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TWR-13009

SPACE SHUTTLE

[redacted]

REPLACEMENT OF PBNA IN HB AND HC POLYMERS USED IN SRM PROPELLANT AND LINER

1 July 1981

DR NO. [illegible]

PREPARED FOR

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Thiokol

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TITLE

FINAL SUMMARY REPORT-REPLACEMENT OF PBNA IN HB & HC POLYMERS USED IN SRM PROPELLANT & LINER

1 July 1981

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CONTENTS

ILLUSTRATIONS

ILLUSTRATIONS (CONT)

TABLES

GLOSSARY OF TERMS

This report makes use of a number of acronyms and abbreviated forms to describe the complex chemicals referred to in the text. The following list presents these terms:

HB polymer is Poly (butadiene, acrylic acid, acrylonitrile) terpolymer, also referred to in the industry as PBAN, manufactured by the American Synthetic Rubber Co. (ASRC). HB polymer is part of the binder in TP-H1148 and TP-H1178 propellants.

HC polymer is carboxyl-terminated polybutadiene copolymer, manufactured by Thiokol Corporation (TC), Chemical Division. HC is part of the liner and inhibitor formulations.

AOs (Antioxidants) covered in this report are specially listed in Table 1 - (Page 6).

ERL 510 is the epoxide curing agent used in liner and inhibitor. It is [4-(2, 3-epoxy) propoxy - N, N-bis (2, 3-epoxy propyl)- aniline]. It is also sometime referred to as, simply, ERL.

MAPO is the azirdine curing agent used in the liner and inhibitor. It is methyl azirdinyl phosphine oxide.

AP is the oxidizer in the propellant, and is ammonium perchlorate.

ECA is the epoxy curing agent in the propellant and is the commerical epichlorohydrin of bisphenol-A.

Thixcin E is hydrogenated castor oil used to impart thixotropic properties to the UF-2137 liner.

1.0 INTRODUCTION

The antioxidant phenyl-beta-naphthylamine (PBNA) has been used in both HB and HC polymers. The sole (domestic) supplier of PBNA has withdrawn this product from the market, primarily because of suspected health hazards. Commercially available substitute(s) must be selected and qualified for use in the two polymers. Because the two polymers are chemically different, it was not presumed that one antioxidant would be suitable.

2.0 OBJECTIVES

This program has three major objectives: (1) selection and qualification of a new antioxidant for the HB polymer; (2) selection and qualification of a new antioxidant for HC polymer; and (3) verification of the selection and qualification on the liner-inhibitor-propellant bond interfaces and in the propellant, liner and inhibitor themselves.

3.0 SUMMARY AND CONCLUSIONS

3.1 POLYMER

Initial polymer studies narrowed the possible field of antioxidants to Agerite Stalite, Vanox 13 and AO2246 for HB polymer, and AO2246 and Agerite Geltrol for HC polymer.

HB polymer was then manufactured with Agerite Stalite and Vanox 13. Because AO2246 was difficult at least (and most likely uncontrollable) to get into the polymer, it was discarded as a candidate. After looking at polymer and propellant properties, Agerite Stalite emerged as the preferred choice.

For the HC polymer, AO2246 emerged as the preferred choice after the polymer was manufactured with no antioxidant and various AOs individually added to drums of the manufactured production-size batch.

A three-batch "lot" of HB polymer was then manufactured using Agerite Stalite. The lot was within all SRM HB lot specifications. This material was used for all subsequent qualification tests.

A sizeable lot of HC polymer was manufactured with AO2246 for another program. This polymer, also within the SRM HC lot specification, was also used for some tests.

HB with 1.1% PBNA (10% and 1%) were mixed with HB with 1.1% Stalite (90% and 99%) to check for compatibility, as would occur in the tank farms (5% heel at both TC and vendor). There was no evidence of incompatibility.

An analytical method was developed to independently measure both Stalite and PBNA in the polymer (DAP 0285 Rev A). Both the HB and HC polymers with the new AOs aged at least as good as the polymers with the old AOs.

3.2 PROPELLANT

A 5-gallon standardization of TP-H1148 propellant was made in preparation for the 600-gallon full qualification mix. A slightly higher end-of-mix viscosity was evident. All other mix data were normal.

The 600-gallon mix was made and the multitude of mechanical property specimens were cast without problems; however, the end-of-mix viscosity was again higher than normal.

A 5-gallon rheology study followed, showing that the flow rate of the new AO HB propellant would have a (calculated) flow rate of 123.4 lb/min, whereas the control propellant, with PBNA HB, has a flow rate of 162.2 lb/min. This should not significantly affect a motor casting because the new flow rate still exceeds the casting time requirement.

Extensive mechanical property testing was performed on the samples from the 600-gallon mix. Generally, the Stalite HB propellant correlates well with the control propellant, with PBNA HB.

The tests were run at zero time, after one year of ambient aging, and after six months at 150°F aging. In all cases the Stalite propellant aged as well as the PBNA propellant.

The tests run were uniaxial tensile, biaxial tensile, ambient and pressurized, long-term constant strain, stress relaxation, ambient and pressurized, and cohesive fracture energy, pressurized and unpressurized.

3.3 LINER

Several iterations of liner and liner - propellant bond tests were made. It was apparent after the first test series that the new AO HC liner was curing faster, and because of that mechanism the liner-propellant bond was poorer than the control.

The direct substitution of HC Polymer with AO2246 antioxidant to replace the present PBNA antioxidant system in UF-2137 liner, results in the following:

a. Liner cure rate is increased which will require process changes.
b. Decrease in liner strain capabilities with increase in stress and hardness.
c. Lower peel strength to propellant and possible shorter shelf life of lined motor (i.e. time between liner precure and propellant cast).

Three approaches were tested decreasing the total curing agents (MAPO + ERL), decreasing the cure catalyst, and decreasing only the ERL-510.

The reduction of the curing agents to polymer equivalents ratio while maintaining the MAPO/ERL equivalents ratio resulted in softer liner and higher peel strengths if the propellant was cast upon the 68 hour precured liner. The peel strength decreased to almost half the value when the propellant was cast on the 168 hour precured liner, which implies a liner storage life problem.

It was postulated that PBNA interferes with the epoxy/carboxyl (ERL/HC) reaction. Reaction rate tests of AO2246 and PBNA with the ERL-510 using infrared spectroscopy indicated that PBNA reacted with the ERL-510 at 3.5 times the rate at which AO2246 reacted. The reactions of both PBNA and AO2246 with MAPO were negligible. This implies that the liner formulation should be modified by changing the MAPO/ERL equivalents ratio to accommodate the lower reaction rate of the AO2246 with the ERL-510.

Tests conducted with liner formulated at modified MAPO/ERL equivalents ratios further verified the PBNA, AO2246, ERL-510 reaction rate tests. The highest peel strength at both the 68 hour and 168 hour liner precure times were attained with liner made with a MAPO/ERL equivalents ratio of 4.67 (standard UF-2137 has a MAPO/ERL equivalents ratio of 2.06). This formulation also resulted in a liner cure rate and liner physical properties that approximate that of the standard UF-2137 liner.

Of these three approaches, then, decreasing the percent ERL in the formulation most closely approached the zero time characteristics of the liner

and liner-propellant bond of PBNA HC liner. Decreasing the amount of ERL does not necessarily increase the moisture sensitivity because the extra ERL was being used up by the PBNA. Reducing the ERL readjusts the stoichiometry to achieve the same overall properties.

4.0 RECOMMENDATIONS

Propellant made with Stalite HB is perfectly acceptable (by itself) from all mechanical properties and aging standpoints. Although it has a slightly higher viscosity, it is still acceptable for casting motors. It is therefore recommended that the Stalite be used as a direct replacement for PBNA.

The use of AO2246 in liner and inhibitor requires that the liner formulation be adjusted to compensate for the reaction of PBNA with ERL. The present data indicate the MAPO:ERL ratio of 2.06:1.0 should be changed to about 4:1, while keeping the MAPO:HC at about 0.7:1.

The chemical resistance of the liner is enhanced by the ERL. Without the ERL the liner readily hydrolyzes. To date the selection of the ERL content has been done through empirical process tests. In order to select the proper ERL content, reaction rate tests of the various liner constituents should be made in relation to the resultant chemical resistance of the liner.

After this selection, various process tests should be conducted to regain the data base for future problem resolution. Examples of these tests are as follows:

a. Liner specification studies, including tensile adhesion to NBR, tensile adhesion to steel, peel strength to propellant, peel strength after 1,000 hours, and Shore A hardness

b. Liner production process studies, including HC polymer preheat time limitations, slinger application life, liner-liner bond strength (time limit between brush application and sling lining), and maximum time at ambient prior to 135°F cure initiation

c. Other special liner requirements, including moisture tolerance during processing, aging quality of the liner-propellant bond, aging at high humidity, and tolerance to oils and greases (seven kinds, per TWR-12998)

d. Inhibitor specification studies, including viscosity, tensile adhesion to TP-H1148 propellant, and Shore A hardness

e. Inhibitor production process studies, including HC polymer preheat time limitations, storage and template application life from end of mix, and inhibitor degas and application properties

f. Other special inhibitor requirements, including peel strength vs cure (both propellant and inhibitor cure), moisture tolerance during processing, aging quality of inhibitor-propellant bond, aging at high humidity, and tolerance to oils and greases (again per TWR-12998)

5.0 TECHNICAL DISCUSSION - POLYMER STUDIES

5.1 INITIAL POLYMER STUDIES AND ANTIOXIDANT SCREENING

Polymer free of PBNA was prepared by stripping production polymer through an ASCO 2-in. diameter film still under high vacuum. In the case of HB, other volatile constituents which were removed along with PBNA were reconstituted and blended back into the polymer. Candidate antioxidants were blended into PBNA-free polymer by dissolving both polymer and antioxidant in chloroform and then removing the solvent using a Rinco evaporator. Antioxidants were tested in HB at the one percent level and the HC at the two percent level. Two antioxidants tested are given in Table 1, plus PBNA as a control. It should be noted that when PBNA was added back to stripped HB polymer, the mixture did not exhibit the same viscosity as the starting material, indicating the viscosity data should not be compared to normal production polymer but only the PBNA "control" used in this study.

Polymer-antioxidant mixtures were subjected to the following tests, at zero time, after four weeks, at 150°F, and after eight weeks at ambient storage: (1) visual observation for evidence of insolubility, color change, precipitate formation, etc.; (2) viscosity at 25°C; (3) acid number; (4) molecular weight distribution; and (5) total unsaturation. For the sake of brevity, complete test data are not reproduced here, but may be found in total in TWR-20608. Only viscosity and molecular weight appeared to show significant change with time and temperature, i.e., the measured changes with time in acid number and iodine number were within the three-sigma standard

TABLE 1

CLASSES OF COMPOUNDS SUBJECTED TO PRELIMINARY EVALUATION AS ANTIOXIDANTS IN HB POLYMER

1. Hindered Phenols

   *a. Cyanox 2246 (also referred to as AO2246)
   2,2'-methylene bis (4-methyl-6-tert-butyl) phenol
   *b. Agerite Geltrol
   Modified High Molecular Weight Hindered Phenol
   *c. Vanox 13
   Modified Polyalkyl Phosphited Polyphenol
   *d. Irganox 1076
   High Molecular Weight Hindered Phenolic Ester

2. Phenylene Diamines

   a. Antozite 1
   N,N'-dioctyl-p-phenylene diamine
   b. Antozite 67
   N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylene diamine
   c. Flexzone 6H
   N-cyclohexyl-N'-phenyl-p-phenylene diamine

3. Quinolines

   a. Agerite Resin D
   Polymerized 1,2-dihydro-2,2,4-trimethylquinoline

4. Alkylated Diphenyl Amines

   a. Vanox 12
   Octyl Diphenyl Amine
   *b. Agerite Stalite (MOD)
   Mixed Octylated Diphenylamines

* Also evaluated in HC polymer.

deviation of the test method itself. In the case of acid number, there was some impact on the absolute magnitude of the test data, depending on whether the antioxidant is itself acidic or basic, i.e., the antioxidant may or may not influence the test method. However, in terms of polymer stability, the acid number test did not discriminate among the antioxidants. The same was true of number average molecular weight ($\bar{M}_n$) in HB polymer, whereas weight average molecular weight ($\bar{M}_w$) showed some discrimination among the antioxidants in both HB and HC.

General results for each polymer-antioxidant system are as follows:

HB-Cyanox 2246 - Good ambient polymer stability was noted, comparable to but slightly inferior to PBNA ambient protection. Very poor polymer stability was noted at 150°F as measured by viscosity changes. Molecular weight properties were stable. Because of this antioxidant's known compatibility with propellant and liner systems it was selected as a backup choice for further study in spite of the viscosity instability.

HB-Agerite Geltrol - Very good polymer stability was noted, both ambient and 150°F. Viscosity properties, however, averaged 125 poises higher than the PBNA control. Because of potential impact on SRM propellant processing, this antioxidant was not recommended for further study in HB.

HB-Vanox 12 - Polymer stability comparable to PBNA was noted. Viscosity properties averaged a little higher than the PBNA control. The $\bar{M}_w$ showed a small increase at 150°F. Polymer color was notably lighter than the PBNA control. This antioxidant was recommended for further study, however, because Vanox 13 is a more commercial product (than the specialized Vanox 12), this AO was held back in favor of Vanox 13.

HB-Antozite 1 - Somewhat lower polymer stability than PBNA was noted. The polymer color was extremely dark. This antioxidant eventually precipitated out under ambient conditions and was not recommended for further study.

HB-Irganox 1076 - Poor ambient polymer stability was noted. This antioxidant was not recommended for further study.

HB-Antoxite 67 - poor polymer stability was noted. The polymer showed an extremely dark color. This antioxidant eventually precipitated out and was not recommended for further study.

HB-Flexzone 6H - Polymer stability comparable to PBNA was noted. Viscosity properties averaged nearly 90 poises higher than the PBNA control. Polymer color was darker than for PBNA. This antioxidant was not recommended for further study unless the initial choices proved to be unsuitable.

HB-Agerite Resin D - Polymer stability somewhat inferior to PBNA was noted, as measured by molecular weight changes. Viscosity properties averaged about 50 poises higher than for the PBNA control. Polymer color was comparable to PBNA. This antioxidant was not recommended for further study unless the initial choices proved to be unsuitable.

HB-Vanox 13 - Excellent polymer stability was noted, superior to the PBNA control. Viscosity properties averaged about 75 poises higher than for the PBNA control. Because of potential impact on propellant processing, this antioxidant was not recommended for futher study at this time. However, Vanox 13 is the first backup choice should the initial choices prove unsuitable.

HB-Agerite Stalite - Good polymer stability was noted, especially at 150°F. Polymer color was much lighter than PBNA. Viscosity properties averaged about 50 poises higher than for PBNA. This antioxidant (a less pure form of Vanox 12) was recommended for futher study in HB polymer.

HC-Cyanox 2246 - Good polymer stability was noted, and polymer properties were similar to the PBNA control. This antioxidant was recommended for futher study in HC polymer.

HC-Agerite Gel ol - Good polymer stability was noted, and polymer properties were similar to the PBNA control. This antioxidant was recommended for further study in HC polymer.

HC-Vanox 13 - Poor polymer stability was noted at 150°F. This antioxidant was not recommended for futher study in HC polymer.

HC-Agerite Stalite - Poor polymer stability was noted at 150°F. This antioxidant was not recommended for futher study in HC polymer.

5.2 INITIAL HB POLYMER MANUFACTURE

In order to provide an actual HB polymer completely manufactured with new antioxidants, two candidate antioxidants were chosen to make polymer in production-size batches at the vendor's plant (American Synthetic Rubber Corp). Because the antioxidant is added after polymerization, but prior to final polymer processing, it was necessary to manufacture the polymer to assure that the new antioxidant would protect the polymer during processing, in addition to providing sufficient material for further experiments.

The three candidate antioxidants selected for the production batches were Agerite Stalite, Vanox 13, AO2246, per the initial polymer studies described in the previous section. Because the program needed to reduce the number of experimental batches to conserve expenditures, only two of the three candidate antioxidants were chosen. Because the AO2246 is a fine white powder that will not dissolve or slurry with soap and water, as required by the manufacturers' process for HB, Agerite Stalite and Vanox 13 were chosen.

Observation of the polymerization, addition of the AOs into the reactors after polymerization, and subsequent polymer processing indicated no difficulties.

After processing, the batches were tested and loaded into drums. Sufficient polymer was sent to Thiokol/Wasatch for continued qualification testing.

The viscosity of the first batch with Agerite Stalite was 478 poises and the second batch with Vanox 13 was 348 poises. Both these viscosities are within normal variation and the differences are not attributed to the AO at this time.

5.3 HC POLYMER MANUFACTURE

One 3,000-lb batch of HC polymer was manufactured at Thiokol, Chemical Division, with no antioxidant. Five drums were then prepared and shipped to Thiokol as follows.

Drum 1 - No antioxidant
Drum 2 - PBNA
Drum 3 - AO 2246
Drum 4 - Agerite Geltrol
Drum 5 - Irganox 1076

5.4 HB POLYMER TESTS

Analytical characterization was performed on the two production size batches of HB polymer manufactured with candidate antioxidants for replacement of PBNA. Resulting data are presented in Table 2 and Figures 1 through 4. Also included are data from two previous lots of HB manufactured with PBNA to provide a baseline for comparison. These baseline data were generated during the qualification of new source DDM, as reported in TWR-20182. The following conclusions were drawn from the data in Table 2:

SUMMARY OF ANALYTICAL DATA FOR NEW ANTIOXIDANT BATCHES OF HB POLYMER

Batch Number Antioxidant	9999-1616 Vanox 13	9999-1617 Agerite Stalite	7227-0020* PBNA	9404-0146** PBNA
Viscosity (Poises at 25°C)				
As received	351	488	382	416
Stripped	550	764	633	711
Acid No. (Equiv/100g)				
As received	0.065	0.065	0.065	0.064
Stripped	0.052	0.052	0.052	0.053
$\overline{M}_n$				
As received	2700	2900	2800	2600
Stripped	2800	3000	3000	3100
$\overline{M}_w$				
As received	6300	7500	6900	6600
Stripped	6700	8000	7400	7500
Total Volatiles, %	3.6	4.4	4.5	5.0
Free Acid, %	17.8	15.3	17.0	17.2
Moisture, %	0.02	0.01	0.01	0.02
Ammonyx G, %	4.90	4.83	4.63	4.53
Chloride, %	0.31	0.31	0.44	0.36
Sulfur, %	1.08	1.10	1.06	1.00
Acrylonitrile, %	10.8	10.9	11.1	11.4
Average Functionality	2.17	2.12	1.91	2.13
Functionality Distribution				
Non, %	12	11	12	8
Mono, %	18	14	19	16
Di, %	16	22	27	30
Tri, %	40	34	35	34
Tetra, %	9	0	0	5
Specific Gravity (25°C/25°C)	0.934	0.941	0.936	0.939

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* New DDM batch selected during qualification studies as target for future production (see TWR-20182).

