

AD-759 972

HYDROLYTIC STABILITY OF TWO NEW POLY-
URETHANE POTTING COMPOUNDS

Richard D. Ezell, et al

Naval Ordnance Laboratory

Prepared for:

Naval Air Systems Command

4 April 1973

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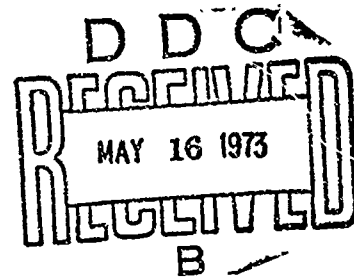
NOLTR 73-42

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NATIONAL TECHNICAL
INFORMATION SERVICE

NOLTR 73-42

15

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D		
<i>Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified</i>		
1. ORIGINATING ACTIVITY (Corporate author) Naval Ordnance Laboratory Silver Spring, Maryland 20910		2a. REPORT SECURITY CLASSIFICATION Unclassified
		2b. GROUP
3. REPORT TITLE Hydrolytic Stability of Two New Polyurethane Potting Compounds		
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)		
5. AUTHOR(S) (First name, middle initial, last name) Richard D. Ezell Joseph M. Augl		
6. REPORT DATE 4 April 1973	7a. TOTAL NO. OF PAGES 815	7b. NO. OF REFS 4
8a. CONTRACT OR GRANT NO. A320-5203/WF00-543-201	9a. ORIGINATOR'S REPORT NUMBER(S) NOLTR 73-42	
b. PROJECT NO.	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) None	
c.		
d.		
10. DISTRIBUTION STATEMENT Approved for public release; distribution is unlimited		
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY Naval Air Systems Command Washington, D. C. 20360
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DD FORM 1 NOV 65 1473

(PAGE 1)

S/N 0101-897-6801

19

UNCLASSIFIED

Security Classification

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Security Classification

KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Potting Compounds Polyurethanes Hydrolytic Stability 1,2-polybutadienes						

16

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NOLTR 73-42

4 April 1973

HYDROLYTIC STABILITY OF TWO NEW
POLYURETHANE POTTING COMPOUNDS

This report describes two new polyurethane potting compounds which have excellent hydrolytic stability. A possible limitation is the fact that isocyanates, which are toxic, are used as the cross-linking agent. The particular isocyanates used, however, have low volatilities, thus reducing the hazard. In any case, there is no danger from the potting compounds as long as all of the isocyanate is consumed in the cross-linking reaction.

Neither this report nor the work it describes should be considered an endorsement by the U. S. Government of these materials or the Hystl. Company.

This work was carried out with funding from the Naval Air Systems Command under Task No. A320-5203/WF00-543-201 in FY1972.

ROBERT WILLIAMSON II
Captain, USN
Commander


ALBERT LIGHTBODY
By direction

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INTRODUCTION

During the last few years the Navy has had difficulties with proprietary potting compounds used in many of the electrical components of F-4 aircraft. For example, during the period from 1958 to 1965 a potting compound called Pro-Seal 777 was used. In 1963, after several years of presumably satisfactory performance, this material occasionally "reverted" from a solid to a viscous liquid. Since the electrical properties of the resin deteriorated along with its physical properties, the electrical connectors were either replaced, or depotted and then repotted, on a per case basis for several years. By 1965 a very large number of such failures was occurring, particularly in aircraft being operated in humid tropical climates. During this period the defective Pro-Seal 777 was being replaced by a more reversion resistant material made by the 3M Company known as EC-2273. In 1965 it was decided to use EC-2273 in all future F-4 construction.

This change in potting compounds, however, did not solve the problem. In 1968, 1969, and 1970 occasional failures occurred even with the EC-2273. Certain blocks of aircraft had a relatively large number of documented cases of reversion, the pattern of failure being very similar to that experienced with the Pro-Seal 777. Thus, in late 1969 EC-2273 was replaced by an even more reversion resistant polysulfide in all new F-4 construction. But the Navy had every reason to expect, on the basis of previous experience, an oncoming costly repair bill on all F-4 aircraft having EC-2273 potted electrical components. On the basis of data available at that time, it was expected that the problem would peak in 1972 or 1973. Thus, there seemed to be ample time to investigate methods of reducing or possibly avoiding the costly depotting-repotting procedure used with the Pro-Seal 777 and which had failed in the earlier F-4's.

