ASD TECHNICAL REPORT 61-144

ACCELERATED STORAGE STABILITY TESTS

ELDRED N. CART, JR., 1/LT. USAF
DIRECTORATE OF MATERIALS AND PROCESSES

SEPTEMBER 1961

AERONAUTICAL SYSTEMS DIVISION

Fuels and Lubrication Branch, Non-Metallic Materials Laboratory, Directorate of Materials and Processes, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.
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SEPTEMBER 1961

PROJECT No. 3044

AERONAUTICAL SYSTEMS DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

300 - December 1961 - 14-568 & 569

FOREWORD

This report was prepared by the Lubrication Section, Fuels and Lubricants Branch, Non-Metallic Materials Laboratory, Directorate of Materials and Processes, Aeronautical Systems Division.

Acknowledgement is made for the assistance given by Dr. A.H. Popkin of the Esso Research and Engineering Company who volunteered to run tests requested by this Division at his company's expense. The spirit of cooperation is greatly appreciated.

ABSTRACT

The storage life of MIL-L-7808 oils has been improved by the use of amine type additives. This report describes the accelerated storage tests used to arrive at qualification limits. The time-temperature relationships for the storage life of MIL-L-7808 oils are given. From these relationships, the storage life can be estimated at any temperature from data at one given temperature.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. P. Dunnam
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Chief, Fuels & Lubricants Branch
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I. INTRODUCTION AND BACKGROUND

The problem of storage instability of MIL-L-7808 oil originated in April 1955 when a di-ester oil qualified to this specification was found to have a lead corrosion value exceeding 200 mg/in^2 after one year's storage. The lead corrosion value was determined by the Standard Oil Development Lead Corrosion Test (SOD Test) by AMC Quality Control. The maximum value for qualification to MIL-L-7808 is 6 mg/in^2.

To determine the effect of an oil having a high SOD value on engine operation, nine turbojet engines were run for approximately 100 hours each on oils having SOD values ranging from 17 to over 250 mg/in^2. Details of these tests were reported at an Air Force conference in November 1957 (1). In one of these tests, lead corrosion was found. Also, in a B-47 flight test with the J-57 engine, complete removal of the lead was found along with severe corrosion of the silver and bronze. This flight test was run at 225°F oil-in, initial SOD of 175 mg/in^2, oil change every 25 hours, and a total of 434 flight test hours on the engine. General operation and condition of these engines were normal except for the corrosion noted above. With the J-57, the fact that the lead-overlay is removed is not too critical, since it is there as a thin, soft wear-in surface for a new bearing. However, the B-47 flight test showed that other metals could be attacked by some oils once the lead was removed. Corrosion of the silver and bronze could cause a flight safety problem.

The Navy also engine tested an oil having an SOD of 205 mg/in^2 (2). This J-57 had slight lead removal on the number 2 bearing and complete lead removal on the number 4 1/2 bearing. The Navy concluded that satisfactory results were obtained as far as general engine operation and condition were concerned. However, a lead removal problem could exist on the bearing cages in a turbojet if a high SOD oil were used and there was no protective coating over the lead plating. A high SOD oil in a new or newly overhauled J-57 engine could cause extensive lead removal in the first 20 hours of operation. An allowable SOD value of 150 maximum was placed on Specification 7808 lubricants in storage, since certain factors, such as deterioration rate and the 3-month recheck period, could bring a storage oil above the average 230 SOD value of the oil tested (2).

Work has been performed by various investigators on the mechanism of the breakdown of di-ester oils in storage. (3), (4), (5). James, Murphy, and O'Rear, state (4) that acidolysis of di-ester and sebacic acid to form a half-ester is not the mechanism responsible for the formation of this compound in stored MIL-L-7808 oils. Murphy, Ravner, and Timmons (5) found that lead corrosion was due to the progressive formation of the acid half-ester resulting from the hydrolysis of sebacic acid di-esters. Di-ester hydrolysis is catalyzed by acid phosphates present in or formed from tri-cresyl phosphate (5).

Work was performed for the Air Force by the Denver Research Institute (3). They found no correlation between water content and storage life. A summary of their data is given in table 1 showing the basis for this conclusion. They concluded the mechanism was almost entirely oxidation. The auto-oxidation process proceeds through the formation of peroxide-like materials which are decomposed to form agents corrosive to lead.

Manuscript released by author April 1961 for publication as an ASD Technical Report.

Based on the results of the work at Denver, a program was initiated at ASD to find an additive that would improve the storage life of qualified 7808 oils (6). Different additives were tested and aliphatic amines were found to cause increased storage life. One of the additives, 2,6-di-tert-butyl- α -dimethylamino-p-cresol (abbreviated 26AC), was tested in three different qualified 7808 oils, (representing different base stocks), and definitely improved the storage life of all 3 oils. Satisfactory results were obtained on specification tests on these oils containing 0.1 percent 26AC. Four 100-hour J-57 engine tests were run on oils containing 0.1 percent of the above amine with satisfactory results. The additive did not adversely affect the engine performance of the oils in any way.

Since the results on the effect of concentration of 26AC were reported in WADC TR 59-379, additional data have been obtained. These data are plotted in figure 1. Increasing the amount of 26AC improves the storage life of the oil. In general, increasing the concentration of 26AC increases the induction period, with the rate of breakdown after the induction period remaining about the same.

The effect of the increased amount of 26AC on the Deposition Number is shown below:

Concentration of 26AC	Deposition No. (4 tests)
0%	4.10
0.1%	3.18
0.2	2.74
0.4	1.84

With this oil, addition of more 26AC improved the deposit forming tendencies. Too much 26AC could increase the -65°F viscosity beyond the 13,000 cs. limit. The effect on other properties of greater concentrations of 26AC would have to be determined for each oil. These data show that some oils will have longer storage life when the amount of 26AC is increased above the 0.1 percent level.

II. STORAGE TESTS

Two storage tests have been used in the qualification program. An ASD Tropical Room storage program and a 185°F oven test. The 185°F oven test method is given in appendix I. Repeatability of this test is described in Reference 6. It was designed to correlate with the storage life data on oil in the ASD Tropical Room which is maintained on a cycle of 8 hours at 115°F, and 4 hours to cool down to 70°F, where it remains for 8 hours, and then 4 hours are allowed to heat up to 115°F. The cycle is then repeated. For correlation purpose, the oil in the Tropical Room is assumed to be at an average temperature of 92°F.

SOD tests were periodically run on samples from the Tropical Room. Curves of time versus SOD lead loss were determined for the 16 oils listed in table 2. The time to reach a SOD of 25 and 150 mg/in^2 for the two storage conditions is also given in table 2.

Based on previous data, a straight line was obtained when the time to reach a SOD of 25 or 150 was plotted against 1/T (T = absolute temperature). The slope for each oil was calculated and is listed in table 2. A sample calculation is shown in appendix II. The average slopes from table 2 are 1.25×10^4 and 1.22×10^4 for 25 and 150 SOD limits respectively. The slope for doubling the rate of reaction for every 18° is 1.25×10^4. The average percent difference in the slopes for 25 and 150 SOD values is about 10 percent. All storage tests are based on the assumption that SOD is a linear function of time after the induction period to at least a SOD of 150 mg/in^2.

Using an average slope, a relationship between days in the 185°F oven test and days in the Tropical Room was developed and is given in figure 2. The method used is given in appendix II.

For the Air Force there are two limiting SOD values. The first is a maximum SOD of 25 mg/in^2 for the T-34 engine and 150 mg/in^2 for all other gas turbine engines. The limit for the T-34 engine is required because it contains plain lead bearings. The limits of one year and 3 years for a SOD of 25 and 150 were chosen as the desired goals for storage of 7808 oils under tropical conditions. Based on these goals and figure 2, the limits of 14 and 45 days at 185°F for SOD's of 25 and 150 mg/in^2 respectively were included in MIL-L-7808 as a qualification requirement. Actually, the three-year limit relates to 42 days at 185°F as noted on figure 2. However, due to the inherent inaccuracies of this type of testing, three days were added as a safety factor giving the established limit of 45 days.

III. COMPARISON OF PREDICTED VALUES TO ACTUAL DATA

At the time of requalification of all 7808 oils, samples were placed in the Tropical Room to check our predicted values. These samples were from the same batches tested in the 185°F oven test. SOD tests are being run on these samples every 15 weeks. Most of the samples were in the Tropical Room for about one year. Thus, only limited results are available to compare the predicted values to actual values. The predicted storage life is based on the 185°F requalification test and figure 2. The results available to May 1960 are compared in table 3 and figures 3 and 4. All of these samples are "as received." The same samples with 26AC have not started to deteriorate.

In general, the predicted values approximate the actual values; well within the limits of the test and estimation method.

IV. TROPICAL ROOM DATA ON DIFFERENT BATCHES OF MIL-L-7808 OIL

The Air Force is storing in the Tropical Room a case of every production batch of MIL-L-7808 oil delivered to the Air Force. Quart cans are taken from the case every 2 months and tested in the SOD test. Charts are made for each batch so that a prediction can be made when the oil will reach 25 and 150 mg/in^2. Field activities are then notified that certain batches must be used by a specified date or be discarded. This method allows a poor oil to be used before it must be disposed of, thus reducing the amount of discarded oil.

Four different oils which have been included in this program are reported here. The time to reach a SOD of 150 for each batch is given in tables 4 and 5. Since some of the batches have been in normal storage before being placed in the Tropical Room, this time must be corrected so that all batches are on the same basis. This was done by using a "corrected" time to reach a SOD of 150, calculated as:

Corrected Time = Months to reach 150 - (Total months - Months in Tropical Room)

Therefore, only the months in the Tropical Room are taken as being responsible for the breakdown of the oil.

Table 6 gives the average time to reach 150 SOD values for the four oils, the maximum and minimum time for any batch and the average between batches. These data serve to indicate the wide variation between batches of the same oil and also the wide range of storage life of MIL-L-7808 oils. None of these samples contained 26AC. These results show the need for a batch acceptance test.

V. VARIOUS ACCELERATED TESTS INVESTIGATED

Several different accelerated storage tests were tried by Esso Research and Engineering Company to see if they could differentiate between oils A and B, a very good and a very poor oil respectively in storage. An ASTM Bomb Oxidation Test shows the same pressure drop on both oils at the end of 10 days. Generally, all formulated oils will show this same trend.

An extended SOD lead corrosion test was run on the two oils, A and B. This test is run as shown in appendix III except the test was continued beyond 12 hours. Table 7 contains the results of this test. While this test did separate the two oils it gave an increase in the lead weight on Oil A and a loss with Oil B. It was decided that work would be required on this test in addition to simply increasing the temperature of the present 185°F test.

Work by Denver Research (3) indicated that ultraviolet light may be used as an accelerated storage test. A test was run on Oil B to check this out. A one gallon sample of oil was placed in a 5 liter - 4 neck flask. UV light from a 450 watt source was focused into the oil through one of the necks in the flask. Air at 1750 cc/min. was added to the oil which was maintained at 125°F. Samples were taken periodically for acid number determination. Three runs were made using this equipment. In the first 2 runs dry air was used, while in Run No. 3 saturated air was used and 0.1 percent water was added to the oil. The results are given in table 8. In Run No. 3 the oil showed an increasing acid content with time, but the SOD lead loss was low, even at the end of 250 hours with an acid number of 3.59. It appeared the oil was breaking down by a different mechanism than it does during normal storage. For this reason, no future work is contemplated for this method.

VI. HIGH TEMPERATURE OVEN BATCH ACCEPTANCE TEST

Tests at various oven temperatures were run by ASD and Esso Research. Six oils coded A through F were selected as representative for the development of a test method. Oils A and E are very good in storage and along with Oil F meet the MIL-L-7808 requirements for the 185°F test. Oils B, C, and D failed the 185°F test and represent different levels of unstable oils in storage.

