidity of test room, if other than 23 ± 2°C and 50 ± 5%, and

19.1.5 Temperature of testing, if other than 23 ± 2°C (73.4 ± 3.6°F).

19.2 In the case of a dispute or referee tests are being made, the following items shall also be reported:

19.2.1 All observed and recorded data on which the calculations are based

19.2.2 Type of testing machine

19.2.3 Date of test, and

19.2.4 Date of vulcanization of the rubber, if known.

20. Precision-Method A

20.1 Tensile Stress, Tensile Strength, and Ultimate Elongation (Note 9)—Section 3 of Table 1 contains the coefficients of variation that have been estimated at a minimum of 612

degrees of freedom (DF) for the ranges of property levels in Column 2. In Column 4 are the differences (ranges) that should be considered acceptable for two results. These precision data are based on tests conducted exclusively with vulcanized sheets prepared by the National Bureau of Standards.

NOTE 9—These precision data are based on results from 69 (67 to 72) laboratories. 10 materials (9 for tensile stress) covering the range of properties tabulated above, and short-term replication, consisting of tests on 2 sheets of essentially the same material, usually on the same day rather than on separate days. Therefore, any day-to-day variability due to differences in set-up, etc., over and above the variability sheet-to-sheet on the same day, is largely reflected in the multilaboratory precision rather than in the single operator precision.

20.2 *Tension Set and Set After Break*

(Note 10)—Column 3 of Table 2 contains the coefficients of variation estimated for the ranges indicated in Column 2 of the properties listed in Column 1. In Column 4 are the differences (ranges) that should be considered acceptable for two results. The data are based on tests conducted with vulcanized sheets prepared by different laboratories.

NOTE 10—These precision data are approximations based on limited data from tests with 6 specimens from each of 9 materials at 7 laboratories. The minimum degrees of freedom are estimated at 76 and 10 for single-operator and multilaboratory precision, respectively.

TABLE 1 Precision of Tensile Strength, Tensile Stress, and Ultimate Elongation (Method A)

| Property | Applicable Range of Property | Coefficient of Variation, % of mean, S % | Acceptable Range of Two Results, % of mean, D2S % |
|-------------------------------------|--|--|---|
| Single-operator (repeatability): | | | |
| Tensile strength | from 16.5 to 32.0 MPa (2400 to 4600 psi) | 3.0 | 8.0 |
| Tensile stress, at 300 % elongation | from 5.5 to 17.0 MPa (800 to 2500 psi) | 3.0 | 8.0 |
| Ultimate elongation | from 450 to 740 % | 3.0 | 7.0 |
| Multilaboratory (reproducibility): | | | |
| Tensile strength | from 16.5 to 32.0 MPa (2400 to 4600 psi) | 6.0 | 16.0 |
| Tensile stress, at 300 % elongation | from 5.5 to 17.0 MPa (800 to 2500 psi) | 6.0 | 17.0 |
| Ultimate elongation | from 450 to 740 % | 5.0 | 13.0 |

TABLE 2 Precision of Tension and Permanent Set

| Method | Applicable Range, % | Coefficient of Variation, % of mean, S % | Acceptable Range of Two Results, % of mean, D2S % |
|------------------------------------|---------------------|--|---|
| Single-operator (repeatability): | | | |
| Permanent set after break | from 4 to 10 | 31 | 30 |
| | from 11 to 18 | 6 | 17 |
| | from 19 to 32 | 5 | 13 |
| Tension set | from 2 to 7 | 8 | 23 |
| | from 8 to 17 | 3 | 8 |
| | from 18 to 26 | 3 | 8 |
| Multilaboratory (reproducibility): | | | |
| Permanent set after break | from 4 to 10 | 23 | 66 |
| | from 11 to 18 | 14 | 44 |
| | from 19 to 32 | 11 | 33 |
| Tension set | from 2 to 7 | 31 | 92 |
| | from 8 to 17 | 17 | 53 |
| | from 18 to 26 | 13 | 40 |

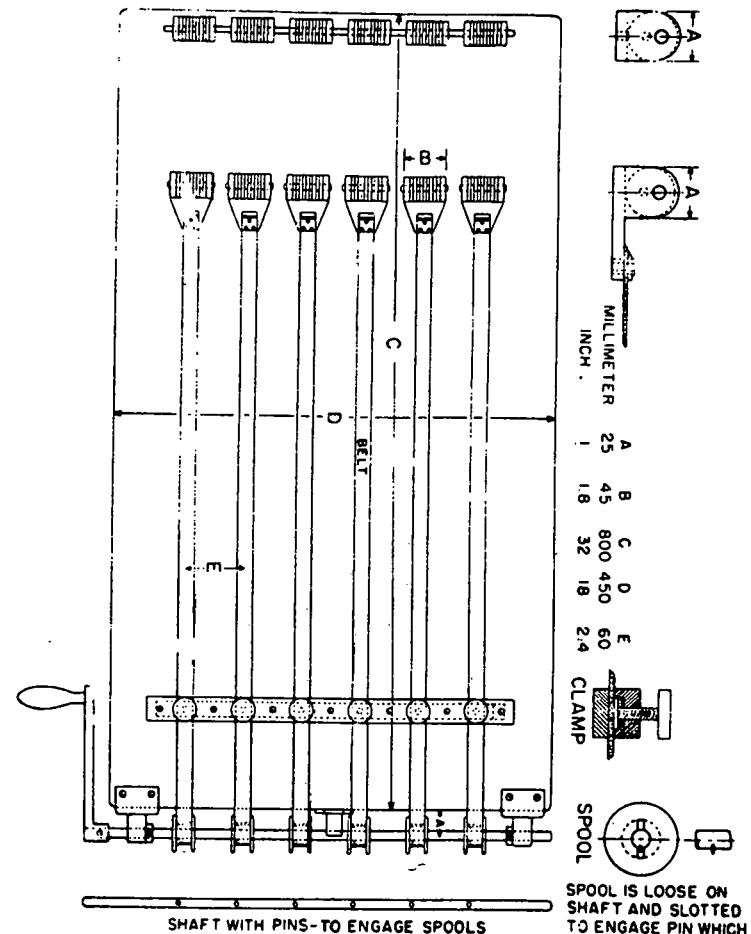
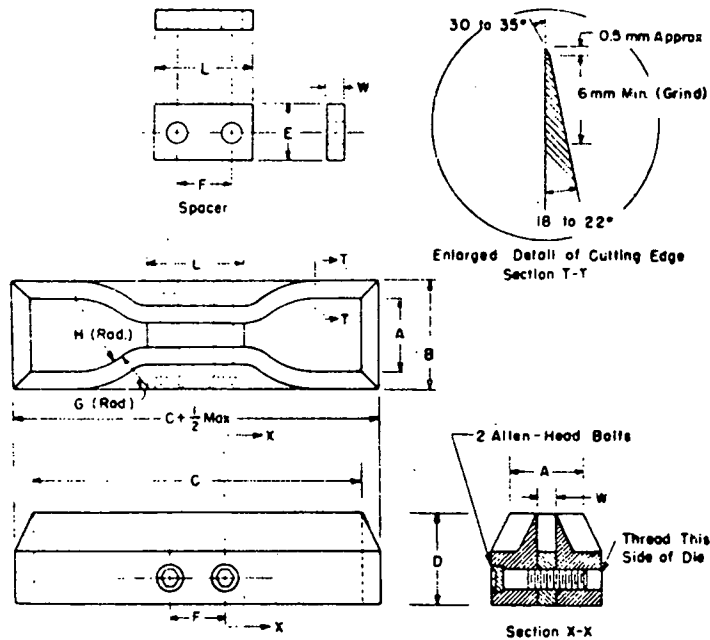


FIG. 1 Apparatus for Tensile Set Test.

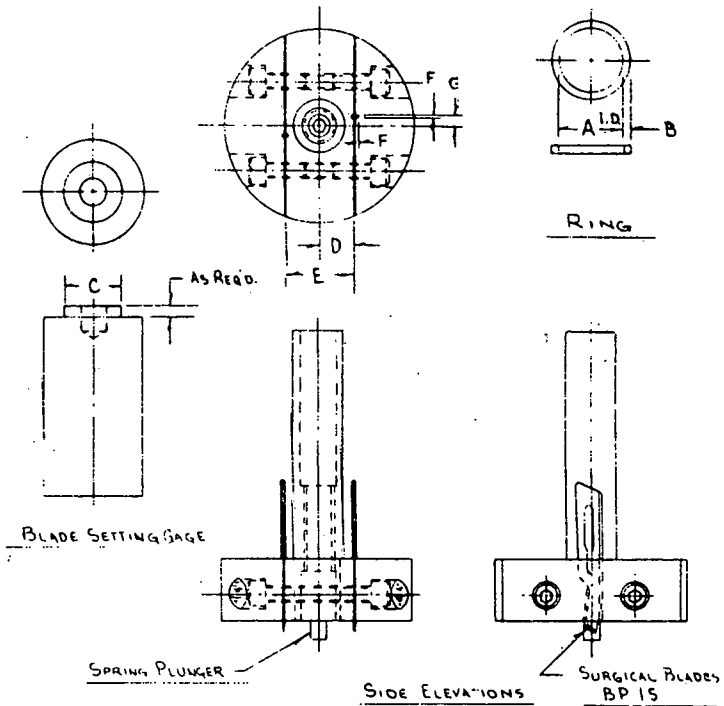


Dimensions of Standard Dumbbell Dies*

| Dimension | Units | Tolerance | Die A | Die B | Die C | Die D | Die E | Die F |
|-----------|-------|-----------|-------|-------|-------|-------|-------|-------|
| A | mm | ±0.04 | 25 | 25 | 25 | 16 | 16 | 16 |
| B | mm | max | 40 | 40 | 40 | 30 | 30 | 30 |
| C | mm | max | 140 | 140 | 140 | 125 | 125 | 125 |
| D | mm | ±0.04 | 13 | 13 | 13 | 13 | 13 | 13 |
| D-L | mm | ±0.04 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 |
| F | mm | ±0.04 | 14 | 14 | 14 | 14 | 14 | 14 |
| G | mm | ±0.04 | 0.56 | 0.56 | 0.56 | 0.56 | 0.56 | 0.56 |
| H | mm | ±0.04 | 25 | 25 | 25 | 16 | 16 | 16 |
| L | mm | ±0.04 | 2.32 | 2.32 | 2.32 | 2.32 | 2.32 | 2.32 |
| W | mm | ±0.04 | 12 | 12 | 12 | 6 | 6 | 6 |

* For dies used in slicing machines it is preferable that this tolerance be ± 0.5 mm or ± 0.01 .

