

Shouldn't Grease Upper Operating Temperature Claims Have a Technical Basis?

Abstract:

Grease high-temperature claims based on different standards can vary widely. The range of approaches commonly used in the industry to define the maximum temperature at which a grease will provide adequate lubrication can be confusing for customers wishing to select the best product for their application. A lubrication decision based upon a published grease temperature range can lead to undesired consequences unless the user understands the basis for the high-temperature limit being claimed.

Factors limiting grease high-temperature performance include degradation due to oxidation, and / or the loss of base oil from bleed and evaporation. In general, dynamic grease life determinations based on standardised bearing tests, better represent what occurs in the field, providing a measure of grease high-temperature performance limits, which is more realistic than claims based on dropping point.

A test program was conducted on a variety of commercial greases, which included DIN 51821 FAG FE9 Life, ASTM D4290 Wheel Bearing Leakage, ASTM D2265 Dropping Point, and ASTM D5483 PDSC testing, and the test results were compared to product data sheet claims. Interesting discrepancies were found between product high temperature claims and their relative ratings based on FE9 or Wheel Bearing Leakage testing.

An industry standard approach to high temperature claims would be preferable to the various claims made by suppliers today. Such a basis would be far superior to the "rule-of-thumb" guidance provided in the NLGI Lubricating Grease Guide (Maximum Usable Temperature in the Grease Application Guide table), which is based solely on thickener type, and would benefit consumers and producers alike, reducing confusion in the marketplace. For example, not all lithium complex greases are the same. Complexing agents, manufacturing methods and base oil type can influence the high-temperature performance of a lithium complex, or for that matter, any high-temperature grease.

Introduction:

Historically, grease high temperature operating claims have been based on Dropping Point, thickener type, actual field experience, various laboratory bench or rig tests, or a combination of the

above. When basing upper operating temperature limits on Dropping Point, a margin of safety is usually applied, such that the recommended limit is some number of degrees below the dropping point. Various "rules of thumb" have been applied, such as 50oC below the dropping point.

Background:

Table 1 is an example of industry "generic" guidance, based on thickener type, extracted from the NLGI Grease Application Guide 1. Table 2 is a similar example, extracted from an ExxonMobil grease training module 2.

Table 1 Example: NLGI Grease Application Guide

Thickener Type	Dropping Point °C	Maximum Usable Temperature, °C
Simple Lithium	175	135
Lithium Complex	260+	177
Polyurea	243	177
Organic Clay	260+	177
Conventional Calcium	96 - 104	93
Anhydrous Calcium	135 - 143	110
Calcium Complex	260+	177
Aluminum Complex	260+	177
Sodium	163 - 177	121

Table 2 Historical ExxonMobil Guidance

Thickener Type	Dropping Point °C	Maximum Service Temperature °C
Simple Lithium	175	120 to 135
Lithium Complex	250+	150 to 175
Polyurea	250+	180 to 200
Modified Clay	280+	190 to 220
Calcium Sulfonate	260+	150 to 175
Lime Soap	90	60 to 70
Anhydrous Calcium	140	90 to 110
Calcium Complex	260+	150 to 175
Aluminum Complex	260+	150 to 175
Sodium	190	135 to 150
Sodium Complex	250+	150 to 175

Examination of the "deltas" between the reported typical dropping points and the recommended maximum service temperatures in these two examples illustrates the inconsistency in this approach:

In the NLGI guidance, some thickener types are given a “conservative” maximum usable temperature of more than 83°C below the dropping point. For other thickener types, the maximum usable temperature is as close as 3 to 11°C below the dropping point. Excluding conventional and anhydrous calcium greases, the average delta is about 56°C.

In the ExxonMobil guidance, some thickener types are given a “conservative” maximum service temperature of between 85 to 110°C below the dropping point. For other thickener types, the maximum service temperature is as close as 20 to 30°C below the dropping point. Excluding lime and anhydrous calcium greases, the average delta is about 80°C.

So, even by these more traditional approaches, there is considerable difference in the maximum operating temperature recommendations given by different industry sources. It is no wonder that end users may be a bit confused by these conflicting recommendations.

A selection of Product Data sheet claims was assembled to examine how various grease marketers represent the upper temperature limitations of their products. Table 3 is a summary of the claims made for nine commercial greases. The claims are made in a variety of ways, including statements of both upper and lower operating temperature limits, sometimes including a test result as the basis for the claims, DIN 51825 Classifications, and in some cases, no upper operating temperature limits were listed.

Table 3 Selection of Product Data Sheet Claims

It is apparent that the range of approaches commonly used in the industry to define the maximum temperature at which a grease will provide adequate lubrication can be confusing for customers wishing to select the best product for their application.