The ASD oven test results at 185, 215, 230 and 260°F on oils A through F are shown in figures 6 through 9. Data obtained by Esso on Oils A through F at 230 and 250°F are shown in figures 10 and 11. Comparison of the data obtained by Esso and ASD is given in tables 9 and 10. The two laboratories do not agree on the time to reach a specific SOD value. Esso is more severe in both the induction time and the rate of breakdown after the induction period.

a. Repeatability of the Oven Tests

The repeatability of the 230°F oven test on Oils A and B was determined by Esso with the results given in table 11 and figures 12 and 13. In general, the repeatability was good.

In an attempt to determine whether differences in operating procedure could account for this difference, ASD ran a 215°F oven test on Oil B under several different conditions. One container was shaken before taking the 500 cc sample and the other can was not shaken. Results are shown in table 12 and figure 14. This variable (shaking the can) had no effect on the test results. A check was also made during this same series of runs to determine the effect, if any, of letting the sample set a number of days before running the SOD test. This had little effect on the results. Work is still in progress to find the reason for the difference of time in Esso Laboratory and ASD to reach a specific SOD value.

A modified oil test was run on Oils A and B. In this test 12 liters/hour of pure oxygen were bubbled into 550 cc of oil at 95°C. Catalysts of lead and copper were used. Because of the addition of lead as a catalyst, neutralization numbers were used as the criteria of change. Results are shown in table 7. This test did not show promise as an accelerated storage test.

The effect of adding water to the oil to decrease the oven test time was determined by ASD. Results are shown in figure 5. Adding water does increase the severity of the oven test. However, the effect this would have on all qualified MIL-L-7808 oils would have to be determined.

It was decided to use the present 185°F oven test, with the given temperature increased in order to give an answer in less than one week.

VII. TIME-TEMPERATURE RELATIONSHIPS

On analysis of all the ASD oven test and Tropical Room data, several curves have evolved to predict the effect of temperature and time on storage life.

One of the assumptions in this study was a linear relationship between SOD and time at one temperature between the induction period and a SOD of 150. The time-temperature slopes for SOD values of 25, 50, 100, and 150 are given in table 13. Since the average slope for these oils, and the oils in table 2 from the Tropical Room test all have a slope of about 1.2×10^4 it was decided to use the value of 1.25×10^4 for all future work.

Based on this slope, the time-temperature relationship for SOD values of 25 through 150 is given in figure 15. From this figure the effect of increasing or decreasing the storage temperature can be estimated for any given SOD value. For example a SOD of 50 at 185°F can be used to estimate the time to reach the same SOD value at 90°F or 250°F.

This same approach was taken to determine the time relationship for any SOD at any two given temperatures. The method is given in appendix II. The relationship between 185°F and the Tropical Room was given in figure 2. The relationship between 185 and 230°F is given in figure 16. Using these figures (more could be developed for any temperature range) the value of any SOD at one temperature can be estimated at any other given temperature. For example, the SOD's at 60 and 120 were taken from 185°F data for oils B, C, and D and estimated at 230°F for the ASD data. A comparison is given in table 14. The same procedure was used for the Esso data between 230 and 255°F and is given in table 14.

Using the 185°F oven test limits as given in figure 17, oven test limits for any oven temperature and any time limit can be set. For example, for a 5-day test at 230°F, the oil should have a SOD of 35 mg/in^2 or less to correspond to the 185°F limits, which in turn corresponds to the Tropical Room limits.

VIII. CONCLUSIONS AND RECOMMENDATIONS

1. Regardless of the mechanism responsible for oil deterioration in storage (oxidation and/or hydrolysis), a test method is available for predicting a storage life at any temperature up to 260°F, the highest temperature investigated.

2. The oven test is satisfactory for a batch acceptance test. However, additional work is needed to improve the reproducibility between various laboratories, particularily the wide difference between Esso and ASD (Esso being almost 3 times more severe).

3. Precise temperature control is very important at these high oven temperatures and for the short times involved. Methods of maintaining the desired temperature other than an oven may be investigated. However, the same basic method of breakdown must be maintained.

4. Once the reproducibility between Esso and ASD is resolved, a Coordinating Research Council cooperative test should be initiated on two reference oils.

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1. Cart, E.N., Jr., "Qualification Testing and Service Problems," WADC High Temperature Aircraft Gas Turbine Engine Lubricants Meeting, 20 November 1957.

2. NAMCAEL Report 1549, Project TED-NAM-PP-5101, "High SOD Value MIL-L-7808 Oil Turbine Engine Test," February 1958.

3. Bohner, G.E., Frederickson, G.D., & Schmidt, J.J., "Development of an Accelerated Storage Test for Dibasic Ester Type Lubricants," WADC TR 58-572, March 1959.

4. James, F.L., Murphy, C.M., & O'Rear, J.G., "Acidolysis Reactions for the System Bis(2-ethylhexyl) Sebacate, Sebacic Acid, and 2-Ethylhexyl Hydrogen Sebacate," NRL Report 5181, August 1958.

5. Murphy, C.M., Raver, H., & Timmons, C.D., "Factors Influencing the Lead Corrosivity of Diester Base Oils," NRL Report 5451, February 1960.

6. Cart, E.N. Jr., "Improvement of the Storage Life of MIL-L-7808 Oils," WADC TR 59-379, June 1959.

APPENDIX I

185°F OVEN TEST PROCEDURE

This test is designed to predict the storage life of MIL-L-7808 oils under accelerated storage conditions. The following equipment is needed:

Oven, capable of maintaining a temperature of 185 ± 2°F

SOD bath and test equipment

1 gallon tinplated cans

1. Place the oil to be tested in a gallon can.

2. Place the can in the oven. At 0, 7, 14, and every 14 days thereafter, run a SOD lead corrosion test on a 500 cc sample from the can. The can is returned to the oven after each sample for the SOD test is removed. A new 500 cc sample is taken from the can for each test, so that the volume of oil in the can decreases by 500 cc every 7 or 14 days, as the case may be.

3. The test procedure is continued until the oil reaches a SOD of 160 mg/in^2 or greater, or until all the oil in the gallon can is used, whichever occurs first.

4. A plot of SOD versus time in the oven is made. From this curve the time to reach a SOD of 25 and 150 is obtained. Duplicate tests shall be run for greater accuracy and the average values used to determine the storage life under tropical conditions.

APPENDIX II

SAMPLE CALCULATIONS

Method 1:

If a straight line is obtained when Ln Y vs X is plotted, the equation for this line is $Ln\ Y = BX + Ln\ A$, $Ln\ Y - Ln\ A = BX$, $Ln\ Y/A = BX$, $Y/A = e^{BX}$, $Y = Ae^{BX}$. In this equation, Y is a specific SOD value, B is the slope of the line, X is a temperature function (in this case, $X = 1/T$, T = °Rankine) and A is assumed to be 1.

Example:

From table 2, for GTO-309*

$$Y = e^{BX}$$

$$Ln\ Y = BX$$

$$\frac{Ln\ Y_1 - Ln\ Y_2}{(1/T_1 - 1/T_2)} = B = \text{Slope}$$

Where: Y_1 = 350 days for SOD of 25 at 92°F ($0.00181°R^{-1}$)

Y_2 = 14 days for SOD of 25 at 185°F ($0.00155°R^{-1}$)

$$\text{Slope} = \frac{Ln\ 350 - Ln\ 14}{0.00181 - 0.00155} = \frac{5.86 - 2.64}{0.00026} = 1.24 \times 10^4$$

Method 2:

Using this same equation but fixing the temperature difference, the relation between Y_1 and Y_2 can be obtained.

$$Ln\ Y_1 - Ln\ Y_2 = \text{Slope}\ (1/T_1 - 1/T_2)$$

Assuming the slope is constant at 1.25×10^4

$$1/T_1 - 1/T_2 = 0.00026 \text{ (92°F and 185°F data)}$$

Then: $Ln\ Y_1/Y_2 = Ln\ \frac{350}{14} = Ln\ 25 = 3.22$

$$Y_1/Y_2 = e^{3.22}$$

$$Y_1 = 26Y_2$$

This gives a straight line when plotted on rectangular coordinates with a slope of 26 and intercept at 0. This method can be used for any temperature difference between 92°F and 260°F.

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* GTO is the Gas Turbine Oil Code Number.

APPENDIX III

EXTENDED SOD LEAD CORROSION TEST*

The extended SOD Lead Corrosion Test is a modification of the one-hour SOD Lead Corrosion Test which is a specification test for all synthetic aircraft engine lubricants. This modification was developed as a means of predicting storage stability under field conditions. The extended periods give a far more realistic picture of the corrosive characteristics of an oil than does the one-hour Lead Corrosion value. The test differs from the usual MIL-L-7808 test in the following respects:

(1) After the MIL-L-7808 one-hour Lead Corrosion Test is completed, a new lead strip is added to the same oil and the test is run for an additional 3 hours. The four-hour result consists of the cumulative values in mg./in^2 of the one-hour plus the three-hour results.

(2) A new strip is now added to the same oil and the test is continued for an additional 4 hours. The sum of the previous 4 hours and this period comprises the eight-hour results.

(3) The final phase consists of using the same oil plus a new strip and again running the test for 4 more hours to give a total of 12 hours. The twelve-hour cumulative result is thus obtained on the same sample of oil, using four different strips and adding the four period times:

(a) 1st period = 1 hour

(b) 2nd period = 3 hours

(c) 3rd period = 4 hours

(d) 4th period = 4 hours

12 hours total time

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* Courtesy of Esso Research and Engineering Company.

Table 1

Comparison of Water Content and Storage Life			
GTO	SOD after 22 months Storage at Room Temp.	Days to Reach 150 mg/in^2 in Tropical Room	% Water in Dec. 1956
301	-144	91	0.06
303	0.4	-	-
309	0.7	350	0.05
339	3.7	350	0.03
340	0.5	406	0.08
341	67	133	0.04
342	0.4	252	0.09
363	28	133	0.03
369	0.5	-	0.11
370	37	126	0.04
371	14	-	0.21
SOD and Water values from Ref. 3, Pages 66 and 88.			

Table 2

Tropical Room Data and 185°F Oven Data on MIL-L-7808 Oils				
GTO	Days to Reach SOD of 25		Days to Reach SOD of 150	
	Tropical Room	185°F	Tropical Room	185°F
301	91	1.05	175	7
303	-	14	-	31.5
305	140	8.6	210	18.2
309	350	14	1160	36.5
317	329	8	1050	31
321	94	3	-	-
332	455	15.4	1460	43.5
339	350	10.5	980	-
340	406	19.6	1040	56
341	133	7.7	260	15.4
342	252	9.1	810	26.8
368	133	8.8	-	31.5
370	126	8.8	-	37.8
519	77	1.2	168	6.5
570	301	6.3	950	33

Slope of Time-Temperature Curves for Tropical Room
and 185°F Oven Test - 25 SOD Limit

GTO	Slope (x) $\times 10^4$	$x-\underline{x}$ $\times 10^4$	
301	1.72	0.475	
305	1.05	0.20	
309	1.24	0.029	
317	1.26	0.014	Mean = 1.25×10^4
321	1.31	0.058	
332	1.29	0.043	Mean Deviation $(x-\underline{x}) = 0.115 \times 10^4$
335	1.18	0.072	
339	1.34	0.087	% Difference = 9.2%
340	1.15	0.10	
341	1.08	0.17	
342	1.26	0.014	
368	1.08	0.172	
370	1.07	0.184	
570	1.50	0.242	

Table 2 (Cont'd)

Slope of Time-Temperature Curves for Tropical Room and 185°F Oven Test - 150 SOD Limit			
GTO	Slope (x) x 10^4	$x-\underline{x}$ x 10^4	
301	1.24	0.014	
305	0.95	0.027	Mean = 1.22 x 10^4
309	1.32	0.10	
340	1.12	0.10	Mean Deviation $(x-\underline{x})$ = 0.125 x 10^4
341	1.09	0.13	
342	1.35	0.10	% Difference = 10%
519	1.41	0.19	
570	1.29	0.07	

Table 3

Comparison of Predicted Storage Life and Actual Tropical Life				
	25 mg/in^2 Limit*		150 mg/in^2 Limit*	
Oil Code	Estimated	Actual	Estimated	Actual
1	0.39	0.44	0.80	0.83
2	0.39	0.72	1.60	1.20
4	1.30	0.92	-	-
5	1.20	0.90	-	-
7	0.07	0.05	0.26	0.24
8	0.21	0.18	0.90	0.81
9	0.13	0.40	-	-
11	0.50	0.40	0.90	0.90
14	0.55	0.38	0.90	0.94
20	0.24	0.30	1.40	1.44

* Measured in years.