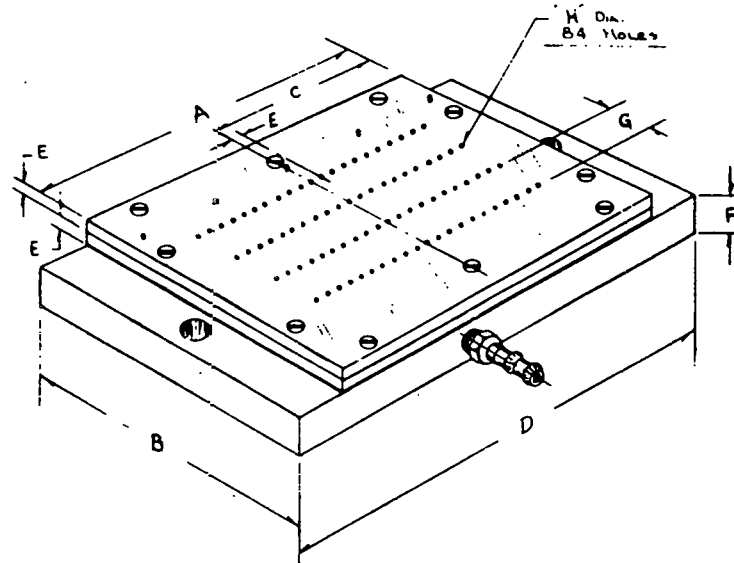
FIG. 2 Standard Dies for Cutting Dumbbell Specimens.



| Dimension | Type 1 | | Type 2 | | Type 3 | | Type 4 (ISO) | |
|-----------|---|----------------|------------|---------------|------------|---------------|--------------|---------------|
| | mm | in. | mm | in. | mm | in. | mm | in. |
| A | 16.18 ± 0.05 | 0.637 ± 0.002 | 29.5 ± 0.1 | 1.161 ± 0.004 | 30.0 ± 0.1 | 1.181 ± 0.004 | 44.6 ± 0.1 | 1.756 ± 0.004 |
| B | 1.02 ± 0.01 | 0.040 ± 0.0007 | 2.0 ± 0.02 | 0.079 ± 0.001 | 2.0 ± 0.02 | 0.079 ± 0.001 | 4.0 ± 0.04 | 0.157 ± 0.002 |
| C | <A | <A | <A | <A | <A | <A | <A | <A |
| D | One-half A dimension less half of thickness of cutting blade. | | | | | | | |
| E | Sum of B and D dimensions. | | | | | | | |

NOTE—The slot for the cutting blade must be positioned so the point is on the diameter perpendicular to the slot. For Bard-Parker blade No. 15, the slot is 5.4 ± 0.1 mm wide, 0.35 ± 0.02 mm deep, and offset from the center line in the direction of rotation by 1.7 mm.

FIG. 3 Ring Cutter.



| Dimension | mm | in. | Dimension | mm | in. |
|-----------|-----|-----|-----------|-----|-------|
| A | 178 | 7.0 | E | 6 | 0.23 |
| B | 152 | 6.0 | F | 19 | 0.75 |
| C | 89 | 3.5 | G | 23 | 0.90 |
| D | 229 | 9.0 | H | 1.5 | 0.062 |

FIG. 4 Vacuum Holding Plate.

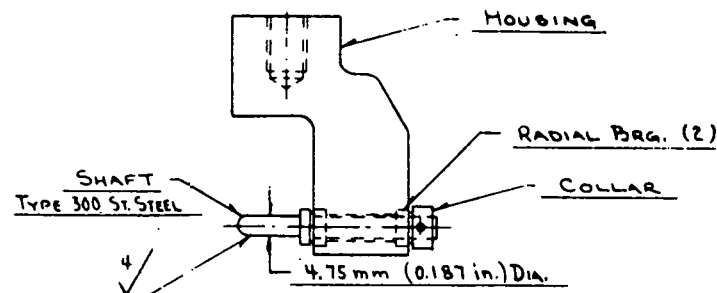


FIG. 5 Assembly Ring Tensile Test Fixture.



Designation: D 518 - 61 (Reapproved 1974)¹

American National Standard Z39.1-1971
Approved July 27, 1971
By American National Standards Institute

Standard Test Method for RUBBER DETERIORATION—SURFACE CRACKING¹

This Standard is issued under the Fixed designation D 518; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

¹ NOTE—Editorial changes were made in Section 7 in October 1974.

1. Scope

1.1 This method covers three procedures that estimate the comparative ability of soft rubber compounds to withstand the effects of normal weathering, or exposure in an atmosphere containing controlled amounts of ozone. It does not apply to testing of electrical insulation or other rubber parts where high concentrations of ozone prevail due to electrical discharge, nor to testing of materials ordinarily classified as hard rubber.

NOTE—This method is not suited for use in purchase specifications, not only because correlation with service life is uncertain but because the results from duplicate specimens tested in different locations do not ordinarily give the same values. No exact relation between the results of the test and actual service performance is given or implied. The test is principally of value when used for comparisons between two or more rubber compounds.

2. Type of Test

2.1 This test consists of continuously exposing rubber test specimens, held under strain, to normal weather conditions or in an atmosphere containing controlled amounts of ozone in accordance with ASTM Method D 1149, Test for Rubber Deterioration—Surface Ozone Cracking in a Chamber (Flat Specimen).² Specimens are exposed for definite periods of time and their deterioration observed as evidenced by the appearance and growth of cracks on the surfaces.

PROCEDURE A. EXPOSURE OF STRAIGHT SPECIMENS

3. Apparatus

3.1 Mounting Block—A rectangular

wooden block shall be used for supporting the extended specimens. The block shall be 140 mm (5.5 in.) wide and approximately 380 mm (15 in.) long and shall have a thickness of not less than 22 mm ($\frac{7}{8}$ in.). The block shall have the grain running lengthwise and shall be suitably reinforced on the back to prevent warping. Both of the 15-in. edges of the face carrying the specimens shall be rounded with a 3-mm ($\frac{1}{8}$ -in.) radius. (Fig. 1 shows the details of construction.) The block shall be planed smooth and painted with two coats of clear lacquer.³

3.2 Fasteners—Aluminum tacks, or other suitable inert material fasteners shall be used for fastening the specimens to the edges of the wooden block.

3.3 Angle Strips—Right-angled aluminum molding strips 13 by 22 mm ($\frac{1}{2}$ by $\frac{7}{8}$ in.) and of approximately No. 22 gage, for shielding the specimens where tacked and bent over the edges of the block. Strips made from commercial aluminum sheet of Alloy No. 2-S are suitable.

4. Test Specimens

4.1 The test specimens shall be rectangular strips 25 mm (1 in.) in width by 150 mm (6

This method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.13 on Degradation Tests.

Current edition effective Sept. 18, 1961. Originally issued 1938. Replaces D 518-40.

¹ Annual Book of ASTM Standards, Part 17.

² duPont clear lacquer, or its equivalent, has been found suitable for this purpose.

in.) in length cut from standard laboratory test slabs having a thickness of 1.9 mm (0.075 in.) minimum and 2.5 mm (0.100 in.) maximum so that the grain will be in the lengthwise direction of the specimen.

4.2 Duplicate test specimens shall be tested whenever possible.

5. Procedure

5.1 Firmly fasten the test specimens at one end to one long side of the test block, spacing them 6 mm ($\frac{1}{4}$ in.) apart and using three aluminum tacks per specimen. Then draw the strips across the face of the block in such a manner as to cause an extension of 20 percent, measured between gage marks that shall be 100 mm (4 in.) apart and centered at the middle of each strip. Fasten the other end of each specimen in the same way to the opposite long side of the block. Mount the aluminum shields by means of screws on each of the long sides so that the 22 mm ($\frac{7}{8}$ -in.) leg covers the tacked ends of the specimens and the 13 mm ($\frac{1}{2}$ -in.) leg covers the specimens at the bend on to the face of the block.

5.2 Expose the extended specimens to the weather and sunlight at an angle of 45 deg facing south, preferably on a roof of a building; or they may be exposed as described in Method D 1149.

5.3 Record the date on which the tests were begun, and examine the specimens daily thereafter or as often as necessary, for the effects of ozone. Observation of cracks shall be made with a hand 7-power magnifying glass. Record the time of the appearance of the first surface cracks on these specimens. If desired, the exposure may subsequently be continued for the purpose of observing the rate of growth of the cracks or the development of any characteristic or unusual surface effects.

6. Report

6.1 The report shall include the following:

6.1.1 Statement of the method used.

6.1.2 Description of the specimens, identifying the rubber compounds and giving the duration, temperature, and date of vulcanization, if known.



D 518

6.1.3 Dates of starting the exposure and the first appearance of checks or cracks, and
6.1.4 Geographical location of specimens exposed to weather.

PROCEDURE B. EXPOSURE OF LOOPED TEST SPECIMENS

7. Apparatus

7.1 Clamping Strips, made of a medium-soft wood for clamping the specimens. Each strip shall be 13 mm (0.5 in.) in thickness, 25 mm (1 in.) in width, and 575 mm (23 in.) in length. Holes shall be drilled through the 13 mm thickness of each strip at intervals of 40 mm (1.6 in.), starting 14 mm (0.6 in.) from one end. The holes shall be made approximately 4 mm (0.160 in.) in diameter, and shall match in paired strips. The strips shall be fastened together using appropriate round-head chromium-plated or galvanized iron machine screws fitted with nuts.

7.2 Base Panel, made of medium-soft wood upon which the clamped specimens are mounted. It shall be 530 mm (24 in.) long, 205 mm (8 in.) wide, and at least 13 mm (0.5 in.) thick. Three cross-pieces, each 8 in. long, 1 in. wide, and 6 mm (0.25 in.) thick, shall be fastened to the base. The cross-pieces shall be mounted 19 mm (0.75 in.) from each end of the panel and at the center. Each cross-piece shall be held in place by three 50 mm (2-in.) round-head chromium-plated or galvanized iron machine screws. These screws shall be fitted with washers on the underside of the base panel and shall fit into a counter-sink. The screws shall pass through the panel and through the cross-pieces, 32 mm (1.25 in.) from each end of the cross-pieces and through the center. These screws shall protrude above the surface of the panel to a height of about 35 mm (1.38 in.) and shall be used to fasten the wooden specimen strips securely to the base as described in 9.2.

7.3 All wooden panels, strips, and cross-pieces, shall be painted with two coats of clear lacquer.³

8. Test Specimens

8.1 The test specimens shall be rectangular

strips 25 mm (1 in.) wide by 95 mm (3 7/8 in.) in length, cut from standard laboratory test slabs having a thickness of 1.9 mm (0.075 in.) minimum and 2.5 mm (0.100 in.) maximum so that the grain will be in the lengthwise direction of the specimen.

8.2 Duplicate test specimens shall be tested whenever possible.

9. Procedure

9.1 Loop the test specimens until their ends meet and then insert these ends between the paired wooden strips until they are flush with the underside of the strips. The minimum distance between the specimens shall be 6 mm (1/4 in.). Clamp the wooden strips together by means of machine screws so that the specimens are firmly held in place. As a result of this procedure, 25 mm (1 in.) of each end of the specimen will be covered by the wooden strips, which will act as a protective shield. The remaining 43 mm (1 3/4 in.) of the specimen shall form a loop having a varying elongation along its length, as shown in Fig. 2.

9.2 Mount the rack of clamped specimens on the cross-pieces attached to the base panel by passing the protruding machine screws of the panel between the paired wooden strips and fastening them with nuts and washers, as shown in Fig. 3.

9.3 Expose the tapered specimens to the weather and sunlight at an angle of 45 deg facing south, preferably on the roof of a building; or they may be exposed in a cabinet having an atmosphere in which the ozone concentration is controlled, as described in Method D 1149.