So, what are the factors which limit grease high temperature performance? Any of the following mechanisms may be involved when a grease fails due to high temperatures 3: -

1. Loss of base oil due to excessive bleeding (separation from thickener) at elevated temperatures, or shear, or a combination of the two.
2. Loss of base oil due to evaporation due to excessive volatility at elevated temperatures.
3. Degradation of base oil or thickener due to oxidation at elevated temperatures.
4. Irreversible fluidisation at or above the dropping point temperature.

Given these different potential mechanisms for failure at high temperatures, what is the best test to predict performance?

Test Methods:

Examining the available industry standard test methods for greases at high temperatures, we can broadly categorize them into static and dynamic tests.

Static heat resistance tests are useful for comparing greases to one another, predicting grease life in storage conditions, and for controlling product quality during manufacture. These tests do not correlate with dynamic service conditions. Static heat resistance is dependent on thickener type, base oil type (and in some tests, viscosity), antioxidant additives and temperature. A listing of the more common static heat resistance tests is given in Table 4.

Table 4 Static Heat Resistance Tests

Test Method	Description
Dropping Point (ASTM D2266 or D566)	Identifies thickener’s high temperature properties
Evaporation Loss (ASTM D972 and D2595)	Measures oil and additive high temperature volatility
Bomb Oxidation (ASTM D942)	Resistance to moderate temperature and oxygen
PDSC (ASTM D 5483)	Resistance to heat and oxygen

The **dropping point** of a grease is the temperature at which the thickener can no longer hold the base oil, under the static conditions of the method.

The **evaporation loss** test measures the amount of weight loss of either volatile additives or base fluid from the grease under the static conditions of the method.

The **bomb oxidation** test evaluates the resistance of a grease to oxidation by measuring the consumption of oxygen under the static conditions of the method.

The **Pressure Differential Scanning Calorimeter (PDSC)** evaluates oxidation stability as the induction time at a selected temperature, under the static conditions of the method.

Dynamic heat resistance as determined in bearing rig tests can provide a good indication of how greases will perform in actual applications. These tests measure the life of the grease rather than the life of the bearing, can simulate a variety of real life applications, and use the same bearings used in the actual applications. Dynamic grease life is dependent on base oil type and viscosity, thickener type, antioxidant additives and grease structural stability/oil release properties. It is also a function of the dynamic test conditions, including bearing geometry, temperature, speed, load, load direction, and seal design. A listing of the more common dynamic life tests is given in Table 5.

Table 5 Dynamic Life Tests

Test Method	Description
FAG FE9 (DIN 51821)	High Speed angular contact ball bearing
High Temperature Wheel Bearing Life (ASTM D3527)	Tapered roller bearing used in automotive applications
SKF R0F (DIN 51806)	Deep groove ball bearings
POPE Test (ASTM D3336)	Deep groove ball bearing

The **FAG FE9** test uses five angular ball bearings, rotating at 6000 rpm, under an axial load (usually 1500N). Test temperature is selected (120 to 200°C), and time to failure is based on a two-fold increase in the power requirement to rotate the bearing. The times at which the bearings have a failure probability of 10% and 50% (denoted by L10 and L50) are calculated from the data by using Weibull analysis. This method is the basis for upper operating temperature claims in the DIN (Deutsches Institut für Normung) 51825 grease classification system.

In the **High Temperature Wheel Bearing Life** test, two automotive type tapered roller bearings are rotated at 1000 rpm, under an axial load (111N). The test temperature is 160°C, and the test continues in repeated cycles of 20 hours rotating followed by 4 hours of no rotation, until the power requirement increases to four times the steady state value. Hours to failure is reported. This test is included in the NLGI GC wheel bearing grease certification test requirements as found in ASTM D4950 4.

The **SKF R0F+** test uses five deep groove ball bearings, rotated

at 10,000 rpm, under a combined axial (100N) and radial (50N) loads. Test temperature is selected between ambient and 170°C, and the test is run until the temperature increases by 20°C over the steady state. The times at which the bearings have a failure probability of 10% and 50% (denoted by L10 and L50) are calculated from the data by using Weibull analysis.

The **POPE** test is similar to the SKF R0F test, in that it uses five deep groove ball bearings, rotated at 10,000 rpm. However, it uses a light axial load (22N), and a 20 hour on, 4 hours off operating cycle (like the Wheel Bearing Leakage test). End of test occurs when the power requirement increases to three times the steady state value. The times at which the bearings have a failure probability of 10% and 50% (denoted by L10 and L50) are calculated from the data by using Weibull analysis.

A comparison of the test conditions and data analysis methodology of these four dynamic life tests is shown in Table 6. It should be noted that the tests all differ from one another in bearing type, speed, load, load direction, operating cycle or data treatment.