Previous work at this laboratory (reference 1,2) resulted in two solutions to the problem of reversion of the EC-2273. One involved a simple static depotting procedure which reduced the time, manpower, and equipment requirements for depotting and repotting the connectors. The second procedure involved rehardening either virgin connectors or those that had begun to deteriorate, forming a protective skin which prevented the center from running out of the connector when it liquefied. The latter procedure extended the usable life of the potting compound up to 100% in laboratory tests.

During and following these tests, other potting compounds were being investigated at the Naval Ordnance Laboratory. Two new

compounds, based on a precursor resin manufactured by the Hystl Development Company, Redondo Beach, California, were conceived and developed in a joint effort by the Hystl Company and the Naval Ordnance Laboratory. These compounds are polyurethanes prepared by cross-linking an OH-terminated 1,2-polybutadiene with tri-functional isocyanates. The low volatility of the isocyanates used in the process reduces the toxicity and preparation hazards considerably. The results of tests on these compounds indicate that they have much greater hydrolytic stability than previously used materials.

EXPERIMENTAL

The manufacturer's designations of the new compounds are as follows:

<u>Compound</u>	<u>Manufacturer's Designations</u>	
	<u>Name</u>	<u>Number</u>
Hystl I	Desmodur N Formulation	6793-223
Hystl II	Isonate Formulation	6793-224

One property of prime importance when considering the usefulness of a potting compound is its hydrolytic stability. To test the stability of the new compounds, specimens 5 x 3 x 1 cm were kept in desiccators over a saturated potassium sulfate solution, which maintained a 95% relative humidity inside the desiccators. The cured specimens were obtained from Hystl. The desiccators were kept in ovens at 97°C, 85°C, and 71°C. A fourth set of specimens was kept at ambient conditions (23°C, 50% R.H.). The hardness of each specimen was measured at intervals varying from once every other month for the controls to three times per week for the specimens at 97°C. Measurements were made according to the procedure described in reference 3, using duplicate specimens at each temperature.

The electrical resistances of the samples were measured at 50 and 500 v in order to detect any deterioration of the electrical properties of the resins during the high humidity exposure. The measurements were made using a megohmmeter and copper electrodes having a constant 1 cm separation.

RESULTS AND DISCUSSION

The hydrolytic stability of the resins is indicated in Figures 1-4, in which the change in Shore A hardness of the resins is plotted against time. Both of the Hystl resins show a slight increase

in hardness over an extended period. For Hystl II, this occurs only after passing through a minimum hardness, which varies from Shore A of 48 at 71°C to a Shore A of 35 at 97°C. The increase in hardness is easily explainable in terms of additional cross-linking occurring between the unsaturated parts of the compounds. (Because of the humid environment, it is unlikely that any isocyanate would still be present to effect additional cross-linking after the period of time shown in Figures 1-4.) After passing through a minimum hardness, Hystl II increases in hardness, reaching its original value somewhere around 300 days. In comparison, EC-2273 undergoes complete reversion in only 12 days at 97°C (references 2, 4).

The purpose of following the change in hardness at more than one temperature was to be able to derive an Arrhenius plot for the failure time as determined by a sharp decrease in hardness. Since there was no failure in the materials during the test period, however, such plots could not be made. Furthermore, the lifetime of these compounds is sufficiently long, even under laboratory conditions, that it is reasonable to believe that under normal usage conditions their lifetime would far exceed that of the aircraft.

No change could be detected in the electrical resistances of the resins at the end of the 300 day test period.

CONCLUSIONS AND RECOMMENDATIONS

Because of the obvious stability of these resins under accelerated aging conditions, it is expected that they will easily outperform previously used electrical potting compounds in actual aircraft use. Their lifetime is such that they would easily outlast the aircraft in which they could be used for potting electrical components. It is recommended, therefore, that further laboratory tests be conducted on these materials, including determination of handling characteristics and bonding characteristics (both to the connector and to a primer), further testing of the electrical properties by standard test methods, and finally determination of temperature limitations in terms of their useful lifetime and mechanical and electrical properties.