9.4 Record the date on which the tests were begun, and examine the specimens daily thereafter, or as often as necessary, for the effect of sunlight and weather. Record the time of the appearance of checking as well as the time of appearance of initial surface cracking for each specimen. Observation of cracks shall be made with a hand 7-power magnifying glass. If desired, the exposure may subsequently be continued for the purpose of observing the rate of growth of the cracks or the development of any characteristic or unusual surface effect.

10. Report

10.1 Report the results in accordance with

Section 6.

PROCEDURE C—EXPOSURE OF TAPERED SPECIMENS

11. Apparatus

11.1 *Mounting Frames*—Wooden frames for mounting the test specimens shall have the following dimensions: inside width, 100 mm (4 in.); over-all width, 175 mm (7 in.); inside length, 300 mm (12 in.); over-all length 380 mm (15 in.). For constructing the frames, 25-mm (1-in.) thick cypress shall be used, and the members shall be joined with dowels using waterproof glue for the bond. The frame shall be planed smooth and painted with two coats of clear lacquer.

11.2 *Fasteners*—Aluminum tacks, size No. 6, made from Alloy No. 31-S, or stainless steel staples for fastening the specimens to the wooden frame.

11.3 *Angle Strips*—Aluminum strips 38 mm (1 1/2 in.) wide and of approximately No. 22 gage, for shielding the specimens. Strips made from commercial Alloy No. 2-S are suitable.

12. Test Specimens

12.1 The test specimens shall be die-cut tapered strips having outside dimensions as shown in Fig. 4, cut from standard laboratory test slabs having a thickness of 1.9 mm (0.075 in.) minimum and 2.5 mm (0.100 in.) maximum so that the grain will be in the lengthwise direction of the specimen.

12.2 Duplicate test specimens shall be tested whenever possible.

13. Procedure

13.1 Place an identification mark on the broad end of each test specimen, using materials that will not have a deleterious effect on the specimens during aging and that shall not extend beyond the area covered by the aluminum angle strips.

13.2 Superimpose upon the specimen a template conforming to the dimensions shown in Fig. 4, and mark the specimen with a sharp-pointed pencil to conform with the holes in the template.

13.3 Draw parallel lines on the frame at the desired distance to give the required over-all elongation in accordance with the following table:

| Over-all Elongation, percent | Distance Between Parallel Lines, mm (in.) |
|------------------------------|---|
| 10 | 140 (5.5) |
| 15 | 146 (5.75) |
| 20 | 152 (6.0) |

Fasten the stretched specimen to the frame in such a manner that the fasteners or staples driven through the pencilled dots on the specimen will coincide with the awl points on the two lines. These awl points are spaced according to the holes in a template for the required elongation, as shown in Fig. 5. Because of the taper, the elongation for any one area will vary with the width of the strip. By placing bench marks at regular intervals along the center line of the tapered strip prior to stretching, it is possible to determine the percentage elongation for any given area by measuring the distance between the bench marks after the strip has been elongated, and applying the following formula:

$$\text{Elongation, percent} = [(L_s - L_o)/L_o] \times 100$$

where:

L_o = length after stretching, and

L_o = length before stretching.

13.4 After the specimens have been mounted, mount the aluminum shield on each of the long sides so that the tacks holding the specimens and the markings are covered.

13.5 Expose the extended specimens to the weather and sunlight at an angle of 45 deg facing south, or in a cabinet with controlled ozone concentration, as described in Method D 1149.

13.6 Record the date on which the tests were begun, and examine the specimens daily thereafter, for the effects of ozone. Observation of cracks shall be made with a hand 7-power magnifying glass. Record the time of the appearance of the first minute surface cracks on each specimen. If desired, the exposure may subsequently be continued for the purpose of observing the rate of growth of the cracks or the development of any characteristic or unusual surface effects.

14. Report

14.1 Report the results in accordance with Section 6.

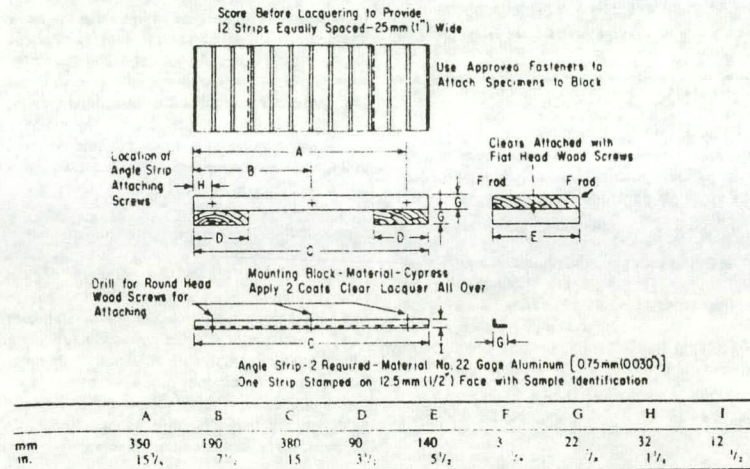


FIG. 1 Apparatus for Mounting Straight Rubber Test Specimens, Procedure A.

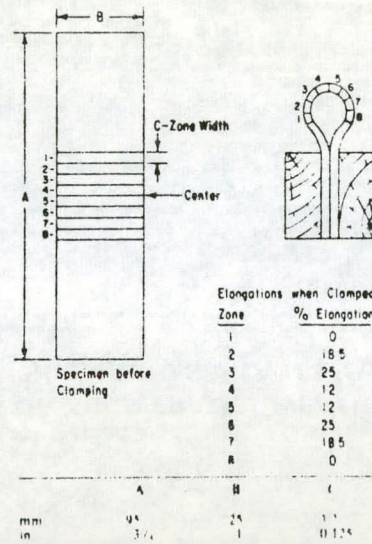


FIG. 2 Cross-Sectional View of Mounted Looped Specimen Showing Elongation at Different Parts, Procedure B.



FIG. 3 Assembled Panel, Showing Method of Mounting Looped Specimens.

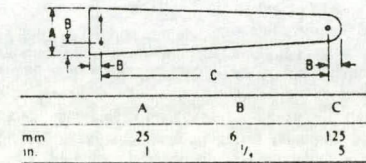


FIG. 4 Die-Cut Tapered Specimen.

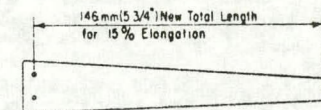


FIG. 5 Template for Spacing Holes in Frame for Mounting Elongated Specimens. (Template shown is for 15 percent elongation.)

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Standard Test Method for EFFECTS OF ACCELERATED WEATHERING ON ELASTOMERIC JOINT SEALANTS¹

This Standard is issued under the fixed designation C 793, the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval.

1. Scope

1.1 This method covers a laboratory procedure for determining the effects of accelerated weathering on cured-in-place elastomeric joint sealants (single- and multicomponent) for use in building construction.

2. Applicable Documents

2.1 *ASTM Standard*.

G 23, Recommended Practice for Operating Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Exposure of Non-metallic Materials²

3. Summary

3.1 Three sealant specimens are spread on aluminum plates and exposed to 250 h of ultraviolet radiation with intermittent water spray in a standard accelerated weathering machine. Following this treatment the specimens are exposed for 24 h in a freezer maintained at $-26 \pm 2^\circ\text{C}$ ($-15 \pm 3.6^\circ\text{F}$). At the end of the cold exposure the specimens are bent over a mandrel within 1 s at the specified temperature.

4. Significance

4.1 It is known that ultraviolet radiation contributes to the degradation of sealants in exterior building joints. The use of a laboratory accelerated weathering machine with ultraviolet radiation and intermittent water spray appears to be a feasible means to give indications of early degradation by the appearance of sealant cracking. The effect of the test is made more sensitive by the addition of the bending of the specimen at cold temperature.

5. Apparatus

5.1 *Exposure Apparatus*—An accelerated weathering machine, twin-enclosed carbon arc with 102-18 light/water spray cycle (102 min of light, followed by 18 min of light and deionized water). It shall conform to Type D of Recommended Practice G 23.

NOTE 1—There are several other weathering machines described in Recommended Practice G 23 available for use, and these may or may not give different results from the one described under Type D.

5.2 *Freezer or Cold Box* having a temperature controlled at $-26 \pm 2^\circ\text{C}$ ($-15 \pm 3.6^\circ\text{F}$).

5.3 *Rectangular Brass Frame*, with inside dimensions 130 by 40 by 1 mm (5 by 1 1/2 by 1/8 in.).

5.4 *Aluminum Plates*, three, each 152 by 80 by 0.3 mm (6 by 3 by 0.01 in.).

5.5 *Steel Mandrel*, 12.7 mm (1/2 in.) in diameter and about 102 mm (4 in.) long.

5.6 *Thin-Bladed Knife*.

5.7 *Straightedge*, metal or plastic, about 152 mm (6 in.) long.

5.8 *Spatula*, steel, about 152 mm (6 in.) long.

6. Standard Test Conditions

6.1 Unless otherwise specified by those authorizing the test, standard conditions of temperature and relative humidity shall be $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and 50 \pm 5%, respectively.

¹ This method is under the jurisdiction of ASTM Committee C-24 on Building Seals and Sealants, and is the direct responsibility of Subcommittee C24.32 on Test Methods for Chemically Cured Compounds.

Current edition approved Jan. 14, 1975. Published April 1975.

² Annual Book of ASTM Standards, Parts 32, 35, and 41.



7. Procedure

7.1 Test of Multicomponent Sealants:

7.1.1 Condition at least 200 g of base compound and appropriate amount of curing agent in a closed container for at least 24 h at standard conditions; then mix thoroughly for 5 min.

7.1.2 Fill the brass frame, after centering it on the aluminum plate, with a portion of the mixed compound and strike it off flat with a straightedge. Immediately lift the frame from the sealant after separating it by running a thin-bladed knife along the inside of the frame (Note 2). Prepare three such specimens and cure them for 72 h at standard conditions.

7.1.3 At the end of the curing period, leave one (control) specimen at standard conditions and place the other two in the drum of the weathering machine and expose them for 250 h. The temperature at the specimen during operation shall be $60 \pm 2.8^\circ\text{C}$ ($140 \pm 5^\circ\text{F}$) and the water temperature shall be $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$). Change carbons and clean glass globes daily during the exposure period.

7.1.4 At the end of 250 h of exposure, remove the specimens from the machine and note changes in appearance as compared with the control specimen.

7.1.5 Place all three specimens and the mandrel in the freezer, controlled at $-26 \pm 2^\circ\text{C}$ ($-15 \pm 3.6^\circ\text{F}$) for 24 h. At the end of this period, while in the freezer at this temperature, bend each specimen, with sealant side outward, across its width, 180 deg around the mandrel within 1 s. Examine each specimen for cracks developed over the bent area.

NOTE 2—In the case of pourable grade compound, do not lift the brass frame until the sealant is sufficiently set that it will retain its rectangular shape.

7.2 Test of Single-Component Sealants:

7.2.1 Condition at least 200 g of compound in a closed container for at least 24 h at standard conditions.

7.2.2 Follow the same procedure as specified in 7.1.2 through 7.1.4.

8. Report

8.1 The report shall include the following information for each sample tested:

8.1.1 Identification of the sealant tested.

8.1.2 Description of the type of sealant, such as single- or multicomponent, nonsag or pourable, color, etc.

8.1.3 Name and description of accelerated weathering machine.

8.1.4 Description of specimens after 250 h of accelerated weathering, as compared to the control specimen. Figure 1 includes examples of cracking obtainable after the ultraviolet test. Number 0 represents no cracks.