** Old DDM Lot.

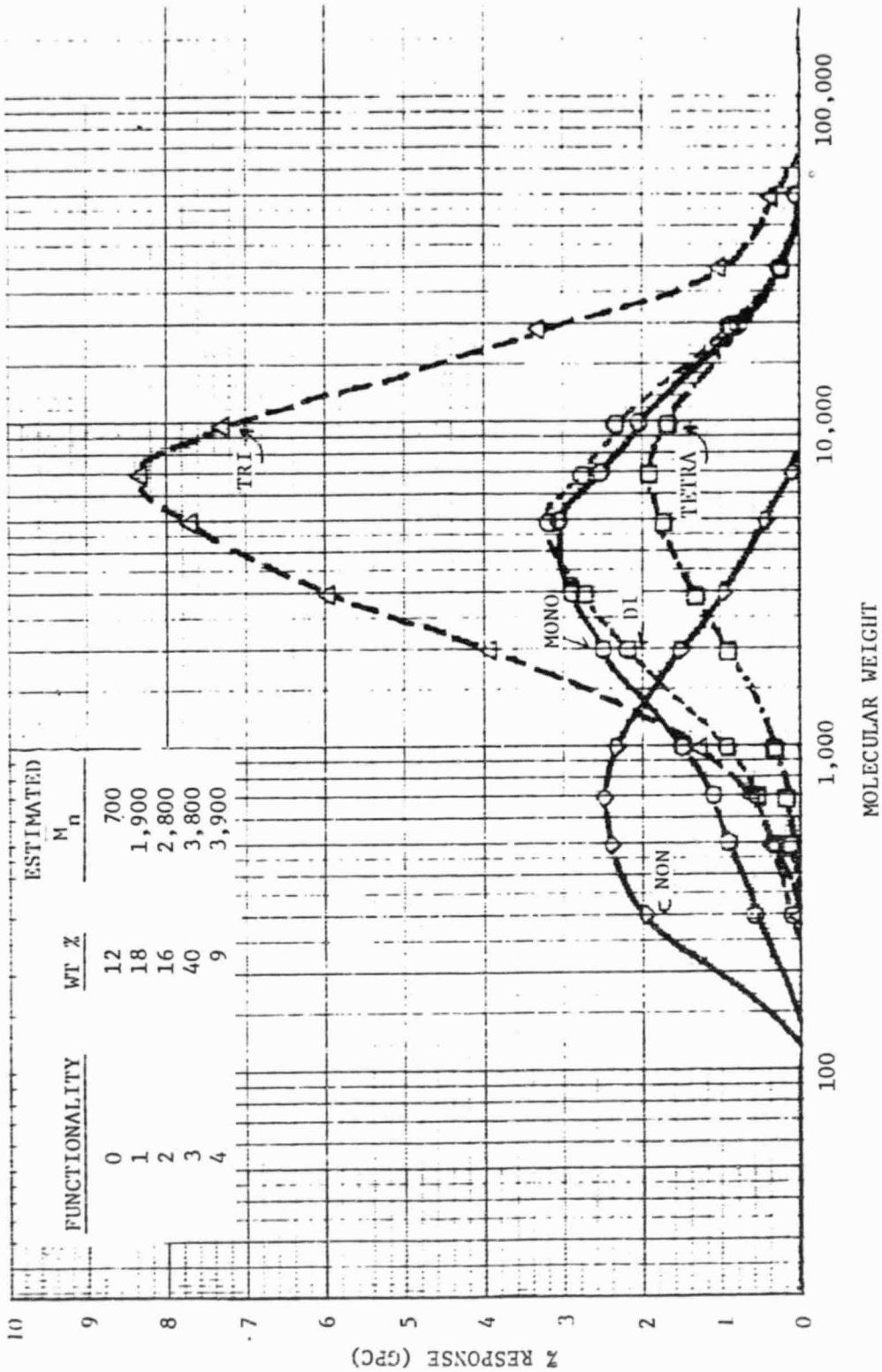

Figure 1. HB Polymer Batch 9999-1616 Vanox 13 Antioxidant

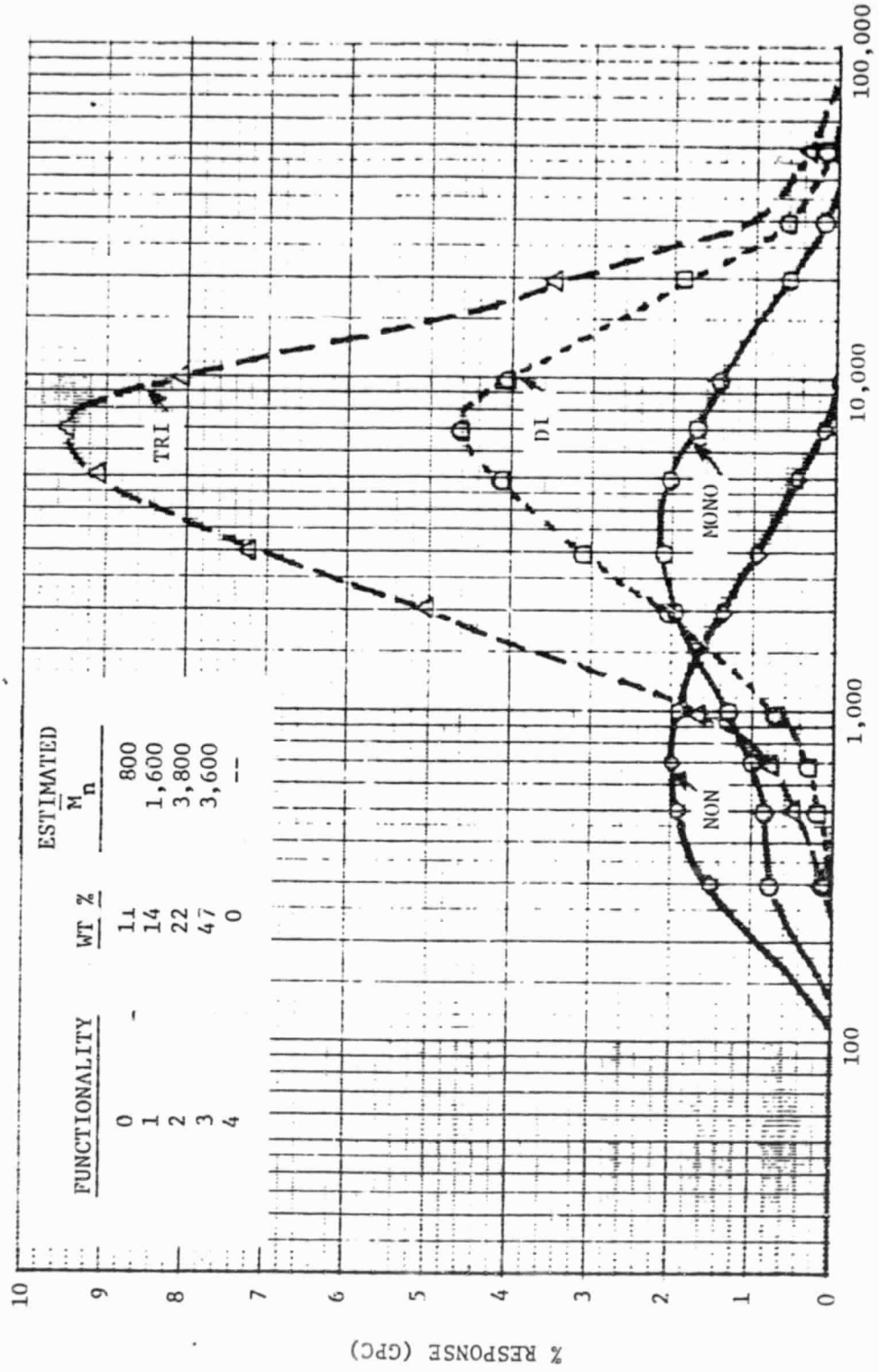

Figure 2. HB Polymer Batch 9999-1617 Agerite Stalite Antioxidant

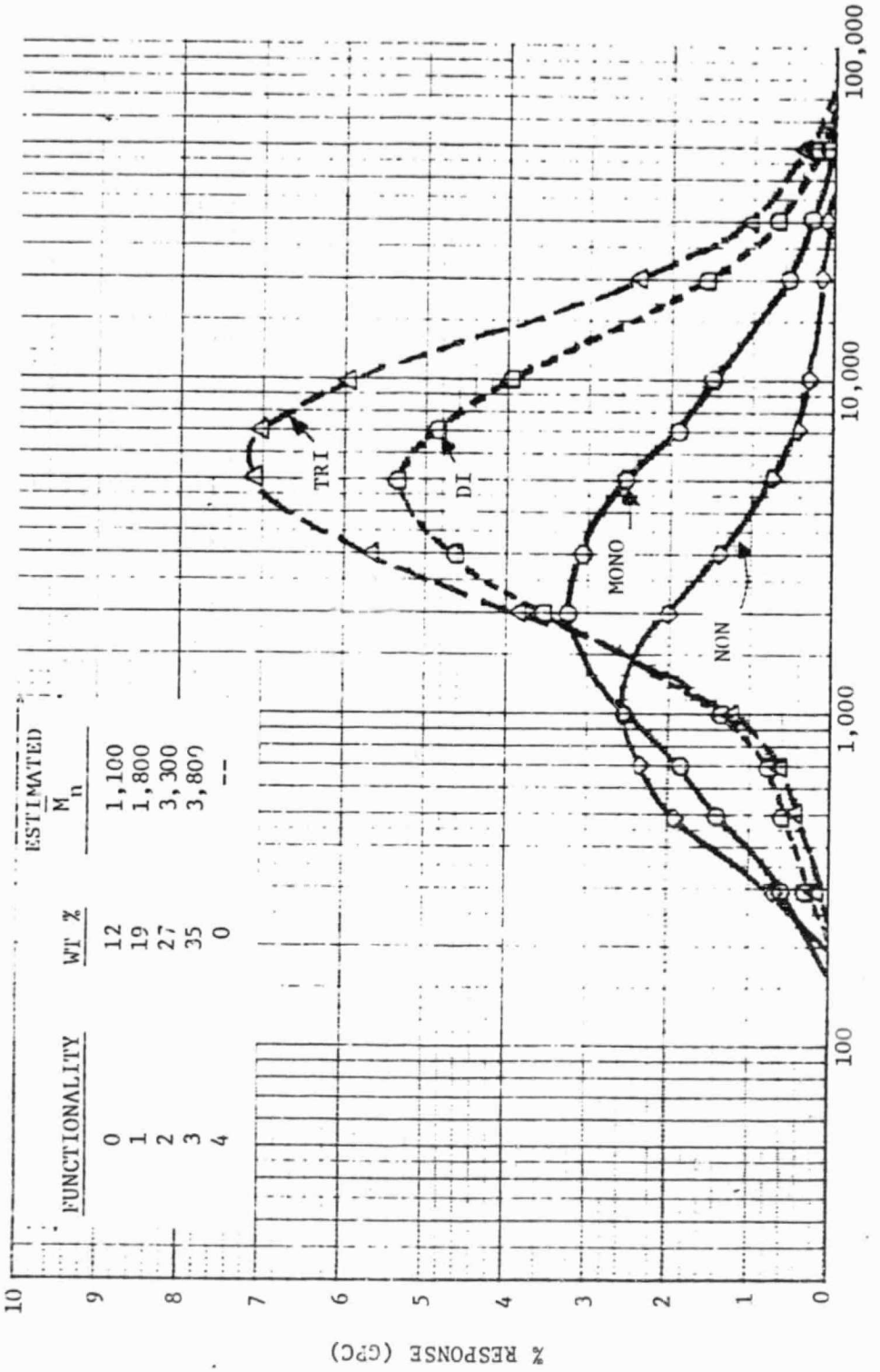

Figure 3. HB Lot 7227-0020 New DDM Target Batch PBNA Antioxidant

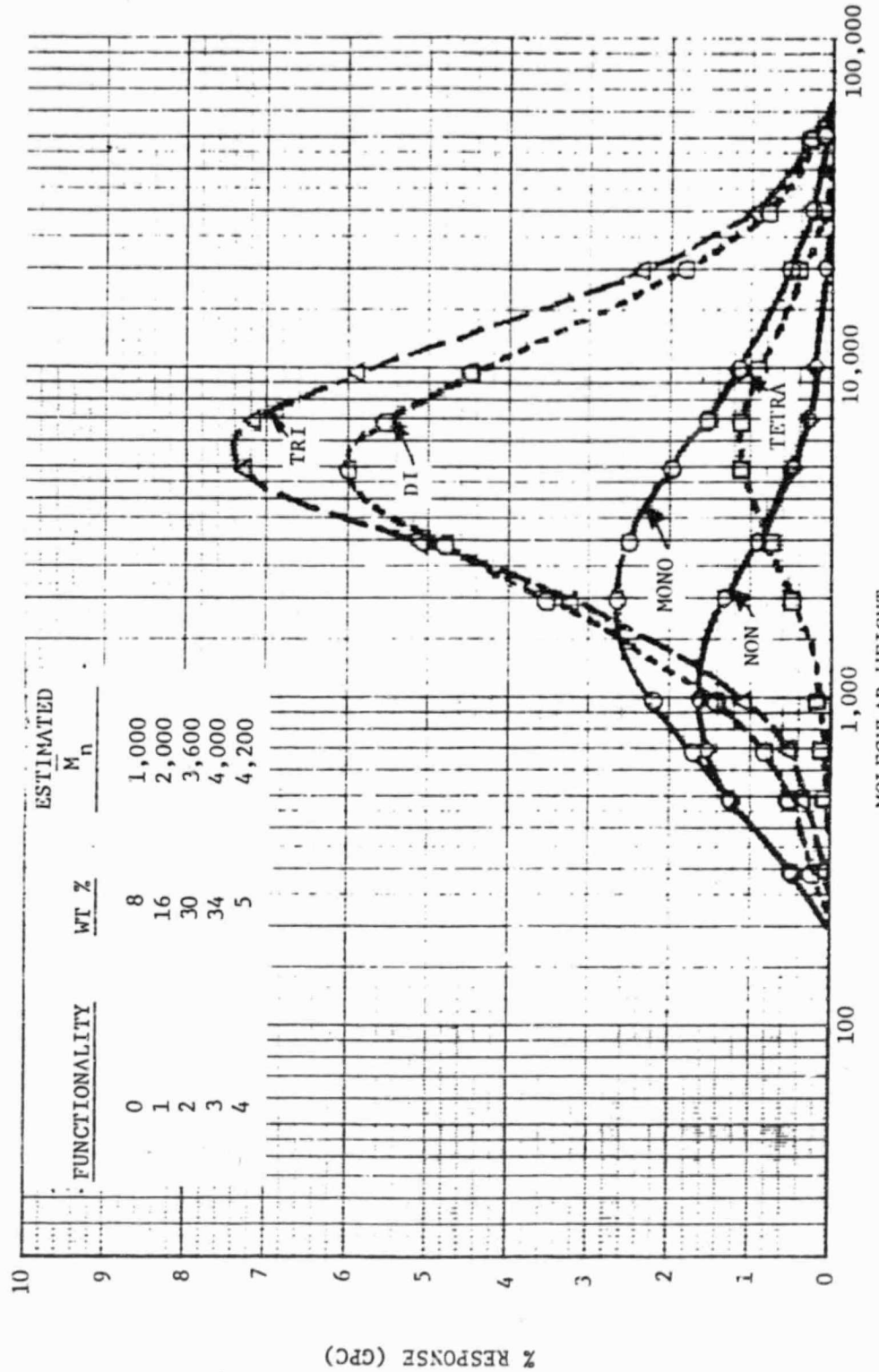

Figure 4. HB Lot 9404-0146 Old DDM Production Lot PBNA Antioxidant

1. Both experimental batches of polymer fall within the normal batch-to-batch variation for HB polymer. Until additional batches have been manufactured and tested, the differences between the two batches (9999-1616 and 1617) cannot necessarily be ascribed to the two different antioxidants.
2. Based strictly on polymer properties alone, either Vanox 13 or Agerite Stalite should be satisfactory as a replacement for PBNA. Final choice between these two should be based on propellant properties.
3. Since no clear preference emerged from the propellant data (paragraph 5.6) Agerite Stalite was recommended for further testing for the following reasons:
   a. The functionality distribution pattern seen in batch 9999-1617 is somewhat more typical of past experience.
   b. Agerite Stalite is less expensive than Vanox 13.
   c. Agerite Stalite is a less complex mixture than is Vanox 13 and should lend itself more readily to analytical determination and subsequent quality control.

5.5 HC POLYMER TESTS

Into separate 30-gallon portions of the same HC polymer batch (9999-1618), different antioxidants were blended as follows:

Antioxidant	% by Wt	Drum No.
PBNA	2	2
AO2246	2	3
Agerite Geltrol	2	4
Irganox 1076	2	5
None (control)	0	1

Analytical data generated for these five drums at Thiokol/Wasatch immediately after receipt are summarized on the following page.

	Drum 1 No. AO	Drum 2 PBNA	Drum 3 AO2246	Drum 4 Ag Geltr	Drum 5 Irganox
Viscosity at 26°C, Poises	236	224	238	232	225
Acid No. (equiv/100g)	0.056	0.054	0.057	0.056	0.055
$\bar{M}_n$	4,070	4,040	4,130	*	*
$\bar{M}_w$	7,260	7,350	7,210	7,440	7,300
Specific Gravity 25°/25°	0.905	0.910	0.908	0.909	0.905

*No test data due to interference from the AO

5.6 ONE-GALLON MIXES (INITIAL HB POLYMER)

Three 1-gallon mixes of Stalite-HB and three 1-gallon mixes of Vanox-13 HB were made to check propellant properties prior to deciding which antioxidant to use. Mechanical properties of the TP-H1148 propellant are as follows:

Polymer	% HB	$E^{2.6}$ (psi)	σ_m (psi)	ϵ_m 2.6 (%)	ϵ_r (%)
Stalite	86	745	153	33	42
Stalite	87	470	111	36	48
Stalite	88	224	70	42	68
Vanox	86	740	151	34	43
Vanox	87	468	110	37	50
Vanox	88	227	72	42	66

Target values for standard TP-H1148 are: $\epsilon^{2.6}$ (psi) = 535, σ_m(psi) = 110, $\epsilon_m^{2.6}$ (%) = 35

From this data, it is apparent that there is no significant difference between propellant made from these polymers as compared to standard TP-H1148 propellant.

Brookfield viscosities ranged from 12.4 to 15.8 kps, which is also normal for this propellant.

Finally, the percent HB required for the target stress of 110 psi is around 87.0% HB, which is also within the normal range required by PBNA polymers.

5.7 FIVE-GALLON MIXES (INITIAL HB POLYMER)

In order to provide liner specimens and verify the 1-gallon mix data, three 5-gallon mixes were made at the target percent HB determined by the 1-gallon mixes. Target mechanical properties were obtained and the remaining loaf cartons used for the initial liner investigation as described below.

The three 5-gallon mixes were a control mix with PBNA-HB polymer, a mix with Agerite Stalite-HB polymer and a mix with Vanox 13-HB polymer.

5.8 SELECTION OF FINAL CANDIDATE ANTIOXIDANTS

For HB polymer, the selection of Agerite Stalite was made after looking at the data reported thus far. A clear choice was either Agerite Stalite or Vanox 13. Because propellant mixes showed no particular preference, Agerite Stalite was chosen because (a) the functionality distribution in the Stalite batch is somewhat more typical of past experience, and (b) Stalite costs less than Vanox 13 and lends itself more readily to analytical determination of percentage in the polymer and, therefore, better subsequent quality control.

A survey was made of the Agerite Stalite supplier, R. T. Vanderbilt Co., with very positive results. Vanderbilt was most cooperative and pulled out 15 years of Stalite manufacturing history, showing a complete set of adequate quality control records of every lot manufactured.

For HC polymer, the field of antioxidants was quickly narrowed to Agerite Geltrol and Cyanox 2246 on the polymer-antioxidant compatibility basis only. The selection of 2246 was then made on the basis of its having been used in another propellant liner system successfully for many years, thus implying both compatibility and adequate supply.

5.9 FINAL QUALIFICATION BATCHES MANUFACTURE

HB Polymer - After finally selecting Agerite Stalite as the antioxidant for HB polymer, four batches of polymer were manufactured at the vendor's plant (American Synthetic Rubber Co.).

The first batch was high in viscosity (466 poise). At this point, the second batch was in progress but the third batch had not been started. Because the first batch was higher in viscosity than expected, starting the third batch was held until the results of the second batch were obtained.

The second batch was 344 poise viscosity, more in line with what was expected. Subsequent investigation indicated that the first batch was much darker than the second, indicating that some sludge of PBNA polymer was picked up in the lines.

The third batch plus a replacement batch for the first one were then manufactured. Viscosities of these two batches were 407 poise and 311 poise respectively.

Viscosity of the blend, designated 9999-1742, was 359 poise, within the specification limits for a lot.

HC Polymer - Because HC polymer can be manufactured and processed without an antioxidant, a single batch was manufactured as described in Section 5.3. Subsequently, a large lot of AO2246 HC polymer was manufactured for other programs and designated as stock-lot number 6296-0007. This material will be used for final data and aging.

It was felt that the manufacture of this polymer was fortuitous and beneficial for the SRM new AO qualification program, because it removes the stigma of a single batch of HC polymer, which has been shown to be a problem in the past. Normally, a lot, consisting of many batches, of HC polymer is used in the SRM motors and our past data and control mixes are based on lots, not batches. Data for lot 6296-0007 are as follows:

	TC Wasatch Analysis	TC Moss Point Analysis
Viscosity (25°C)	220 poises	222
Specific Gravity (20°C/20°C)	0.909	0.908
Acid Number	0.555 (gm/gm equiv)	0.555
Moisture	0.01%	0.020

The lot is well within specification requirements.

5.10 POLYMER PROPERTIES AND AGING

A 15-month polymer shelf life study was conducted to confirm that 1.1 percent Agerite Stalite would protect HB polymer during normal storage. Lot 9999-1742 of HB containing a nominal one percent Agerite Stalite (the full scale manufacturing blend) was stored in glass under air at 120°F and at ambient temperature in glass under air. Lot 7227-0007, containing a nominal one percent PBNA, was stored under identical conditions as a control.

Samples were withdrawn for determination of viscosity, molecular weight, and antioxidant level at zero time, 3 months, 6 months, 9 months, 12 months, and 15 months. Results are given in Table 3. These data indicate that, compared to the PBNA control, Agerite Stalite is entirely satisfactory as an antioxidant for purposes of polymer protection, and there is some suggestion that Agerite Stalite may be slightly superior.

5.11 MIXING OLD AND NEW HB POLYMER

HB with PBNA is nearly opaque and HB with Agerite Stalite is honey-colored and transparent. The tank farms (at the HB vendor's plant and at Thiokol/Wasatch) will each have about a five percent (by weight) heel, so the two polymers were mixed in the laboratory to check for compatibility. Ten percent of PBNA-HB was added to 90% of Agerite Stalite-HB, and a second mixture of one percent PBNA-HB was added to 99% Agerite Stalite-HB.

It should be mentioned that no incompatibility is theoretically expected because PBNA and Agerite Stalite are similar chemical species. This experiment was performed mainly as a final check and to assess the color/transparency changes.

The polymers were simply heated up to 135°F and individually weighed into clear glass sample vials. No mixing was done at first. The two polymers remained essentially separated, as indicated by the color difference. The vials were turned upside down, then returned, many times. The dark-colored PBNA HB did not ever actually mix. The vials were then let set for about 3 weeks. The two phases then became one and remained so.

This indicates that when the Agerite Stalite-HB polymer is introduced into the tank farms at the vendor's plant and at Thiokol/Wasatch there will be a trace impurity of PBNA-HB polymer in the Agerite Stalite-HB polymer for some time.

An analytical method is now available to analyze separately the amounts of Agerite Stalite and PBNA in HB polymer, should this become necessary.

6.0 PROPELLANT DATA AND ANALYSIS

6.1 STANDARDIZATION OF TP-H1148 PROPELLANT WITH AGERITE STALITE ANTIOXIDANT

Five mixes of TP-H1148 propellant were processed in the 5-gallon vertical mixer using raw material evaluation A94 (Agerite Stalite) ingredients.

TABLE 3

POLYMER SHELF LIFE STUDY

	Viscosity at 25°C (Poises)[1]		Number Avg. Mol. Wt. ($\bar{M}_n$)[2]		Weight Avg. Mol. Wt. ($\bar{M}_w$)[3]		Antioxidant Level, %	
	Agerite Stalite[4]	PBNA[5]	Agerite Stalite[4]	PBNA[5]	Agerite Stalite[4]	PBNA[5]	Agerite Stalite[4]	PBNA[5]
Zero Time	359	373	2380	2530	6340	6080	1.25	1.07
3 Months								
Ambient	363	368	2780	2640	6760	6270	1.28	1.06
120°F	359	372	2660	2910	6470	6790	1.30	1.06
6 Months								
Ambient	325	351	2520	2400	6450	6010	1.32	1.10
120°F	330	341	2420	2420	6000	6090	1.35	1.09
9 Months								
Ambient	330	347	2550	2860	6440	6350	1.24	1.08
120°F	354	352	2610	2870	6690	6630	1.40	1.08
12 Months								
Ambient	355	369	2460	2750	6370	6960	1.38	1.09
120°F	376	382	2440	2750	6370	7060	1.30	1.12
15 Months								
Ambient	361	375	2750	2970	6490	6320	1.32	1.08
120°F	379	391	2670	3330	6740	6770	1.29	1.09

1 Cannon-Manning Viscometer
2 GPC Calibrated against VPO
3 GPC Calibrated against VPO
4 HB Lot 9999-1742, Nominal 1% Antioxidant
5 HB Lot 7227-0007, Nominal 1% Antioxidant

Thiokol/WASATCH DIVISION

Three one-half gallon cartons and three TU-131 motors were cast from each mix to evaluate mechanical and ballistic properties.

Raw Materials - The raw material lots used in the standardization mixes are listed in Table 4. The particle size distribution of the ground AP is summarized in Table 5. It is observed that grind BUD A94-10026 is coarse and not totally representative of SRM history. Note also the inconsistency between thief and flap samples.

Rheology - The EOM propellant viscosity data obtained from the standardization mixes are listed in Table 6. Brookfield viscosities (TD spindle, 1 rpm) ranged from 19.1 to 20.8 kp.

Mechanical Properties - Mechanical property data obtained from the standardization are summarized in Table 7. These data are presented graphically in the following figures.

Figure 5 - Mechanical Properties vs HP Polymer Content

Figure 6 - Propellant Mechanical Properties

The response of maximum stress, strain at maximum stress and modulus to variations in the HB polymer content is presented below:

1. σ_m = 4,012.4-45.00 (HB)
2. $\varepsilon_{m-}^{2.6}$ = 96.74-0.7143 (HB)
3. $E^{2.6}$ = 23,665.6-266.79 (HB)

The mechanical property requirements defined in the propellant specifications are summarized below:

Parameter	Specification Requirement	Standardization Target
σ_m (psi)	80	110
ε_{m-}2.6 (%)	30	--

Solution of equation (1) for the target stress of 110 psi results in an HB polymer content of 86.7 percent. Corresponding solutions for equations (2) and (3) result in the following predicted properties:

HB	86.7%
σ_m	110 psi
ε_{m-}2.6	35%
$E^{2.6}$	535 psi

TABLE 4

RAW MATERIAL EVALUATION A94
TP-H1148 PROPELLANT

Ingredient	Lot Number	Grind Number
HB Polymer	9999-1742	
Aluminum	7228-0005	
Ferric Oxide	7226-0004	
AP - Ground	7229-0004	BUD A94-10026
AP - Unground	7229-0004	
ECA	7225-0007	

TABLE 5

AP PARTICLE SIZE DISTRIBUTION

	Screen (%)		Coulter Counter (μ)		
Grind Number	200 Mesh	325 Mesh	10%	50%	90%
BUD A94-10026			7.3	25.8	64.0
A Flap	6.1	27.0			
B Flap	6.1	29.4			
Thief	3.3	21.0			

TABLE 6

UNCURED PROPELLANT PROPERTIES
TP-H1148 PROPELLANT
EVAL A94

				EOM Rheology		LSBR at 1500 psig	
Mix Number	HB (%)	Fe_2O_3 (%)	TS (%)	Visc* (kp)	Temp (°F)	Break Wire (in./sec)	Acoustical (in./sec)
A947001	87.7	0.29	85.79	20.8	144	0.551	0.544
2	87.7	0.09	85.97	19.7	142	0.498	0.481
3	87.0	0.19	85.93	18.1	144	0.548	0.528
4	86.3	0.29	86.00	18.4	141	0.566	0.543
4	86.3	0.09	85.96	19.2	141	0.504	0.486

* Brookfield Viscometer, TD Spindle, 1 rpm.