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ACKNOWLEDGEMENT

The authors wish to express their appreciation to Mr. Thomas Wade for the laboratory work involved in making the hardness measurements.

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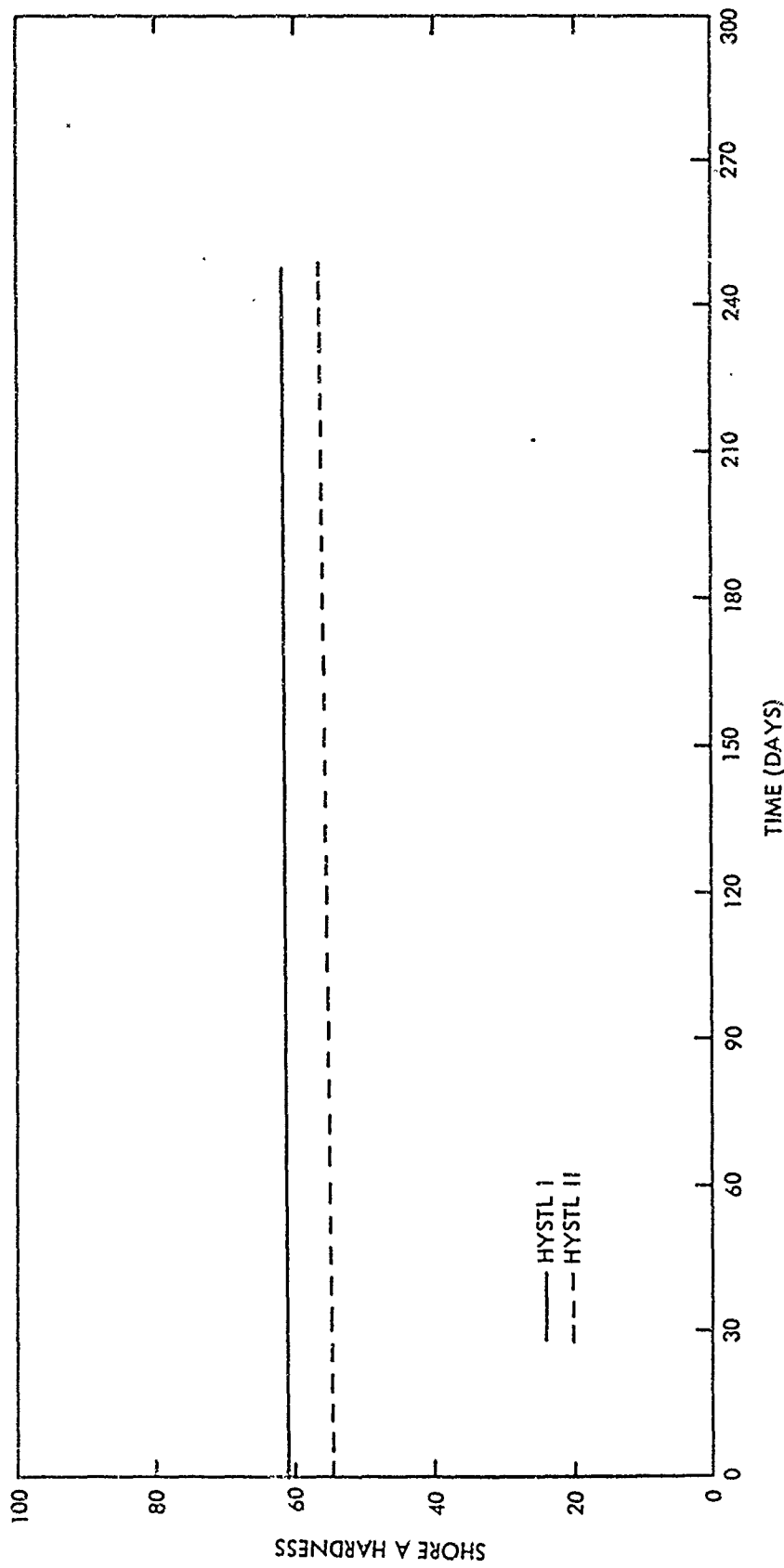


FIG. 1 CHANGE IN HARDNESS WITH EXPOSURE TO AMBIENT CONDITIONS (23° C, 50% R. H.)