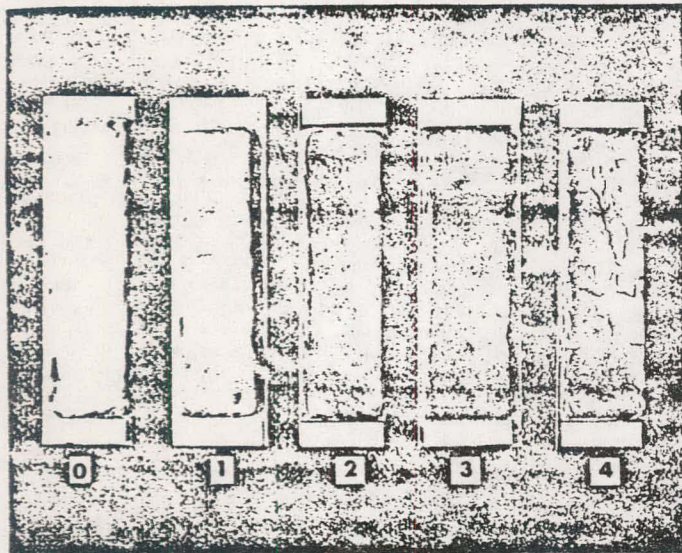
8.1.5 Description of specimens after bend test. Figure 2 includes examples of cracking obtainable after the bend test. Number 0 represents no cracks.

8.1.6 Variations, if any, from the specified test procedure.

9. Precision

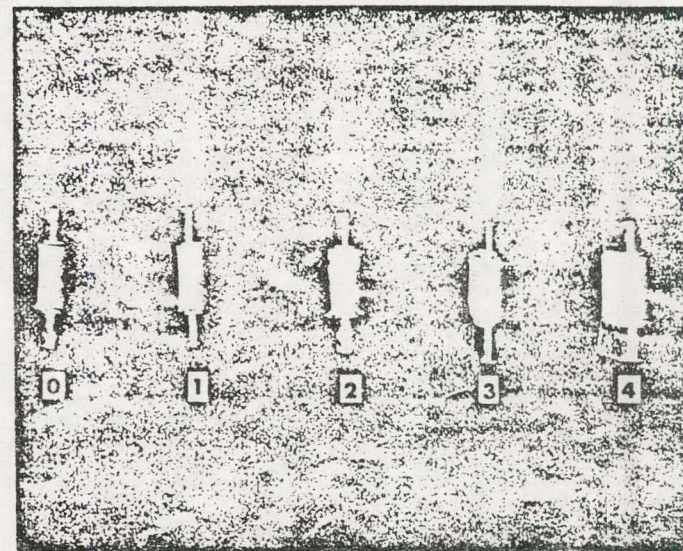
9.1 In a round-robin test in which each of four laboratories tested eight sealant samples to determine the effect of ultraviolet radiation on cracking as prescribed in the test, the laboratories agreed on 31 of the 32 determinations.

9.2 In a round-robin test in which each of three laboratories tested eight sealant samples to determine the effect of bend test at -26°C (-15°F) after ultraviolet exposure, as prescribed in the test, the laboratories agreed on 22 of the 24 determinations.



NOTE—Number 0 represents no cracks.

FIG. 1 Examples of Cracking Obtainable After the Ultraviolet Test.



NOTE—Number 0 represents no cracks.

FIG. 2 Examples of Cracking Obtainable After the Bend Test.

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Standard Test Method for ADHESION-IN-PEEL OF ELASTOMERIC JOINT SEALANTS¹

This Standard is issued under the fixed designation C 794; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval.

1. Scope

1.1 This method covers a laboratory procedure for determining the strength and characteristics of the peel properties of a cured-in-place elastomeric joint sealant, single- or multicomponent, for use in building construction.

2. Applicable Documents

- 2.1 *ASTM Standards*
D 1191, Testing Concrete Joint Sealers²
- (2.2) Recommended Practice for Operating Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Exposure of Non-metallic Materials³

3. Summary

3.1 The method consists of preparing test specimens by embedding a strip of cloth in a thin layer of the sealant being tested, on several substrate materials, curing these specimens for a certain length of time under specified conditions, then placing them in a tension-testing machine in such a way that the embedded cloth is peeled back from the substrate at 180 deg. and measuring the force exerted as well as the nature of the separation of the sealant from the substrate.

4. Significance

4.1 There are differences in opinion among those concerned with sealant technology whether or not this adhesion-in-peel test is intended to simulate the conditions encountered by a sealant in normal use. Nevertheless, since it represents a test to destruction, the value of the test denotes the ability of the cured sealant to maintain a bond to the substrate under severe conditions.

4.2 Many sealant manufacturers utilize the adhesion-in-peel test for determining the adhesive characteristics of sealant/primer combinations with unusual or proprietary substrates.

5. Apparatus and Materials

5.1 *Testing Machine* with tension grips capable of pulling at the rate of separation of 51 mm (2 in.) min. and having a chart indicator calibrated in 0.45-kg (1-lb) units.

5.2 *Standard Substrates*

5.2.1 *Aluminum Alloy*, Type 6063-T5 or 6061-T6, with a clear anodized finish of not less than 0.0075-mm (0.3-mil) thickness over a scale-free finish, 2 pieces, 152 by 76 by 6.3 mm (6 by 3 by $\frac{1}{4}$ in.).

5.2.2 *Mortar*, portland cement, 2 slabs, 152 by 76 by 9.5 mm (6 by 3 by $\frac{3}{8}$ in.) prepared as described in Method D 1191, with each slab having one of its 152 by 76-mm surfaces smoothed by wet grinding on a cast iron lap with No. 60 silicon carbide or aluminum oxide grain.

5.2.3 *Plate Glass*, polished, clear, 152 by 76 by 6.3 mm (6 by 3 by $\frac{1}{4}$ in.).

NOTE 1—Because of the fact that adhesive properties of a joint sealant are related to the nature of the substrate, it is strongly recommended that whenever possible the peel test be made with the substrates that are to be used in the building under consideration in addition to or in place of the specified substrates.

¹ This method is under the jurisdiction of ASTM Committee C-24 on Building Seals and Sealants, and is the direct responsibility of Subcommittee C24.32 on Test Methods for Chemically Cured Compounds.
Current edition approved Jan. 30, 1975. Published April 1975.

² Annual Book of ASTM Standards, Parts 14 and 15.

³ Annual Book of ASTM Standards, Parts 32, 35, and 41.



described in 5.2.1, 5.2.2, and 5.2.3. Such substrates include brick, marble, limestone, granite, stainless steel, plastic, quarry tile, and others. For practical reasons the specimen dimensions may be changed from the standard sizes provided the thickness of the sealant remains as specified.

5.3 *Spacer Strips*, four, of hard wood, metal, or glass as follows: two 152 by 76 by 6.3 mm (6 by 3 by $\frac{1}{4}$ in.) for preparing the test specimens on aluminum and glass, and two of the same length and width but 9.3 mm ($\frac{3}{8}$ in.) thick for preparing the test specimens on mortar.

5.4 *Glass Rod*, about 12.7 mm ($\frac{1}{2}$ in.) in diameter and about 305 mm (12 in.) long.

5.5 *Stainless Steel or Brass Rods*, two, 1.6 mm ($\frac{1}{16}$ in.) in diameter, about 305 mm (12 in.) long.

5.6 *Masking Tape*, paper, roll, 25.4 mm (1 in.) wide.

5.7 *Airplane Cloth*, Grade A, desized, 4.28 oz/yd, 80/84 count, 6 pieces at least 178 mm (7 in.) long and 76 mm (3 in.) wide, or suitable wire cloth, 30-mesh, 0.254-mm (10-mil) thickness.

5.8 *Putty Knife*, stiff, about 38 mm (1 $\frac{1}{2}$ in.) wide.

5.9 *Knife* with sharp razor-type blade.

6. Test Specimens

6.1 Two test specimens shall be prepared on aluminum, two on cement mortar, two on glass, and two on each of any other substrate materials specified, using the following procedures:

6.1.1 Condition not less than 280 g of sealant (and portion of catalyst, if a multicomponent) in a closed container for 24 h at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 5\%$ relative humidity.

6.1.2 Clean the test surfaces of all metal and glass substrates with methyl ethyl ketone or similar solvent followed by a thorough cleaning with a detergent solution (Note 2), a final rinse with distilled or deionized water, and air dry. Clean masonry surfaces with a dry stiff fiber bristle brush.

6.1.3 Apply primer to the clean dry test surfaces only when specified and supplied by the sealant manufacturer and agreed upon by the purchaser.

6.1.4 Place a strip of masking tape 25 mm (1 in.) wide across the test surface of the substrate so that the lower edge of the tape is parallel and at least 76.2 mm (3 in.) from the lower short edge of the substrate (Fig. 1.1).

6.1.5 Spread a portion of the conditioned compound, after being mixed thoroughly for 5 min (if multicomponent), over the 102 by 76-mm (4 by 3-in.) area, which includes the masking tape, to a depth slightly more than 1.6 mm ($\frac{1}{16}$ in.), as shown in Fig. 1B.

6.1.6 Smear one piece of cloth with the compound at one end over an area of 102 by 76 mm (4 by 3 in.), forcing it into both sides of the cloth with a putty knife until the sealant has thoroughly penetrated the cloth.

6.1.7 Lay the impregnated cloth over the layer of compound and place the spacer bars of proper thickness (see 5.5) on each side of the specimen.

6.1.8 Place a 1.6-mm ($\frac{1}{16}$ -in.) metal rod lengthwise on top of each spacer bar and squeeze the compound to 1.6 mm ($\frac{1}{16}$ in.) thick by rolling the glass rod over the metal rods (starting from the taped end), and simultaneously pressing on the cloth and sealant beneath it. Trim off the excess amount that is squeezed out (Fig. 1C).

6.1.9 Cure the specimens containing multicomponent compounds 14 days at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$). Cure those containing single-component compounds 21 days as follows (Note 3): 7 days at $23 \pm 2^\circ\text{C}$ ($73 \pm 3.6^\circ\text{F}$), $50 \pm 5\%$ relative humidity; 7 days at $37.8 \pm 2^\circ\text{C}$ ($100 \pm 3.6^\circ\text{F}$) and $95 \pm 5\%$ relative humidity, and finally 7 days at $23 \pm 2^\circ\text{C}$ ($73 \pm 3.6^\circ\text{F}$) and $50 \pm 5\%$ relative humidity.

6.1.10 After the specimen has cured for about 7 days, coat the cloth with a layer of the compound about 1.6 mm ($\frac{1}{16}$ in.) thick to help minimize cloth failure (Fig. 1D).

6.1.11 Immediately following the full curing period, make four cuts with a sharp blade lengthwise of the specimen, cutting completely through to the substrate surface, and remove excess material so as to leave two 25.4-mm (1-in.) wide strips of cloth-covered sealant separated by a space about 9.5 mm ($\frac{3}{8}$ in.) wide (Fig. 1E) (Note 4).