Table 4

Tropical Room Storage Tests on Oil 20			
Months in Tropical Room	Total Months	Predicted Months to Reach SOD of 150	Corrected Months
12	14	20	18
33	39	50	44
31	37	50	44
33	39	50	44
15	39	50	26
26	30	34	30
29	35	40	34
29	29	38	38
8	31	38	15
25	31	38	32
8	31	36	13
12	31	38	19
25	31	38	32
25	31	39	33
25	25	34	34
20	22	29	27
20	22	31	29
23	29	31	25
23	29	28	22
19	20	26	25
19	20	30	29
18	20	25	23
18	26	26	18
16	20	28	24
4	7	16	13
21	27	33	27
9	27	33	15
21	27	33	27
21	27	33	27
21	27	38	32
21	21	34	34
17	18	23	22
16	18	30	28
14	18	28	24
14	18	24	20
14	18	31	27
14	18	25	21
17	23	36	30
17	23	37	31
15	17	40	38
15	17	42	40
14	17	28	25
14	17	40	37
14	17	30	27

Table 4 (Cont'd)

Months in Tropical Room	Total Months	Predicted Months to Reach SOD of 150	Corrected Months
14	17	27	24
13	14	24	23
13	14	39	38
13	14	25	24
10	14	24	20
10	14	20	16

Table 5

Tropical Room Storage Tests on Oil 9			
Months in Tropical Room	Total Months	Predicted Months to Reach SOD of 150	Corrected Months
14	20	29	23
21	22	28	27
21	22	28	27
17	38	47	26
16	38	47	25
17	38	45	24
12	17	23	18
12	17	26	21
12	16	23	19
12	14	22	20
15	36	44	23
13	36	42	19
16	17	23	22
10	15	22	17
8	12	21	17
11	32	39	18
11	12	20	19
6	11	20	15
4	30	35	9
9	30	35	14
4	9	14	9
4	8	16	12

Table 5 (Cont'd)

Months in Tropical Room	Total Months	Predicted Months to Reach SOD of 150	Corrected Months
Tropical Room Storage Tests on Oil 7			
10	14	18	14
6	9	12	9
6	8	12	10
8	10	14	12
8	10	14	12
8	10	13	11
8	10	14	12
8	10	14	12
8	10	13	11
8	10	11	9
4	8	15	11
4	7	12	9
6	8	13	11
Tropical Room Storage Tests on Oil 8			
8	8	13	13
5	5	10	10

Table 6

Summary of Tropical Room Results on Batch Samples of MIL-L-7808 Oils

		Months to Reach SOD of 150 (Corrected)			
Oil	Number of Batches Tested	Avg.	Max.	Min.	Avg. Spread
7	13	11.0	14	7	1.3
8	2	11.5	13	10	1.5
9	22	19.3	27	9	4.1
20	50	27.4	44	13	6.3

Table 7

325°F Extended SOD Test		
	SOD mg/in^2	
Time-Hours	Oil A	Oil B
16	7	0
20	9	0
24	12	-1
47	17	-2
70	24	-8
93	30	-18
115	34	-100
123	37	-345

Modified Oil Tests*		
	Neut. Nr.	
Time-Hours	Oil A	Oil B
24	0.16	0.63
72	0.16	0.63
96	0.32	0.63
120	0.32	0.63
144	0.30	0.60
168	0.30	0.60
192	0.30	0.60
240	0.30	0.75
264	0.30	0.75
288	0.30	0.45

* Catalysts of lead and copper were used. Lead added at 96 hours with no weight loss up to 192 hours.

Table 8

UV Test on Oil B		
Run #1		
Time-Hours	Neut. Nr.	SOD
0	0.5	
1.25	0.46	
3.25	0.48	
5.25	0.55	
12.25	0.79	
18	0.86	
Run #2 - Dry Air		
4	0.42	
19.75	0.55	
27	0.55	
47.25	0.54	0.0
52.5	0.92	-3.0
Run #3 - Saturated Air Plus 0.1% Water Added to Oil		
4	0.41	
10	0.54	
16	0.59	
21.5	0.66	
27	0.68	
32	0.70	
38	0.76	
45	0.81	
48	0.81	
52	1.00	
64	1.14	
68	1.01	
75	1.43	
83	1.29	
85	1.37	-4.0
94	1.39	
102	1.48	-5.0
110	1.58	
115	1.84	
127	1.93	
139	1.93	
165	2.14	
188	2.32	
202	2.66	
226	2.66	
250	3.59	-13.0

Table 9

Comparison of Esso and ASD Data on Number of Days to Reach SOD Nr.									
	Lead Loss,	185°F	215°F		230°F		255°F		260°F
Oil	mg/in^2	ASD	ASD	ASD	ASD	Esso	Esso	Esso	ASD
A	25	33	-	> 10	> 7	3.3	3.9	1.65	6.8
B	25	5	-	2.3	1.6	1	0.3	0.15	-
C	25	6	1.8	1.7	1.4	1.3	0.4	0.4	0.6
D	25	10	-	3.7	2.0	0.9	0.35	0.35	0.6
E	25	-	> 14	> 10	> 4	> 7	4.4	3.25	8.2
F	25	32.5	13.4	7.2	3.6	1.8	0.2	0.9	-
A	150	> 60	-	> 10	> 7	11.6	12	5	21
B	150	11	-	5.2	3.3	1.7	1.1	0.8	-
C	150	26	15.3	10.8	8.1	3.4	1.8	2.5	7
D	150	30.5	-	15	8.8	3.3	1.6	2.2	5.7
E	150	-	> 14	> 10	> 7	> 4	11.4	5.2	16
F	150	57	-	9.5	7	2.6	0.8	1.5	-

Table 10

Oil	185°F	215°F		230°F		255°F		260°F
	ASD	ASD	ASD	ASD	Esso	Esso	Esso	ASD
Comparison of Induction Time in Days								
A	14	-	7	7	2	2.4	1.2	5
B	4	-	2	1	1	0	0	-
C	0	-	0	0	0	0	0	0
D	6	-	2	0	0	0	0	0
E	-	14	10	4	7	3	3	7
F	28	7	7	3	1.5	0	0.8	-
Comparison of Slopes after Induction Period Slope - mg/in^2 per day								
A	1.3	-	NB*	NB	20	16	54	9.2
B	20	-	47.5	75	150	180	190	-
C	5.4	9.2	14	18.5	58	82	60	18.5
D	6.2	-	11	18	50	96	70	23
E	-	NB	NB	NB	NB	16	68	16
F	6.4	3.5	60	36	160	184	215	-

* NB - No break in curve.

Table 11

Repeatability of 230°F Oven Test Results

Esso Data

	Oil A			Oil B			
Days	Can 1	Can 2	Can 3	Can 1	Can 2	Can 3	Can 4
1	0	0	0	17	13	18	10
2	8	10	9	175	207	183	203
3	-	18	16	-	304	291	295
4	-	35	32	-	279	242	270
5	78	-	-	285	-	-	-
7	95	110	75	293	-	-	-
8	-	-	-	-	267	266	-
9	71	-	-	262	-	-	-
16	83	230	224	343	236	294	-

Oil A:

Cans 2 and 3 were run at the same time, side by side.
Can 1 was run at a different time.

Oil B:

Cans 2 and 3 were run at the same time, side by side.
Cans 1 and 4 were run at different times.

Table 12

215°F Oven Test on Oil B to Check Procedure					
		Can 1		Can 2	
Time Days	Previous Test	Shaken	SOD after Setting	Not Shaken	SOD after Setting
0	-4.0	-6.1	-	-5.0	-
1	-	-10.5	2 days -9.0	-12.2	2 days -9.2
2	-11.2	-	-	-	-
3	-65.5	-	-	-	-
4	-	-123.7	3 days -127.3	-129.7	3 days -146
7	-216.5	-201.5	-	-205.7	-

Representative Temperatures in 215°F Oven Test on Oil B*			
Time of Day	1st Day	2nd Day	3rd Day
8 am	221°F	212°F	215°F
11 am	218	216	216
2 am	214	214	214
3:30 pm	216	212	216

* Oil temperature in can was same as the oven temperature.

Table 13

Time-Temperature Slopes at Various SOD Values				
Oil	25 mg/in^2 x 10^4	50 mg/in^2 x 10^4	100 mg/in^2 x 10^4	150 mg/in^2 x 10^4
A	-	-	-	-
B	1.14	1.15	0.9	1.20
C	1.46	1.17	1.17	1.26
D	1.62	1.46	1.50	1.22
E	-	-	-	-
F	2.30	2.10	2.13	2.19
Avg.	1.63	1.47	1.43	1.47
Avg. without F	1.41	1.26	1.19	1.23

Temperature range 185°F -230°F

GTO 570 Temperature range 92-185-220°F 1.20 x 10^4

Double Rate for every 18°F rise 1.25 x 10^4

Table 14

Test on ASD Data*						
	SOD -60			SOD -120		
		Predicted	Actual		Predicted	Actual
Oil	185°F	230°F	230°F	185°F	230°F	230°F
B	7	2.1	2.0	10	3	3
C	11.5	3.5	3.3	20.3	6.2	6.6
D	16	4.9	4.0	26.2	8.0	7.4

Test on Esso Data*

SOD 150 mg/in^2				
	230°F	Predicted 255°F	Actual 255°F	
A	11.6	6.4	12	5.0
B	1.7	0.9	1.1	0.8
C	3.4	1.8	1.8	2.5
F	2.6	1.4	0.8	1.4

