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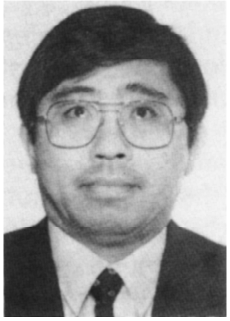

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Most military greases are formulated for use in severe service environments and multipurpose applications. To meet these requirements, the specifications usually require a wide operational-temperature range; excellent water and storage stability; good shear and oxidation stability; good antiwear and load carrying capacity; and rust and corrosion protection. These physical and chemical properties can change during storage and use. Most of these changes are interrelated and can eventually lead to lubrication failure.

Background: One of the most important grease properties is their oxidation stability. This chemical property is often used to determine grease service life in high-temperature applications.[1] The American Society for Testing and Materials (ASTM) D 942 test method, *Oxidation Stability of Lubricating Greases by the Oxygen Bomb Method*, has been widely used to assess storage and service oxidation stability of these military greases for several decades.

This test method takes a long time and has been criticized because the results have limited validity in predicting oxidation stability under service conditions.[2,3] For these reasons, it has become necessary to seek the development of a new oxidation stability test that would be capable of representing field performance within a short testing time.

Stability Evaluation: Pressure Differential Scanning Calorimeter (PDSC) is a thermal analytical technique developed to evaluate oxidation-thermal stability of polymer materials using the differential heat flow between sample and reference thermocouples under various temperatures and pressures. This technique is widely used in polymer quality control and research applications and is particularly useful for determining vapor pressures and the oxidation stability of lubricants.[4,5] The major advantages of the technique are:

- only a small sample required
- rapid procedure
- good sensitivity
- easy operation
- automatic data analysis
- flexibility

Because of these advantages, a program was initiated within the Army to examine PDSC as a standard oxidation stability test procedure for lubricating greases.[6] In preliminary investigations, it was found that numerous PDSC methods had been developed to determine the oxidation stability of engine oils, hydraulic fluids, and greases.[7,8,9,10] Common to all of these PDSC techniques were varying degrees of precision problems and difficulty in the measurement of oxidation stability.

To resolve these problems, a research effort was directed toward resetting the test parameters, improving the test procedures, and developing a theoretical approach. This paper describes the US Army's PDSC test method, its round robin test results, the kinetic study, and findings.

Test Method: The Army's method as developed was based on a *DuPont* dynamic pressure operation technique.11 The configuration of this test apparatus is a thermodynamically semi-open system, which consists of the *DuPont* Thermal analyzer, a PDSC cell, an oxygen cylinder, and a flowmeter. The schematic diagram of this test unit is shown in Figure 1.

The test procedure was designed for completion between 10 to 120 minutes using a test temperature which is selected from three specified temperatures — 210°C, 180°C, 155°C — depending on the type of lubricating grease being tested. In this procedure, the degree of oxidation stability at a given test temperature is determined by the induction time. Due to the chemical complexity of grease formulation, the oxidation stability of greases tends to depend upon the thermal stability and anti-oxidant level of the greases. Because of this, no single test temperature can measure the induction times for all greases.

Initially, 180°C was chosen as the test temperature, but many grease samples had induction times outside of the 10 to 120 minute range that was desired. Therefore, the additional upper test temperature (210°C) was selected to give reasonable induction times for greases having high oxidation stability.

This temperature, in fact, exceeds the occasional excursion temperature of wheel bearings observed in vehicles which operate under frequent stop-and-go service, or under severe braking service.[12] The additional lower temperature of 155°C was selected to accommodate greases with lower oxidation stability so their induction times would fall within the desired range of 10 to 120 minutes.

Prior to the test, the sample and control thermocouples were calibrated at the test conditions (oxygen pressure: 500 psig, purge rate: 100 ml/min). At this elevated pressure, there was some change in the melting point of indium, which was used for the standard calibration temperature, from that found at ambient pressure (156.6°C). Because of this occurrence, the sample thermocouple was calibrated at 157.6°C.