6.1.12 Immediately following step 6.1.11 (except as explained in Note 4), completely immerse the specimen in distilled or deionized water for 7 days.

NOTE 2—At the request of the sealant manufacturer the detergent cleaning step shall be omitted from the specified cleaning procedure.

NOTE 3—The manufacturer can suggest other curing conditions for the single component sealants.

when may be used provided (a) the curing time does not exceed 21 days, and (b) the temperature does not exceed 70°C (158°F).

NOTE 4—If peel adhesion is to be tested on glass substrate specimens, after ultraviolet exposure through glass, after completing step 6.1.11 and before proceeding with 6.1.12, place such specimens with the sealant surface facing away from the light source on the drum of an accelerated weathering machine as specified in Method G 23 (see description of Type D apparatus). Expose the specimens to the ultraviolet radiation for 200 h without water spray and continue as stated in 6.1.12.

7. Procedure

7.1 Immediately following the 7 days' immersion, prepare the specimen for testing by wiping it dry, releasing the sealant from the tape undercutting the sealant 12.7 mm (1/2 in.) and leaving a 63.5-mm (2 1/2-in.) length adhered to the substrate.

7.2 Place the specimen in the testing machine and peel the cloth back at an angle of 180 deg at a rate of separation of 50.8 mm (2 in.)/min (Fig. 2). Peel the sealant for about 1 min and record the average force in newtons (or pounds-force) indicated by the dial or recording chart of the machine. If the cloth peels clean from the sealant, disregard the values. In such instances, undercut the compound with a sharp blade to produce separation at the interface to the test surface and continue the test.

7.3 Test four strips for each of the substrate materials specified.

8. Report

8.1 The report shall include the following information for each sample tested:

- 8.1.1 Identification of sample
- 8.1.2 Identification of the type of sealant, such as single- or multicomponent, color, etc.
- 8.1.3 Average peel strength in newtons (or pounds-force) for the four strips tested on each substrate.
- 8.1.4 The percentage loss in bond and cohesion for each strip tested.
- 8.1.5 Any indication of cloth failure.
- 8.1.6 Variation, if any, from the specified test procedure.

9. Precision

9.1 Round-robin studies of the peel test conducted by members of ASTM Committee C-24 as well as by individual members of the sealant industry indicate that peel tests made on one sealant sample with a specified substrate by a single operator in a single laboratory may yield a range of values that vary by ± 10 to ± 20 % from the mean value.

9.2 The range of values that can be encountered when peel tests are performed by several laboratories on the same sealant sample with the same substrate is commonly ± 60 % from the mean value and is often ± 100 %. The reason for this interlaboratory variation has not yet been determined.

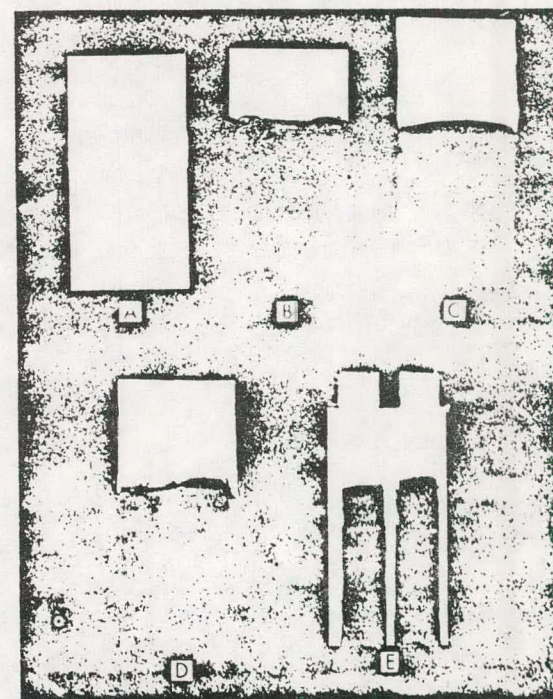


FIG. 1 Stages in the Preparation of the Adhesion-in-Peel Test Specimen.

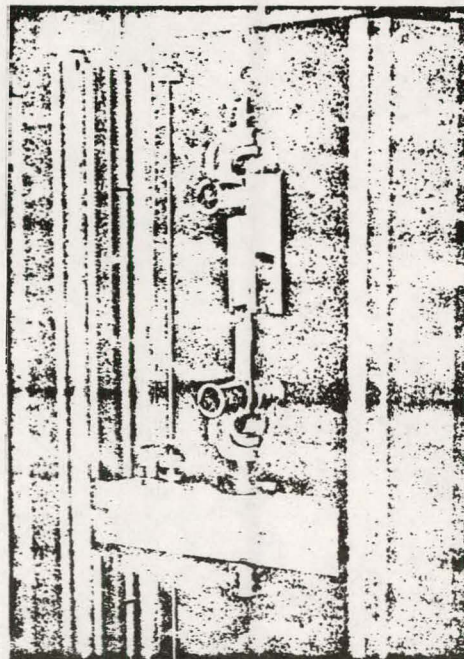


FIG. 2 Extension Machine Used in the Adhesion-in-Peel Test.

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Standard Test Method for RUBBER DETERIORATION—SURFACE OZONE CRACKING IN A CHAMBER (FLAT SPECIMEN)¹

This Standard is issued under the fixed designation D 1149, the number immediately following it indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers the estimation of resistance of vulcanized rubber to cracking when exposed to an atmosphere containing ozone. The rubber specimens are kept under a surface tensile strain, and the ozone concentration in the test chamber is maintained at a fixed value.

1.2 The method may not give results correlating exactly with outdoor exposure tests since the correlation of accelerated ozone tests with outdoor performance is in general not good and is highly dependent upon the specific conditions of both the accelerated and outdoor exposures. Conditions that influence accelerated tests are ozone concentration, relaxation of stress, temperature, and degree of bloom of additives. Conditions that influence outdoor tests in addition to these are the amount of sunshine and rainfall.

2. Summary of Method

2.1 Specimens under tensile strain are exposed in a chamber containing an ozone-air atmosphere at a controllable prescribed temperature. The concentration of the ozone can be varied and is measured with a spray-jet device or a counter-current absorption column. The specimens are examined at intervals and their condition recorded.

3. Ozone Test Apparatus

3.1 **Test Chamber**—Requirements for an acceptable ozone test chamber are sufficient air-ozone throughput rate, sufficient internal

circulation, and sufficient internal volume. A secondary requirement is that of controlling the temperature within acceptable limits. An acceptable ozone test chamber can be custom-made in a particular laboratory or one of the commercial manufactured chambers which are available may be used. The ozone test chambers shall conform to the following requirements.

3.1.1 The test chamber shall be constructed of a material with minimal reaction to ozone.

3.1.2 The volume of the chamber shall be at least 0.11 to 0.14 m³ (4 to 5 ft³).

3.1.3 A means for generating and controlling an air-ozone stream shall be provided. The generating source shall be located outside of the chamber. The source of air can be either drawn directly from the laboratory or from a compressed air supply. In either case adequate filtration of foreign matter from the stream must be provided. The air-ozone stream shall be introduced into the chamber in such a manner that stratification of ozone is prevented.

3.1.4 The air-ozone replacement rate or throughput rate must be of a magnitude such that no appreciable reduction in ozone concentration results from the introduction of test specimens. This minimum replacement rate will vary with the ozone concentration, tem-

perature, number of test specimens introduced, and their reaction with ozone.² For many chambers operating under normal conditions (approximately 50 parts per 100,000,000), an air-ozone replacement rate of a three-fourth change per minute is an acceptable and adequate value. For thorough and accurate work, especially under unusual conditions, the minimum or safe replacement rate should be determined.

3.1.5 A means of providing adequate internal circulation shall be provided. The air-ozone velocity in the chamber shall be at least 0.6 mm (2 ft)/s. Where it is doubtful that such velocities exist, the installation of an ordinary 1700 rpm electric motor and fan blade of approximately 150-mm (6-in.) diameter and 20 to 30-deg pitch will produce such air velocities. The motor itself shall not be in the chamber. An extension shaft shall be used with an appropriate seal.

3.1.6 A means of controlling the temperature of the chamber from ambient to 70°C (158°F) shall be provided. The temperature regulation should be capable of maintaining the test temperature within $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$).

3.1.7 It is often advantageous to have a glass window or glass front door as part of the chamber. This feature is optional.

3.1.8 **Ozone Generator**—The mercury vapor lamp is the most common source for generating ozone.³ With such a lamp the ozone concentration can be easily controlled by means of a variable transformer.⁴ This will transform the voltage fed to the primary of the transformer to that required by the mercury lamp.

4. Apparatus for Measuring Ozone Concentration

4.1 Apparatus for measuring the concentration of ozone in the test chamber shall be provided. A method for analysis that is satisfactory is absorption of the ozone in buffered potassium iodide solution and titration of the liberated iodine with a standard solution of sodium thiosulfate. One of two alternative absorption devices shall be provided. The first of these is the spray-jet device and the second is a counter-current absorption column.

4.2 **Spray-Jet Device**

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4.2.1 The spray-jet device is shown in Fig. 1. The glass tube, *A*, is approximately 9.5 mm (0.375 in.) in diameter and 100 mm (4 in.) long, terminating at *B* in a short length of capillary tubing with a bore of 1 to 2 mm (0.04 to 0.08 in.). Concentric within *A* is a smaller glass tube, *C*. (Figure 1(a) is an enlarged view of this part.) The end of *C* is first carefully heated in a blowpipe flame until the bore is reduced in size so as just to admit a wire or drill 0.75 mm (0.03 in.) in diameter. At this thickened end two flats are ground off on a sheet of fine alumina abrasive paper as at *D* in Fig. 1(b). When in position in tube *A*, end *D* fits snugly against the hole in capillary *B*. A rubber tubing connection at *E* holds the two tubes in position. *F* is a trap about 50 mm (2 in.) in diameter and 100 mm (4 in.) long, and *G* is an enlargement in the exit tube about 40 mm (1.5 in.) in diameter, containing glass wool to trap spray passing *F*. *F* is connected to the side tube of *H* is a 1-liter three-neck Woulff bottle or round-bottom chemically resistant flask in which *A* and *F* are secured by standard-taper ground joints. *A* occupying the center opening with *B* protruding just below the neck and *J* reaching to within 13 mm (0.5 in.) of the bottom of the bottle. The third opening serves to introduce and remove the reagent. *A* is connected through plasticized poly(vinyl chloride) tubing and glass tubing to a rotameter⁵ graduated from 0 to 1.0 m³ (0 to 35 ft³) of air/h. The entrance to the rotameter is connected with glass tubing to the sampling tube, and the exit of *F* is connected to a vacuum line. When properly regulated and a vacuum applied at *F*, most of the reagent enters *F*, furnishing a head of reagent at *B*, where the entering air resolves it into a fine mist which fills the entire bottle. The absorption flask shall be mounted in a light-tight box to protect it from light during the time a run is being made.

4.2.2 A modification of the spray-jet method is in current use on some commercial

² Upward concentration adjustments can compensate for a reduction in ozone concentration when specimens are introduced.

³ Hanovia Safe-T-Aire No. 2851 with quartz tube is satisfactory.