* Days

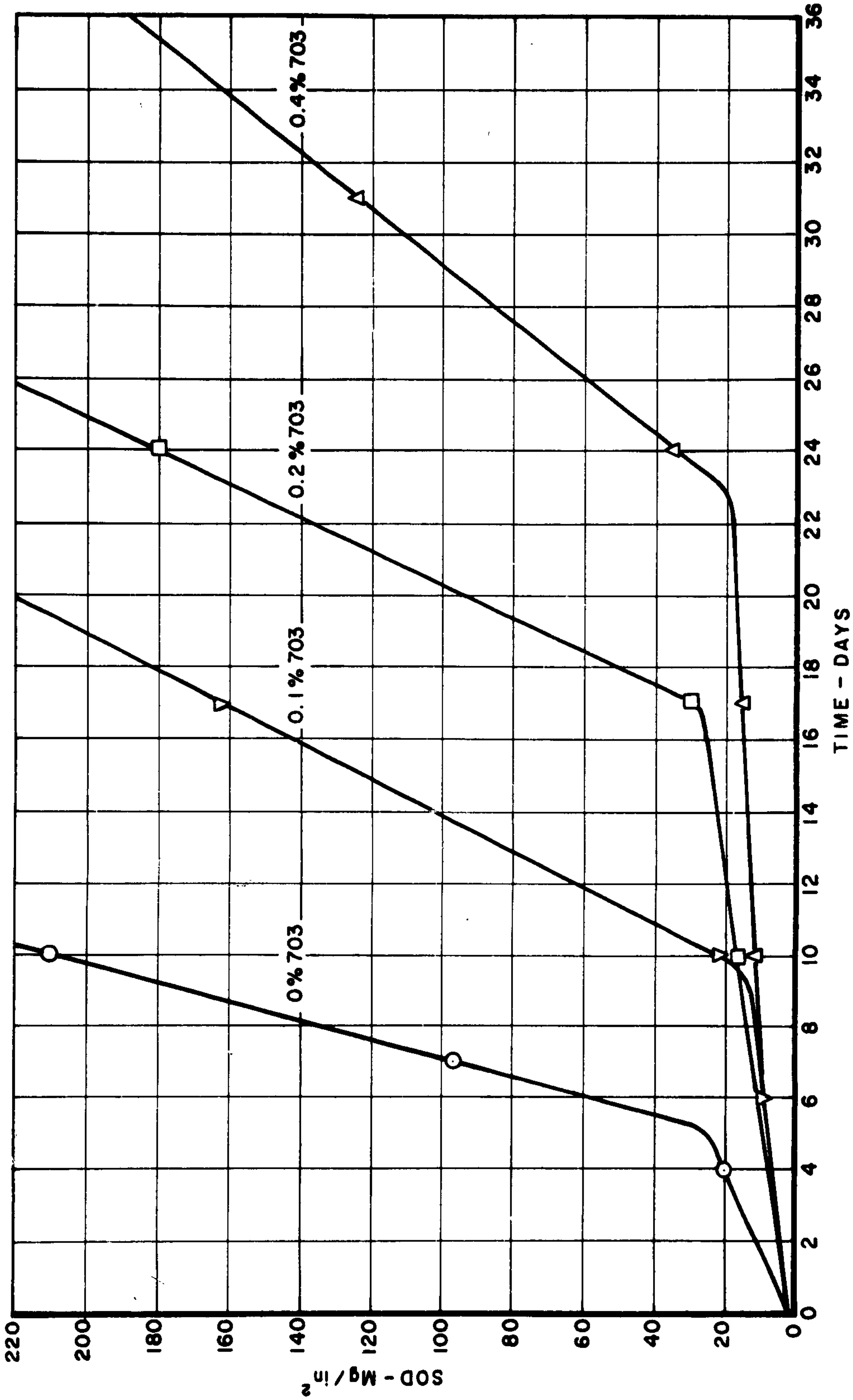

Figure 1. 200°F Oven Test on Oil B with Ethyl 703

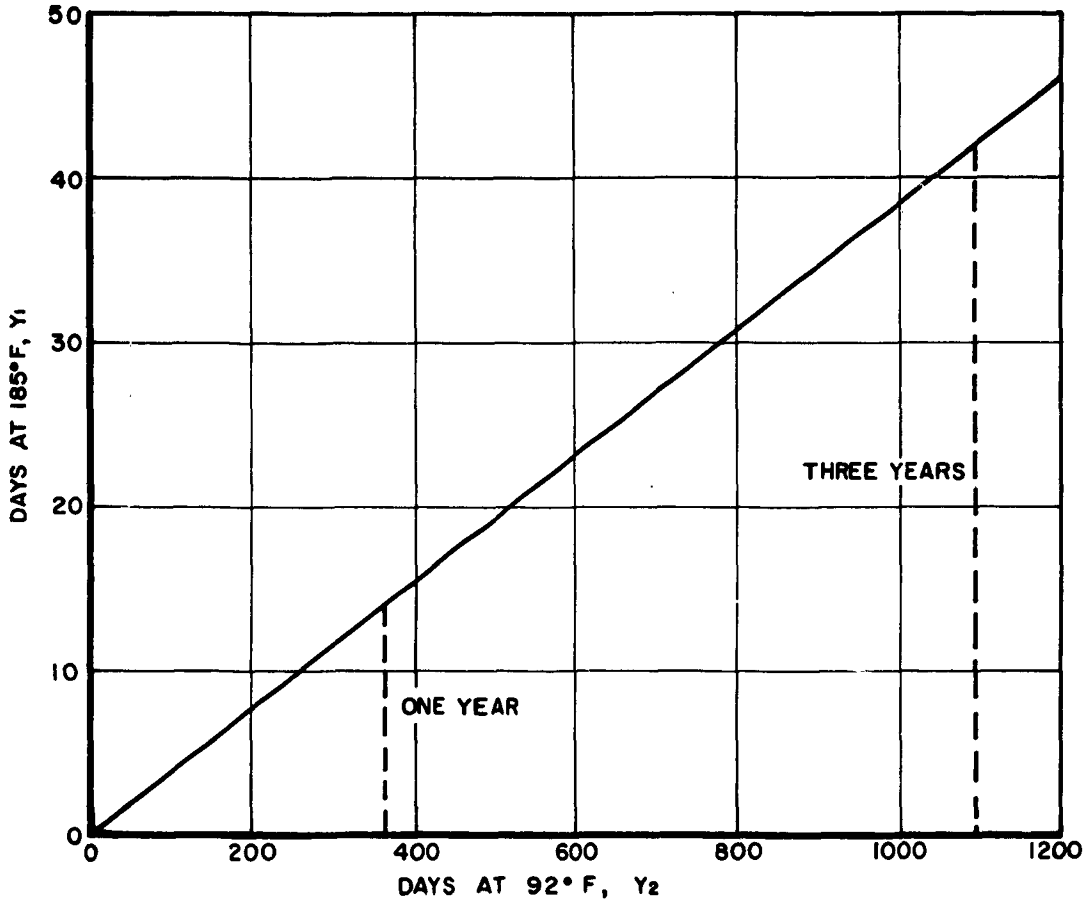

Figure 2. Relationship Between 185°F Oven Test and Tropical Room

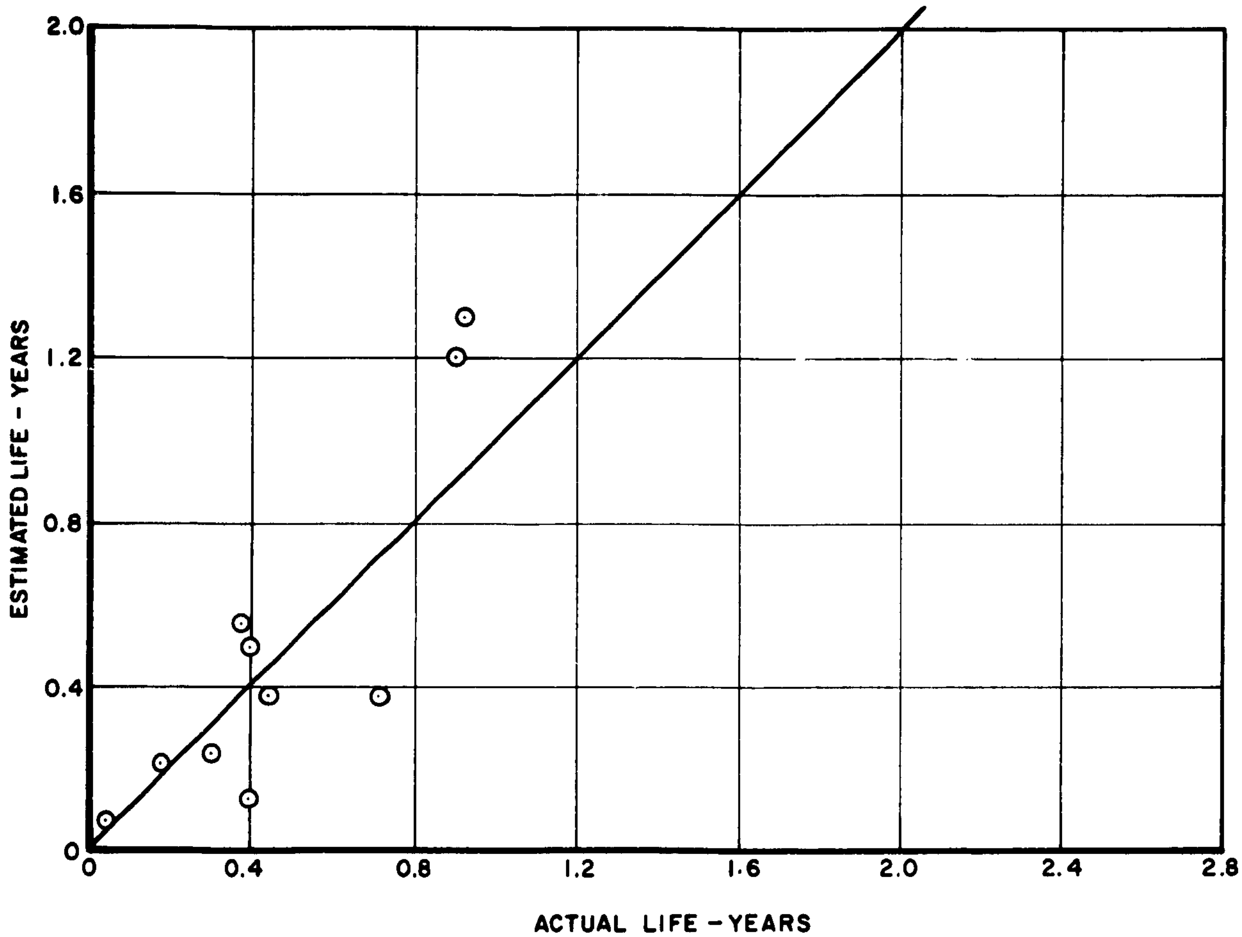

Figure 3. Comparison of Actual and Estimated Life for 25 SOD Limit

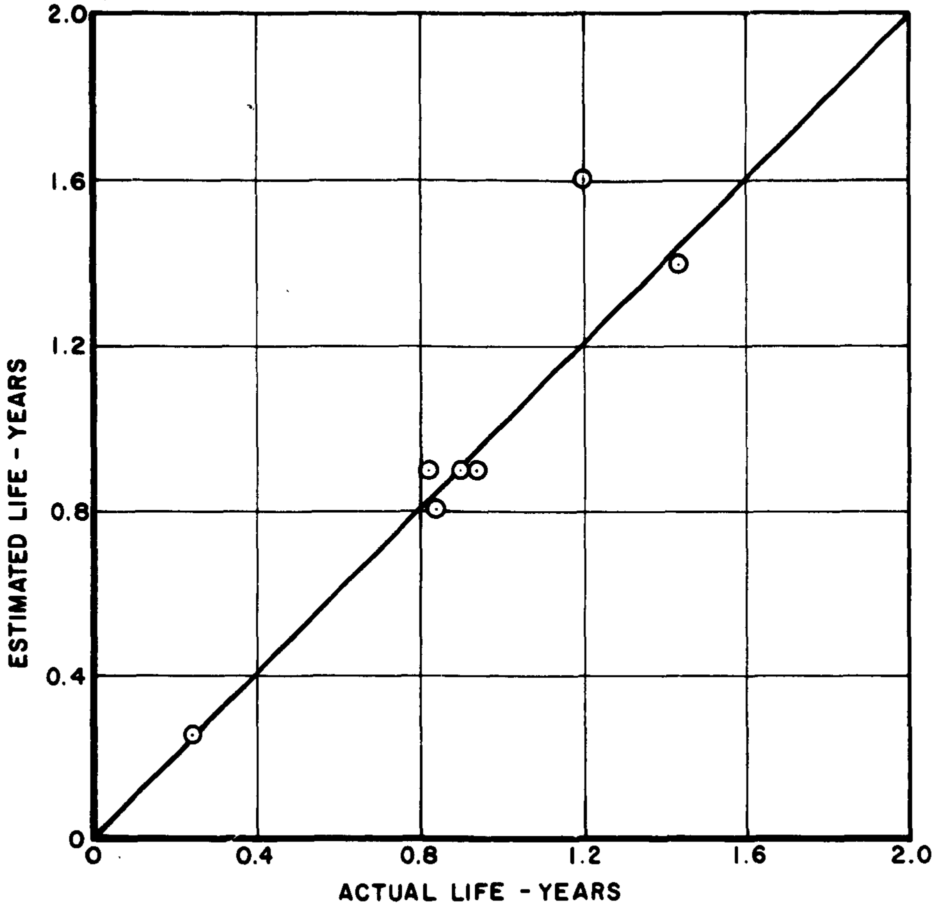

Figure 4. Comparison of Actual and Estimated Life for 150 SOD Limit

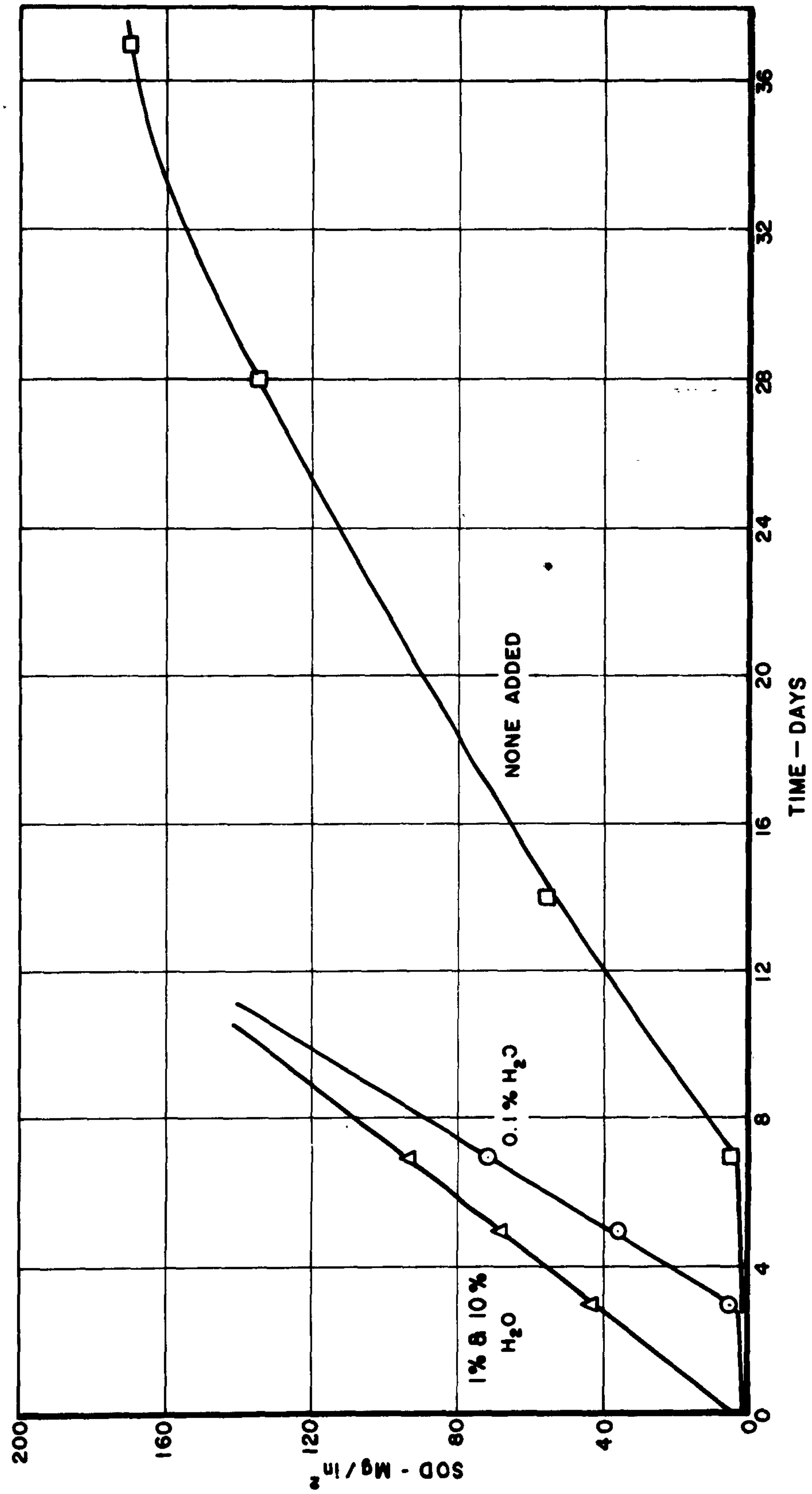