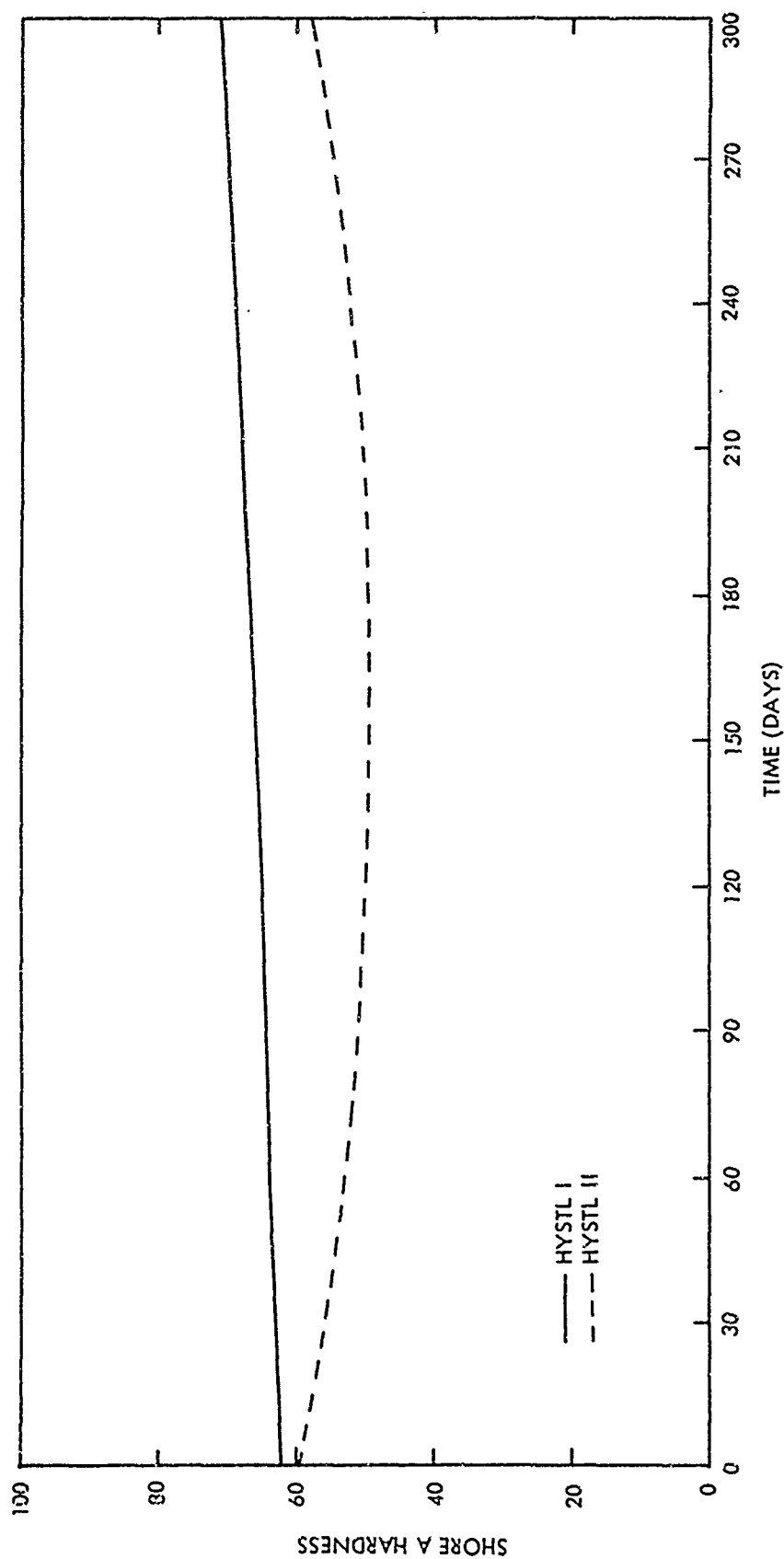


FIG. 2 CHANGE IN HARDNESS WITH EXPOSURE TO 71° C - 95% R. H. ENVIRONMENT