⁴ A Powerstat or Variac is satisfactory for this purpose. A Fischer & Porter Rotameter, obtainable from Fischer & Porter Co., Warminster, Pa., is satisfactory for this purpose.

¹ This method is under the jurisdiction of ASTM Committee D-11 on Rubber and Rubber-Like Materials and is the direct responsibility of Subcommittee D11.13 on Degradation Tests.

Current edition effective Aug. 31, 1964. Originally issued 1951. Replaces D 1149 - 63.

ozone chambers. This involves absorbing the ozone in a buffered potassium iodide solution which contains a measured amount of standard sodium thiosulfate solution. The time for the sodium thiosulfate to be consumed by reaction with iodine is measured. This has the advantage over the unmodified spray-jet method, in that no iodine is volatilized, and no empirical factor is therefore necessary to correct for this loss. To use this modified method, it is necessary to alter slightly the equipment shown in Fig. 1. A round-bottom flask with a bottom drain cock shall be used with four necks or outlets. Two of these are used as depicted in Fig. 1 to house the spray-jet and the upper reservoir return tube, and the other two contain a pair of platinum electrodes and a buret for adding sodium thiosulfate solution. The modified apparatus is shown in Fig. 2. A sufficient quantity of buffer solution containing 15 g of potassium iodide is added so that a pool of solution fully immerses the electrodes when an air stream is drawn through the apparatus. As iodine is liberated, the current increases and the null indicator or microammeter will indicate this increase. A reference point on the scale is chosen, and as soon as the indicator reaches this point a stop watch is started. Immediately afterward a known volume of 0.0020 *N* sodium thiosulfate solution is added from the buret and the time for the current to again reach its original or reference value is noted. For low ozone concentrations, 1 to 2 ml of sodium thiosulfate solution are sufficient; for higher concentrations correspondingly more shall be added. This is the preferred method when using the spray-jet to remove ozone from the air-ozone stream.

4.3 Counter-Current Absorption Column:

4.3.1 This device absorbs the ozone from the air-ozone stream by providing a large surface area for gas-liquid contact.⁴ A buffered solution of potassium iodide percolates down through the column, and this counter-current flow (solution down, air stream up) removes the ozone from the air-ozone stream. Figure 3 is a diagram for the counter-current column. Such a column can be fabricated by a qualified glass blower. The column is filled with clean 3-mm (0.125-in.) glass helices. These must be packed down tightly for efficient operation. Glass beads, 6-mm (0.25-in.), may

be used at the bottom of the column below the inlet to prevent fragments of the helices from entering the stopcock. None of the dimensions on the drawing are critical, but the lower part of the column should be not less than 200 mm (8 in.). It has been found unnecessary to shield this column from artificial lighting. It is not recommended that it be used in direct outdoor light. The advantages of this column over the spray-jet device are (1) the mechanical operation is simpler since no tedious spray adjustments are required, (2) there is no volatilization of iodine since it is carried promptly into the collection flask, and (3) no pressure drop occurs in the analysis apparatus train. Such a pressure drop will cause faulty ozone concentration measurements if a small undetected leak develops.

4.3.2 The air sample should be drawn directly from the ozone chamber. Special precautions to draw air from various levels of the chamber are not necessary, since adequate circulation will be maintained if the preceding chamber specifications are met. Glass lines only should be used to convey the air-ozone stream to the absorbing device. Plasticized plastics are to be avoided, except as short connecting pieces for joining glass tubing.

5. Titration Apparatus

5.1 *Microammeter Method*—The solution and washings from either absorption method may be titrated in a 250-ml wide-mouth flask, or beaker of equal size. An air or magnetic stirrer should be used. The titrating equipment consists of a microburet, microammeter of 0 to 20 range,⁵ a heavy-duty dry cell of 1½ V, one 1000-Ω and one 30,000-Ω resistor, and two platinum electrodes approximately 2.5 mm (0.1 in.) in diameter and 25 mm (1 in.) in length. The resistors are connected in series across the 1½-V battery, and the potential across the 1000-Ω resistor is applied to the electrodes. The microammeter is connected in series with proper consideration for polarity, with the electrodes in this secondary circuit. The platinum electrodes are

⁴ Information on the counter-current absorption column is available from the B. F. Goodrich Research Center, Brecksville, Ohio, Attn: A. G. Veith.

⁵ A 0 to 20 range meter provides somewhat greater sensitivity than a 0 to 50 meter.

imbedded in glass tubing in the usual manner.

5.2 *Null Indicator Method*—A special null meter can be employed that utilizes a one-transistor amplification stage and represents a ten-fold increase in end-point sensitivity.⁶ This null meter may be used with any ozone absorption device. The initial reading is taken for the buffered potassium iodide solution with a potential of 100 mV applied to the electrodes. This potential is automatically applied by the circuitry in the null meter.⁶ After absorbing the ozone in the buffered solution, the free iodine is titrated with standard sodium thiosulfate solution. The end-point is the original or initial meter reading.

6. Reagents

6.1 Reagent grade chemicals and distilled water shall be used in all tests.

6.2 *Buffer Solution*—Prepare a 0.025 *M* solution of anhydrous disodium hydrogen phosphate (Na_2HPO_4) and a 0.025 *M* solution of anhydrous potassium dihydrogen phosphate (KH_2PO_4). To prepare the buffer solution having a pH of 6.7 to 7.1, add 1.5 volumes of 0.025 *M* Na_2HPO_4 solution to 1 volume of 0.025 *M* KH_2PO_4 solution. Shake thoroughly.

6.3 Potassium Iodide (KI).

6.4 *Sodium Thiosulfate Solution (0.020 N)*—Prepare a 0.020 *N* sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) solution. This may be standardized by using a standard 0.0200 *N* potassium bromate (KBrO_3) solution to oxidize an excess quantity of potassium iodide (KI) in acid solution. Titrate the liberated iodine immediately with the $\text{Na}_2\text{S}_2\text{O}_3$ solution. The titration equipment for the Microammeter Method (5.1) may be used to determine the end point in this titration. Store the prepared 0.020 *N* $\text{Na}_2\text{S}_2\text{O}_3$ solution in a cool dark place.

6.5 *Sodium Thiosulfate Solution (0.0020 N)*—Prepare 0.0020 *N* $\text{Na}_2\text{S}_2\text{O}_3$ solution for use in the ozone analysis by diluting the 0.020 *N* solution 10 to 1, using a 10-ml pipet and 100-ml volumetric flask. Redeterminations of the normality of the 0.020 *N* $\text{Na}_2\text{S}_2\text{O}_3$ solution should be carried out weekly.

7. Measurement of Ozone Concentration

7.1 With Spray-Jet Absorber:

7.1.1 Dissolve 15 g of KI in 75 ml of buffer solution. Add this to the absorber flask, adjust

the jet to produce a fine mist, and turn on the vacuum. Adjust the flow to 0.25 to 0.30 m^3 (9 to 11 ft^3)/h. After completion of the absorption run, titrate the solution and washings with 0.0020 *N* $\text{Na}_2\text{S}_2\text{O}_3$ solution. Add the $\text{Na}_2\text{S}_2\text{O}_3$ solution to the buffered solution of liberated iodine until zero current or the initial current value is reached. Add the $\text{Na}_2\text{S}_2\text{O}_3$ solution dropwise when nearing the end point, and wait between each drop to ensure complete reaction.

7.1.2 Calculate the ozone concentration of the chamber in parts per 100,000,000 (pphm) of air by volume as follows when the unmodified spray-jet absorption method is used:

Concentration of ozone, pphm

$$= (3470 \times B \times N \times T) / (F \times P \times t / 60)$$

where:

B = milliliters of $\text{Na}_2\text{S}_2\text{O}_3$ solution required,

N = normality of $\text{Na}_2\text{S}_2\text{O}_3$ solution,

T = absolute temperature, K (deg C + 273),

P = barometric pressure, mm Hg,

F = flow rate, m^3/h , and

t = time of run, min.

7.1.3 For the modified spray-jet method, calculate the ozone concentration as follows:

Concentration of ozone, pphm

$$= (3120 \times B \times N \times T) / (F \times P \times t / 60)$$

7.2 With Counter-Current Absorption Column:

7.2.1 Prepare 150 ml of buffer solution containing 15 g of KI. Use part of this to flood and wet the column. Drain the solution out of the column and return to the reservoir *R*. Keep stopcock 4 closed during this operation (Notes 2 and 3).

7.2.2 Lubricate ground-glass joints *I* and *II* with water and fit both caps in place. Use clamp on joint *I*. Close stopcock *I* and open stopcock 2.

7.2.3 Adjust stopcock 5 so that approximately 20 drops per minute (dpm) of solution are flowing into the column from *R*. Open stopcock 3, apply the vacuum, open stopcock 4, and start the clock. Adjust the flow rate to 0.3 m^3 (11 ft^3)/h. A slight readjustment of the solution flow rate may be necessary due to the vacuum. At least 20 dpm should be

⁶ Such a null meter is available from the Masi Development Co., 2212 East 12th St., Davenport, Iowa.

used. Continue the run for at least 30 min.

7.2.4 After the elapsed time, close stopcock 4, stop the clock, shut off the vacuum, and close stopcock 3. Close stopcock 2, open stopcock 5, and allow the column to fill up above the helices. If sufficient solution is not available in *R*, distilled water may be used. Open stopcock 1 and drain the solution in *CF* into a clean 250-ml beaker, open stopcock 2, and allow the column to drain into the same beaker. Open cap *H* and flush with approximately 5 ml of distilled water from a wash bottle. Titrate the solution and washings. Follow the same precautions during titration as in 7.1.

NOTE 1—Do not use any type of stopcock grease on the column. The efficient operation of the column depends upon good contact between the air stream and fully wetted glass helices. If grease is used, the helices will gradually become coated, and efficient air-liquid contact will be lost. Since the column does not cause a sharp pressure drop in the line or analysis train, leaks can be easily prevented by water lubrication of the glass joints of caps *I* and *H* when the clamps are used.

NOTE 2—Do not allow any solution or water to enter the inlet tube. If it does enter, dry the tube interior with a stream of compressed air before starting any test. Keeping stopcock 4 tightly closed will prevent any water from entering the tube.

7.2.5 Calculate the ozone concentration in parts per 100,000,000 of air by volume as follows:

$$\text{Concentration of ozone, ppm} = \frac{3120 \times B \times V \times T}{T_1 P F \times P \times 1000}$$

where *B*, *N*, *T*, *P*, *F*, and *V* are defined as in 7.1. (The factor 3120 is used since there is no correction for iodine loss.)

8. Test Specimens

8.1 Three types of specimens shall be regarded as standard. These are the specimens of Procedures A, B, and C of ASTM Method D 518, Test for Rubber Deterioration—Surface Cracking.¹ They are, respectively, a simple elongation test piece of 20 percent extension, a bent form test specimen whose average surface strain is approximately 18 percent, and a tapered specimen that can be given over-all elongations of 10, 15, and 20 percent. Duplicate specimens shall be tested whenever possible.

8.2 Nonstandard specimens may be tested, but the results cannot be expected to agree with tests carried out on standard specimens.