Figure 5. Effect of Added Water on Storage Life in 185°F Oven Test

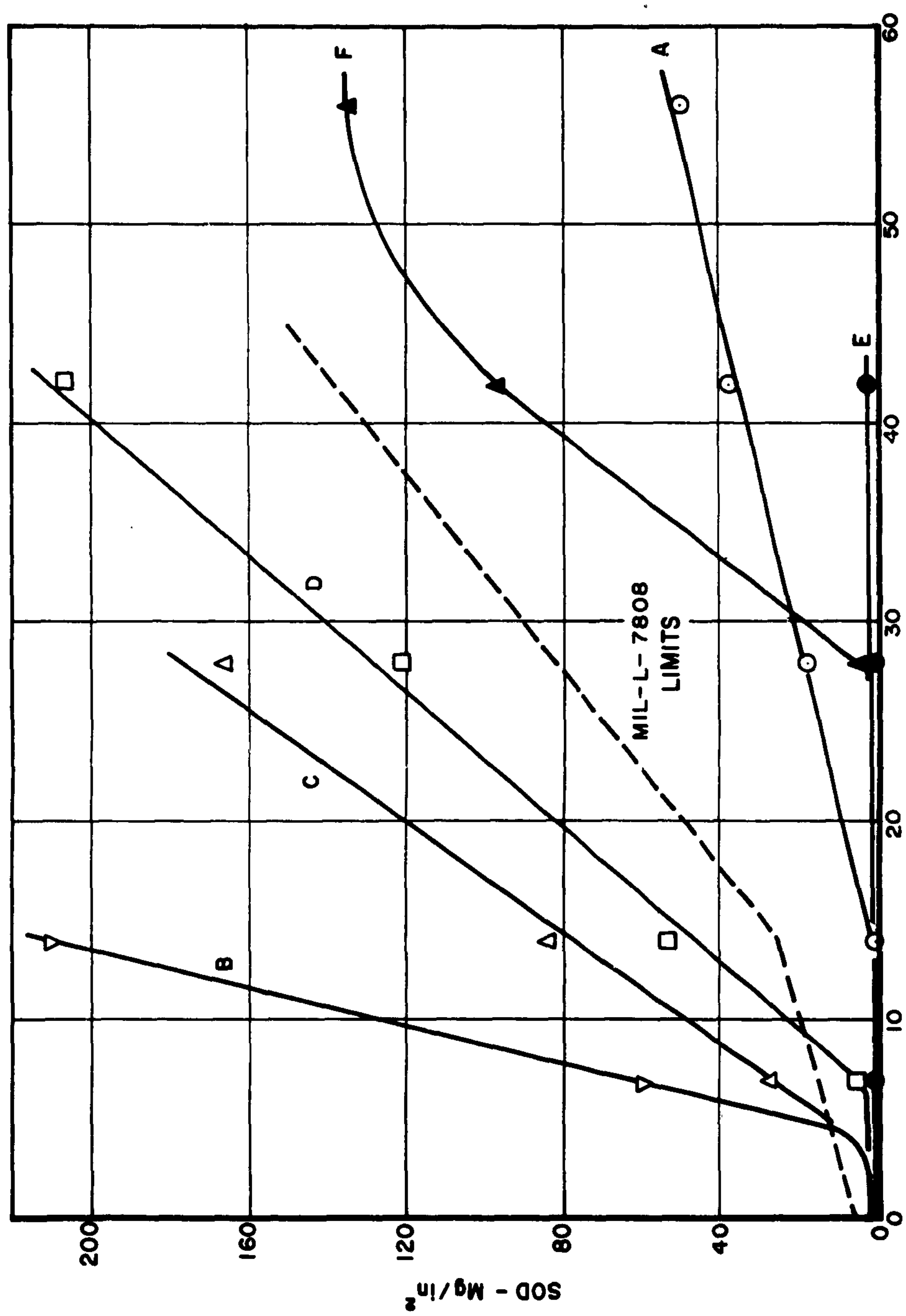

Figure 6. 185°F Oven Tests - ASD Data

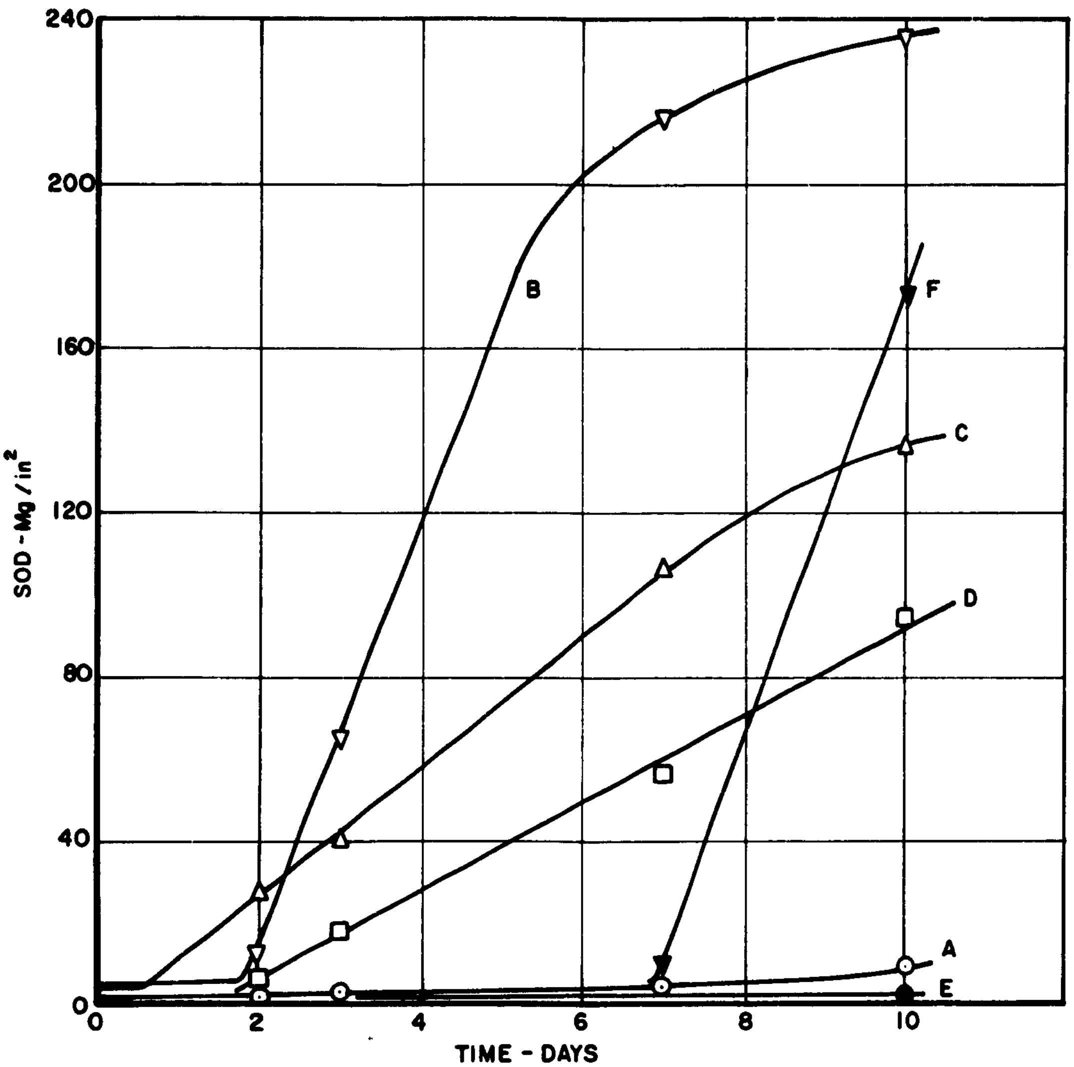

Figure 7. 215°F Oven Tests - ASD Data

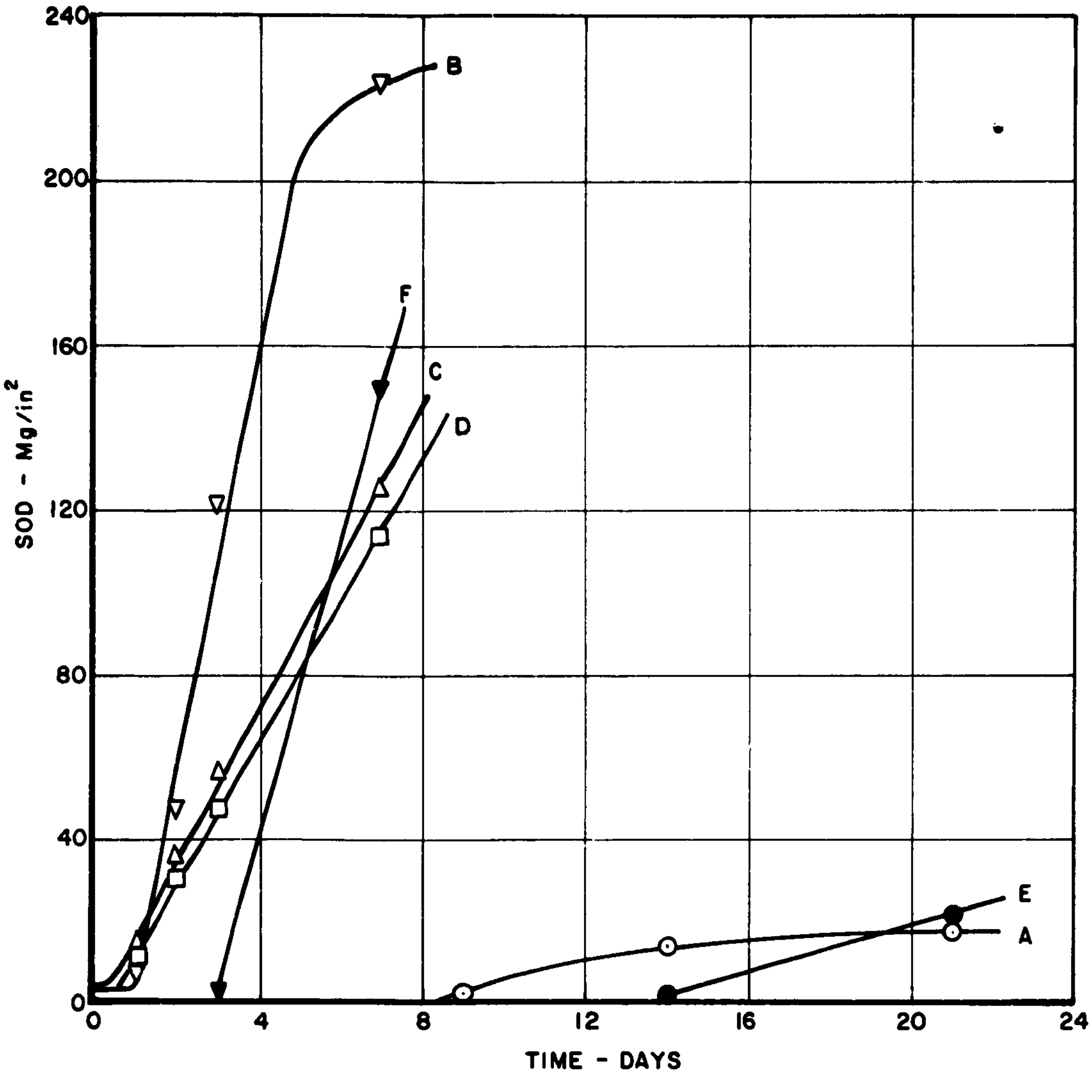

Figure 8. 230°F Oven Tests - ASD Data

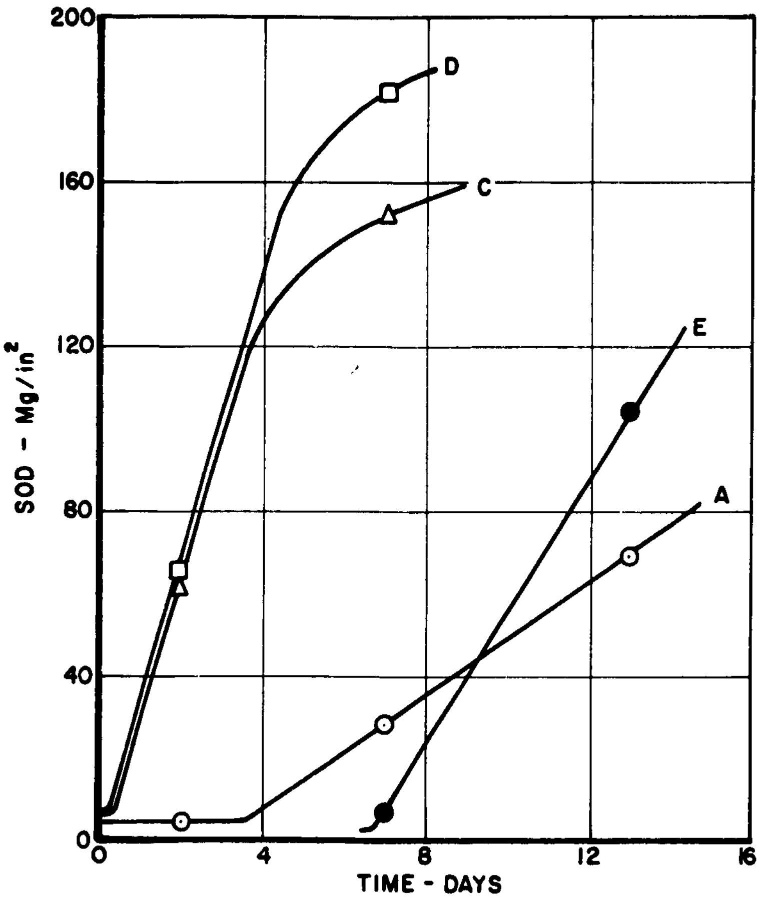

Figure 9. 260°F Oven Tests - ASD Data

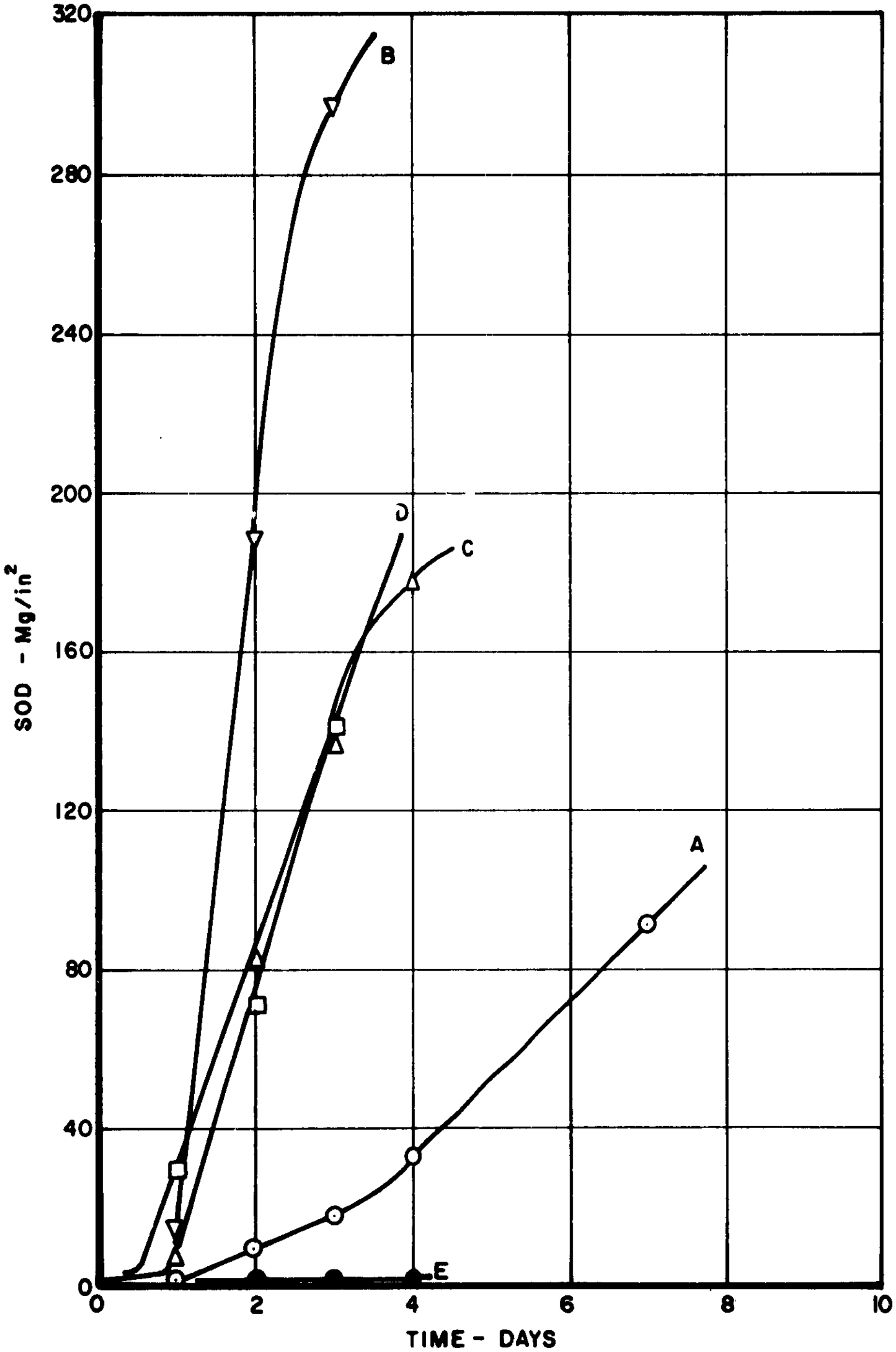

Figure 10. 230°F Oven Tests - Esso Data

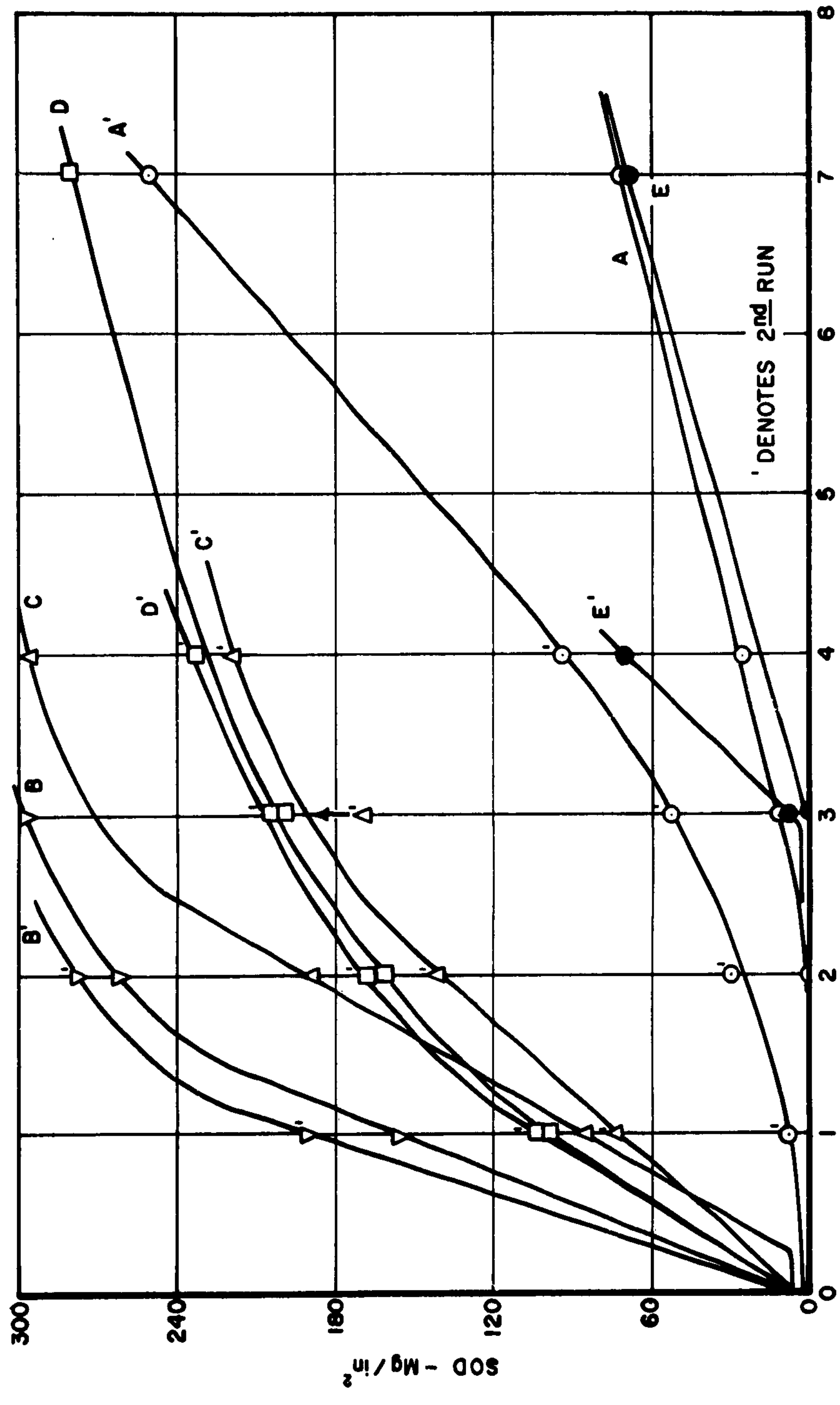