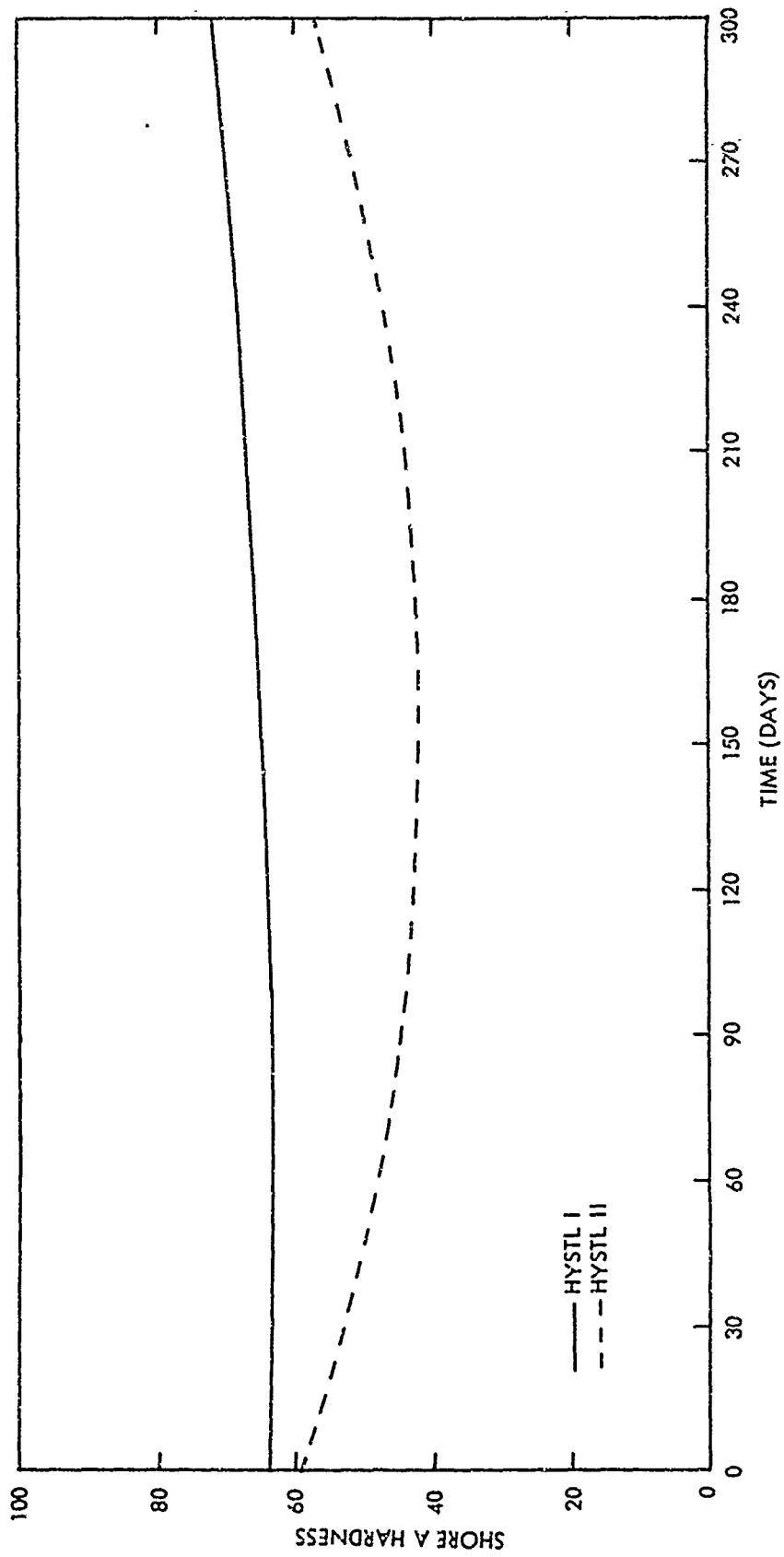


FIG. 3 CHANGE IN HARDNESS WITH EXPOSURE TO 85° C - 95% R. H. ENVIRONMENT