TABLE 7

CURED PROPELLANT PROPERTIES
TP-H1148 PROPELLANT
EVAL A94

			Mech. Properties (2 in/min)					
Mix Number	HB (%)	Fe_2O_3 (%)	σ_m (psi)	$\epsilon_m 2.6$ (%)	$\epsilon_r 2.6$ (%)	$E^{2.6}$ (psi)	Strand R_b (in/sec)	TU-131 R_b (in/sec)
A947001	87.7	0.29	67	34	66	277	0.384	0.371
2	87.7	0.09	63	34	65	251	0.350	0.339
3	87.0	0.19	101	35	53	473	0.371	0.358
4	86.3	0.29	130	36	45	620	0.380	0.368
5	86.3	0.09	126	34	45	655	0.346	0.343

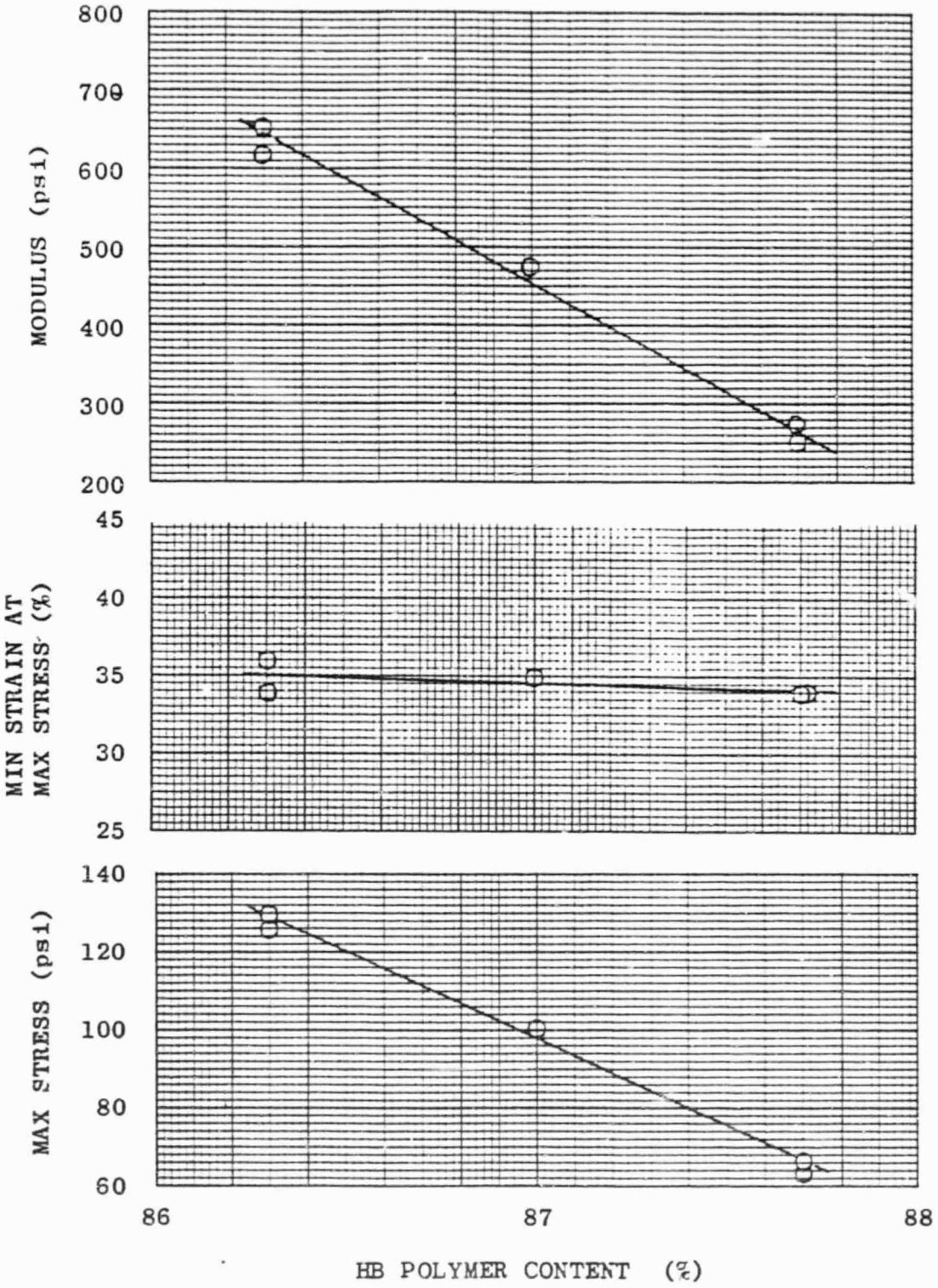

Figure 5. Mechanical Properties vs HB Polymer Content
TP-H1148 Propellant Eval A94

Figure 6. Propellant Mechanical Properties TP-H1148 Propellant Eval A94

Ballistic Properties - Ballistic property data obtained from the standardization mixes are summarized in Table 7. The response of liquid strand and TU-131 motor burn rates to variations in the ferric oxide content is illustrated in Figure 7. The responses may be expressed mathematically as follows:

(4) LSBR at 1,500 psig = 0.4788 + 0.2875 (Fe_2O_3)

(5) TU-131 R_b at 625 psia = 0.3287 + 0.1425 (Fe_2O_3)

Solution of equation (5) for the target burn rate of 0.356 in./sec results in a ferric oxide content of 0.19 percent. Corresponding solution of equation (4) results in an LSBR target of 0.533 in./sec for process control.

It was noted that the viscosity of the propellant is slightly higher than previous standardization mixes with PBNA polymer, but not enough to be significant with this amount of data.

6.2 600-GALLON MIX OF TP-H1148 PROPELLANT WITH AGERITE STALITE ANTIOXIDANT

In order to provide sufficient loaf cartons and strain evaluation samples, a 600-gallon mix of propellant was made. All samples were cast and submitted to various zero time and aging mechanical property tests. The 600-gallon mix had a zero time ambient max stress of 99 psi (11 psi below target, but well within prediction capabilities) and normal strains and modulus values.

A slightly higher end-of-mix viscosity was noted in this mix, and on the 5-gallon standardization mixes. For this reason, additional 5-gallon mixes were made for a flow study (next paragraph).

6.3 ADDITIONAL 5-GALLON MIXES TO STUDY PROPELLANT RHEOLOGY

Two TP-H1148 5-gallon mixes were completed for this study. Mix 209-5-0489 contained Agerite Stalite antioxidant and Mix 209-5-0512 contained PBNA antioxidant. All other ingredients and the formulation were identical for the two mixes.

The propellant from the two mixes was cast through a 5/32 in. slit plate at 135°F. The viscosity of the propellant was monitored during casting. These data are shown in Table 8 and Figures 8 and 9.

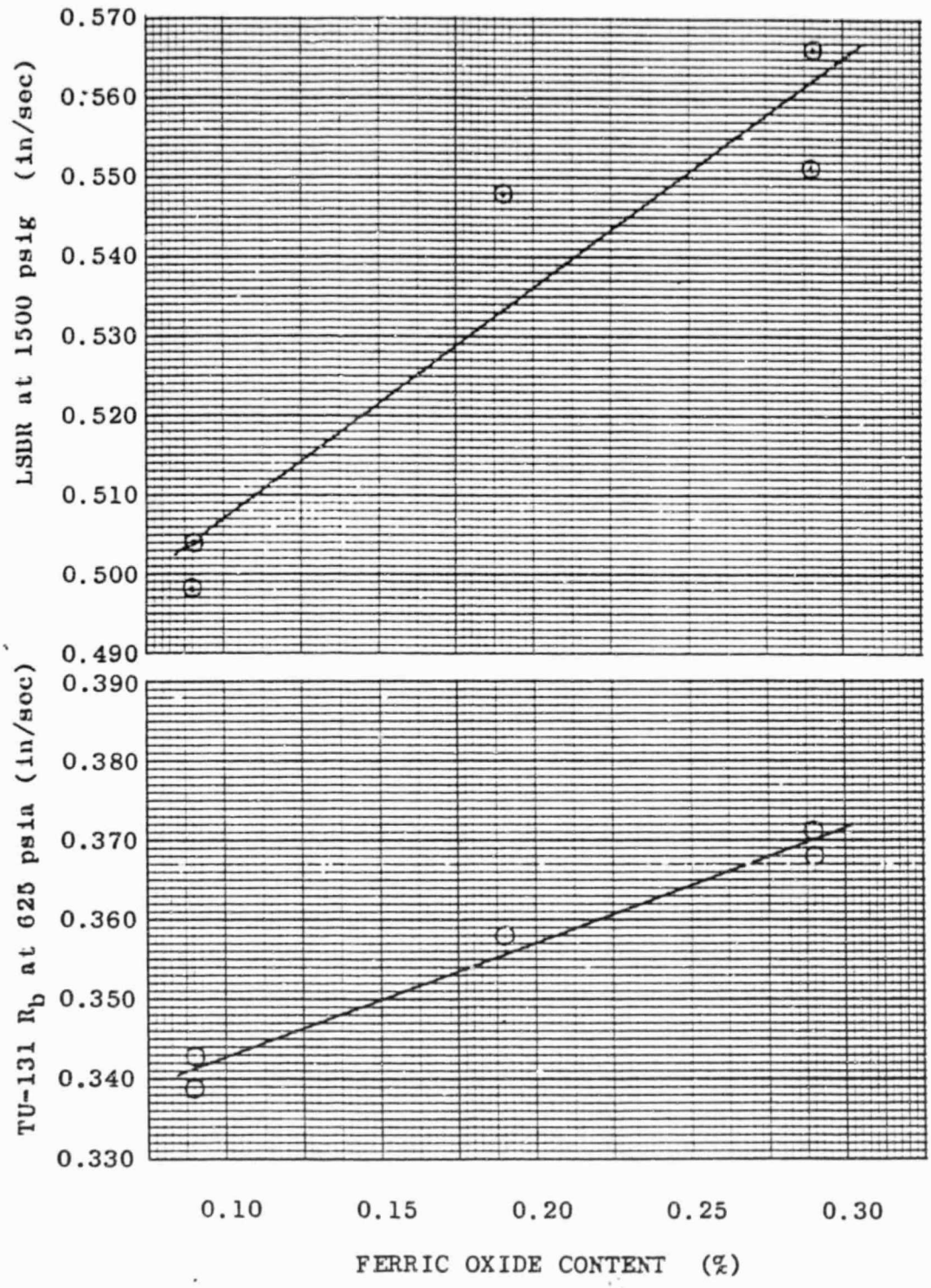

Figure 7. Ballistic Property Summary TP-H1148 Propellant Eval A94

TABLE 8

VISCOSITY AND FLOW RATE DATA FOR TP-H1148
PROPELLANT CAST THROUGH A 5/32 IN. SLIT PLATE AT 135°F

Mix 209-5-0489 New Agerite Stalite AO			Mix 209-5-0512 Old PBNA AO		
Time From EOM* (Hr)	Brookfield Viscosity (KP)	Flow Rate (lb/min/linear in.)	Time From EOM (Hrs.)	Brookfield Viscosity (KP)	Flow Rate (lb/min/linear in)
.67	18.4	.488	.67	19.3	.492
1.17	27.2	.301	1.08	22.3	.390
1.75	28.2	.230	1.58	25.5	.323
2.17	32.9	.197	2.08	29.1	.280
2.67	35.0	.182	2.58	31.5	.258
3.17	37.1	.145	3.08	35.5	.202
3.67	40.1	.146	3.58	37.2	.206
4.17	44.8	.134	4.08	43.7	.197
4.67	49.8	.130			
5.17	54.1	.118			

*End of Mix

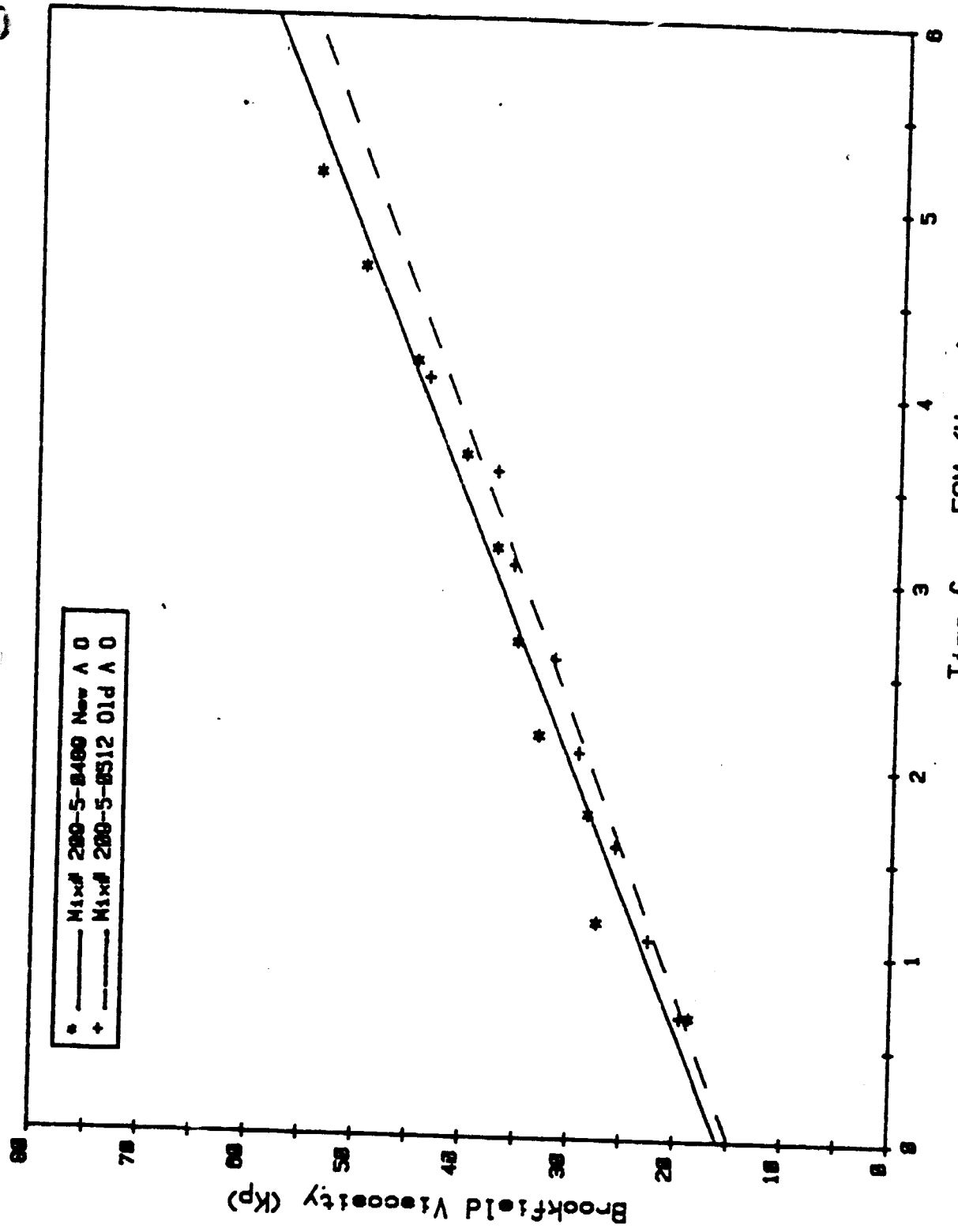

Figure 8. Brookfield Viscosity vs Time From EOM for Old and New Antioxidant Propellants

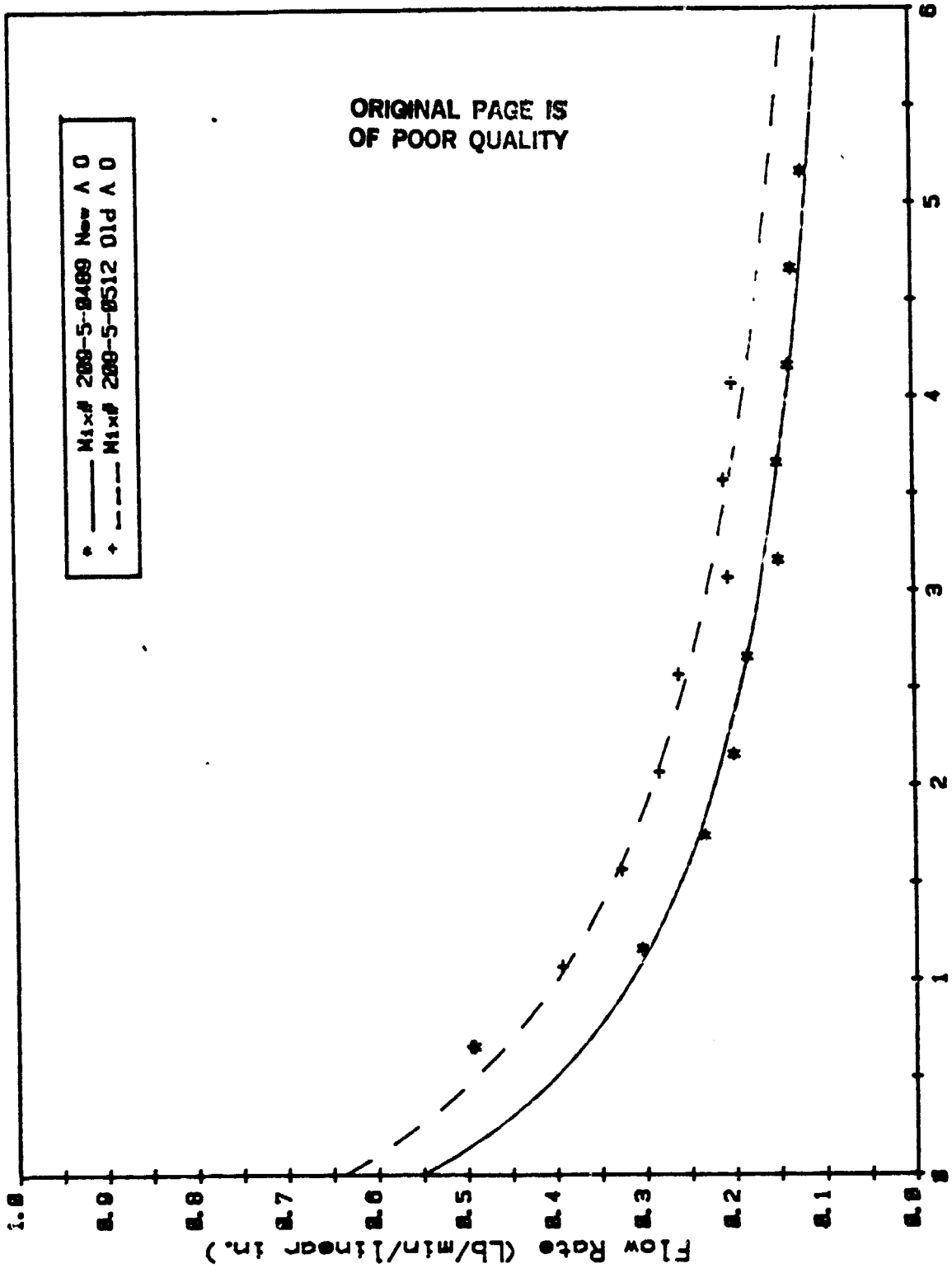

Figure 9. Flow Rate vs Time From EOM for Old and New Antioxidant Propellants

As can be seen by examining the data, there are differences in viscosity and flow rate for the two different antioxidants.

In order to show these differences more clearly, the effect the different propellants have on casting a Shuttle segment will be calculated.

Regression of the data from Figure 9 yields the following flow rate equations:

$$Q_{new} = 1/(1.817 + 1.349\ EOM)$$

$$Q_{old} = 1/(1.568 + 0.933\ EOM)$$

Where

Q_{new} = flow rate of Agerite Stalite AO propellant (lb/min/linear in.)

Q_{old} = flow rate of PBNA AO propellant (lb/min/linear in.)

EOM = time after EOM (hr)

A slit plate for casting a Shuttle segment contains 557 linear inches of slit. Using a value of two hours after EOM for comparison and equations 1 and 2, flow rates are calculated.

$$Q_{new} = [1/(1.817 + 1.349 \times 2)]\ 557$$

$$= \underline{123.4\ lb/min}$$

$$Q_{old} = [1/(1.568 + 0.933 \times 2)]\ 557$$

$$= \underline{162.2\ lb/min}$$

This shows that the new Agerite Stalite antioxidant propellant has a 38.8 lb/min lower flow rate than the old PBNA antioxidant propellant when using a single slit plate two hours after EOM.

6.4 TP-H1148 MECHANICAL PROPERTIES AND AGING

Summary - An extensive mechanical properties characterization was conducted on this TP-H1148 propellant. The baseline test matrix is found in Table 9 while Table 10 contains the accelerated aging test matrix. Table 11 outlines testing conducted on two mixes containing PBNA and one containing Agerite Stalite.

Data and/or plots from five different TP-H1148 propellant mixes are contained in this section of the report. The mixes are:

1. Mix A947006 - Experimental and developmental mix containing Agerite Stalite antioxidant. This was cast in March 1979.

TABLE 9

SRM PROPELLANT CHARACTERIZATION TEST MATRIX

Test	Crosshead Rate (in./min)	Test Temperature, °F -30	0	30	75	100	145	165	Number of Tests
Uniaxial Tensile	0.2	X	X	X	X	X	X	X	60
	2	X	X	X	X	X	X	X	
	20	X	X	X	X	X	X		
Pressurized Tensile (1000 psi)	0.2			X	X	X	X		36
	2			X	X	X	X		
	20			X	X	X	X		
LTCS % Elongation 5, 10, 15% (Hold 90 Days then test)	2				X	X			54
	20				X	X			
	200				X	X			
Biaxial	0.1	X	X	X	X	X	X		54
	1	X	X	X	X	X	X		
	10	X	X	X	X	X	X		
Biaxial Pressurized (1000 psi)	0.1			X	X	X	X		36
	1			X	X	X	X		
	10			X	X	X	X		
Stress Relaxation (to 10^4 second) Strain, % 2		X	X	X	X	X	X	X	21
10			X	X	X	X	X		15
Pressurized (1000 psi), % 2			X	X	X	X	X	X	18
10			X	X	X	X	X		15
Y_c	0.1	X	X	X	X	X	X		51
	1	X	X	X	X	X	X		
	10		X	X	X	X	X		
Y_c Pressurized (1000 psi)	0.1			X	X	X	X		36
	1			X	X	X	X		
	10			X	X	X	X		
Y_a	0.02		X	X	X	X	X		45
	0.2		X	X	X	X	X		
	2		X	X	X	X	X		
Y_a Pressurized (1000 psi)	0.02		X	X	X	X	X		45
	0.2		X	X	X	X	X		
	2		X	X	X	X	X		

TABLE 9 (Cont)

SRM PROPELLANT CHARACTERIZATION TEST MATRIX

Test	Test Temperature, °F 50	75	100	Number of Tests
Dynamic Shear* (5-600 Htz)	X	X	X	9
TCLE Range (-150 → +150°F)				3
Bulk Modulus		X		3
Poisson's Ratio (Ambient Pressure and Temperature only)		X		6
SECs (2-in. dia.) (3 bore diameter x 3 samples each to failure (135°F, 110°F, ambient, -5°F per day to failure) Bore Dia. - .252" Bore Dia. - .313" Bore Dia. - .376"				9
(1 bore diameter x 3 samples)(cycle to failure) (cycle between 100°F and 30°F) Bore Dia. - .376 in.				3

*Low frequency only.