Test Procedure: To perform the test, 2.0±0.1 mg of grease is weighed into an aluminum sample pan (DuPont "SFI"). After the grease is spread evenly across the flat portion of the sample pan using the tips of the forceps, the sample pan is placed on the front platform in the cell. An empty pan also is placed as a reference on the rear platform. Then, the PDSC cell and pressure release valve are closed. To control the oxygen flow, the inlet valve on the cell front is opened approximately 1/8 turn, and a pressure of 500±25 psig is set, using a pressure regulator attached to the oxygen cylinder. The test is then started, using the isothermal mode. As soon as the test temperature

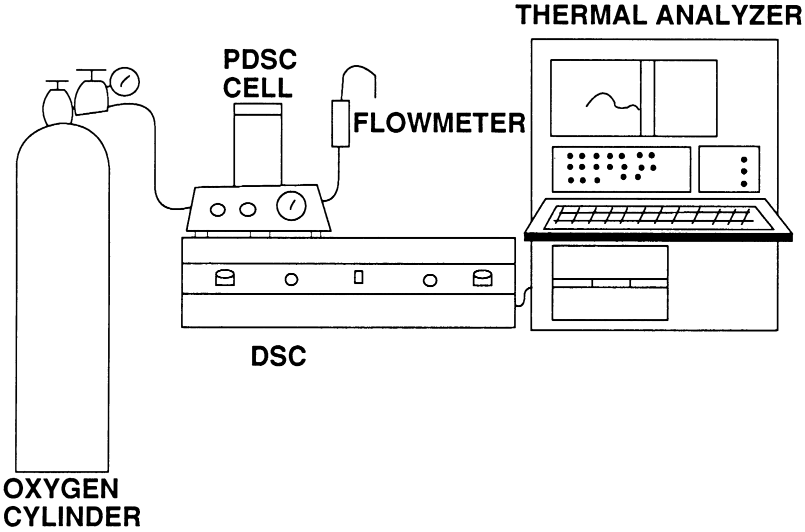

Figure 1 - PDSC T*est unit*

Table 1
Grease Identification and Properties

Code	Grease	NLGI Consistency Number	Dropping Points (^{o}C)	Base Oil Type	Thickener
A	MIL-G-10924F[16]	2	250	PAO+M	Li-Complex
B	MIL-G-23827B[17]	2	185	Diester	Li
C	MIL-G-81322D[18]	2	321	PAO	Clay
D	NLGI Reference System A, EP	1	199	M	Li
E	Commercial Grease I	2	265	M	Polyurea
F	MIL-G-10924[19]	2	149	M	Ca-Anhydrous
G	Commercial Grease II	2	247	M	Li-Complex
H	VV-G-671D[20] (Graphite)	2	130	M	Ca
I	MIL-G-81827[21] (MoS$_2$)	2	>340	PAO	Clay
J	Commercial Grease III, EP	2	295	M	A1-Complex
K	MIL-G-25537B[22]	2	150	M	Ca-Anhydrous

M: Mineral Oil
Li: Lithium 12-Hydroxystearate
Ca: Calcium Conventional
Li Complex: Lithium Complex
A1 Complex: Aluminum
PAO: Polyalphaolefin

reaches 500±25 psig, oxygen is slowly introduced into the test cell over 2 minutes, by slightly opening the cylinder valve. Then, the purge rate is adjusted to 100±10 ml/min, using the outlet valve. The test is continued until an oxidation exotherm is observed on the thermal analyzer scan. After the test is completed, the oxygen cylinder valve is closed and the pressure is slowly released by opening the pressure release valve.

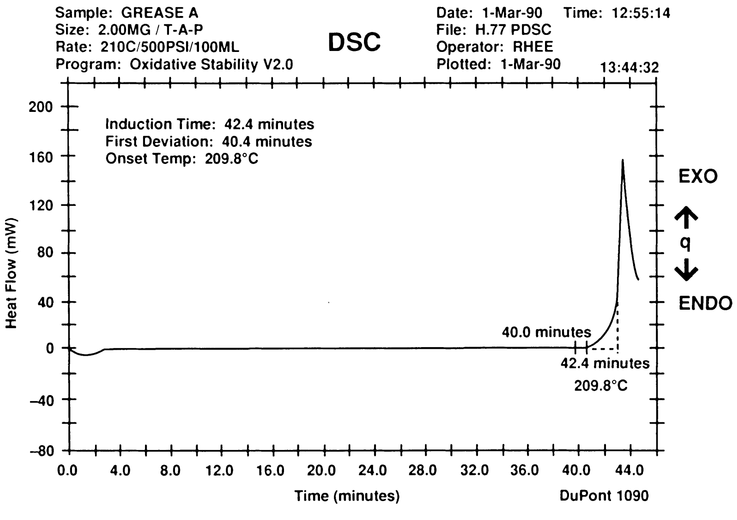

Figure 2 - Typical PDSC Thermograph

The induction time is determined from the extrapolated onset time, as shown for a typical thermogram in Figure 2. If the induction time was less than 10 minutes, the test was reconducted at the next lower temperature. For the next test, the cell was completely cooled to ambient temperature and cleaned, using a tissue wetted with toluene.