8.3 Molded, extruded, or sponge triangular specimens may also be tested. These specimens are fully described in ASTM Method D 1171, Test for Rubber Deterioration—Surface Ozone Cracking Outdoors or Chamber (Triangular Specimen).¹

9. Procedure

9.1 Mount the specimens in accordance with Procedures A, B, or C of Method D 518 or mount triangular specimens as described in Method D 1171. Expose all mounted specimens for 24 h in an ozone-free atmosphere prior to the test.

NOTE 3—It is strongly suggested that all rubber sheets scheduled for ozone testing be cured between aluminum foil 0.05 mm (0.002 in.) in thickness or A 200 Mylar. This will mildly adhere to most commercial rubbers. At the time specimens are cut for ozone testing the foil can be easily stripped off. This furnishes a "fresh" surface, and the 24 h of exposure can be reckoned from the formation of this fresh "bloom free" surface.

9.2 The standard ozone concentration shall be 50 ppm by volume. Other concentrations may, of course, be selected according to the particular aims of any testing program. These shall be referred to as optional concentrations. The ozone concentration shall be measured once a day for routine work and more often for special test conditions. This analysis, moreover, shall be conducted with the chamber loaded with test specimens. If the number of specimens is kept at a minimum, "empty" and "loaded" ozone concentrations will closely agree. The magnitude of the reduction of ozone concentration for "empty" versus "loaded" must be determined for each set of operating variables employed. This will vary for each laboratory.

9.3 The standard temperature shall be either 40°C (104°F) or 50°C (122°F) as desired. Higher temperatures cause a mild acceleration in the rate of ozone attack. These higher temperatures shall be optional.

9.4 Make observations for detecting the appearance of cracking with sufficient frequency to be able to detect the first appearance of ozone cracking. This frequency will depend on the resistance to ozone attack of the rubbers being tested. Recommended observation magnification is 7X, except in the case of the triangular specimen of Method

¹ Annual Book of ASTM Standards, Part 13.

D 1171, where the magnification shall be 2X. When comparisons are being made with a standard reference material, exposures may be made for a fixed time and comparisons made of the degree of cracking.

NOTE 4—This is a static test under one set of conditions. Other conditions encountered in actual use and dynamic tests in conjunction with static tests are desirable, but no agreement has yet been reached on a standard method or methods.

10. Report

10.1 The report shall include the following:

- 10.1.1 Identification of the materials tested,
- 10.1.2 Ozone concentration, both nominal and that actually measured on a daily basis,
- 10.1.3 Temperature of the test,
- 10.1.4 Method used for mounting the specimens, and
- 10.1.5 Time to the first observed cracking or a description of the character of the ozone cracks at various periods of exposure.

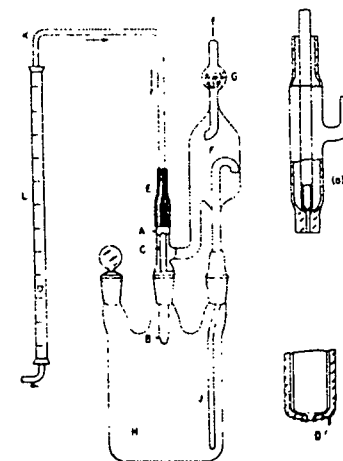


FIG. 1 Ozone Absorbing Device (Spray Jet).

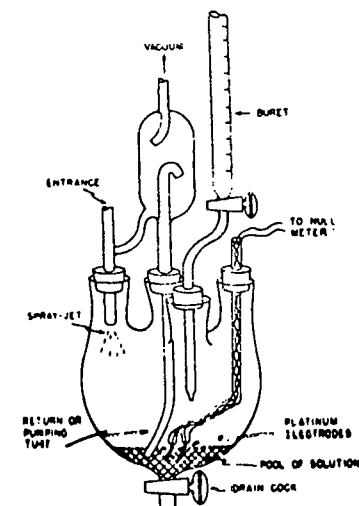


FIG. 2 Modified Spray-Jet Apparatus.

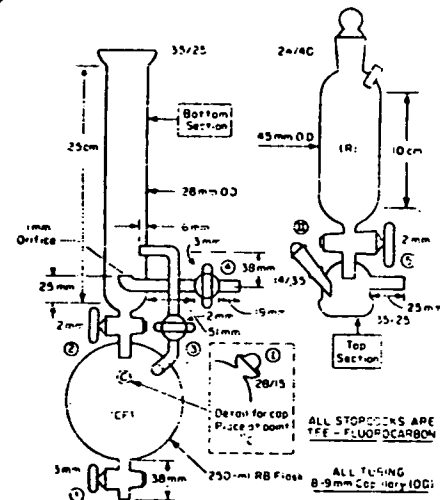


FIG. 3 Counter-Current Ozone Absorption Column.

By publication of this standard no position is taken with respect to the validity of any patent rights in connection therewith, and the American Society for Testing and Materials does not undertake to insure anyone utilizing the standard against liability for infringement of any Letters Patent nor assume any such liability.



Designation: D 1229 - 62
(Reapproved 1975)

American National Standard Z2 21-1971 (R 1975)
Reaffirmed Oct 31, 1975
By American National Standards Institute

Standard Test Method for RUBBER PROPERTY—COMPRESSION SET AT LOW TEMPERATURES¹

This Standard is issued under the fixed designation D 1229; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers evaluation of the ability of vulcanized elastomers that have been compressed at room temperature and then subjected to low temperature (air or carbon dioxide atmosphere), to recover from deformation when taken from the clamping device while still at the low temperature. This characteristic of vulcanizates is important in such applications as hydraulic seals on aircraft, submarine hatch gaskets, and hydraulic brake cups.

2. Compression Set

2.1 For the purpose of this test, compression set of vulcanizates shall be the loss in thickness of the specimen expressed as a percentage of the original deflection. This set, unlike conventional compression set at elevated temperatures, is temporary since the specimen will regain its original dimensions when brought back to room temperature or slightly above.

3. Apparatus

3.1 *Compression Set Jigs* with suitable steel spacer bars as for Method B in Section 10 of ASTM Methods D 395 for Rubber Property—Compression Set.²

3.2 *Dial Gage*, as described in 4.4 of Methods D 395.

3.3 *Cold Box*, cooled by solid carbon dioxide (dry ice), liquid carbon dioxide, or mechanically refrigerated, preferably by the top-opening type, and capable of temperature control

within $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$) of the specified testing temperature as prescribed in ASTM Recommended Practice D 832 for Rubber Conditioning for Low-Temperature Testing.³ The test chamber shall be equipped with a vise, "C" clamp, or other suitable device for holding the compression set jig.

4. Standard Test Specimen

4.1 The standard test specimen shall be a cylindrical disk 28.7 mm (1.129 in.) in diameter and 12.7 mm ($\frac{1}{2}$ in.) in thickness, as specified in Section 5 of Methods D 395.

5. Procedure

5.1 *Original Thickness Measurement*—Measure the original thickness, t_0 , at the center of the specimen to the nearest 0.025 mm (0.001 in.).

5.2 *Application of Load*—Place a test specimen between clean, unlubricated plates of the compression device with the spacers on each side of it. Use only one specimen with each pair of steel plates, and place in the center between the plates. Tighten the bolts so as to draw the plates together uniformly until they are in contact with the spacers. The compression employed shall be 25 percent for all hard-

nesses.

5.3 Within 30 min after the jigs are loaded, place them in the low-temperature cabinet, and maintain at the service temperature for which the vulcanizates are being evaluated. In the event the service temperature is unknown, the following temperatures are suggested: -40°C (-40°F) and -55°C (-67°F). The conditioning period shall be either 22 or 94 h. At least 1 h before the conditioning period is over, place the dial gage in the test chamber and clamp one of the set jigs in the vise or "C" clamp provided in the low-temperature chamber. Use suitable gloves for all operations in the test chamber. At the end of the conditioning period, remove the nuts from the jig, after which release the vise or "C" clamp, and start the stop watch simultaneously. Measure the thickness of the specimens 10 s and again 30 min after release from the vise or "C" clamp and record as t_{10} and t_{30} , respectively. Since the test is conducted at a specific temperature, within $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$), the schedule of opening the jigs shall be such that the test chamber will stay within the permissible variations in temperature.

5.4 *Check Test*—Run tests in duplicate.



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The results should agree within 5.0 percent.

6. Calculation

6.1 Calculate the compression set, expressed as a percentage of the original deflection, as follows:

$$C = [(t_0 - t_{10})/(t_0 - t_i)] \times 100$$

or

$$C = [(t_0 - t_{30})/(t_0 - t_i)] \times 100$$

where:

C = compression set expressed as a percentage of the original deflection, and

t_i = thickness of the spacer bar used.

7. Report

7.1 The report shall include the following:

7.1.1 The original thickness of the test specimen.

7.1.2 The percentage compression of the specimen actually employed.

7.1.3 The thickness of the test specimen 10 s, t_{10} , and 30 min, t_{30} , after removal from the clamp, and

7.1.4 The compression set expressed as a percentage of the original deflection.

By publication of this standard no position is taken with respect to the validity of any patent rights in connection therewith, and the American Society for Testing and Materials does not undertake to insure anyone utilizing the standard against liability for infringement of any Letters Patent nor assume any such liability.

This method is under the jurisdiction of ASTM Committee D-11 on Rubber and Rubber-Like Materials and is the direct responsibility of Subcommittee D11.14 on Low-Temperature and Resistance Tests.
Current edition effective Sept. 28, 1962. Originally issued 1952. Replaces D 1229-55.
² Annual Book of ASTM Standards, Part 37.

MIL -F-13927A(Ord)
22 August 1957
SUPERSEDING
MIL -F-13927(Ord)
23 December 1954

MILITARY SPECIFICATION
FUNGUS RESISTANCE TEST;
AUTOMOTIVE COMPONENTS

1. SCOPE

1.1 Scope. - This specification covers methods of testing automotive components for resistance to fungus attack to determine conformance to requirements specified in the item specification.

1.2 Classification. - For the purposes of this specification, specimens, to be tested shall be classified in one of the three following classes (see 6.2) and tested by one of the two following methods (as specified):

- Class 1 - Permanently sealed component assemblies
Class 2 - Unsealed assemblies and sealed assemblies normally disassembled for servicing
Class 3 - Separate components not used in assemblies, or separate components used as replacement parts in assemblies
Method A - Tropical room
Method B - Incubation cabinet.

2. APPLICABLE DOCUMENTS

There are no applicable documents.

3. MATERIALS, EQUIPMENT, AND TEST SPECIMENS

3.1 Materials.

3.1.1 Test organisms. - Except as otherwise specified, following species of fungi, propagated in conformance to pertinent preparatory procedures specified in Section 4 shall be used for fungus-resistance tests:

| <u>Test Organisms</u> | <u>ATCC No.</u> | <u>QMC No.</u> |
|--|-----------------|----------------|
| <u>Aspergillus flavus</u> | 9643 | 380 |
| <u>Aspergillus niger</u> | 6275 | 458 |
| <u>Penicillium citrinum</u> | 9849 | 1226 |
| <u>Trichoderma sp.</u> | 9645 | 385 |
| <u>Spicarea violacea</u> (<u>Penicillium lilacinum</u>) | | 1034 |

(ATCC cultures may be secured on application to American Type Culture Collection, 2029 M. Street, N. W., Washington, D. C.)