TABLE 10

ACCELERATED AGING TESTS
PROPELLANT AGING TEMPERATURES - 75, 110, 135, and 150°F
TESTS AT END OF SIX MONTH AGING PERIOD

Test	Crosshead Rate (in/min)	Test Temperature, °F 30	75	145	165	Number of Tests
Uniaxial Tensile	0.02		X	X	X	45
	0.2	X	X	X	X	
	2.0	X	X*	X	X	
	20.0	X	X	X	X	
Biaxial	0.1	X	X	X	X	36
	1.0	X	X	X	X	
	10.0	X	X	X	X	

* Uniaxial tests at 75°F and 20 in./min crosshead rate were pulled at monthly intervals excluding the fifth month..

TABLE 11

SRM NEW AND OLD AO TP-H1148 TEST MATRIX

Test	Number of Specimens per Test Point	Crosshead Rate (in/min)	Temperature, °F 75	100	120	Number of Tests
Uniaxial Tensile	3	0.002	X	X	X	69
	5	0.02	X	X	X	
	5	0.2	X	X	X	
	5	2.0	X	X	X	
	5	20.0	X	X	X	
Plane Stress, Y_c	3	0.002	X	X	X	27
	3	0.02	X	X	X	
	3	0.2	X	X	X	
Stress Relaxation	5		X*	X	X	15
Creep	5		X	X	X	45
	Select three loads to fail at 20,100 and 1000 hrs.					
Strain Endurance	5		X	X	X	75
	Strain to failure, then test at lower strains to find strain level which will hold 28 days.					

*Repeat twice more after storing samples at 75, 100, and 120°F for 10 and 30 days.

2. Mix 9915006 - Standardization mix containing PBNA antioxidant. This mix was cast in January 1977 and tested shortly thereafter.
3. Mix 9915008 - Standardization mix containing PBNA antioxidant. This mix was cast in January 1977 and tested shortly thereafter.
4. Mix B160002 - Production mix containing PBNA antioxidant. This mix was cast in March 1980.
5. Mix A455020 - Standardization mix containing PBNA antioxidant. This mix was cast in January 1978.

In general, the mechanical properties of Mix A947006 correlate very well with other Space Shuttle mixes. The differences are slight and probably due somewhat to experimental error. However, the uniaxial data indicate that Mix A947006 has slightly higher strain capabilities at test temperatures ranging from 0° to 145°F while maximum stress values are correspondingly lower. The failure envelopes are also very similar with Mix A947006 showing a slight shift to the right and down at high strain.

The biaxial failure envelopes are even more closely correlated than the uniaxial data with Mix A947006 again being shifted somewhat to the right.

Accelerated aging of the propellant does have some effect on mechanical properties. Although there were not enough tests run on the aged propellant to plot a complete failure envelope, a comparison was made using the available data for both the uniaxial and biaxial tests. In both situations, the failure envelope shifted slightly to the left with increased aging temperature.

Mix A947006 was allowed to age for one year at ambient conditions and then tested again to confirm that the new antioxidant will have little effect on aging properties of the propellant. In addition, two mixes containing PBNA (B160002 and A455020) were tested simultaneously with mix A947006.

These data confirm that the mechanical properties of SRM propellant depend very little on which of the two antioxidants are used in the HB polymer.

Conclusions - All indications are, that from a mechanical properties point of view, TP-H1148 propellant with the new antioxidant Agerite Stalite in the HB polymer, should perform at least as well as previous mixes with PBNA antioxidant in the polymer.

Discussion - The curves generated for this report show very little data scatter. The data, therefore, should be considered valid and meaningful. However, extracting all of the meaningful information from the data is difficult. The data from the mix with the new antioxidant (A947006) compares very favorably with data from previous standardization mixes (9915006 and 9915008).

Figure 10 compares uniaxial data of the unaged propellant with the same propellant aged six months at four different temperatures. As expected, this propellant loses some of its strain capabilities at high aging temperatures. It should be pointed out, that this propellant, aged six months at 150°F, is comparable to propellant aged about eight and nine years at ambient temperature.[1] Figures 11 and 12 are similar comparisons using biaxial data. The results are almost identical.

Figure 13 demonstrates how uniaxial data obtained from propellant with the new antioxidant compares with the original standardization mixes. One can see that the strain capabilities are slightly higher in the new propellant while maximum stress values are correspondingly lower. In other words, the propellants are essentially identical, the major difference stemming from the difference in initial stress values. (Mix A947006 was 99 psi while mixes 9915006 and 9915008 were 117 and 118 psi, respectively.)

Figure 14 demonstrates the effects of ambient conditioning on TP-H1148 propellant. The propellant ages independently of the antioxidant. It hardens slightly with time. The creep data were added to this plot to demonstrate that the curves actually do turn back down at the lower end. No baseline creep testing was conducted on Mix A947006. Note that the creep data for all three mixes fall pretty well on top of each other, which indicates that the aging processes have even smaller effects on very slow rate testing.

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[1] Layton, L. H., Bennett, S. J., and Breitling, S. M., "Advanced Surveillance Technology for Service Life Analysis," Special Report, AFRPL-TR-77-51, Contract F04611-75-C-0031, September 1977.

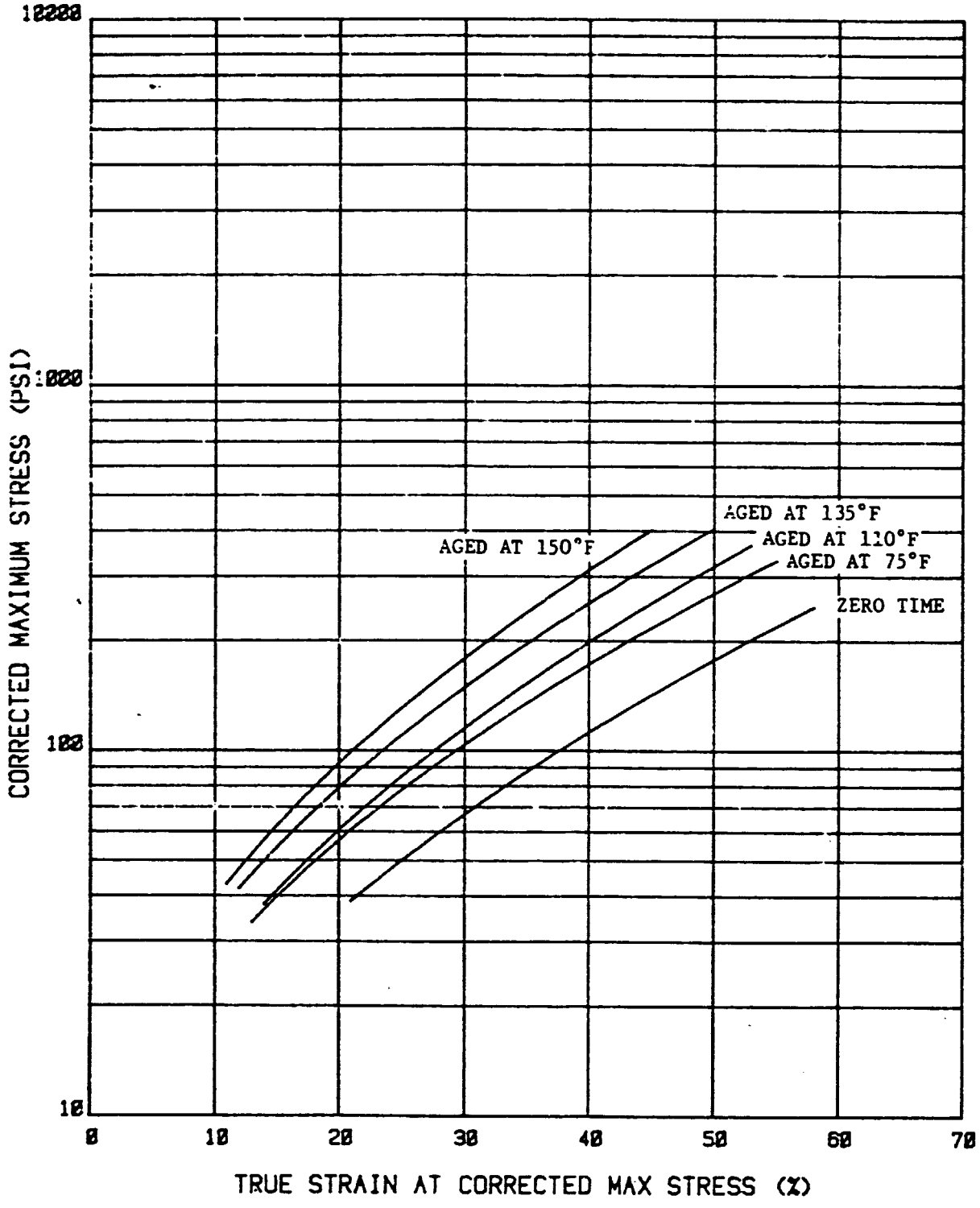

Figure 10. Uniaxial Failure Envelopes for TP-H1148 Propellant Mix A947006, Zero Time and Six-Month Aging

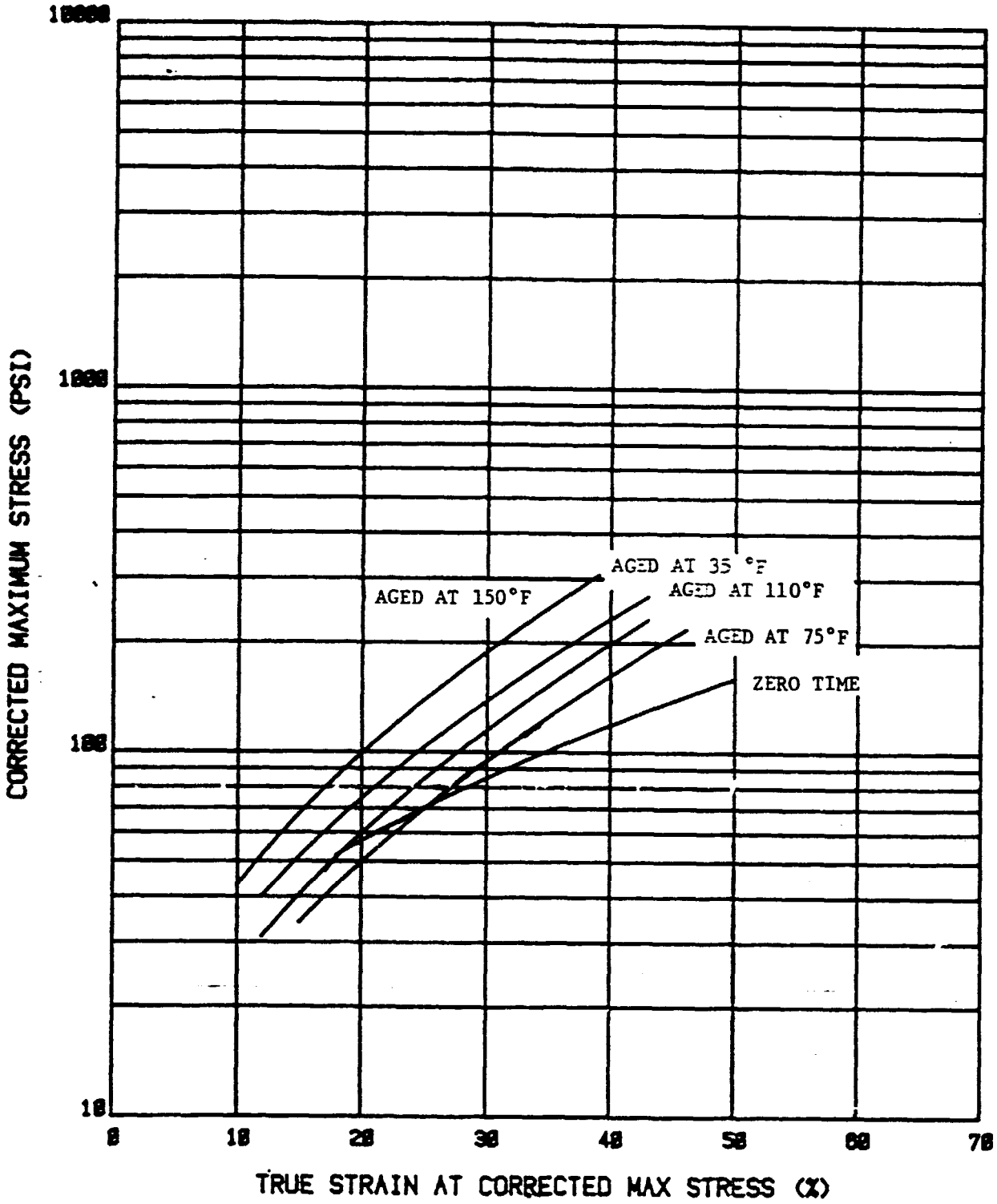

Figure 11. Biaxial Failure Envelopes for TP-E1145 Propellant Mix A947006, Zero Time and Six-Month Aging

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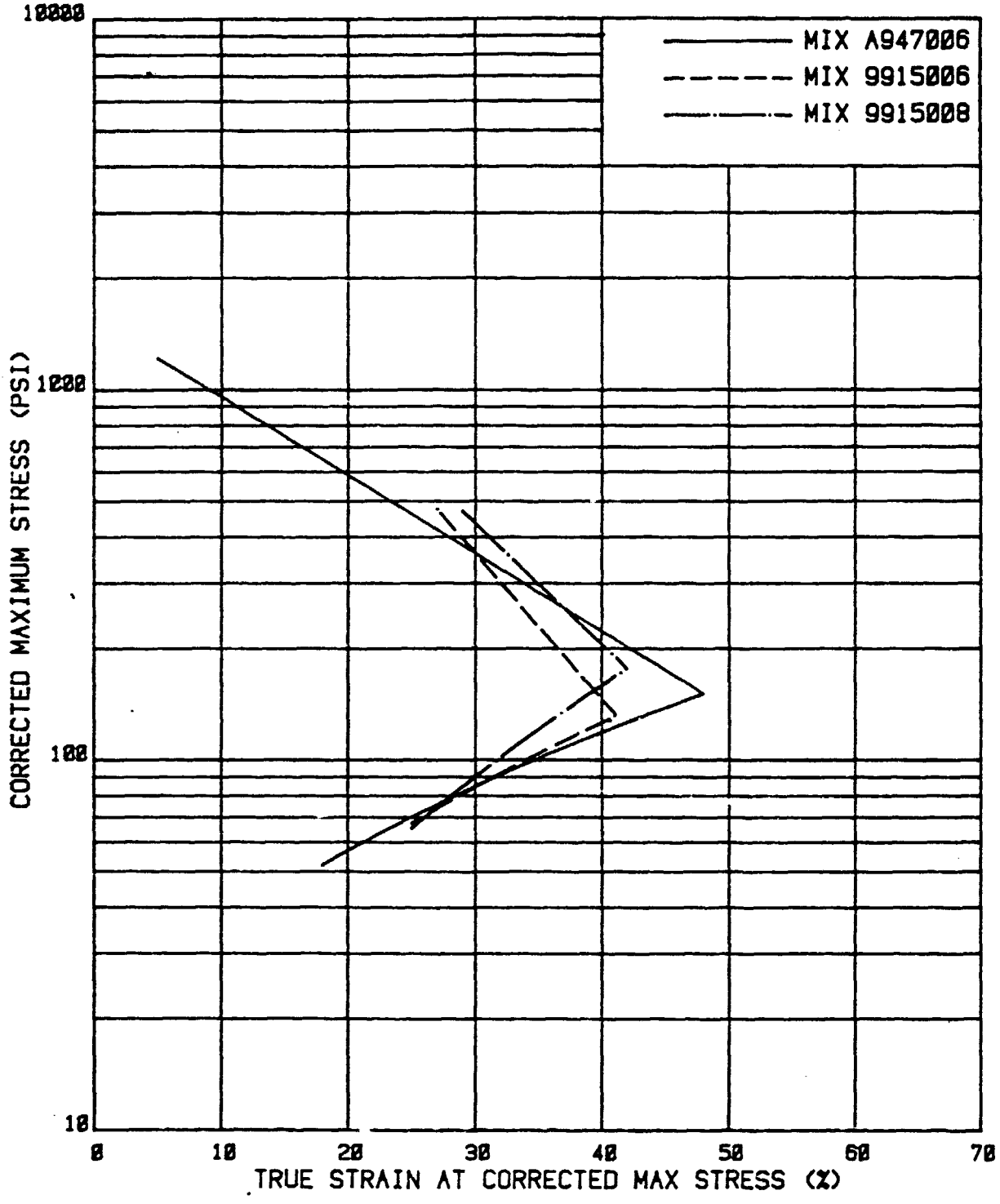

Figure 12. Comparison of TP-H1148 Propellant With New Antioxidant (Mix A947006) to TP-H1148 Propellant Standardization Mixes 9915006 and 9915008 Biaxial Failure Envelope

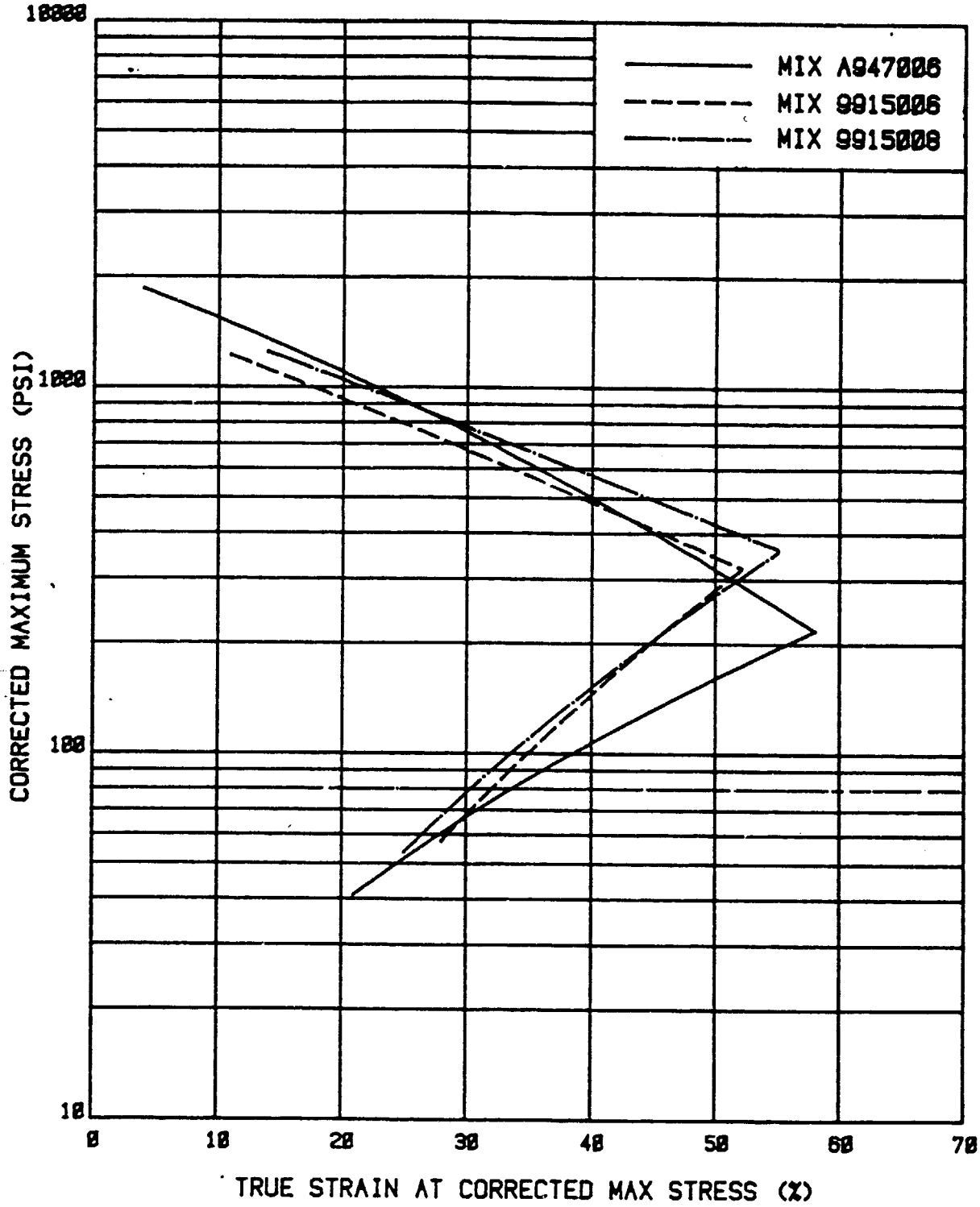

Figure 13. Comparison of TP-H1148 Propellant With New Antioxidant (Mix A947006) to TP-H1148 Propellant Standardization Mixes 9915006 and 9915008 Uniaxial Failure Envelope

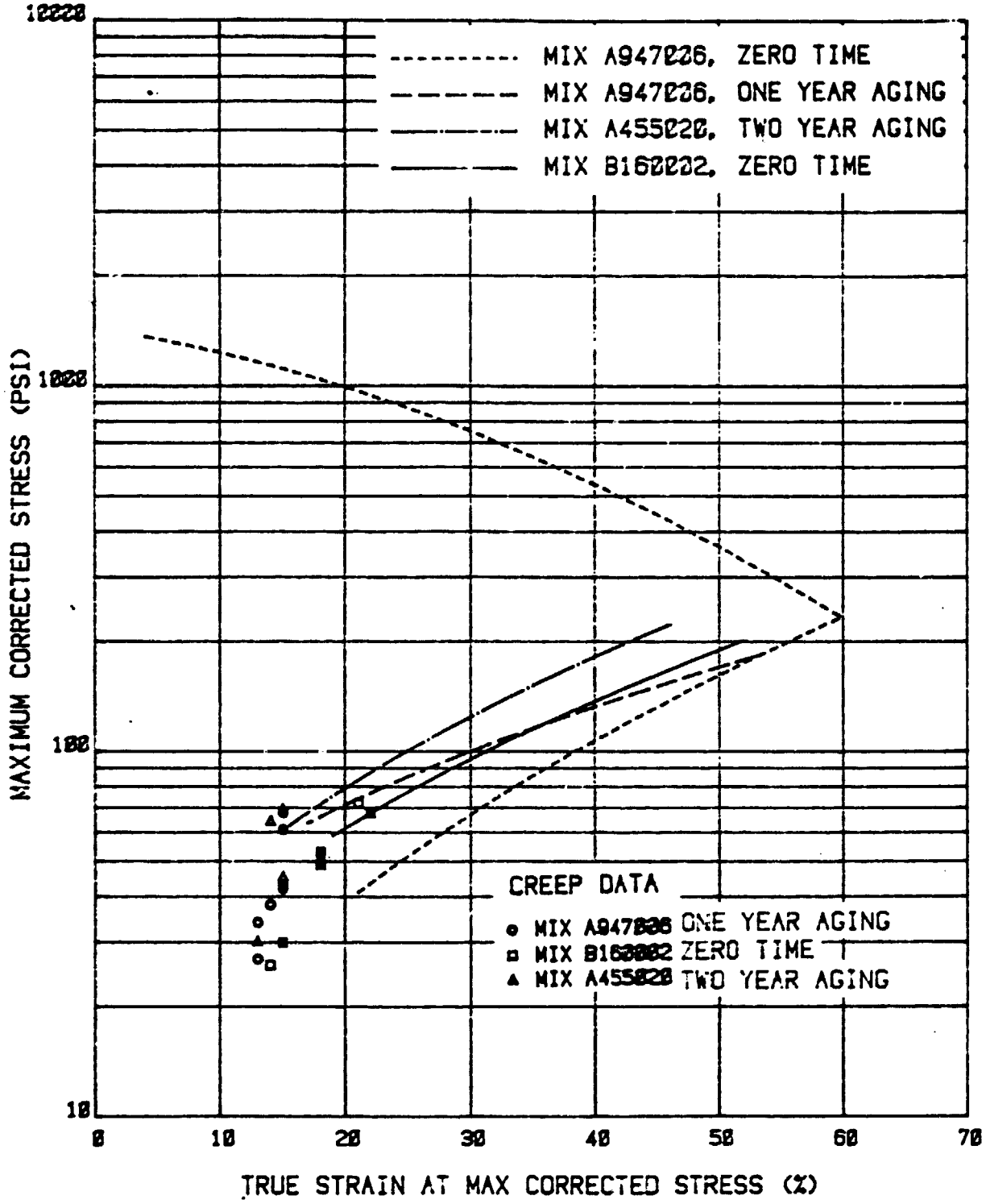

Figure 14. TP-H1148 Propellant Uniaxial Failure Envelopes
Mix Numbers and Propellant Ages Indicated

Figures 15 and 16 are failure envelopes generated from 1,000 psi pressure data. No corresponding plots from the other mixes are available.