Equation Search: Oxidation of lubricants is an exothermic, thermodynamically irreversible reaction.[13] The oxidation stability test procedure developed for lubricating grease is based on thermodynamic theory and designed to measure induction times at various temperatures in a pressurized oxygen environment. The detailed oxidation mechanism of grease is not yet established, because of the chemical complexity of grease formulations and the complexity of oxidation reactions. For this reason, the rate equation of grease is usually determined by graphical methods which estimate the goodness-of-fit to the data.

In searching for a rate equation to fit a set of experimental data, four grease samples — A, B, F, and G (listed in Table 1) — were pretested at various temperatures. All the data values shown in Table 2 were plotted in Figure 3, using the Arrhenius model, which has a reciprocal scale for absolute temperature and a natural logarithmic scale for induction time.

This Arrhenius plotting technique is often used to determined whether the reaction can be fitted into the first-order rate equation which gives a reasonably straight line on this plotting.

Kinetic Equation: The resulting graphs show that the data points fall on straight lines and the slopes of these lines, proportional to the activation energy, are almost identical.

Based on this graphical method, the PDSC procedure developed for oxidation stability test was found to follow a first-order kinetic equation which is independent of the initial concentration of sample.[14] This kinetic equation may be expressed in terms of fractional conversion, X, and the rate constant, k, which depends on the temperatures.

$$t = \ln(1-X)/k \qquad [1]$$

where

t=induction time

In this reaction, the rate constant (k) is usually associated with the Arrhenius' law, which is based on a theoretical relationship between chemical reaction rates and temperatures.[15]

$$k = Z \exp(-E/RT) \qquad [2]$$

where

Z=frequency factor

E=activation factor

R=gas constant, 8.314 J/mol°K

T=absolute temperature, °K

To develop an oxidation kinetic equation for lubricating greases, Equation 2 is substituted into Equation 1, resulting in Equation 3.

$$t = -(1/Z) \ln(1-X) \exp(E/RT) \qquad [3]$$

The term $-(1/Z) \ln(1-X)$ is assumed constant and denoted as A. After taking the natural logarithm, the above equation can be expressed in Arrhenius form:

$$\ln t = \ln A + E/RT \qquad [4]$$

This kinetic equation actually represents the straight lines shown in Figure 3. To define the slope, the Equation 4 is differentiated with respect to 1/T. The differential equation becomes:

$$d \ln t/d(1/T) = E/R \qquad [5]$$

Activation Energy: E/R represents a slope of the Arrhenius equation and can be calculated, based on the experimental data in Table 2, using regression analysis. The statistical analysis shown in Table 3 clearly indicates that the greases tested provide almost identical activation energy and the only difference between them is in the value of constant A, which is the interception the Y (induction time) axis.

Based on these findings, it can be considered that the activation energy does not vary with the type of grease, in this exothermic reaction. Each grease has its own value of constant A, which is independent of temperature and totally dependent on oxidation stability. It therefore can be denoted as the oxidation coefficient of grease. From the average slope, 17500 K^{-1}, an approximate activation energy (E) of greases may be calculated to be 146 KJ/mol. Then, the oxidation kinetic equation can be rewritten from the Equation 4.

$$t = A \exp(17500/T) \qquad [6]$$

This kinetic equation was developed in Arrhenius form and the induction time (t) varies only with the temperature. For practical purposes, the oxidation coefficient of grease may be found by solving the Equation 6 for A, using a known induction time.

$$A = t_k / \exp(17500/T_k) \qquad [7]$$

where

t_k is a known induction time obtained at temperature T_k

Correlation of Induction Times: To verify the oxidation kinetic equation developed, numerous greases were examined and a correlation was made between the actual induction times and the predicted induction times. Table 4 demonstrates this kinetic study using grease A, and shows the predicted induction times for a series of temperatures. All the measured values for grease A (Table 2) were plotted on