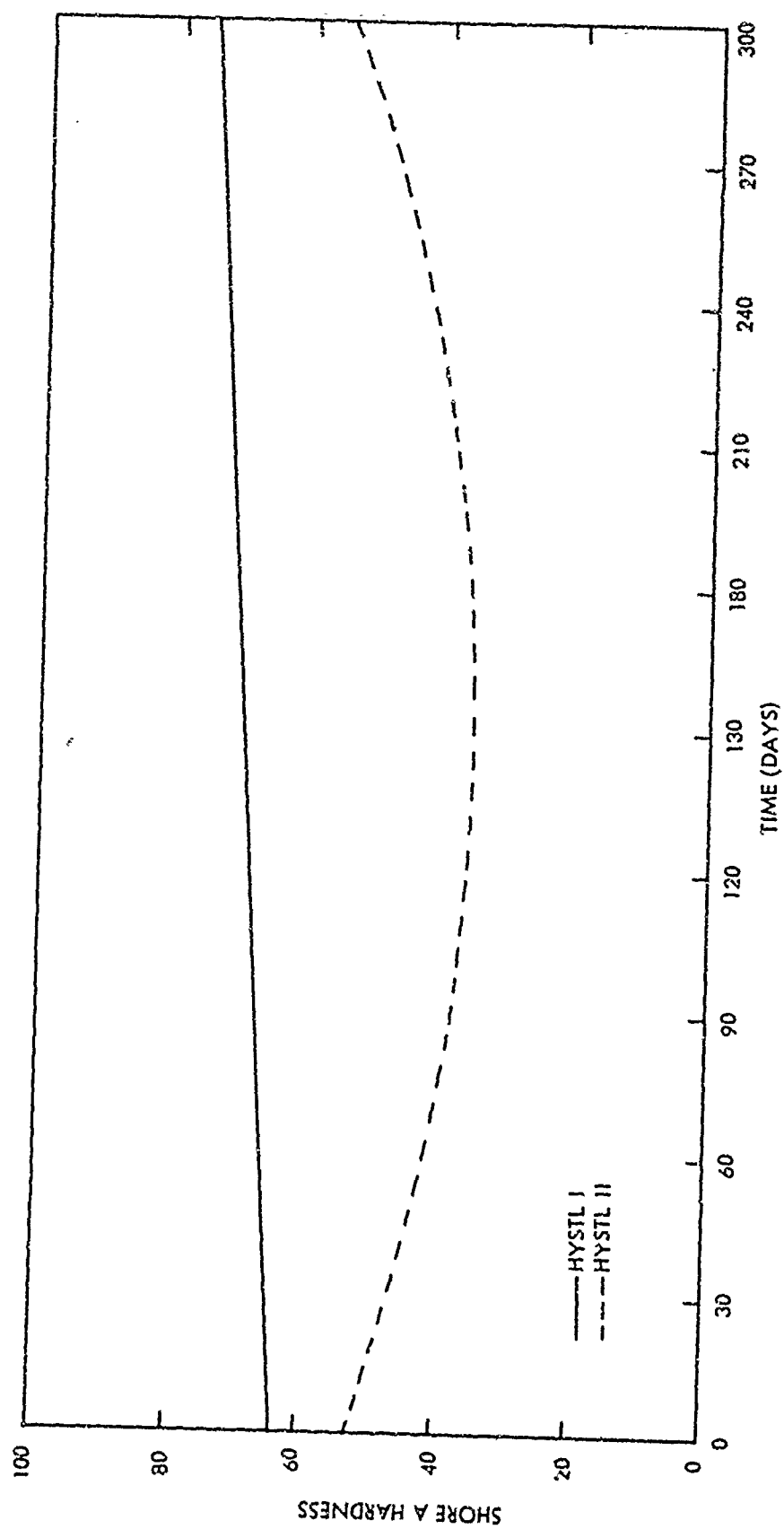


FIG. 4 CHANGE IN HARDNESS WITH EXPOSURE TO 97°C - 95% R. H. ENVIRONMENT