Figure 17 is a comparison of the relaxation modulus curves of three mixes. The relaxation modulus of Mix A947006 (new AO) has increased over the one year aging period, but no more than would normally be expected. Even smaller changes in modulus can be expected from here on because the rate of aging slows down with time. Figure 18 is a plot of cohesive fracture energy and demonstrates the same aging trends as Figure 17. Figure 19 is a relaxation curve generated from 1,000 psi pressure data.

Only the most pertinent figures are included in this report. All data tables and corresponding plots are contained in TWR-12559, Revision A.

7.0 LINER DATA AND ANALYSIS

7.1 INITIAL NEW AO LINER TESTS

The first series of liner (UF-2137) tests were run using PBNA, Agerite Geltrol, and AO2246 antioxidants (one in each of three samples) in the HC polymer and using PBNA-HB in TP-H1148 propellant. Three 5-lb batches of UF-2137 (Table 12) were made and the following tests were conducted:

1. End-of-mix viscosity - Brookfield HBT, TA spindle at 5 rpm.
2. Viscosity vs time - liner placed in 135°F, Brookfield HBT, TD spindle at 5 rpm.
3. Penetrometer - liner cured 24 and 32 hr at 135°F, 0.392 in. dia ft, 100 g, 60 sec readings.
4. Hardness (Shore A) versus cure time at 135°F.
5. Adhesion to steel - cured 168 hr at 135°F and pulled at 0.5 in./min.
6. TP-H1148 bond strength to liner - UF-2137 lined (65 mils nominal) adhesion plates and broadcloth was cured 3 hr at ambient plus 68 hr at 135°F for one set and 168 hr at 135°F for a second set. TP-H1148 (9970089) was cast and cured 96 hr at 135°F.

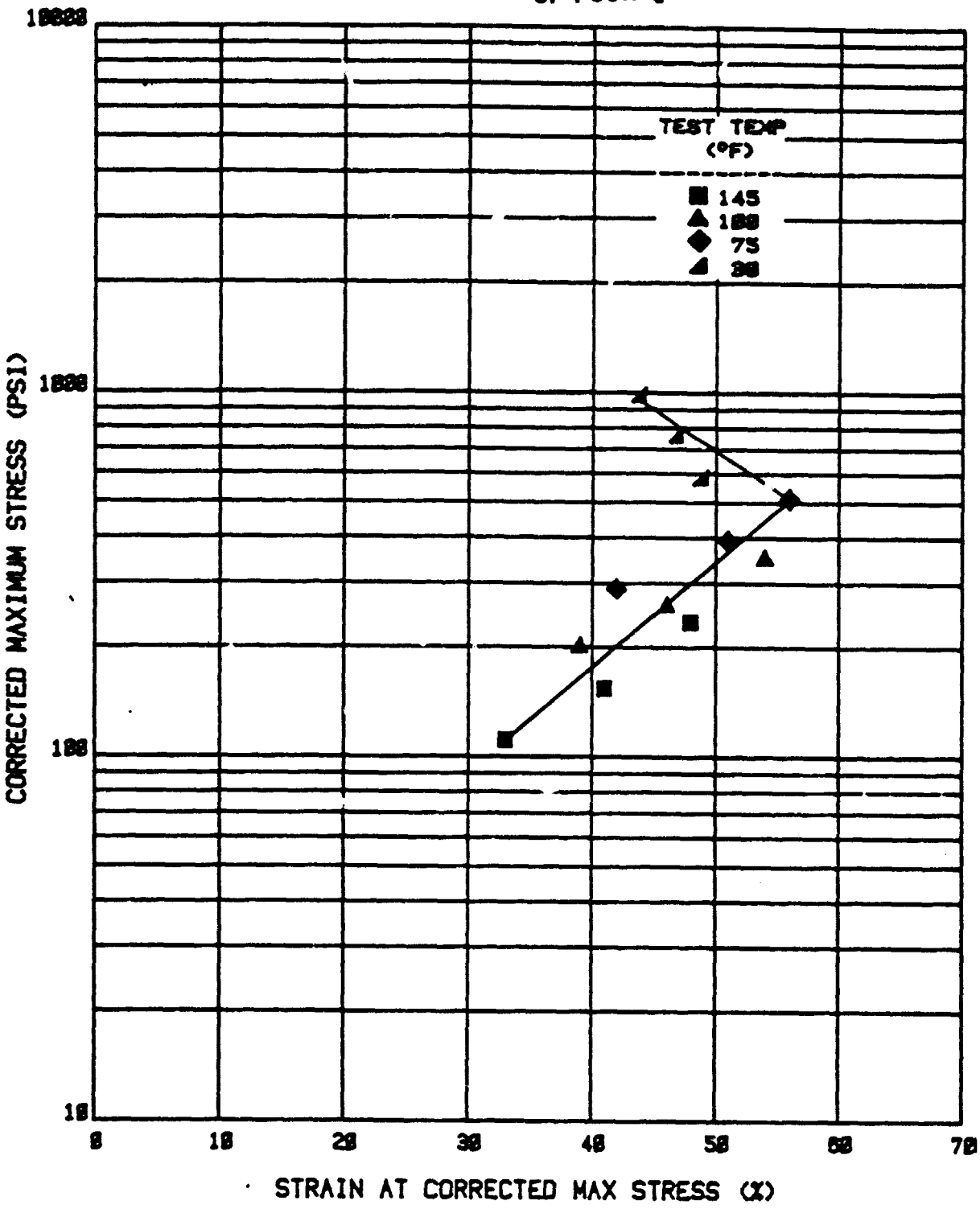

Figure 15. Uniaxial Failure Envelope for TP-H1148 Propellant Mix A947006, Zero Time, 1,000 psi

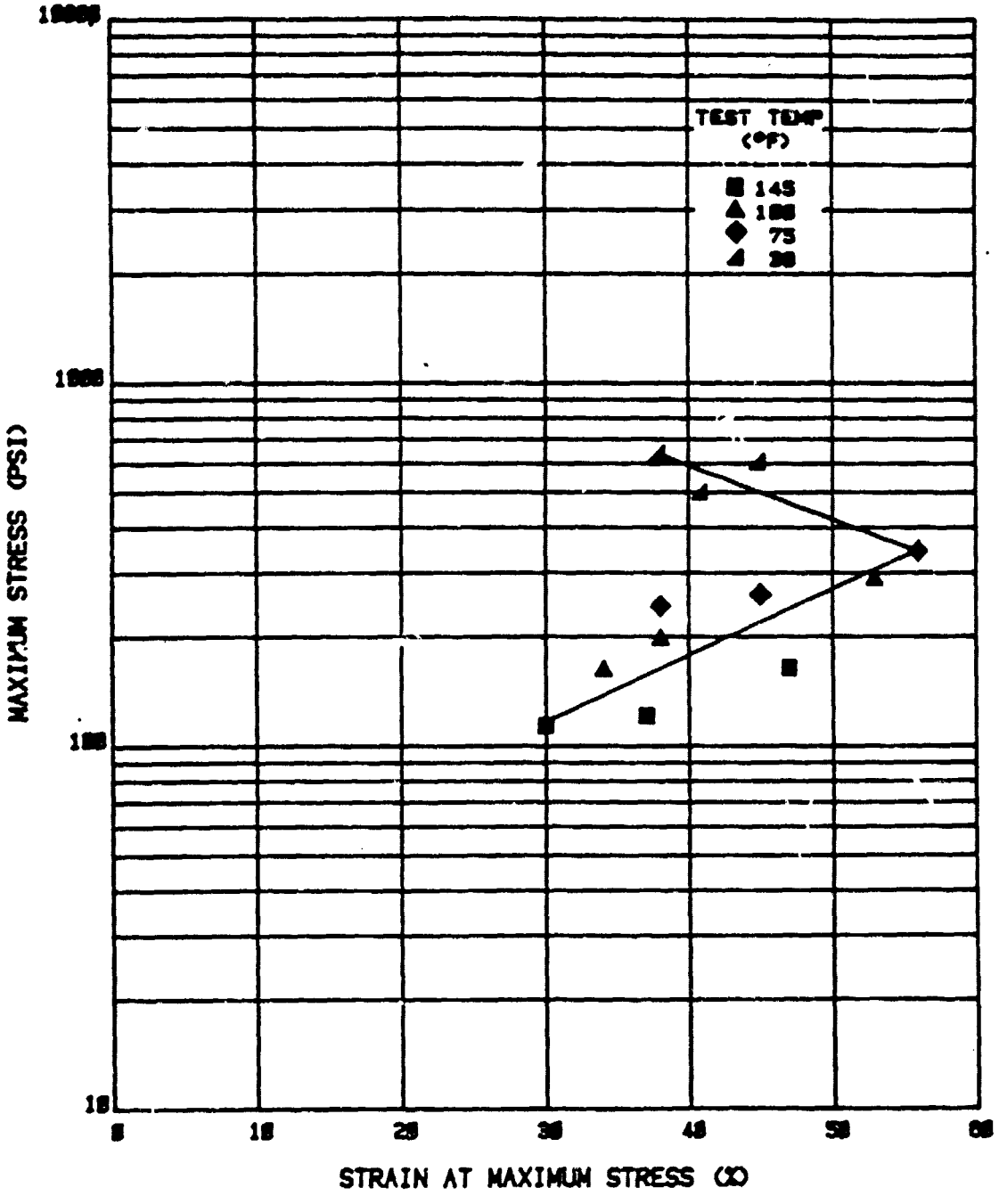

Figure 16. Biaxial Failure Envelope for TP-H1148 Propellant, Mix A947006 Zero Time, 1,000 psi

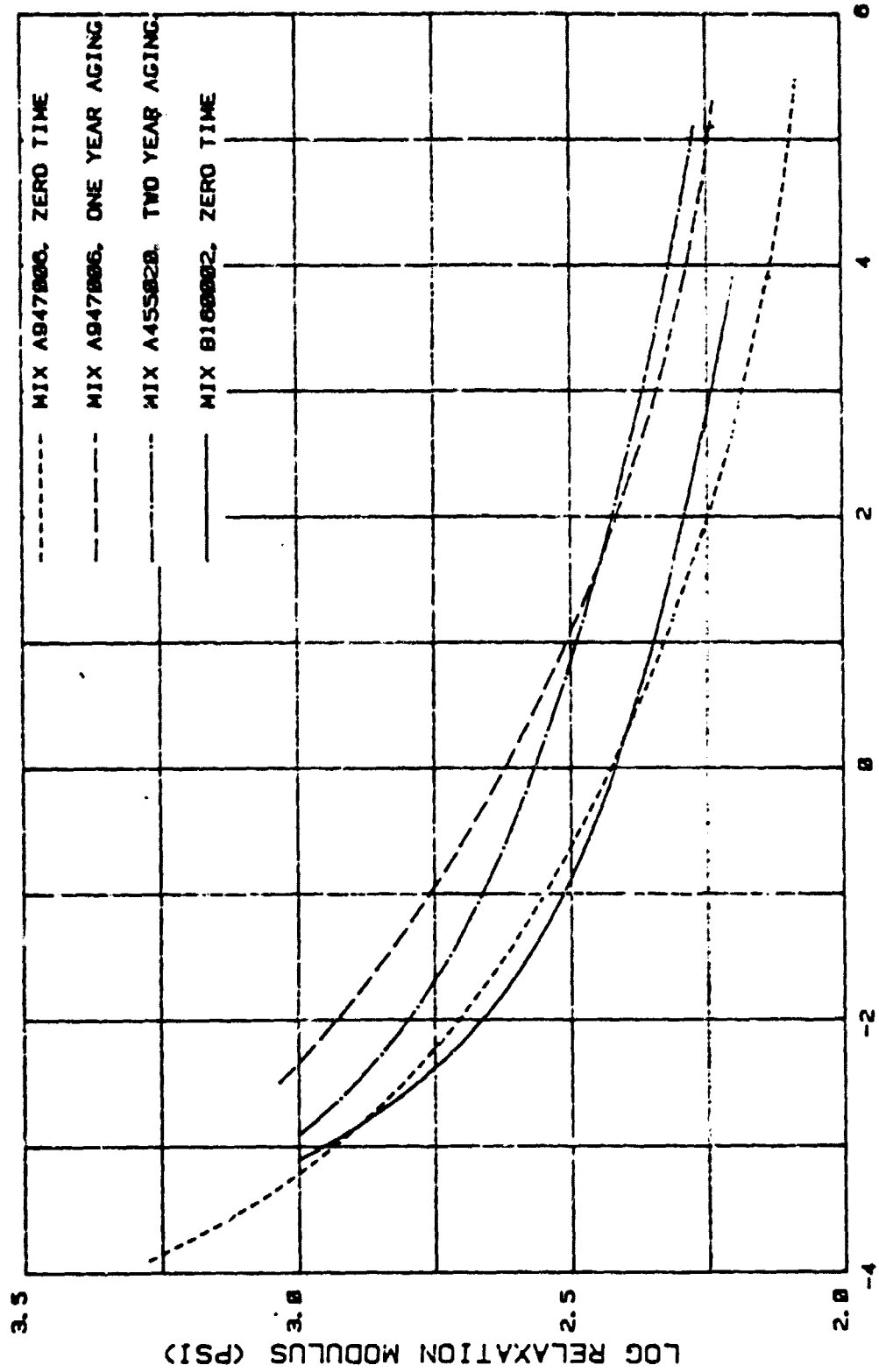

Figure 17. TP-H1148 Propellant Stre[illegible]ation Master Curves
Mix Numbers and Propellant [illegible] Indicated

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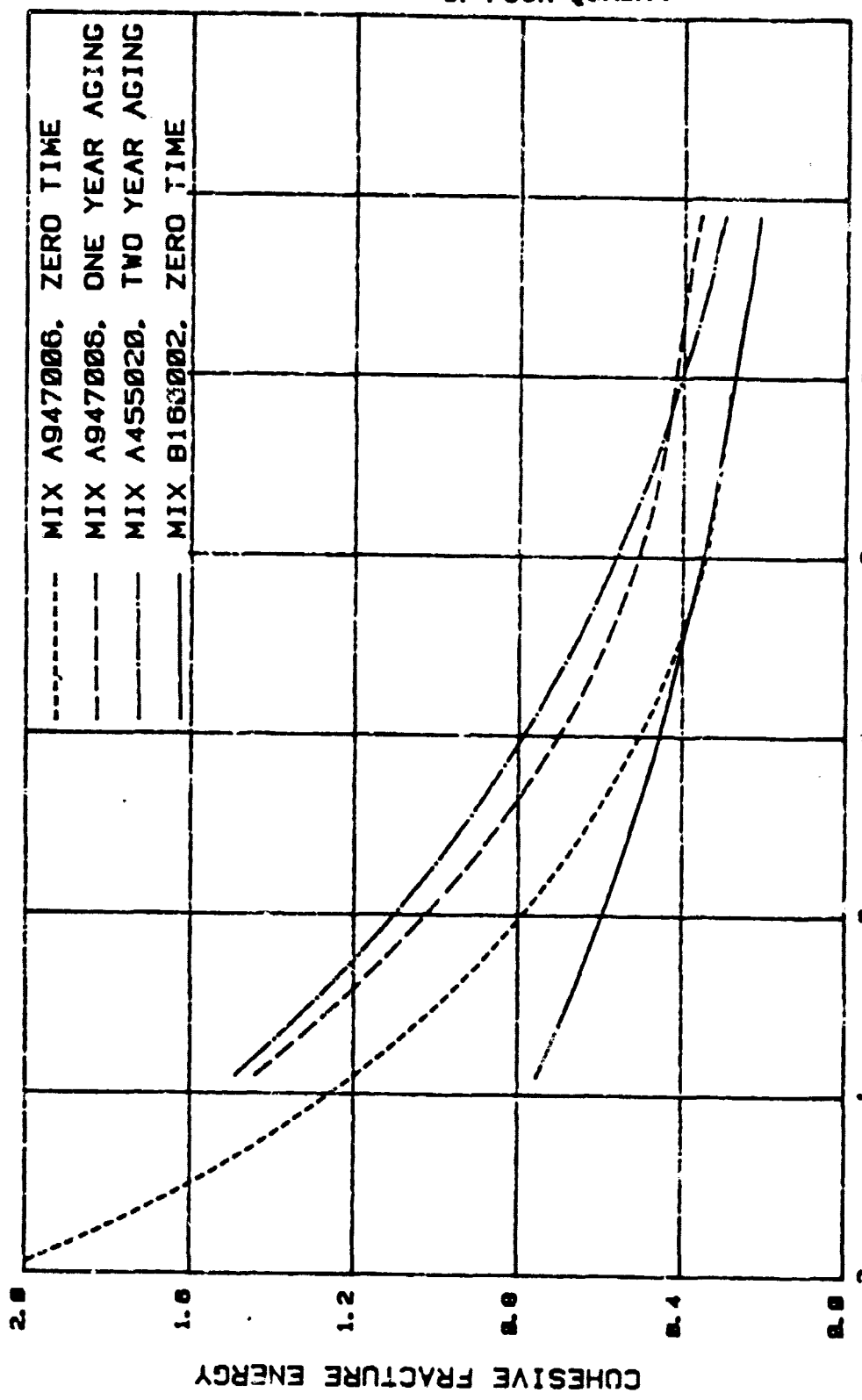


Figure 18. TP-H1148 Propellant Cohesive Fracture Energy Master Curves Mix Numbers and Propellant Ages Indicated

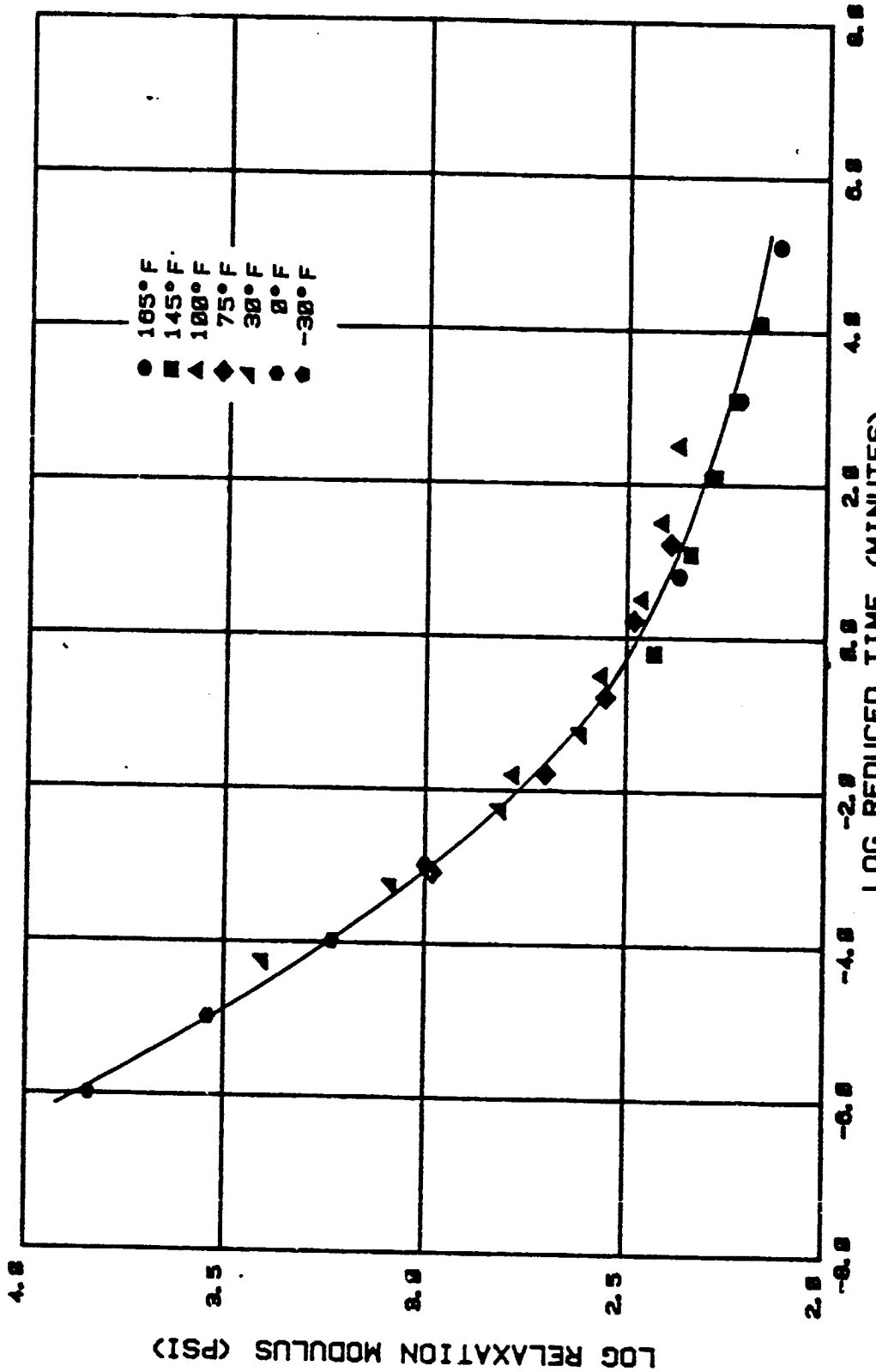


Figure 19. Master Stress Relaxation Curve for TP-H1148 Propellant, Mix A947006 Zero Time at 2% Strain and 1,000 psi

TABLE 12

UF-2137 MIXES

Raw Material	Stock No.	Mix 1 PBNA - Control	Mix 2 Agerite Geltrol	Mix 3 Cyanox 2246
HC Polymer	9999	1618-2	1618-4	1618-3
ERL-0510	9297	0002	→	→
MAPO	7319	0002	→	→
Thixcin E	7317	0002	→	→
Iron Octoate	7318	0002	→	→
Asbestos Floats	7320	0002	→	→
Liner Mix No.		185349	185350	185351
End of Mix				
Viscosity (Poise)		528	554	632
Temp (^{o}F)		152	152	150

Adhesion samples were pulled at 0.5 in./min and 180° peel samples at 12.0 in./min.