Table 2
Oxidation Stability Test Results at Various Temperatures

Temperature	Induction Time (minutes)			
	Grease			
	A	B	F	G
230	11.2	–	–	–
220	22.1	8.1	–	–
210	43.8	16.5	–	4
205	66.2	22.7	–	–
200	102.9	35.7	–	8.6
195	152.3	–	–	–
190	–	75.2	5.9	19.6
185	320.9	121.1	–	–
180	496.6	194.6	13.3	43.0
175	740.2	–	20.6	68.3
170	–	468.9	31.5	105.0
165	–	–	49.1	163.6
160	–	–	83.7	–
155	–	–	133.9	388.3

Figure 4 to make a correlation with its predicted values (Table 4). The correlation coefficient (r) was found to be 0.99.

Round Robin Test: To determine the precision and review the test method, a round robin program was initiated with five cooperators, using eleven grease samples. The round robin was initially sponsored by the Department of Defense (DOD) laboratories and cooperating grease industry laboratories. The round robin samples consisted of six current military greases, three commercial greases, one federal specification grease and one NLGI Reference System A EP grease.

These samples were formulated with variable base oil types, thickeners, and different types of anti-oxidation additives or anti-oxidant levels. No experimental greases were included. Most of the military greases are currently being used in the field and give overall satisfactory performance in military applications. The identifications and physical properties of the selected greases are given in Table 1.

Statistical Analysis: The round robin test results are shown in Table 5. The test temperature for each sample, as used by the cooperators, is also listed, With the round robin data, the statistical analysis was conducted according to ASTM precision method RR D-2-1007.[23] In this statistical analysis, it was found that data obtained from Sample I in Laboratories 3 and 5 have been considered as outliers and rejected by Cochran's and Dixon's rejection criteria, respectively.

Therefore, all data points from sample I have been eliminated in accordance with the ASTM precision method. With ten samples, the precision of this test method was finally determined and its statistical analysis is shown in Table 6. The precision of this method, which is expressed in forms of the power retransformation, is as follows:

Repeatability (r) = $0.60\sqrt{m}$
Reproducibility (R) = $0.82\sqrt{m}$
where
m= the average of two test results

The round robin results showed excellent precision in all determinations, and gave a significant improvement in reproducibility. Previous applications of the PDSC technique to lubricants had a severe reproducibility problem in the oxidation-thermal stability test. A typical example is a draft ASTM procedure which is currently being developed by ASTM D02.09.E subcommittee.[9] The primary differences between the Army and ASTM methods are test temperatures, sequence of oxygen introduction, and method of measuring induction time. It now appears that the procedure developed by the Army has solved the past reproducibility problems that have occurred with the draft ASTM method.

Two Factors: The major factors that improve the reproducibility are the control thermocouple calibration and oxygen introduction time. To minimize the test temperature variation, the control thermocouple was calibrated (within) ±0.2°C. The oxidation introduction time also significantly contributes to the test precision. The developed test procedure requires

Table 3
The Statistical Analysis of the Tested Greases by Regression Analysis

Grease	A*10-16	Slope (E/R)	Activation Energy (E), KJ/mol
A	127	17300	144
B	15.2	17800	148
F	1.42	17700	147
G	11.2	17300	144

Table 4
The Predicted Induction Time of Grease A

Sample Name: Grease A
Induction Time (minutes): 43.8
Test Temperature ($^{\circ}$C): 210

Prediction Temperature ($^{\circ}$C)	Induction Time (minutes)
150	7471.9
155	4608.2
160	2874.0
165	1811.8
170	1154.2
175	742.7
180	482.6
185	316.5
190	209.5
195	139.9
200	94.2
205	64.0
210	43.8
215	30.2
220	21.0
225	14.7
230	10.4
235	7.4
240	5.3
245	3.8
250	2.7

oxygen to be introduced after the test temperature is reached. This approach gave better precision than introduction of oxygen at room temperature.

For these reasons, the Army's PDSC test method was proposed and subsequently adopted as a draft test procedure for ASTM D02.09.E Subsection Committee at the last ASTM meeting, held in June, 1990.[24] Furthermore, there was no evidence that different models of Thermal Analyzers affected the test precision or data. Thermal Analyzers used in this round robin were DuPont models 1090, 2000, 2100, and 9900.

In the round robin test, the data obtained from Sample I showed a large scatter. Particularly, the data generated from Laboratory 5 did not agree with those obtained from the other laboratories. To determine whether the sample preparation affected the test results, a study was conducted using two different sample surface areas. In this experiment, the first sample was uniformly spread across the flat portion of the SFI sample pan, according to the test procedure. The second sample was prepared intentionally on approximately half of the sample pan area with a thickened film.