(QMC cultures may be secured on application to Quartermaster Corps General Laboratories, Natick, Massachusetts.)

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3.1.2 Test reagents. - Sterile mineral salts solution for use when specified, for diluting prepared spore suspensions, shall be of the following composition:

| | |
|---|-----------|
| KH ₂ PO ₄ ----- | 0.7 g |
| K ₂ HPO ₄ ----- | 0.7 g |
| MgSO ₄ ·7H ₂ O----- | 0.7 g |
| NH ₄ NO ₃ ----- | 1.0 g |
| NaCl----- | 0.005 g |
| FeSO ₄ ·7H ₂ O----- | 0.002 g |
| ZnSO ₄ ·7H ₂ O----- | 0.002 g |
| MnSO ₄ ·7H ₂ O----- | 0.001 g |
| Distilled water----- | 1.0 liter |

3.2 Test equipment. - Unless otherwise specified, method A, tropical room incubation (see 1.2 and 3.2.1), shall be used. Method B shall be used only when specifically approved in the detail (product or material) specification.

3.2.1 Tropical room (Method A). - Tropical room shall be provided with means for controlling and for cycling humidity and temperature conditions between limits specified in 4.2.5.2.

3.2.2 Incubation cabinet (Method B). - Incubation cabinet shall be provided with means for maintaining relative humidity between 96 and 100 percent and temperature between 80° and 84° F.

3.3 Test specimens. - Unless otherwise specified, not less than 1 specimen each of classes 1 and 2 items, and not less than 4 specimens of class 3 items, shall be subjected to this test. However, if any of the performance tests required by the detail (product or material) specification to ascertain the effect of this test are destructive in nature, a sample of sufficient size shall be furnished to complete all specified tests.

3.3.1 Preparation. - Prior to specified tests, test specimens shall not be leached, or otherwise conditioned, and there shall be no prior fungi inoculation. Specimens containing rubber, synthetic rubber, or similar materials shall not be subjected to heat-aging tests prior to fungi-resistance tests. Prior to any fungi-resistance tests, specimens shall be subjected to such other tests as are specified in the detail (item or material) specification.

4. PROCEDURES

4.1 Inspection. - Each specimen shall be visually inspected, in accordance with detail specification before testing. After completion of the test specified herein, each specimen shall again be inspected for conformance to detail specification requirements when applicable.

4.2.1 Culture stock maintenance. - Cultures of specified fungi shall be maintained separately on media, such as potato dextrose agar. Stock cultures shall be kept not more than 4 months in a refrigerator at a temperature between 37.4° and 50° F. Subcultures incubated at a temperature between 82.4° and 86° F. for 7 to 20 days shall be used to prepare spore suspensions.

4.2.2 Spore suspensions. - A composite spore suspension shall be prepared from 5 species of fungi specified in 3.1.1. Ten milliliters of sterile distilled water shall be poured into tubes of each of the cultures specified. A sterile needle or other means shall be used to harvest the spore growths on the surface of each culture. The spores and distilled water from the tubes of each species shall be poured into a 125 ml. flask containing 50 ml. of sterile distilled water and 15 - 20 sterile solid glass beads approximately 5 mm in diameter. The flask shall be shaken vigorously to break up the spore clumps and the suspension filtered through a thin layer of sterile glass wool to

remove mycelial filaments. The residue minus the filaments shall be added to 30 ml. of sterile mineral salts solution specified in 3.1.2 and the resultant suspension refrigerated at 40° F. until used. The suspension shall not be kept more than 24 hours.

4.2.3 Specimen inoculation. - The entire surface of each specimen shall be inoculated by spraying with the mixed spore suspension (see 4.2.2) by means of a spray apparatus (see 6.3). A culture dish with potato agar or similar media, shall be inoculated with the same spore suspension that is sprayed on the specimen. The dish shall serve as a control and shall be subjected to the same test conditions as the inoculated test specimens. Failure of the test fungi to produce copious growth on the control dish after 7 days will require the preparation of another spore suspension and a re-inoculation of the test specimens.

4.2.4 Specimen placement.

4.2.4.1 Classes 1 and 3. - Classes 1 and 3 units, assemblies, components, parts, etc., (see 6.2.1), immediately after inoculation (see 4.2.3), shall be placed for method A incubation (see 3.2.1) on open lattice shelves and so arranged as to permit free air circulation around specimens.

4.2.4.2 Class 2. - Class 2 specimens, unless otherwise specified, shall be placed in disassembled condition for incubation as specified in 4.2.4.1. When method B is used specimens shall be placed in the incubation cabinet (see 3.2.2).

4.2.5 Incubation.

4.2.5.1 Period. - Except as otherwise specified, after incubation (see 4.2.3 and 4.2.4), specimens shall be incubated for 90 days.

4.2.5.2 Cycling. - The tropical room shall be operated under the following conditions: Twenty hours with relative humidity between 93 and 97 percent and an ambient air temperature of 80° to 85° F. followed by 4 hours of a nominal 100 percent relative humidity, with condensation and an ambient air temperature of 75° to 80° F.

4.2.6 Performance rating. - Failure of any specimen to pass any of the periodic performance tests specified below shall be considered as failure of the fungus test and shall end the test.

4.2.6.1 Class 1. - Class 1 items shall be subjected to performance tests in accordance with the detail (product or material) specification after 30, 60, and 90 days exposure.

4.2.6.2 Class 2. - Class 2 items, after initial incubation period of 15 days, shall be assembled and subjected to performance tests specified in the detail (product or material) specification. After performance tests, the incubation shall be continued with the specimen disassembled. At the end of 30, 60, and 90 days incubation respectively, the specimen shall be assembled, performance rated and then disassembled for placement back into incubation provided that it does not fail the performance test. Ninety days shall be considered as the termination of the test.

4.2.6.3 Class 3. - Specimens shall be removed from the chamber after 30, 60 and 90 days of incubation and subjected to the tests specified in the item specification. After performance tests the specimen shall not be replaced in the incubation chamber.

5. PREPARATION FOR DELIVERY

There are no applicable requirements.

6. NOTES

6.1 Intended use. - This specification is intended to be used as a test procedure for determining the effects of exposure to fungi and moisture on the physical, electrical, and material characteristics of automotive items, components, and materials.

of components, used in or with military vehicles. The detail (product or material) specification when making reference to this specification would specify the applicable class and the permissible tolerances from specified requirements, if any, after exposure specified herein.

6.2 Classes.

6.2.1 Class 1. - Class 1 items include such sealed items as instruments, circuit breakers, solenoids, switches, etc., which are not normally serviced but are discarded and replaced when defects occur.

6.2.2 Class 2. - Class 2 items include starting motors, generators, unsealed magnetos, open or ventilated distributors, and sealed distributors, and other assemblies that are opened or disassembled for servicing.

6.2.3 Class 3. - Class 3 items include materials or separate components which may be procured as replacement parts of electrical or other assemblies or are not used in assemblies. Typical items in this class are gaskets, cable, hose, insulating material, etc., which require destructive tests such as tensile strength tests, ultimate elongation tests, etc. to determine the effect of fungi.

6.3 Spray apparatus. - Satisfactory spore suspension distribution (see 4.2.3) can be secured with DeVilbiss No. 2 hand-operated spray apparatus, or equal.

Notice. - When Government drawings, specification, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any invention that may in any way be related thereto.

Custodian:

Army - Ordnance Corps

Other Interest:

Army - ESIgTC

Preparing Activity:

Army - Ordnance Corps

MIL-F-13927A(Ord)
AMENDMENT 2
1 June 1967
~~SUPERSEDING~~
AMENDMENT 1
1 March 1962

MILITARY SPECIFICATION

FUNGUS RESISTANCE TEST; AUTOMOTIVE COMPONENTS

This amendment forms a part of Military Specification MIL-F-13927A(Ord), dated 22 August 1957.

Page 1, paragraph 3.1.1. Delete in its entirety and substitute the following: "3.1.1 Test organisms. Unless otherwise specified, the following species of fungi, propagated in conformance to pertinent preparatory procedures specified in Section 4, shall be used for fungus-resistance tests:

| Name of Organism | American type culture collection number | Quartermaster number |
|-----------------------|---|-------------------------|
| Aspergillus flavus | 9643 | 380 |
| Aspergillus niger | 6275 | 458 |
| Penicillium citrinum | 9849 | 1226 |
| Trichoderma sp. | 9645 | 365 |
| Penicillium lilacinum | 13351 | 1034 |

(Cultures of the organisms may be obtained from the following sources: American Type Culture Collection, 2112 M Street, N. W., Washington, D. C. 20037, and Mycology Laboratory, PRD, U.S. Army Natick Laboratories, Natick, Massachusetts 01760)"

Page 3, paragraph 4. Delete "4. PROCEDURES" and substitute:

"4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements."

Page 3, paragraph 4.2.5.1, line 1. Delete "after incubation" and substitute "after inoculation or placement."

MIL-F-13927A(Ord)
AMENDMENT 2

Page 4. Delete paragraphs 4.2.6 through 4.2.6.3 and substitute the following: "4.2.6 Performance tests. When specified in the component specification, performance tests shall be accomplished before, during and after the fungus test as required. Failure of a performance test shall be considered as a failure of the fungus test."

Page 5. Delete "Notice" paragraph in its entirety.

Custodian:

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A P P E N D I X I I

INCANDESCENT LIGHT TRANSMITTANCE MEASUREMENT APPARATUS

Tempered glass cannot be cut into satisfactory size test specimens due to its inherent tendency to shatter into very small pieces. Therefore, relative light transmittance values cannot be obtained using a standard type spectrophotometer.

An apparatus and test method was developed for obtaining relative light transmittance values on solar collector glass glazes without breaking the glass. This apparatus (Fig. 4) consists of a small incandescent light source, a photocell receiver, and a microammeter. The light source and the photocell are positioned and fastened into the ends of a two-pronged metal holder resembling a giant tuning fork, at a fixed distance from each other. The photocell receiver is connected to a microammeter which measures the electrical output of the light being received by the photocell.

The solar collector glass glaze to be evaluated is cleaned in two small 4 in. x 4 in. areas with chloroform, dilute aqueous hydrochloric acid, detergent water and finally, deionized water. These cleaned areas of the glass glaze are then placed between the light source and the photocell. With the light source turned on, the microammeter is adjusted to show a reading of 100 microamps. With the light off, it reads zero microamps.

The apparatus is then placed over the uncleaned areas of the glass glaze and microampere readings taken. Thus, the drop in microamps becomes a relative indication of the loss in light transmittance.

A more absolute value of light transmittance (loss) was obtained by comparing values from the incandescent photocell technique with those obtained using a Coleman Spectrophotometer over the wavelength range of 400-950 nm.

Several pieces of 3 in. x 3 in. soft glass were coated to varying degrees with salt and stearic acid. Light transmittance values were obtained using both the incandescent photocell and the spectrophotometer. The correlation between incandescent photocell values and spectrophotometer values is not perfectly linear. A 40% relative light transmittance loss by the incandescent photocell method is only equivalent to 27% loss by the spectrophotometric method. Figure 9 can be employed to provide correlations between the two methods of light transmittance measurement.