Results of these tests were as follows:

1. Viscosity followed the same general trend for all three mixes except the initial part of the control mix (Figure 20).
2. Penetrometer readings were taken at two intervals as follows:

Cure Time (hr at 135°F)	Penetrometer (MM)		
	Control-PBNA	Agerite Geltrol	Cyanox 2246
24	5.5	2.7	3.1
32	0.4	0.1	0.4

3. Shore A hardness (Figure 21) shows the control mix curing slightly slower than the two new mixes. There was only a slight difference between the two new mixes.
4. UF-2137 adhesion to steel and TP-H1148 propellant adhesion to liner were within the same range for all three mixes (Table 13). Peel strength was lower for both the two new antioxidant mixes (7.2 and 6.2 for the control vs 4.5 to 5.8 lb/in.).

This was the first indication that the new antioxidant in PC polymer could cause a problem, but because this was only one test, there was cause for running additional cure rate studies and liner/propellant bond tests.

7.2 INITIAL NEW HB PROPELLANT, OLD LINER TESTS

The next series of tests was conducted to compare TP-H1148 propellant made with three separate polymers - Agerite Stalite-HB, Vanox 13-HB, and PBNA-HB - to UF-2137 liner made with PBNA-HC polymer.

1. Adhesion plates and broadcloth were lined with UF-2137 (nominal 65 mils thick - Mix 180131). The liner was cured 67 hr at 135°F.
2. Propellant from three mixes (Table 14) was cast on the samples and cured 96 hr at 135°F.

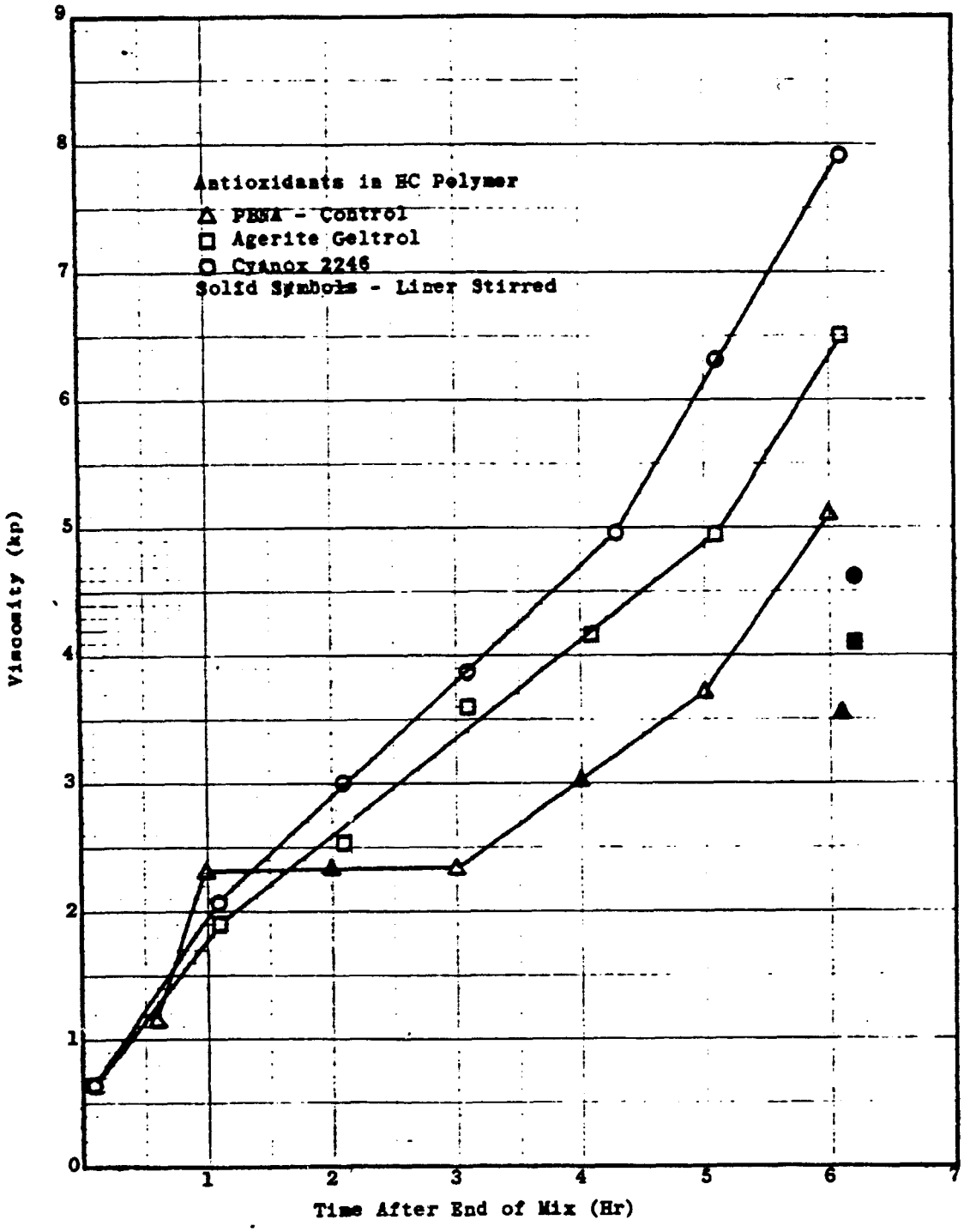


Figure 20. UF-2137 Liner Viscosity - Liner Made With HC Polymer Containing Various Antioxidants

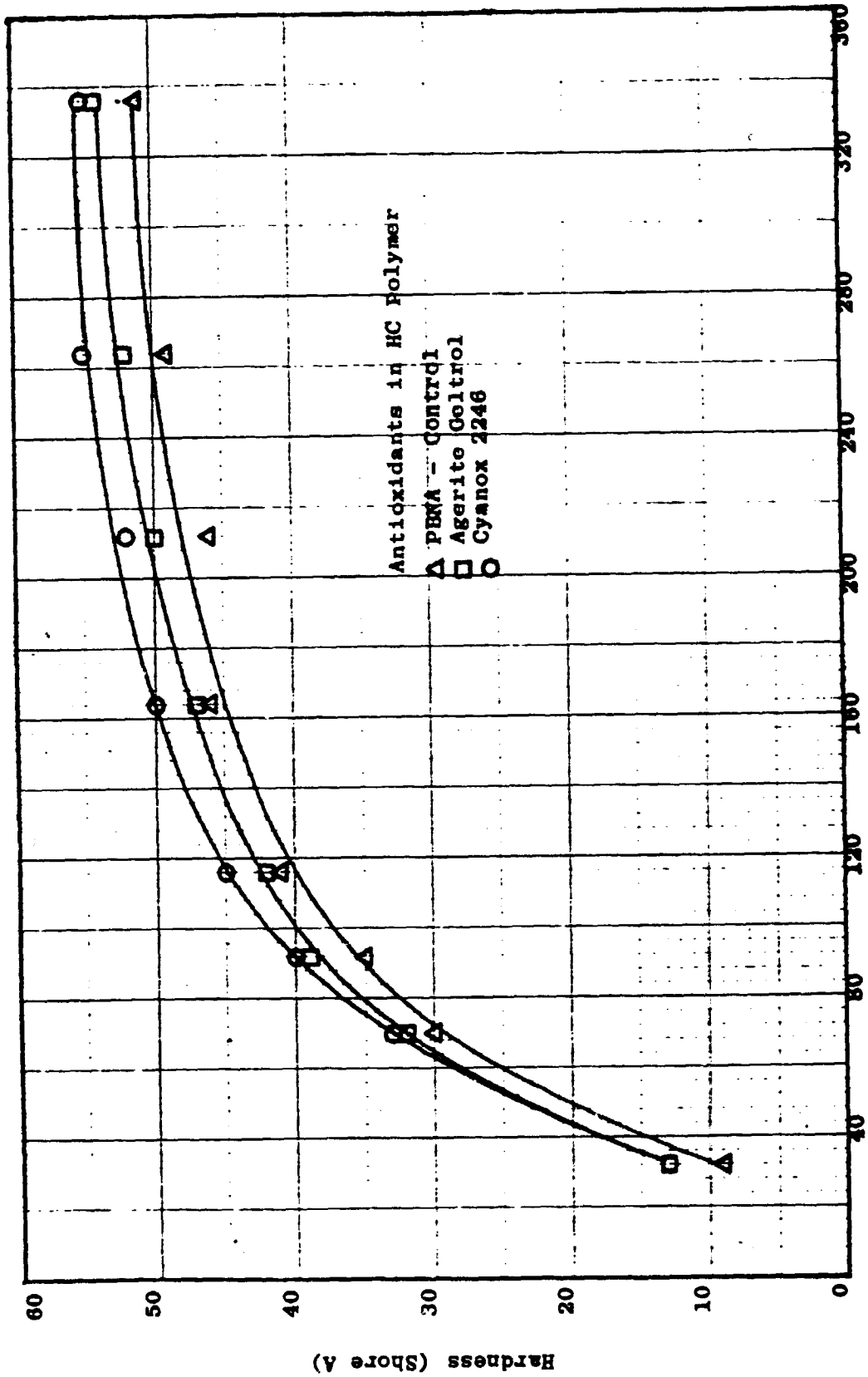


Figure 21. UF-2137 Liner Hardness - Liner Made With HC Polymer Containing Various Antioxidants

TABLE 13

Bond Tests

Mix No.	Antioxidant	UF-2137 to Steel (psi)	Adhesion Failure (% prop)	TP-H1148 to UF-21377 Adhesion (psi) Cure 1*	Adhesion (psi) Cure 2*	180° Peel (lb/in.) Cure 1	180° Peel (lb/in.) Cure 2
1	Control - PBNA	214	100	120	120	7.5	6.2
		200	100	130	118	7.5	6.5
		210	20	113	118	6.7	6.0
		210	100	---	---	---	---
		215	60	---	---	---	---
		200	100	---	---	---	---
		208		121	119	7.2	6.2
2	Agerite Geltrol	205	100	130	121	6.0	5.0
		220	100	122	126	5.8	5.0
		210	100	123	113	5.5	5.0
		206	100	---	---	---	---
		216	100	---	---	---	---
		227	100	---	---	---	---
		214		125	120	5.8	5.0
3	Cyanox 2246	209	100	123	125	5.8	4.5
		209	0	121**	118	6.0	4.5
		215	5	123	124	5.5	4.5
		205	0	---	---	---	---
		210	10	---	---	---	---
		212	100	---	---	---	---
		210		122	122	5.8	4.5

* Cure 1 = 68 hr cure at 135°F
Cure 2 = 168 hr cure at 135°F

** 20% propellant film, all other adhesion samples 100% propellant failure.

Peel samples all had a propellant film on the liner.

TABLE 14

TP-H1148 Peel Strength to UF-2137 Liner

Aging (mo @ °F)	Peel Strength (lb/in.) PBNA-Control 209-5-224	Agerite Stalite 209-5-223	Vanox 13 209-5-220
0	5.0	6.0	5.8
	5.6	5.7	6.0
	6.1	6.0	6.4
	5.6	5.9	6.1
2 @ 150	5.0	4.6	5.1
	4.9	4.6	4.7
	4.8	4.5	4.8
	4.9	4.6	4.9
4 @ 150	5.0	5.4	5.2
	5.0	5.0	5.4
	5.4	5.2	5.3
	5.1	5.2	5.3
6 @ 150	5.4	5.1	5.8
	5.2	5.1	5.4
	5.3	5.2	5.4
	5.3	5.1	5.5
6 @ 75	5.3	5.0	5.8
	5.4	4.7	5.7
	5.2	5.1	5.7
	5.3	4.9	5.7

All samples had a propellant film on the liner

3. Adhesion samples were pulled at 0.5 in./min and 180° peel samples at 12.0 in./min.
4. Additional samples were packaged and were aged two, four and six months at 150°F and six months at 75°F.

Results of these tests were as follows:

1. Peel strength of the Agerite Stalite propellant mix was slightly lower than the control and the Vanox 13 was slightly higher after six months aging as shown in Table 15.
2. Adhesion of the Agerite Stalite propellant mix was slightly lower for the six months aging at 150°F and higher for the six months at 75°F, while the Vanox 13 was higher compared to the control.

Actually, all peel values ranged from 4.9 to 6.1, none of which are significantly different with this number of samples.

7.3 FURTHER TESTS WITH A02246 HC

Because there was some indication that new antioxidants resulted in lower liner propellant peel values and caused an increased liner cure rate, both of which would be detrimental in a motor under aging conditions, further tests with PBNA and A02246 HC liner, and PBNA and Agerite Stalite-HB in propellant were run.

The samples using the "old" polymers in the liner and propellant have higher peel strength than when using "new" polymers (7.8 and 8.0 lb/in. versus 5.5 lb/in., Table 16). When using one "old" and one "new" polymer the peel values are intermediate (6.4 to 7.0 lb/in.).

Liner made with HC polymer containing the new antioxidant cures slightly faster as shown by viscosity (Figure 22) and Shore A hardness (Figure 23). Liner precured 20, 24, 28, and 32 hr at 135°F and then placed under vacuum for a total of 168 hr at 135°F also showed the cure difference when made into peel samples.

TABLE 15

TP-H1148 Adhesion to UF-2137 Liner

Aging (mo @ °F)	Adhesion (psi) and Failure (% Prop) PBNA-Control 209-5-224		Agerite Stalite 209-5-223		Vanox 13 209-5-220	
0	116	100	126	90	132	100
	116	100	125	40	136	100
	116	100	137	90	130	90
	116		129		133	
2 @ 150	147	10	153	20	167	20
	144	0	160	30	151	100
	141	50	154	0	155	100
	144		156		158	
4 @ 150	---		160	0	158	0
	154	0	167	0	156	60
	162	0	163	10	151	0
	158		163		155	
6 @ 150	178	0	173	0	172	0
	175	0	168	0	188	0
	170	0	158	0	187	0
	174		166		182	
6 @ 75	140	0	143	0	158	0
	151	0	148	0	157	80
	143	0	152	20	---	
	145		148		158	

TABLE 16

TP-H1148 TO UF-2137 BOND STRENGTH

UF-2137 Liner	TP-H1148 Propellant	Adhesion (psi) Cure 1**	Adhesion (psi) Cure 2**	180° Peel (lb/in) Cure 1	180° Peel (lb/in) Cure 2
Old*	Old*	110	111	8.0	7.9
		104	114	7.9	7.9
		112	111	8.0	7.6
		109	112	8.0	7.8
Old	New	99	98	7.4	6.9
		96	97	6.9	6.6
		102	98	6.8	6.4
		99	98	7.0	6.6
New	Old	110	111	6.3	6.1
		112	115	6.2	6.6
		111	113	6.5	6.5
		111	113	6.3	6.4
New	New	99	99	5.5	5.4
		98	97	5.5	5.5
		98	98	5.5	5.7
		98	98	5.5	5.5

All adhesion samples were propellant failure.
All peel samples were propellant film on the liner.
* Old is PBNA antioxidant in the HB and HC polymer.
New is Agerite Stalite in the HB and Cyanox 2246 in the HC polymer.
** Cure 1 = Liner precured 68 hours at 135°F prior to casting.
Cure 2 = Liner precured 168 hours at 135°F prior to casting.

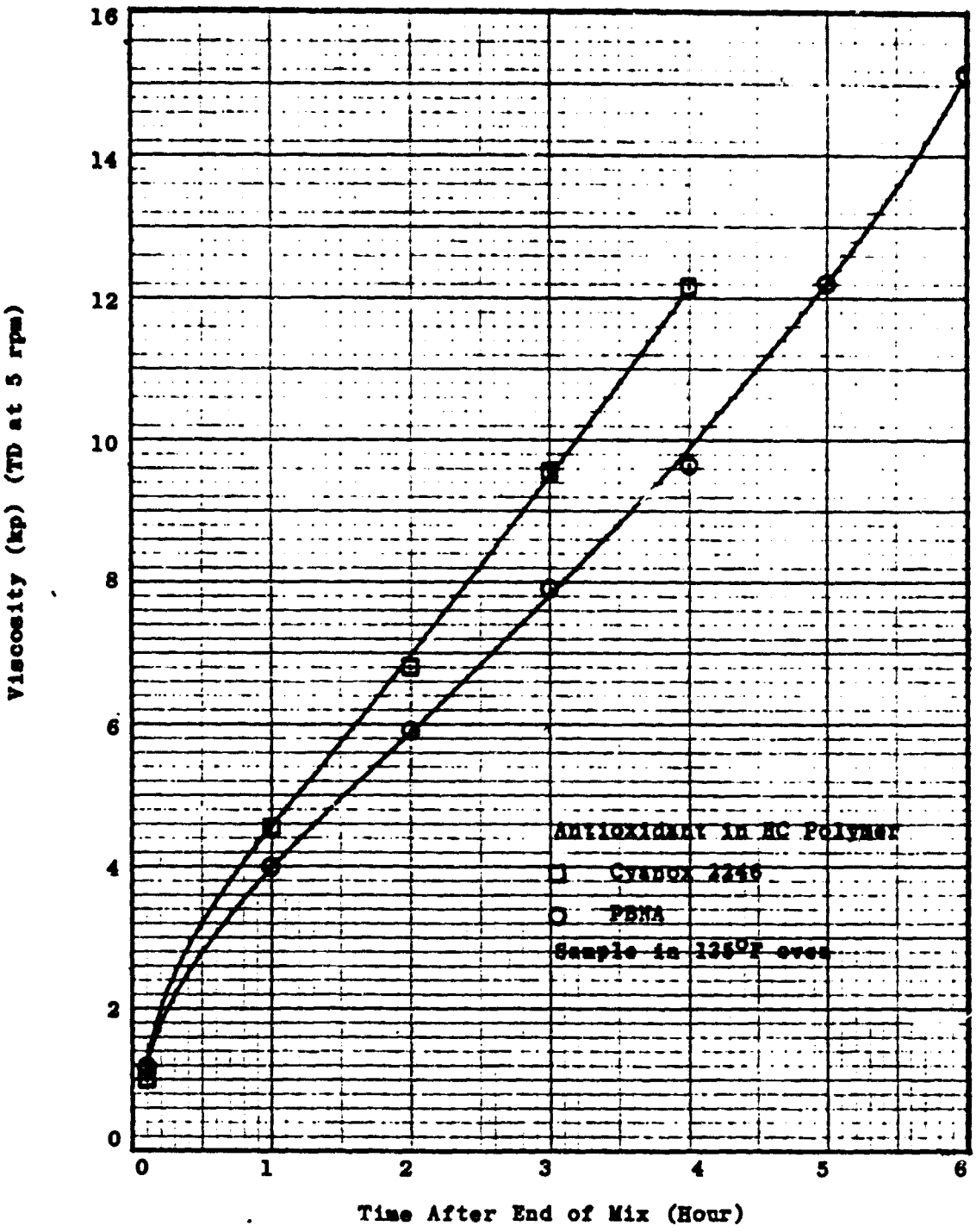


Figure 22. UF-2137 Liner Viscosity - Liner Made With HC Polymer Containing PBNA and Cyanox 2246 Antioxidants

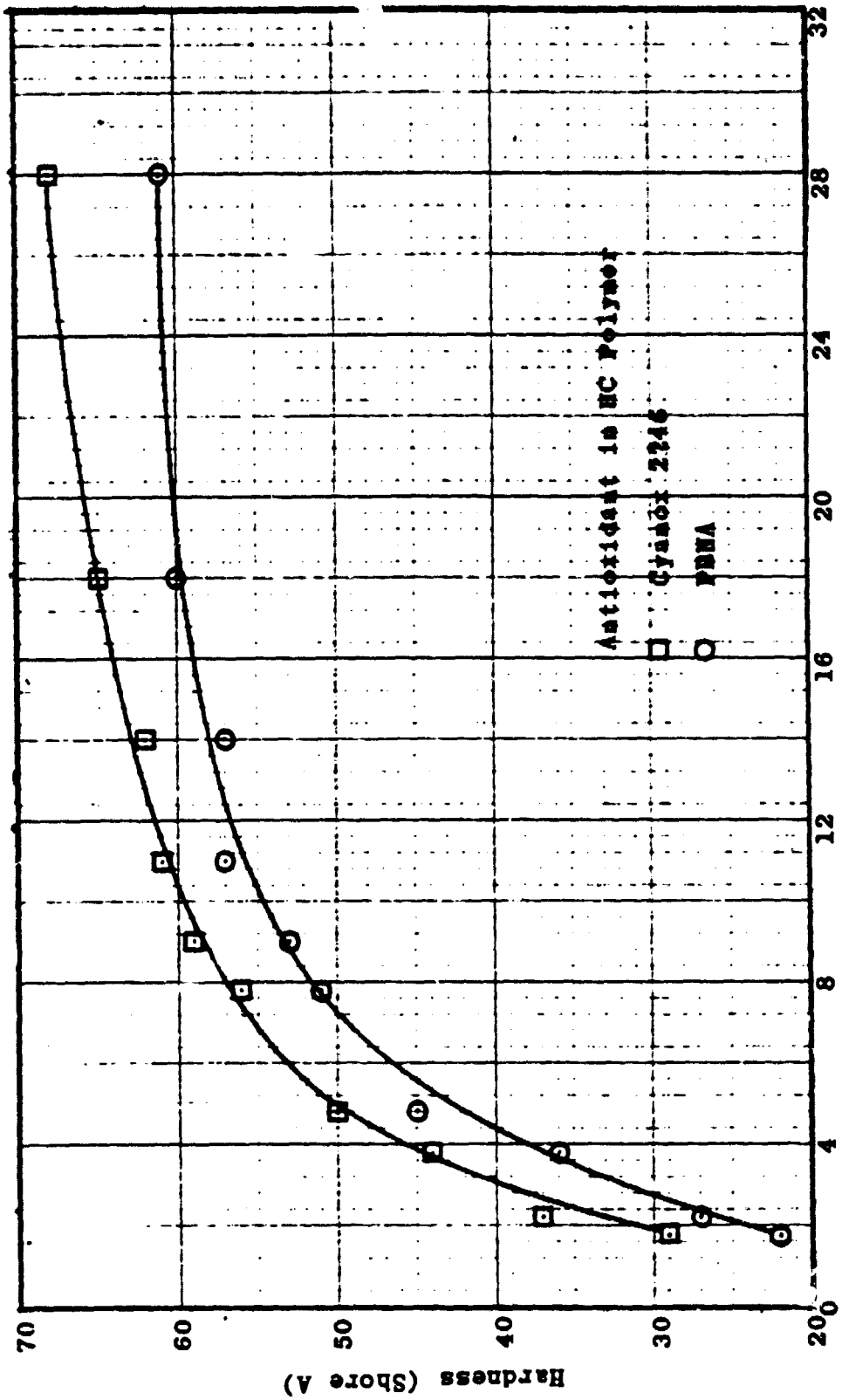


Figure 23. UF-2137 Liner Hardness - Liner Made With HC Polymer Containing PBNA and Cyanox 2246 Antioxidants

Liner Precure (hr)	180° Peel (lb/in.) Old	New
20	4.0	4.0
24	5.0	10.0
28	14.0	18.5
32	20.0	18.6

The low peel strength results from a small layer of foam forming in the liner at the liner-rubber interface. As the liner is cured at longer intervals prior to being subjected to vacuum, the amount of "foam" or small bubbles decreases, thereby adding strength to the liner rubber bond. This data would indicate the "new" liner is about four hours faster curing.