Test Results: The test results clearly showed that the first sample gave a longer induction time than that obtained from the second sample, as shown in Figure 5. Also, the induction time (13.7 minutes) from the second sample agreed well with those of Laboratory 5 (12.9 minutes, 10.9 minutes). It can be deduced that the probable cause of the variable results with Sample I was due to sample preparation technique. Inadequate spreading of the sample will not only affect the test results, but also will increase the sample flammability, which could lead to possible damage of the test cell at high temperatures. Evidently, the second sample was completely consumed during the exothermic reaction.

To clarify whether this test procedure measures oxidation life or its anti-oxidant life, verification tests were conducted using Grease A. Its formulation consisted of a mixture of polyalphaolefin and mineral oil thickened with a lithium complex soap, and with the addition of various inhibitors, including an anti-oxidant. In order to make a comparison, induction times were determined for the neat anti-oxidant used in Grease A, the base oil and thickener without additives, and fully formulated grease.

The test results shown in Figure 6 reveal that Grease A (fully formulated) gave a significant improvement of its oxidation life and was distinct from the anti-oxidant life. Based on these results, it can be concluded that PDSC procedure actually measures the oxidation stability of greases.

Table 5
PDSC Round Robin Data

June 13, 1990

Grease Sample

Lab	A	B	C	D	E	F	G	H	I	J	K
1	40.0	13.2.	51.3	11.6	15.4	13.7	44.8	16.8	36.2	11.6	30.6
	45.3	13.6	48.1	11.3	14.8	16.0	46.8	17.3	31.6	10.3	31.3
2	41.3	15.1	48.8	10.4	15.9	14.0	39.5	17.2	30.5	11.2	31.4
	43.4	15.6	48.8	10.1	14.7	13.7	42.2	15.7	29.0	11.6	31.0
3	44.9	15.5	48.6	9.6	15.9	11.4	44.0	14.6	32.8	10.6	30.8
	41.9	17.2	47.4	9.2	13.9	13.5	43.0	14.9	24.0	9.0	31.2
4	42.4	15.5	48.8	10.2	14.9	13.6	42.7	16.5	28.8	10.7	33.9
	43.8	15.5	48.7	10.5	15.2	13.3	43.0	16.9	28.9	11.1	34.5
5	39.8	13.6	50.1	10.9	17.5	13.0	47.4	16.1	12.9	10.3	28.6
	42.5	13.3	49.7	11.3	15.5	13.9	45.0	14.6	10.5	10.1	28.7
Test temp. (°C)	210	210	210	210	210	180	180	180	210	210	155

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Bomb Tests: The ASTM D 942 test method determines the oxidation stability of grease using the net change in pressure resulting from consumption of oxygen by oxidation and gain in pressure due to formation of volatile oxidation by-products. To examine the significance of the method, the oxygen bomb tests were performed for 600 hours, using Greases A and F. These greases represented "F" and "D" revisions of MIL-G-10924, respectively.

To verify the oxidation of grease samples, their induction times were also measured before and after the bomb tests, using the PDSC procedure. Also, the previously NLGI round robin data for the Bomb Oxidation of Grease D, representing NLGI Reference System A, EP grease, were added into this data set.[25] The bomb test results are plotted in Figure 7 and the PDSC results are shown in Table 7.

Figure 7 clearly shows that Grease D gave less pressure drop than Greases A and F during the test periods. It implies that the Grease D provides a better oxidation stability than the other two greases. On the other hand, the PDSC results indicated that Grease A has a longer induction time (497 minutes) than that of Grease D (112 minutes). In addition, the induction times obtained from the Greases A and F were not significantly changed during the 600 hours of bomb tests.

Bearing Tests: To resolve the apparent contradiction between PDSC and ASTM D 942 results, all three greases were tested according to ASTM D 3527, *Life Performance of Automotive Wheel Bearing Grease*. Currently, this bearing test could be considered to be a dynamic oxidation test, since it is known that failure in this test comes early if the grease used is not stable to oxidation.[1] In ASTM D 3527 tests, the life of Greases A and D was determined to be 300 and 120 hours, respectively, while Grease F lasted only 20 hours. The PDSC induction time ranking of Grease A, D, and F was in the same sequence as the ranking from the ASTM D 3527 life test, clearly showing that, for these greases, long induction time closely corresponds to long life in the bearing test.