Figure 11. 255°F Oven Tests - Esso Data

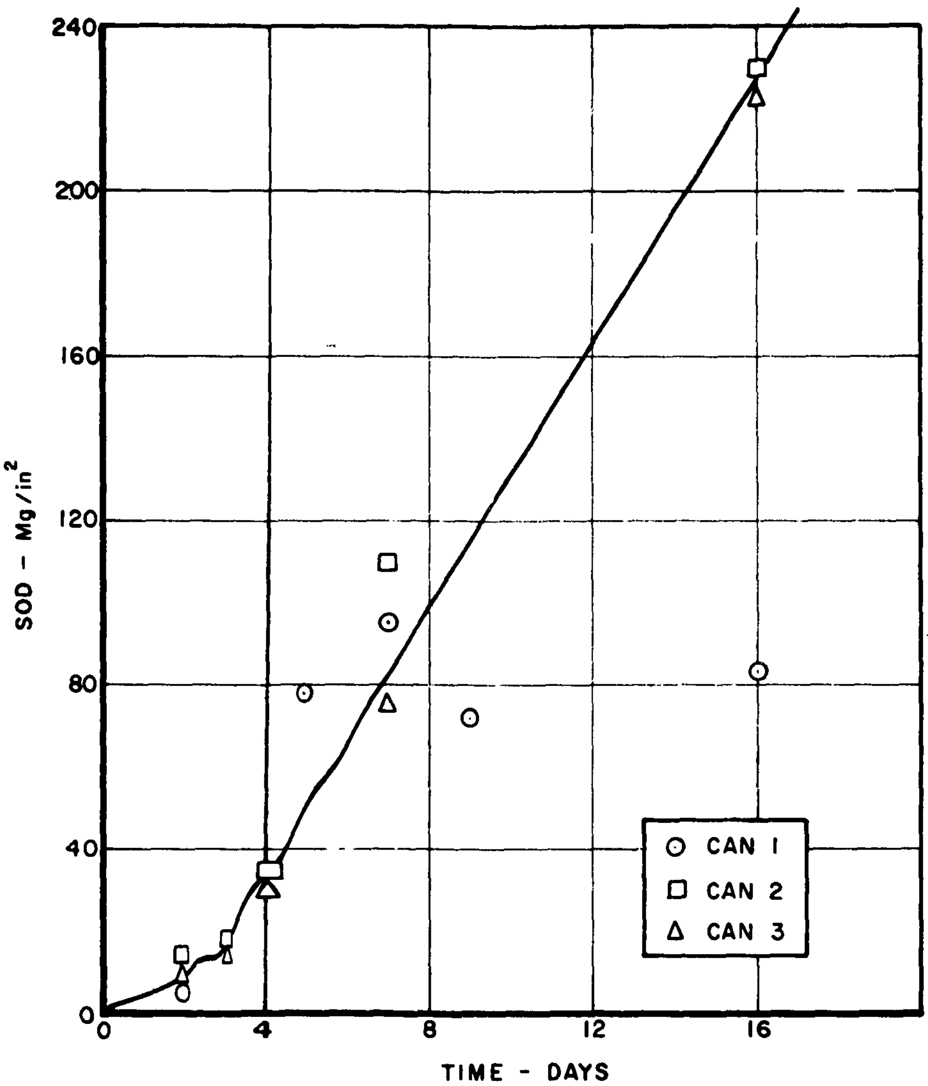

Figure 12. Repeatability of 230°F Oven Test on Oil A - Esso Data

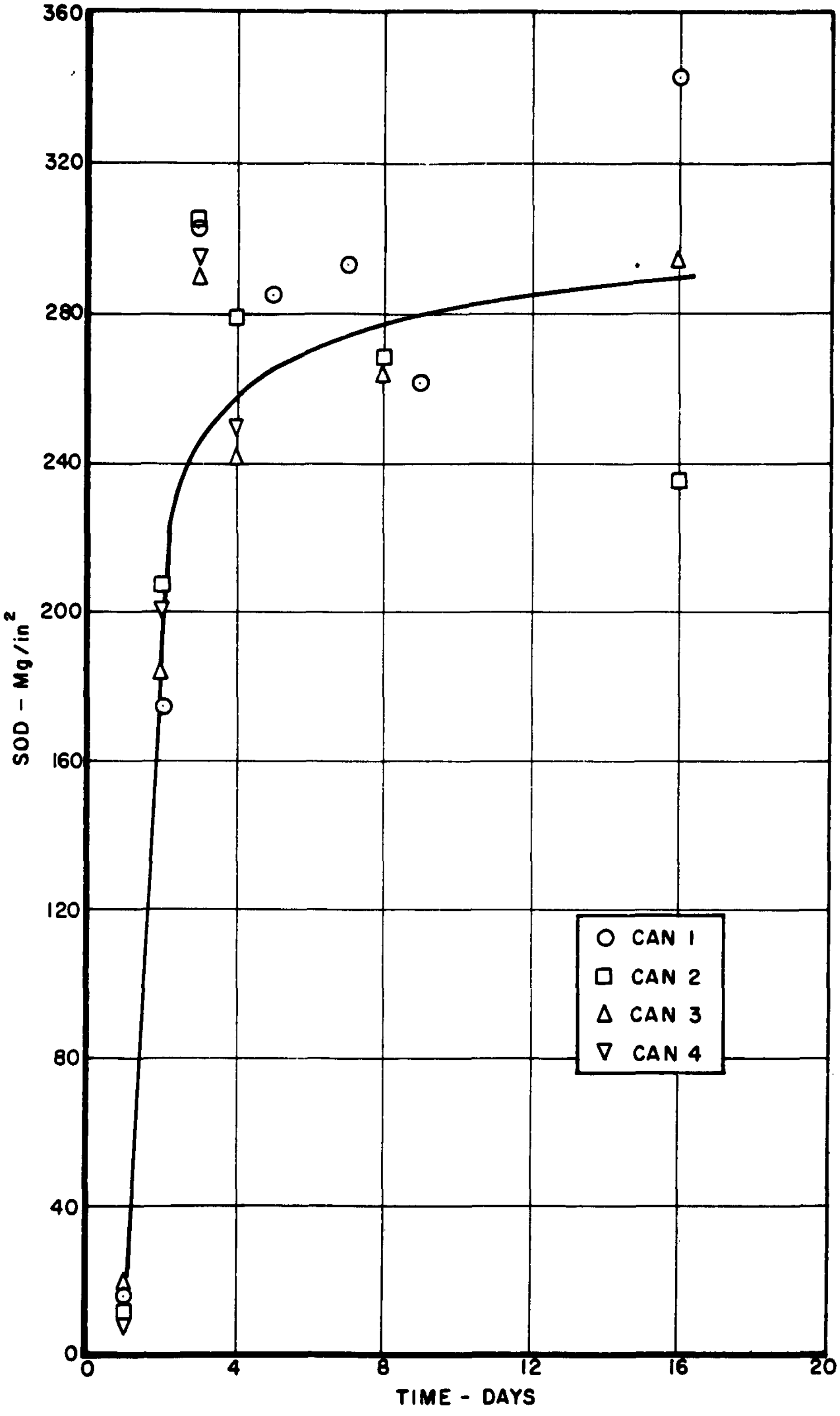

Figure 13. Repeatability of 230°F Oven Test on Oil B - Esso Data

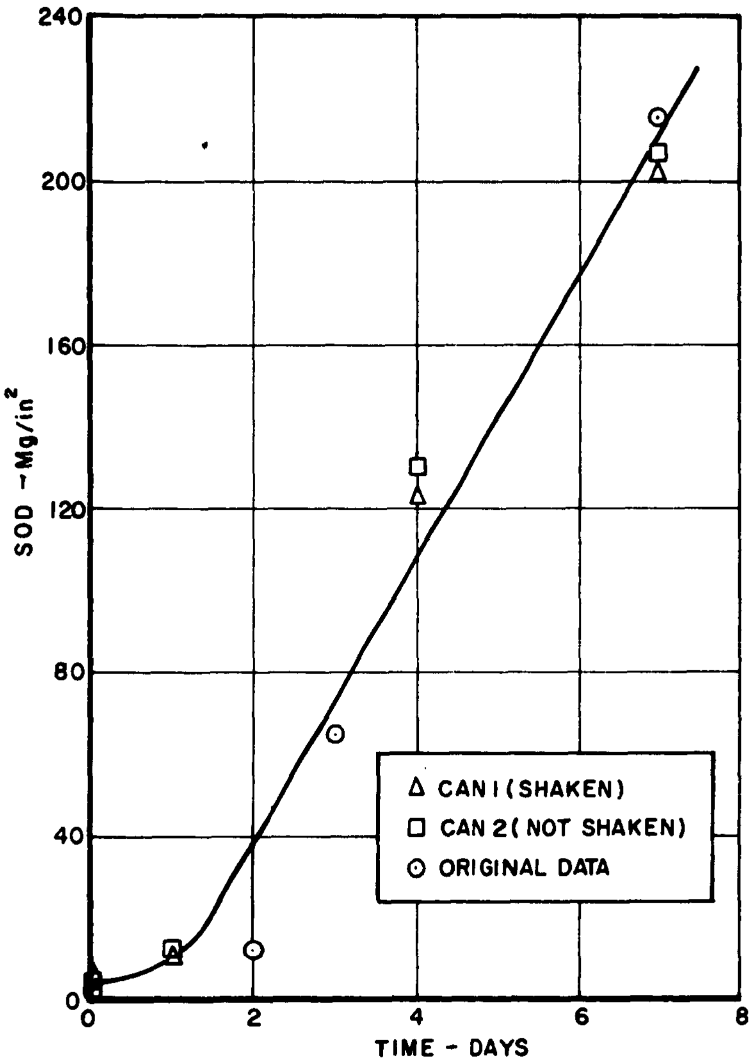

Figure 14. 215°F Oven Test on Oil B - ASD Data

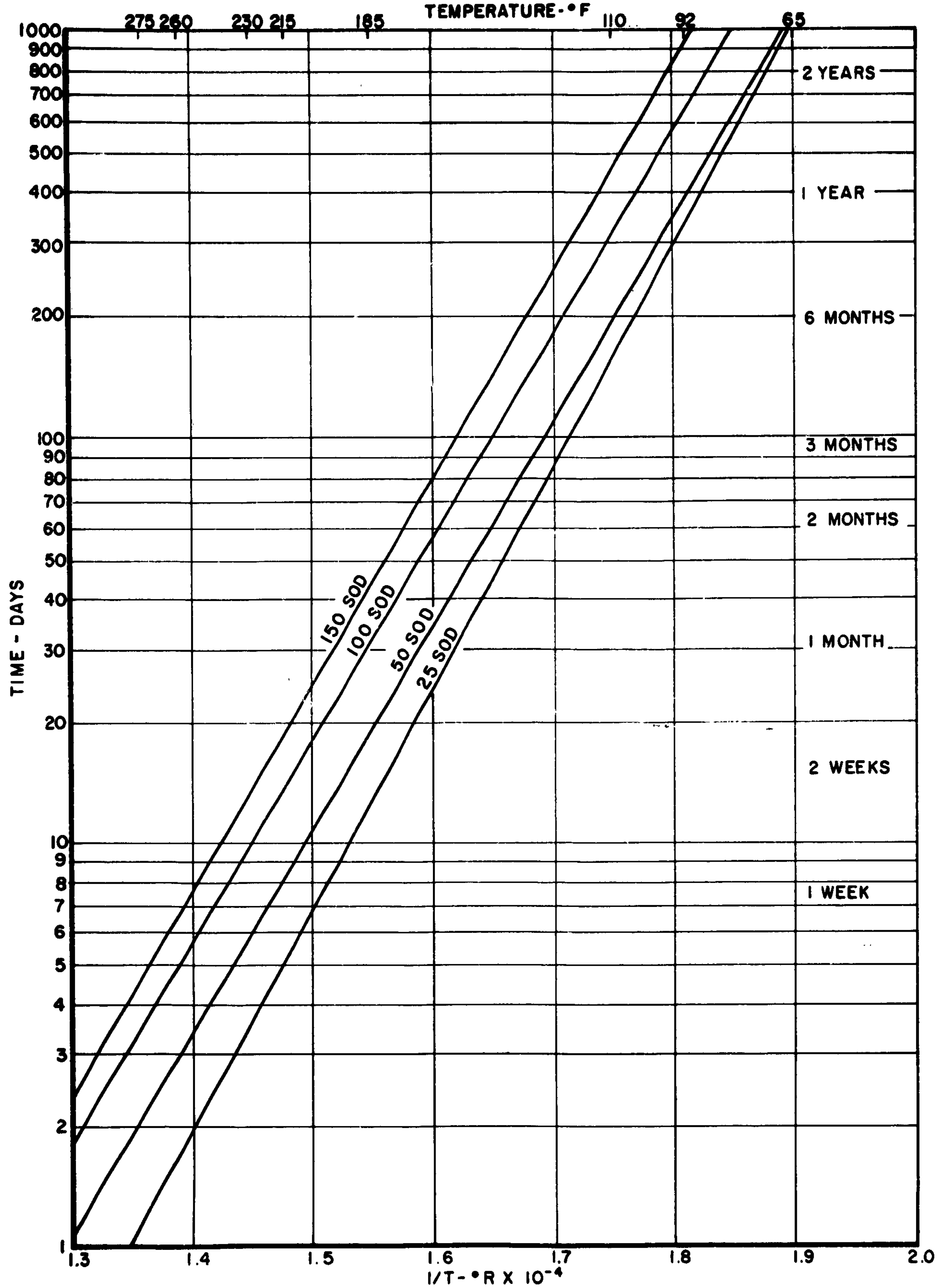

Figure 15. Effect of Temperature on Reaction

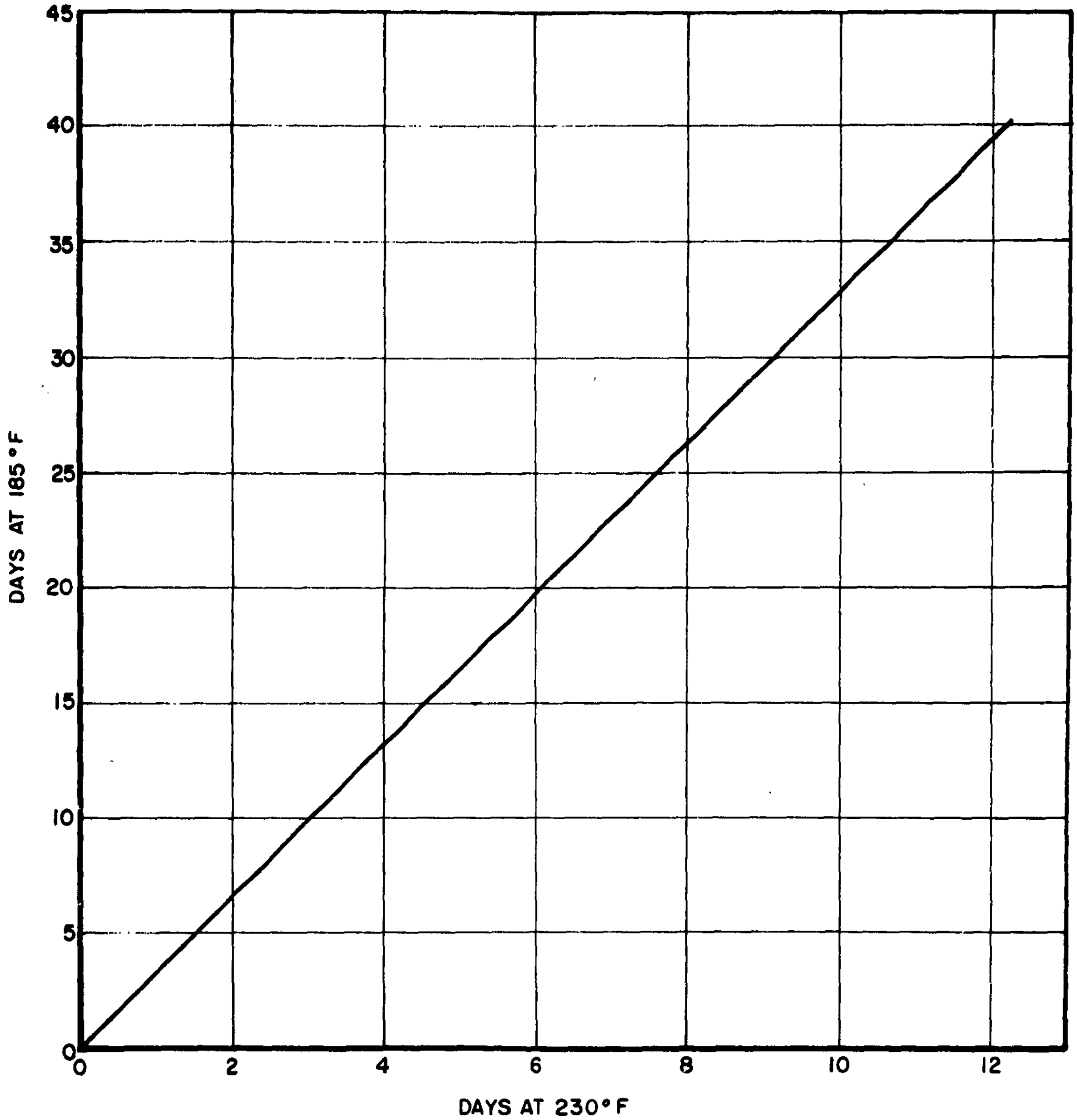