Liner made with HC polymer that did not contain any antioxidant resulted in slightly lower peel strength than when it contained one and two percent Cyanox 2246 (Table 17).

Liner made with no antioxidant in the HC polymer cured slightly slower than the one or two percent antioxidant mixes as shown by viscosity (Figure 24). Shore A hardness did not show any basic difference in the three mixes (Figure 25).

7.4 LINER TESTS WITH THE NEW FULL LOT OF HC WITH AO2246

The blended lot of HC polymer with AO2246 (Paragraph 5.1) was used to check the low peel and fast cure observed in the previous liner and liner-propellant tests.

1. The liner was vacuum mixed the last 10 min of the regular mixing cycle. Adhesion discs and liner molds were cured 168 hr at 135°F. TP-H1148 (Mix B547006) was cured 96 hr at 135°F.
2. Adhesion samples were tested at 0.5 in./min, dog-bones at 2.0 in./min and 180° peel at 12.0 in./min.
3. UF-2137 was formulated using carboxyl, equivalent of 0.055 eq/100 g for the control lot of HC polymer (7220-0008). The new HC polymer (6296-0007) was tested at Thiokol/Wasatch Division as 0.056 eq/100 g (this was used in the liner

TABLE 17

BOND STRENGTH OF UF-2137 LINER CONTAINING HC POLYMER WITH AND WITHOUT ANTIOXIDANTS

Cyanox 2246 Antioxidant in HC	Adhesion (psi) Cure 1	Adhesion (psi) Cure 2	180° Peel (lb/inch) Cure 1	180° Peel (lb/inch) Cure 2
None	102	100	7.2	4.8
	-	98	-	4.8
	107	100	7.3	5.2
	105	99	7.2	4.9
1%	108	102	7.6	5.6
	101	98	7.6	5.7
	102	99	7.7	5.8
	104	100	7.6	5.7
2%	109	105	7.6	5.6
	101	100	7.6	5.9
	107	105	7.7	6.2
	106	103	7.6	5.9

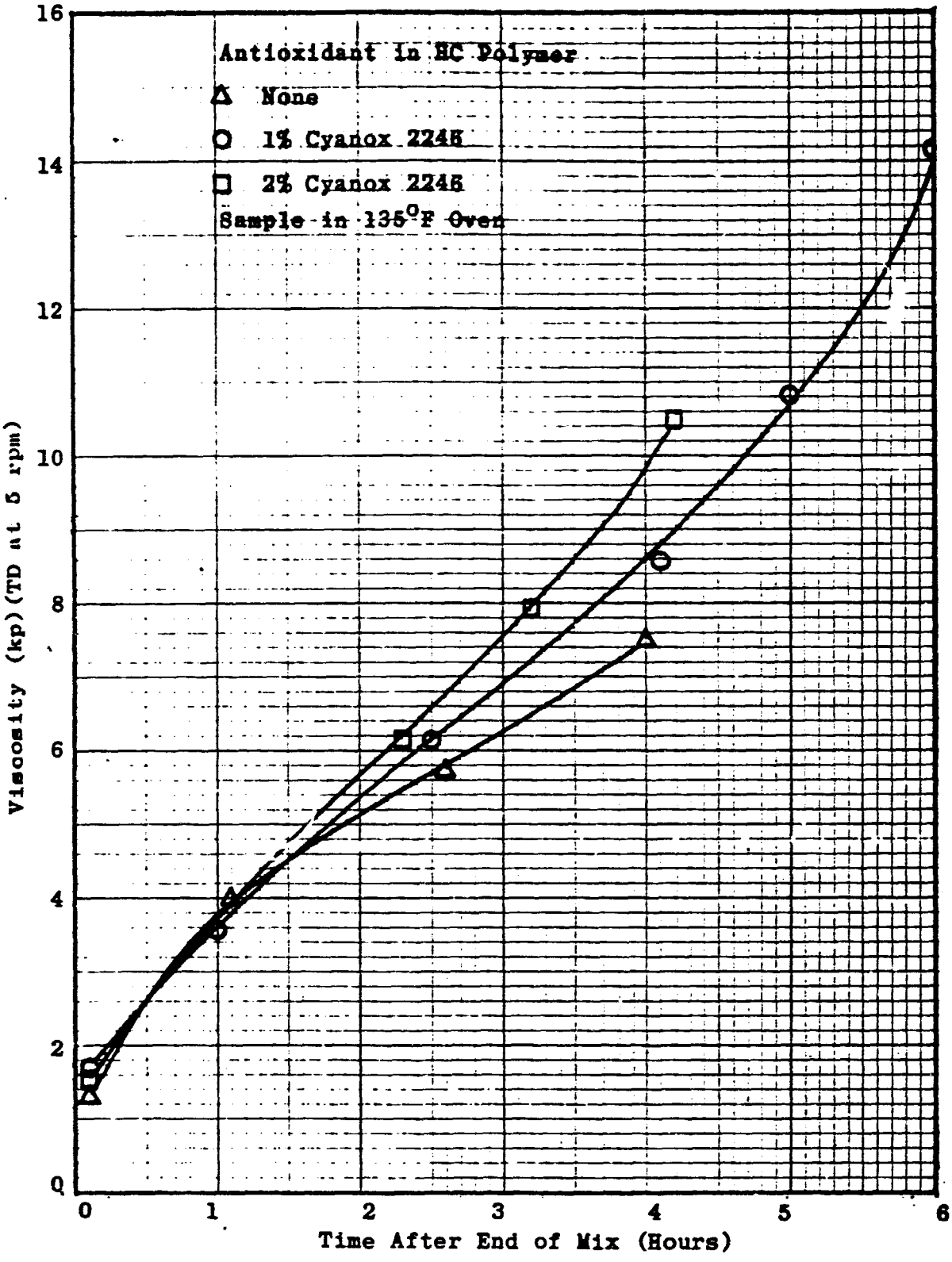


Figure 24. UF-2137 Liner Viscosity - Liner Made With HC Polymer With and Without Antioxidants

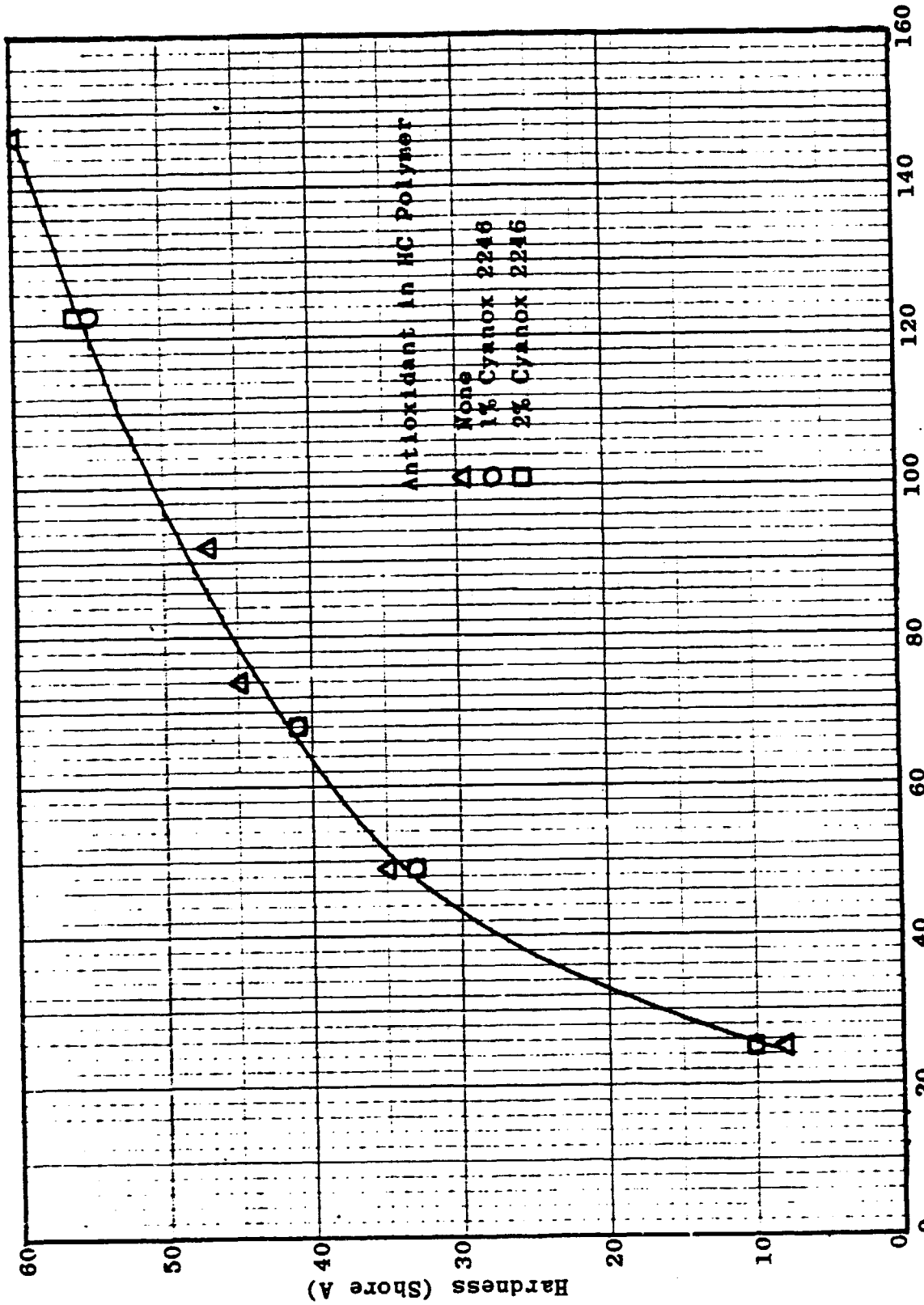


Figure 25. UF-2137 Liner Hardness - Liner Made With HC Polymer With and Without Antioxidants

formulation) but the analysis from Thiokol/Chemical Division was 0.0527 eq/100 g.

UF-2137 (Table 18) was mixed and tested. Viscosity (Table 19) hardness, and penetrometer all show the liner using the AO2246 curing faster than the control mix using PBNA. Liner properties (Table 20) show the AO2246 results in a higher stress and adhesion to steel and lower strain than the control mix.

TP-E1148 peel strength was lower (Table 21) for the AO2246 liner mix.

7.5 REACTION OF PBNA AND AO2246 WITH MAPO AND ERL-510

As a result of the previous liner tests, showing the lower peel values and faster curing rates with AO2246 antioxidant, it was postulated that the PBNA interferes with the epoxy-carboxyl cure reaction.

Mixtures were prepared of PBNA and AO2246 with MAPO and ERL-510 as follows:

Mix No. 1	2.8%	PBNA	97.2%	MAPO
Mix No. 2	2.8%	AO2246	97.2%	MAPO
Mix No. 3	5.6%	PBNA	94.4%	ERL-510
Mix No. 4	5.6%	AO2246	94.4%	ERL-510

Each mix was placed in 135°F oven and the FTIR spectra were obtained at different time intervals on samples removed from each mix.

In the case of MAPO, the aziridine ring deformation peak at 865 wave-numbers was chosen to follow the reaction. The absorbance values were measured and then normalized to the CH_2 absorption at 990 wave-numbers. The normalized absorbance values are plotted as a function of time at 135°F in Figure 26. It is apparent that there was no appreciable decrease in the absorption of the aziridine peak during six days at 135°F.

With ERL-510 the absorbance of the epoxide peak at 820 wave-numbers was divided by the absorption at 1,500 wave-numbers which is due to c = c stretch of the aromatic ring. These values are also plotted in Figure 26. The reaction of ERL-510 with both antioxidants is apparent. The rate of reaction of PBNA is about 3.5 times the rate with AO2246.

TABLE 18

UF-2137 FORMULATION

	Antioxidant in HC Polymer			
	PBNA		A02246	
Raw Material	Stock/Lot No.	Wt %	Stock/Lot No.	Wt %
HC Polymer	7220-0008	82.7910	6290-0007	82.7230
MAPO	9319-0008	2.3966	9319-0008	2.4382
ERL 510	7297-0009	1.5124	7297-0009	1.5388
Iron Octoate	7318-0009	1.0	7318-0009	1.0
Thixcin E	7317-0009	2.0	7317-0009	2.0
Asbestos Floats	7320-0009	10.3	7320-0009	10.3

TABLE 19

UF-2137 PROPERTIES

		Units	AO in HC	
			PBNA	2246
Viscosity* - Time after end of mix (hr @ 135°F)		kp		
	0.1		1.12	1.57
	1.1		3.38	3.17
	2.1		4.14	3.93
	3.1		4.82	4.71
	4.1		6.18	5.72
	5.1		6.64	6.67
	6.1		8.05	8.40
	7.1		9.01	9.56
	8.1		10.68	11.99
Hardness - Cure time at 135°F	(hr)	Shore A		
	48		19	25
	68		32	42
	93		39	49
	117		48	54
	168		50	57
Penetrometer - (0.392" dia foot, 100 gr, 60 sec) Cure time at 135°F	(hr)	mm		
	24		4.5	2.7
	28		2.4	1.1

* Brookfield HBT, TD spindle at 5 rpm

TABLE 20

UF-2137 PROPERTIES

Parameter	Units	AO in HC PBNA	AO in HC 2246
Stress	psi	159	187
		157	184
		152	182
		157	185
		154	184
		156	184
Strain	in/in	2.13	1.63
		1.85	1.72
		1.95	1.38
		1.90	1.65
		1.97	1.61
		1.96	1.60
Adhesion to Steel	psi	258*	289
		245	288
		239	294
		247	294
		250	273
		248	288

* Liner failure, all other samples were bond failure

TABLE 21

TP-H1148 TO UF-2137 BOND STRENGTH

Liner Cure Prior to Casting (hr @ °F)	Antioxidant in HC Polymer	Adhesion (psi)	180° Peel (lb/in)
68 @ 135	PBNA	125	10.3
		122	10.6
		126	10.2
		126	—
		125	10.4
	2246	131	8.0
		126	7.9
		128	8.0
		128	—
		128	8.0
168 @ 135	PBNA	128	11.0
		123	10.6
		130	10.2
		128	—
		127	10.6
	2246	129	7.4
		130	7.4
		126	7.6
		129	—
		129	7.5

All adhesion samples were propellant failure and peel samples had a propellant film on the liner.

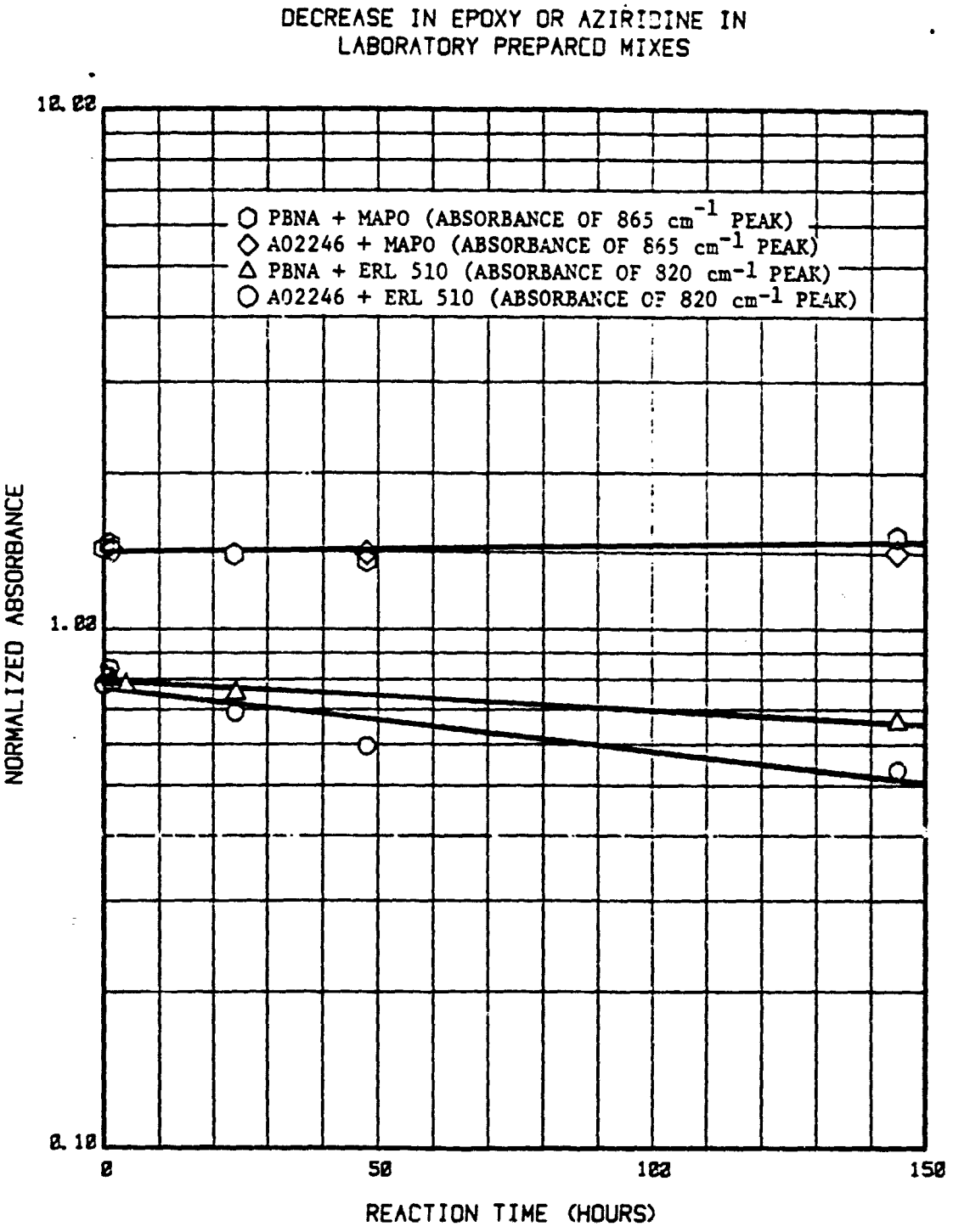


Figure 26. Decrease in Epoxy or Aziridine in Laboratory Prepared Mixes

Thus, it is apparent that PBNA reacts with the ERL-510, thereby reducing the reaction rate of ERL-510 epoxy with the HC polymer carboxyl radicals, by depleting the number of available epoxy radicals.

Subsequent tests were made by mixing PBNA (reagent) with ERL-510 and observing the mixture. PBNA dissolves in the ERL up to and including a 50/50 (by weight) mixture, and cures the epoxy to a soft, but solid mass at 135°F.

In addition to the actual faster cure with AO2246 HC as would be predicted from the above data, it would also be predicted that the liner-propellant adhesion would be somewhat less because the AO2246 HC liner would be in a higher cure state when propellant was cast onto it. Thus, it is now apparent that the liner formulation must be adjusted to compensate for this situation.

There are three approaches to adjusting the liner formulation: (1) decrease the curing agents (MAPO + ERL) to HC polymer ratio (epoxy + imine): carboxyl, now at 1.04:1; (2) decrease the iron octoate cure catalyst (now at 0.1%); and (3) change the percentage ERL in the formulation to provide the desired cure rate and liner-propellant peel values.

These three approaches are covered in the following paragraphs.

7.6 DECREASING THE CURING AGENT:HC POLYMER RATIO

Liner mixes were prepared using 0.7, 0.8, 0.9, 1.0, and 1.04 (control present formulation):one curing agent (imine + epoxy):carboxyl ratios. These liners were then used to fabricate adhesion discs and liner molds. Propellant adhesion samples and 180° peel samples were made from propellant (TP-H1148) with the HB polymer having the new antioxidant. Adhesion samples were tested at 0.5 in./min, dogbones at 1.0 in./min, and 180° peel at 12.0 in./-min. The liner itself was measured by checking viscosity vs time and hardness (Shore A) vs time (cure time of liner).

Results of these tests are as follows.

1. Increasing the curing agent increases adhesion to steel and increases stress, with a corresponding decrease in strain (Table 22).

TABLE 22

MODIFIED UF-2137 STRENGTH

		Curing Agent/HC Polymer Ratio (i.e. 0.7/1)				
Test	Units	0.7	0.8	0.9	1.0	1.04
Adhesion to Steel	psi	170	201	271	290	300
		189*	211	267*	289	286
		158	210	304	290	320*
		158*	210*	285*	295	311*
		167*	221*	257	288	300
		168	211	277	290	303
Stress	psi	99	123	170	175	172
		99	132	170	-	185
		97	139	160	171	174
		100	144	168	179	169
		95	135	168	177	173
		98	135	167	176	175
Strain	%	396	250	220	122	142
		400	250	217	-	176
		391	287	174	119	137
		426	276	212	131	127
		379	267	227	129	146
		398	268	210	125	146

*Liner failure, other samples were bond failure to the steel adhesion disc.

2. Increasing the curing agent shows a trend in increasing adhesion to propellant but drops off again when the ratio is greater than 1.0 (Table 23).
3. The results of the 180° peel tests (Table 23) show similar trends, but somewhat less positive. The "cure 2" results indicate large reduction in peel values at the lower curing agent to carboxyl ratios.
4. The mix viscosity time curve shows a trend of faster rising viscosity with increasing curing agent (Figure 27).
5. The Shore A hardness versus cure time clearly shows a faster rise and harder cure for increasing cure time up to 1.0 ratio. The 1.0 and 1.04 ratios yielded the same results (Figure 28).

The results indicate that the softer liner (i.e., 0.8:1 and 0.9:1 CA:HC ratio) did attain the desired higher peel strength at the low liner cure state but decreased by approximately one-half at the higher liner cure state which is an indication of poor liner storage life.

7.7 CHANGING THE AO2246 HC LINER CURE CATALYST CONTENT AND THE PERCENTAGE ERL

The following tests were run on various liner batches with the new AO2246-HC liner, varying the iron octoate concentration, and varying the amount of ERL in the formulation (separately) along with a PBNA-HC control liner mix.

1. Five lb batches of liner were mixed in a Ross vacuum mixer with the last 10 min of the mix cycle mixed under vacuum.
2. Viscosity (Brookfield GBT viscometer, TD spindle at 5 rpm) was obtained at end of mix and one hour intervals. The liner was stored at 135°F between viscosity readings.