Based on these results, it appears that the variation of pressure in the bomb tests may give misleading indications as to the oxidation of greases, and it can be seen that there is no direct relationship between bomb and PDSC methods. A further indication of the PDSC method's validity is the qualitative observation that greases which are known from field experience to perform well under high temperature conditions all have relatively long induction times. However, correlative studies are planned using military vehicles to establish a more quantitative relationship between PDSC induction time and field performance.

Table 6. Statistical Analyisis of Round Robin Data

Laboratories: 5

Samples: 10

Variance Stability Test	DF	Limit	Table	Result
(a) Laboratory-Sample Means				
Trial Ratio = 0.2079	1, 8	5.318	4.1	Accept
(b) Replicate-Sample Means				
Trial Ratio: 0.0046	1, 8	5.318	4.1	Accept

Conchran Variance Rejection

Lab	Sample	Trial Ratio	DF	Limit	Table	Result
1	A	0.1520	49	0.198/0.252	5.2	Accept
1	All	0.3545	10. 5	0.412/0.470	5.3	Accept

Dixon Outlier Rejection 5 0.642/0.780 5.1

Sample	Lab	High Ratio	Lab	Low Ratio	Result
A	4	0.1272	5	0.5376	Accept
B	3	0.2752	1	0.0183	Accept
C	5	0.1066	3	0.3971	Accept
D	1	0.1638	3	0.4768	Accept
E	5	0.7376	3	0.1064	Accept
F	1	0.3969	3	0.4354	Accept
G	5	0.0727	2	0.3830	Accept
H	1	0.1473	3	0.2634	Accept
J	2	0.2777	3	0.2636	Accept
K	4	0.5298	5	0.4248	Accept

Precision Statement

	Std. Dev	DF	MDF	Power Retransformation
Repeatability:	0.1041	50	2.840	$r(x) = r(x') \mid m^{b}/(b-1) \mid$
Reproducibility:	0.1451	66	2.822	$R(x) = R(x') \mid m^{b}/(b-1) \mid$

Repeatability (r) $= 0.60 \sqrt{m}$

Reproducibility (R) $= 0.82 \sqrt{m}$

where

m: the average of two test results

Table 7

PDSC Test Results at 180°C, minutes

Grease	Fresh Sample	Sample after 600-Bomb Test	600-h Pressure Drop
A	497	497	12
D	112	- - -	4
F	13.3	5.5	17

Conclusion: A new oxidation stability test method for lubricating greases and its kinetic equation were developed using the Pressure Differential Scanning Calorimeter (PDSC). The round robin results showed excellent precision and clearly distinguished between differing formulations. Therefore, this method can be effectively used in quality control, and the research and development of new products. This test method has been adopted as an ASTM draft test procedure by ASTM D02.09.E Subsection Committee.

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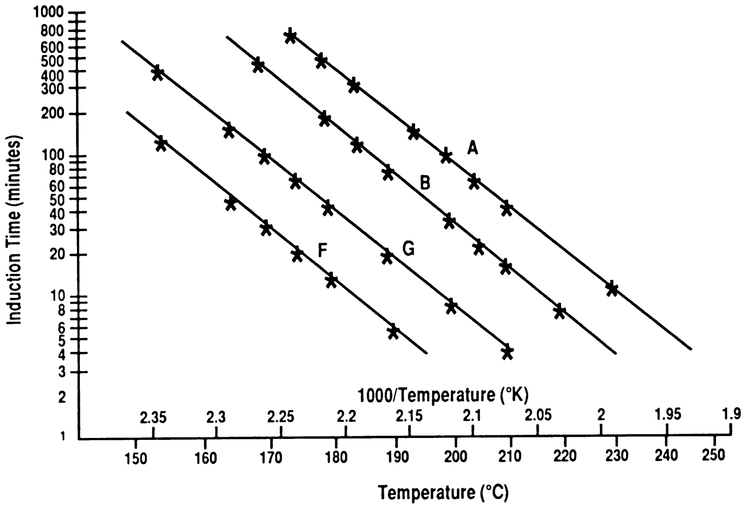