Figure 16. Relationship Between 185°F Oven Test and 230°F Oven Test

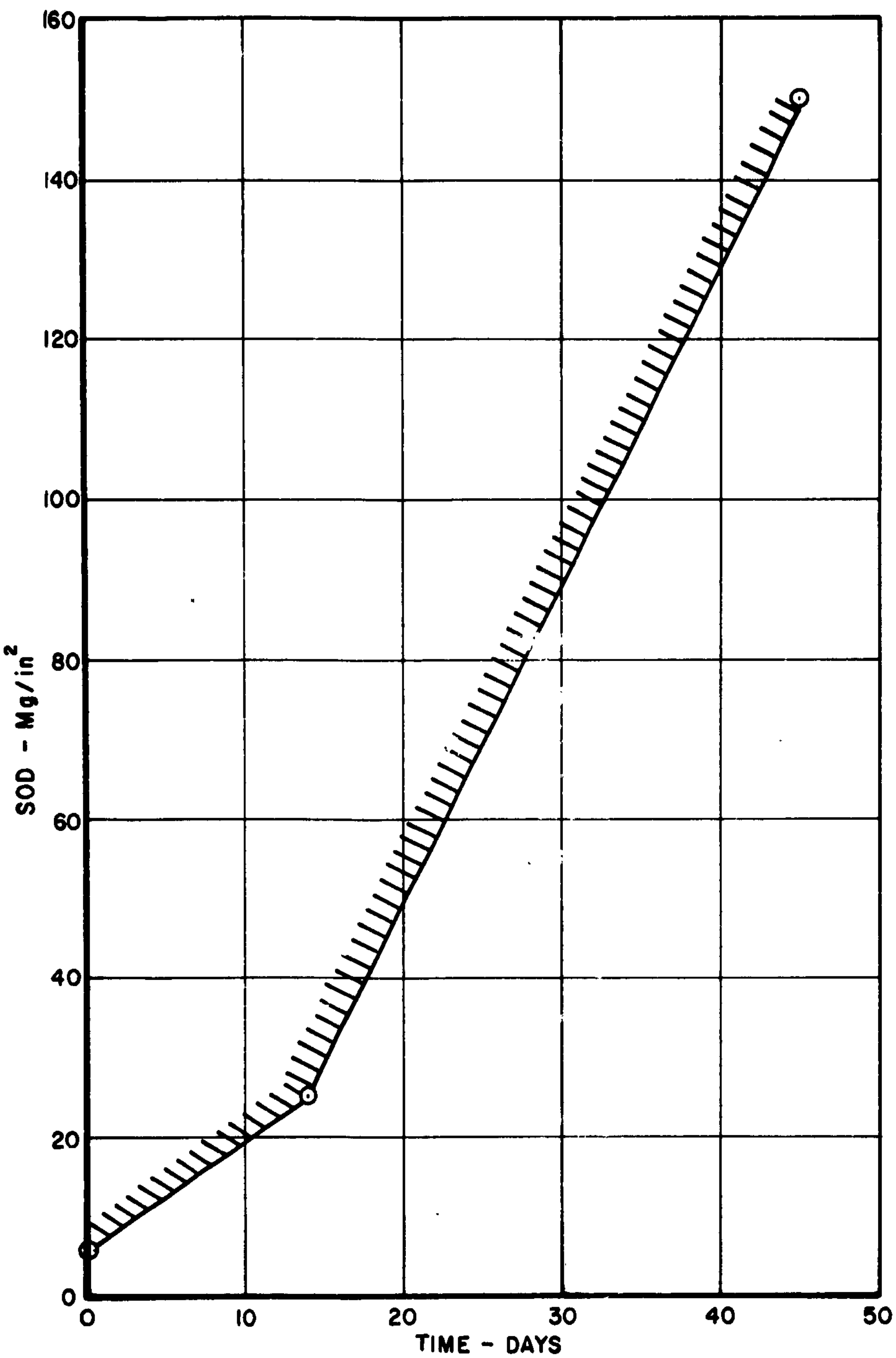

Figure 17. 185°F Oven Test Limits

Fuels and Lubrication Branch, Non-Metallic Materials Laboratory, Directorate of Materials and Processes, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.
ACCELERATED STORAGE STABILITY TESTS, by Eldred N. Cart, Jr., 1/Lt. USAF. Sep 1961. 46 p. incl. illus. (Proj. 3044; Task 30169) (ASD TR 61-144). Unclassified report.

The storage life of MIL-L-7808 oils has been improved by the use of amine type additives. This report describes the accelerated storage tests used to arrive at

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qualification limits. The time-temperature relationships for the storage life of MIL-L-7808 oils are given. From these relationships, the storage life can be estimated at any temperature from data at one given temperature.

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