TABLE 33

TP-H1148 TO UF-2137 (MODIFIED) BOND STRENGTH

Test	Cure	Units	Curing Agent/HC Polymer Ratio (i.e. 0.7/1)				
			0.7	0.8	0.9	1.0	1.04
Adhesion	1*	psi	96	106	107	110	104
			102	103 5***	98 10	108 5	117
			102 50	112	103	111	109
			100	107	103	110	110
	2	psi	92 100	88 100	95 80	102 10	99
			93 100	97 95	102 100	99 10	97
			90 100	98 100	95 90	102	97
			92	94	97	101	98
180° Peel	1	lb/in	10.1**	10.7**	11.1	8.5	8.3
			9.9	10.9	11.4	8.6	7.8
			9.5	9.9	11.2	8.8	7.9
			9.8	10.5	11.2	8.6	8.0
	2	lb/in	6.6**	5.8**	6.8**	8.0	6.9
			6.7	6.1	6.7	7.7	7.1
			6.5	5.5	6.4	7.7	7.2
			6.6	5.8	6.6	7.8	7.1

* Cure 1 = 68 hr @ 135°F liner cure prior to casting propellant
2 = 168 hr @ 135°F liner cure prior to casting propellant

** Heavy propellant film on the liner on these groups, other groups had a light film of propellant

***Percent propellant film, remaining failure is propellant

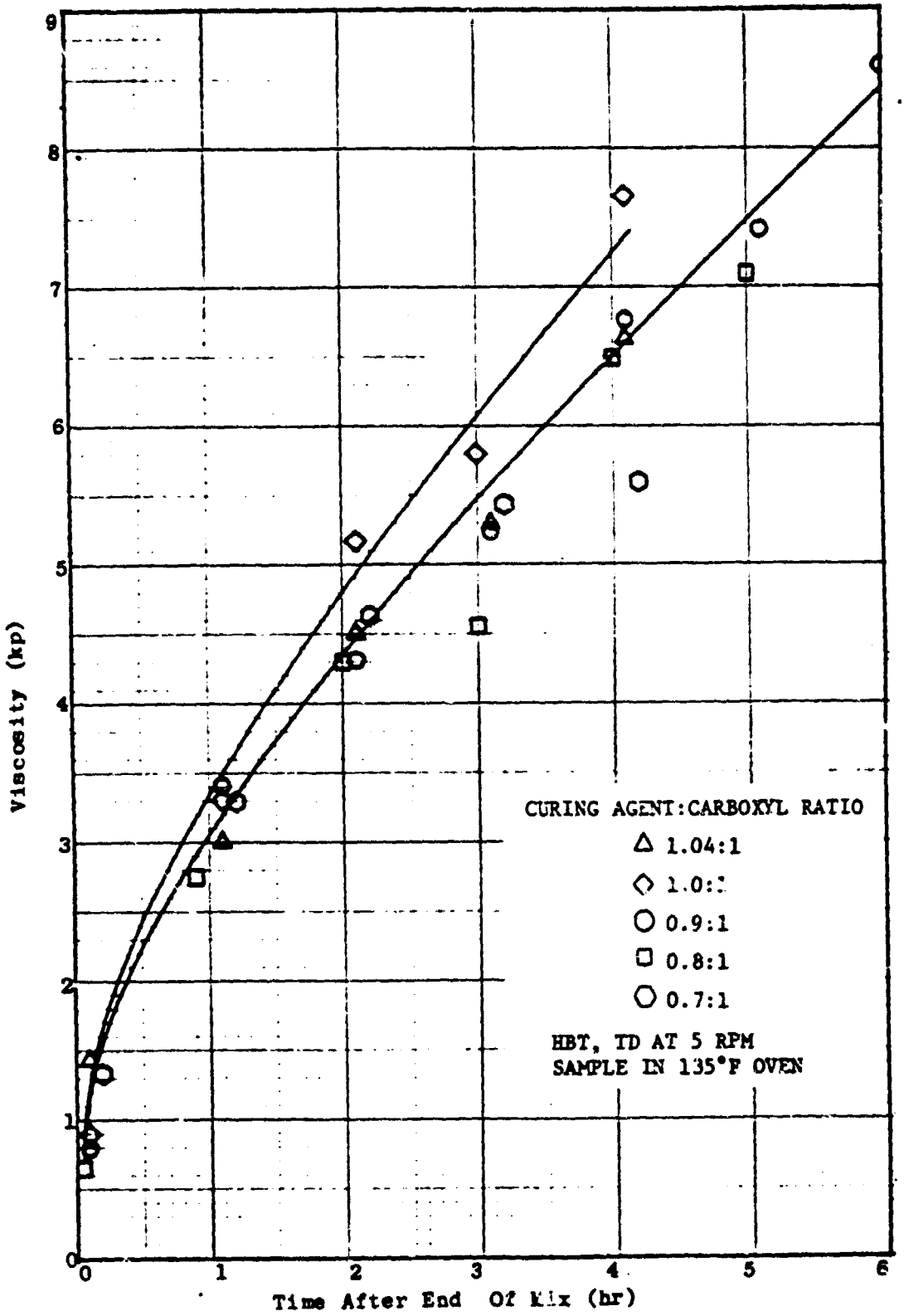


Figure 27. UF-2137 Liner Viscosity - Liner With Various Curing Agent to Carboxyl Ratios

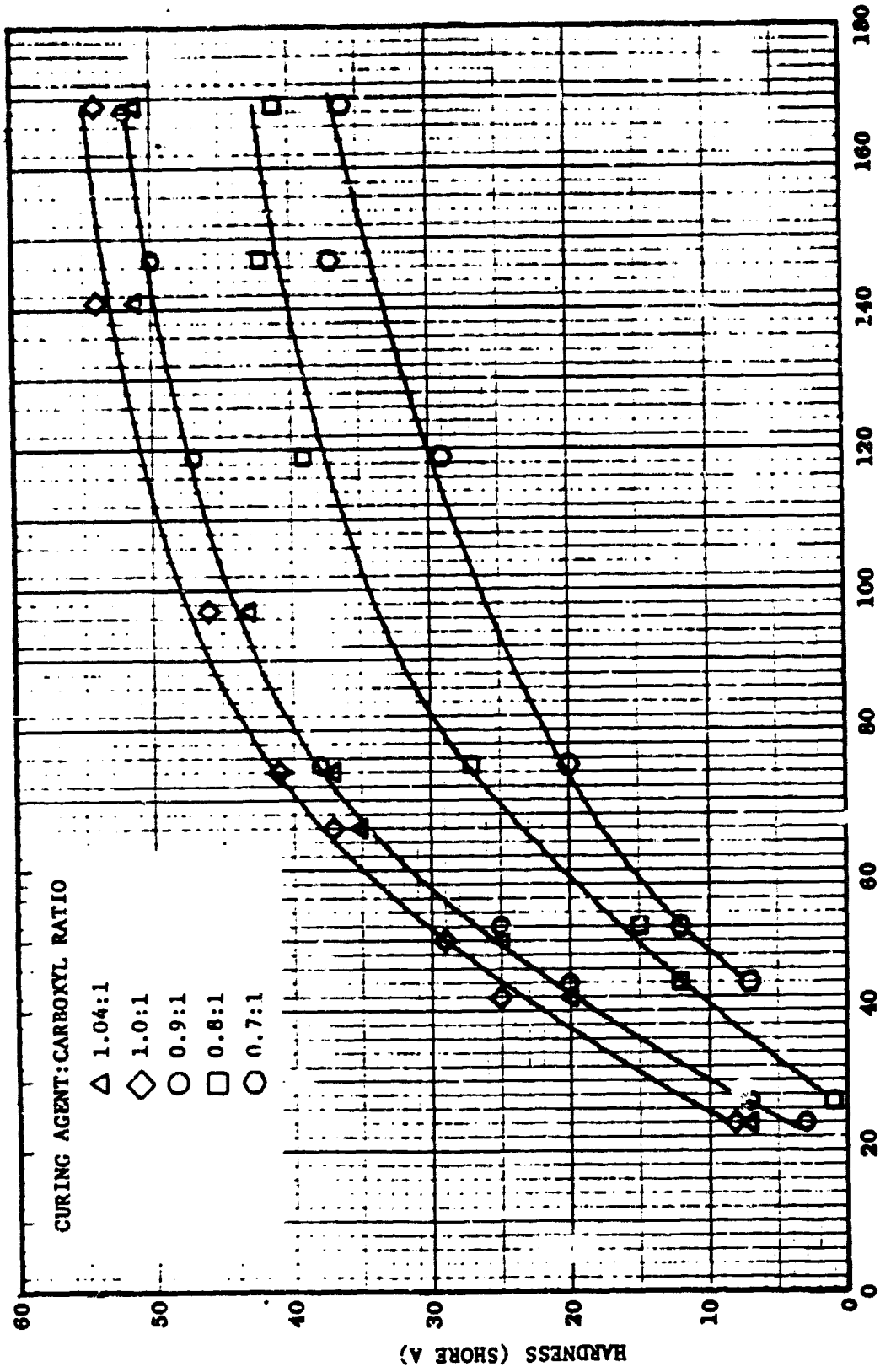


Figure 28. UF-2137 Liner Hardness - Liner With Various Curing Agent to Carboxyl Ratios

3. One liner mold was cured 168 hr at 135°F, die A dogbones were cut and the dogbones were tested at 2.0 in./min.
4. Hardness (Shore A) was measured after 158 hr at 135°F.
5. Adhesion to steel disc samples, cured 168 hr at 135°F, was tested at 0.5 in./min.
6. NBR rubber was degreased with MEK, air dried, lined with liner (65 mils nominal) and cured 68 and 168 hr at 135°F. TP-H1148 propellant was cast on the samples and cured 96 hr at 135°F. Adhesion cup samples were tested at 0.5 in./min and 180° peel samples at 12 in./min.

The control mix is the UF-2137 using the present HC polymer containing the PBNA antioxidant. All other mixes were made using the HC polymer containing AO2246 antioxidant.

Decreasing the iron octoate catalyst content (Table 24) from 1.0 to 0.8% changed the liner properties and TP-H1148 bond properties only slightly (Tables 25 to 27) but did not compare to the control (HC-PBNA).

Increasing and decreasing the amount of MAPO both resulted in lower propellant-liner peel strength than the control. The viscosity on the high MAPO mix increased faster than the control although the physical properties were in about the same range as the control.

Increasing the ERL-510 content produced a harder liner and lower propellant-liner peel strength. Decreasing the ERL-510 produced properties within the range of the control and the peel strength also increased.

Graphs of the varying ERL-510 mixes (Figures 29 and 30) show that the various control mix values compare to the following MAPO:ERL ratios in the liner: Shore A -5.0, adhesion to steel -3.7, stress -4.1, strain -3.9, 68 hr 180° peel -4.1, 168 hr 180° peel -2.9. As the ERL-510 is decreased (increasing MAPO/ERL ratio) the liner becomes softer and propellant-liner peel strength increased within the range tested.

TABLE 24

LINER VARIATIONS

Parameter	Control New Polymer	Low IO	Low ERL	High ERL	Low MAPO	High MAPO	Control Old Polymer	Lowest ERL
Mix No.	1	2	3	4	5	6	7	8
Equivalents								
MAPO-ERL	1.04	1.04	0.90	1.18	0.90	1.18	1.04	0.85
MAPO/HC	0.70	0.70	0.70	0.70	0.56	0.84	0.70	0.70
ERL/HC	0.34	0.34	0.20	0.48	0.34	0.34	0.34	0.15
MAPO/ERL	2.06	2.06	3.50	1.46	1.65	2.47	2.06	4.67
Weight Percent								
HC Polymer	82.7230	82.9138	83.4668	82.1228	[illegible]09	82.2603	82.7908	83.5517
MAPO	2.4382	2.4438	2.4601	2.4205	1.9616	2.9094	2.3966	2.4626
ERL 0510	1.5388	1.5424	0.7731	2.1567	1.5475	1.5302	1.5126	0.6857
Iron Octoate	1.0	0.8	1.0	1.0	1.0	1.0	1.0	1.0
Thixcin E	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Asbestos	10.3	10.3	10.3	10.3	10.3	10.3	10.3	10.3
Stock/Lot No.								
HC Polymer	6296-0007	→					7220-0008	6296-0007
MAPO	7319-0008	→						
ERL 0510	7297-0009	→						
Iron Octoate	7318-0009	→						
Thixcin E	7317-0009	→						
Asbestos	7320-0009	→						

TABLE 25

LINER VISCOSITY

Parameter	Control Old Polymer	Control New Polymer	Lowest ERL	Low ERL	High ERL	Low MAPO	High MAPO	Low IO***
Mix No*	7	1	8	3	4	5	6	2
MAPO/ERL	2.06	2.06	4.67	3.50	1.46	1.65	2.47	2.06
Viscosity (kμ)								
0.1 **	0.87	1.10	1.02	1.00	1.09	1.11	0.96	1.15
1	2.84	2.31	2.72	2.08	2.24	3.65	2.75	2.28
2	3.52	2.94	4.18	2.69	3.58	4.13	4.00	3.24
3	4.15	3.42	4.72	3.94	4.81	5.31	5.53	3.97
4	5.81	3.42	5.78	4.16	5.81	5.70	7.50	4.93
5	—	4.26	6.21	5.15	7.89	7.29	8.23	6.15
6	—	6.07	7.42	6.29	—	8.30	12.56	—

* Reference Table 24

** Time after end of mix (hr), liner stored in 135°F oven

*** Iron Octoate (Cure catalyst)

TABLE 26

LINER PHYSICAL PROPERTIES

Parameter	Control Old Polymer	Control New Polymer	Lowest ERL	Low ERL	High ERL	Low MAPO	High MAPO	Low IO
Mix No *	7	1	8	3	4	5	6	2
MAPO/ERL	2.06	2.06	4.67	3.50	1.46	1.65	2.47	2.06
Shore A (168 hr @ 135°F)	52	59	55	52	61	61	55	59
Adhesion to Steel (psi)	265	326	263	257	320	308	285	325
	277	330	274	254	338	335	293	330
	298	345	274	264	382	323	306	326
	287	337	260	265	344	330	315	322
	275	—	268	—	337	330	302	323
	280	335	268	260	344	325	300	325
Stress (psi)	174	231	159	172	243	263	196	232
	168	232	167	167	254	220	192	235
	166	232	170	172	254	213	195	236
	169	232	165	170	250	232	194	234
Strain (in/in)	1.93	1.20	2.10	2.21	1.39	1.36	1.71	1.43
	1.93	1.17	2.08	2.25	1.22	1.73	1.73	1.19
	2.14	1.16	2.18	2.18	1.40	1.25	1.77	1.35
	2.00	1.20	2.12	2.21	1.34	1.45	1.74	1.32

* Reference Table 24

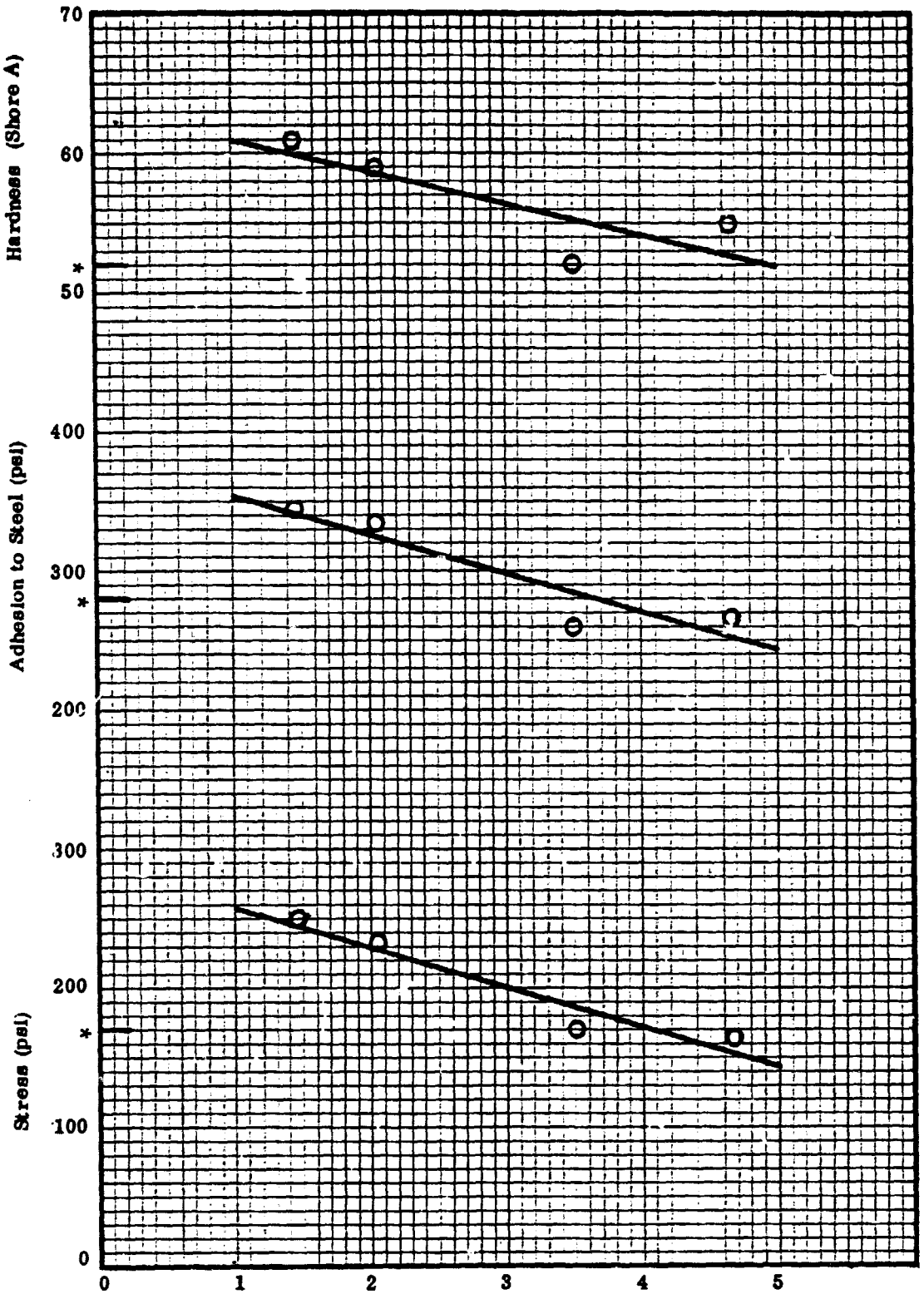
TABLE 27

TP-H1148 TO LINER BOND PROPERTIES

Parameter	Control Old Polymer	Control New Polymer	Lowest ERL	Low ERL	High ERL	Low MAPO	High MAPO	Low IO
Mix No *	7	1	8	3	4	5	6	2
MAPO/ERL	2.06	2.06	4.67	3.50	1.46	1.65	2.47	2.06
180° Peel (lb/in)								
68 hr @ 135°F**	10.2	7.0	10.4	9.7	6.8	7.8	7.0	7.4
	10.0	6.9	11.1	10.0	7.0	7.7	7.2	7.4
	10.2	7.3	10.7	9.7	6.8	8.2	7.1	7.5
	10.1	7.1	10.7	9.8	6.9	7.9	7.1	7.4
168 hr @ 135°F	7.7	6.5	8.1	8.3	6.3	7.0	5.7	7.2
	7.4	7.4	9.2	8.4	6.0	6.3	5.8	6.6
	7.8	7.0	9.0	8.9	6.0	6.2	6.2	6.8
	7.6	7.0	9.0	8.5	6.1	6.5	5.9	6.9
Adhesion								
68 hr @ 135°F	139	136	139	127	132	128	135	138
	125	138	137	132	135	130	134	142
	126	138	134	131	127	124	132	135
	131	137	136	130	131	127	134	138
168 hr @ 135°F	134	137	136	128	132	134	137	140
	139	126	137	131	139	131	135	—
	141	148	135	133	136	130	131	133
	138	137	136	131	136	132	134	137

* Reference Table 24

** Liner precure prior to casting propellant



*UF-2137 Control Mix Values

Figure 29. Liner Properties

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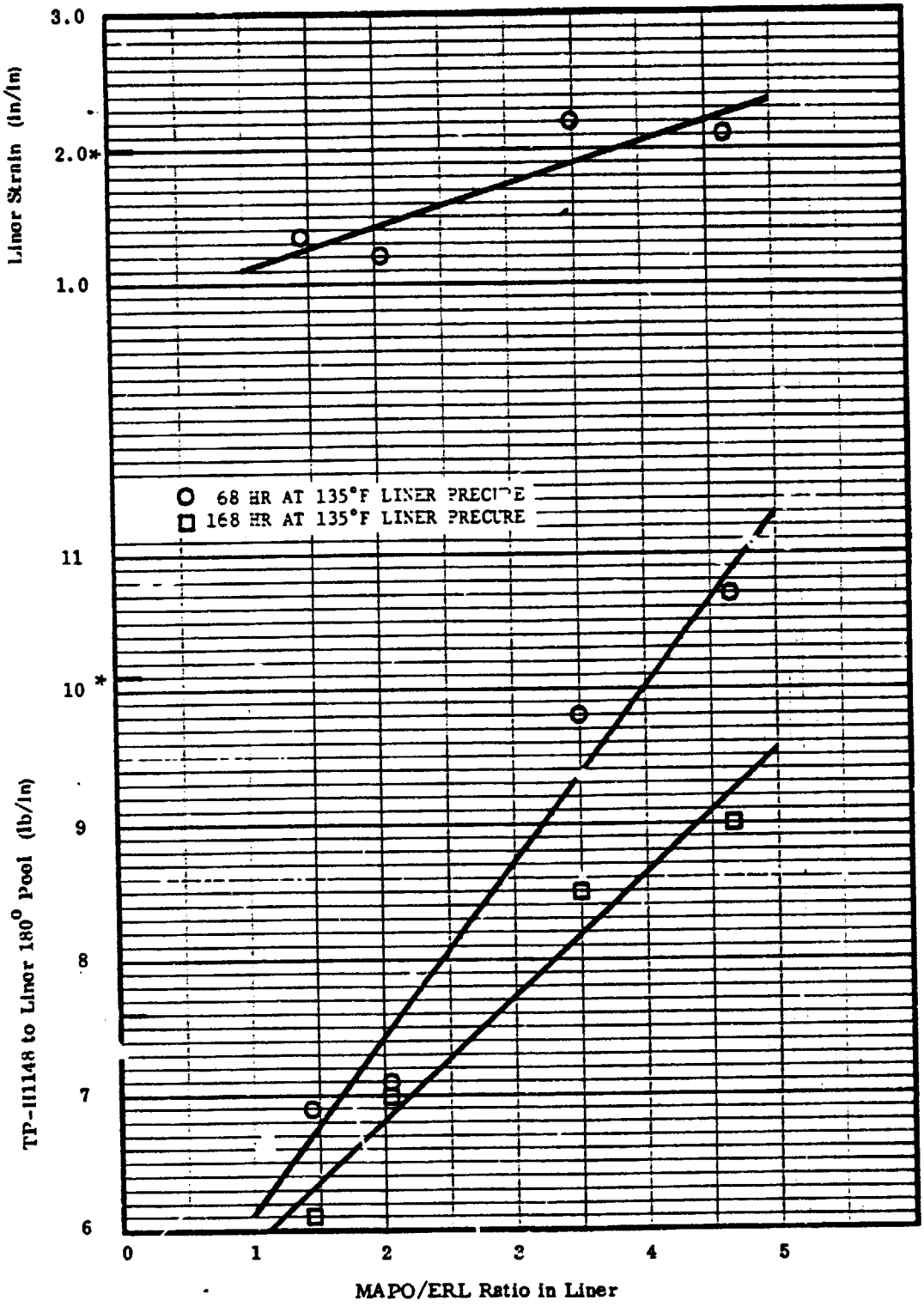

*UF-2137 Control Mix Values

Figure 30. Liner and Propellant to Liner Properties

The aging qualities of liner with a decreased ERL-510 content is not yet known or the properties of inhibitor or the moisture sensitivity of inhibitor and these parameters need to be studied with various ERL-510 levels prior to determining if this produces an acceptable liner or inhibitor formulation. Thus (1) decreasing the amount of ERL-510 in the liner (HC-A02246) produced liner approximating the liner presently used; and (2) varying MAPO and iron octoate did not produce acceptable results.

References:

1. "Qualification Plan for Replacement of PBNA in HB and HC Polymers Used in SRM Liner and Propellant," June 1977, TWR-11293, Revision A.

2. "Preliminary Evaluation of Candidate Antioxidants for Replacement of PBNA," TWR-10608, by R. D. Law.

3. "Qualification for Replacement of PBNA in HB Polymer," D. C. Suisse, February 1981, TWR-12559, Revision A.

4. "Qualification of a New Source DDM in HB Polymer," M. L. Levinthal, TWR-20182.

5. "Compatibility of Lubricants With SRM Propellant, Insulation, and Inhibitor," D. C. Suisse, March 1981, TWR-20182.