Figure 3 - Grease Oxidation Stability

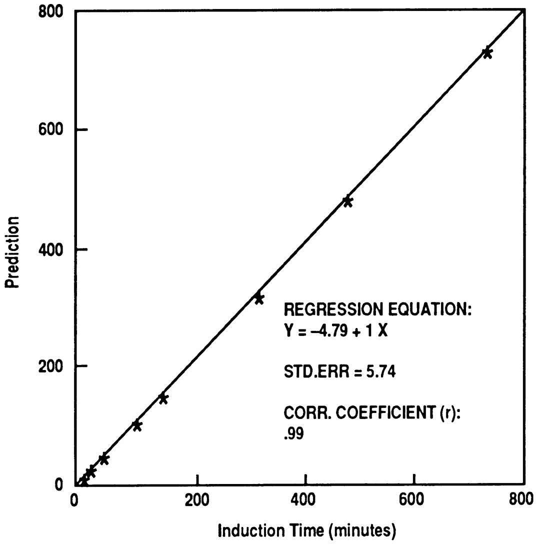

Figure 4 - Correlation of Actual vs. Predicted Induction Times for Grease A

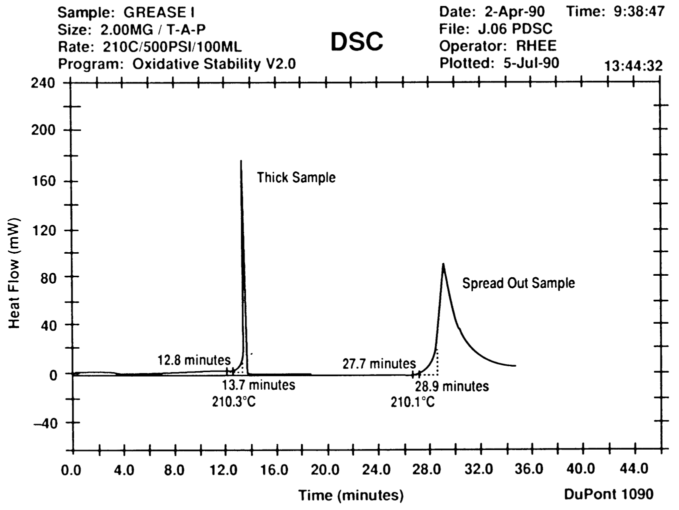

Figure 5 - The Oxidation Stability from Two Different Surface Areas of the Grease I

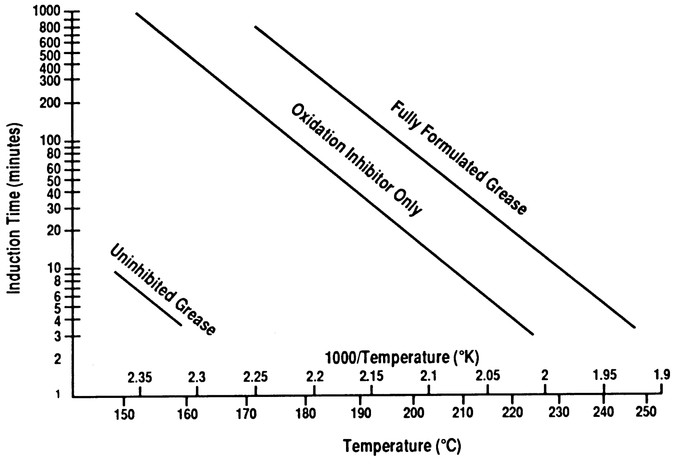

Figure 6 - Comparison of Induction Times for Grease A and its Components

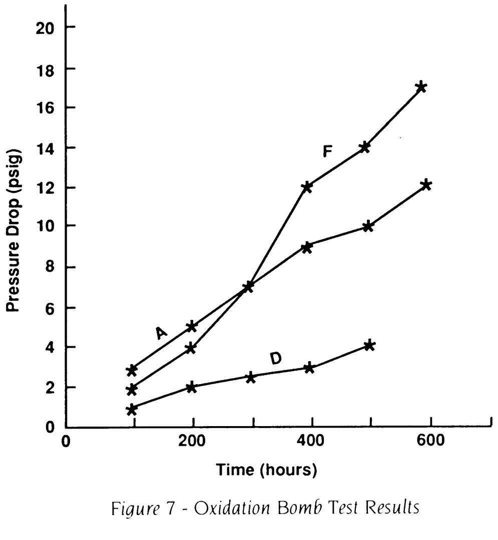

Figure 7 - Oxidation Bomb Test Results

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