Test Program:

A test program was conducted on the nine commercial greases represented in Table 3 to compare Product Data sheet claims with both commonly used static heat resistance test results, as well as several dynamic life tests. The grease matrix for the test program included both lithium complex and polyurea thickeners, mineral and synthetic base fluids, and all had publicly available Product Data sheets. The test matrix included:

Static heat resistance:	Dynamic life:
- ASTM D2265 Dropping Point	- DIN 51821 FAG FE9
- ASTM D5483 PDSC	- ASTM D3527 Wheel Bearing Life

Other static heat resistance tests (bomb oxidation and evaporation loss) were not chosen for this program, since they are not typically used by industry as a basis for upper operating temperature claims. On the other hand, dropping point is widely

Table 6 Comparison of Dynamic Life Tests

Test	Method	Report	Number of samples	Data treatment	Bearing Type	Temperature °C	Speed rpm	Load N.	Load direction	Operating cycle	Criteria for failure
FAG FE9	DIN 51821	L50 Hours @ Temperature	5	Weibull analysis	Angular contact ball	140 ¹	6000	1500	axial	continuous	power increase to 2x steady state
Wheel Bearing Life	ASTM D3527	Hours to failure	2	Averaged?	Tapered roller	160	1000	111	axial	20 Hrs on, 4 off	power increase to 4x steady state
SKF R0F+	DIN 51806	L50 Hours @ Temperature	5	Weibull analysis	Deep groove ball	Ambient to 170	10000	50 100	radial axial	continuous	temperature increase >20°C
POPE (High Speed Spindle)	ASTM D3336	L50 Hours @ Temperature	5	Weibull analysis	Deep groove ball	As specified	10000	22	axial	20 Hrs on, 4 off	power increase to 3x steady state, or temperature increase >15°C

Notes: 1) 140°C used for this study; may be any temperature form 100°C to 200°C in 20°C increments

Table 7 Comparative Test Data

Test	Method	Units	Grease 1	Grease 2	Grease 3	Grease 4	Grease 5	Grease 6	Grease 7	Grease 8	Grease 9
Thickener Type			LIX	LIX	Polyurea	LIX	LIX	LIX	Polyurea	LIX	LIX
Base Oil Type			Synthetic	Synthetic	Synthetic	Synthetic	Mineral	Synthetic	Mineral	Mineral	Mineral
Drop Point	D2265	°C	309	291	301	305	300	308	264	212	309
Worked Penetration											
60x Strokes	D217	0.1 mm	296	313	298	291	297	286	269	293	280 typical
PDSC	D5483										
O.I.T. @210 °C		Mins.	34.8	4.98	41.02	15.61	16.8	31.3	37.6	18.2	120
FAG A/1500/6000/140C	DIN 51821										
L10		Hours	139	85	47	27	15.2	118	21.2	58.1	27
L50		Hours	169	109	230	110	59.7	148	276	202	60
High Temp Wheel Bearing											
Life	D3527	Hours	100	260.5	60.5	120	120	180	560	100	240
PDSC Upper Temp Claims											
Continuous		°C	150	232	180	170	140	150	170	150	150

used as a basis for upper operating temperature claims, and PDSC was also run because it was felt to have some potential for predicting high temperature life as determined by longer dynamic bearing life tests.

The FAG FE9 and Wheel Bearing Life tests were chosen because they each are the basis for industry certifications – FE9 for the DIN 51825 classification system and Wheel Bearing Life for the NLGI GC certification mark. In addition, the SKF ROF+ and POPE tests are both very long tests, usually running for 500 to 1000 hours to failure.

Data Analysis:

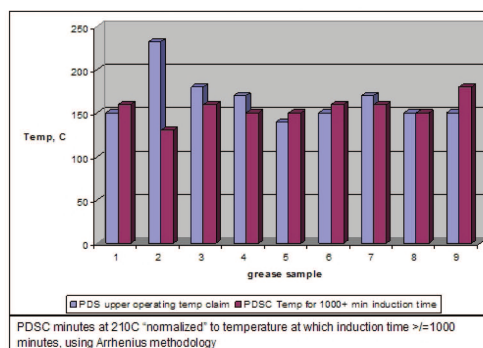
Raw test data from the program are shown in Table 7, including not only the data from the four static and dynamic heat resistance tests, but also thickener type, base oil type and 60 stroke worked penetration.

The nine greases were all in the range of NLGI 1.5 to 3 grade consistencies, with most having a 60 stoke worked penetration in the neighborhood of 300 dmm. Dropping points ranged from 212 to 309°C for the lithium complex greases and from 264 to 301°C for the polyurea greases. PDSC induction times at 210°C ranged from 5 to over 120 minutes. FE9 L50 life at 140°C ranged from 60 to 276 hours. High temperature wheel bearing life at 160°C ranged from 60 to 560 hours. Product Data sheet upper operating temperature claims ranged from 140 to 232°C.

Observations and Conclusions:

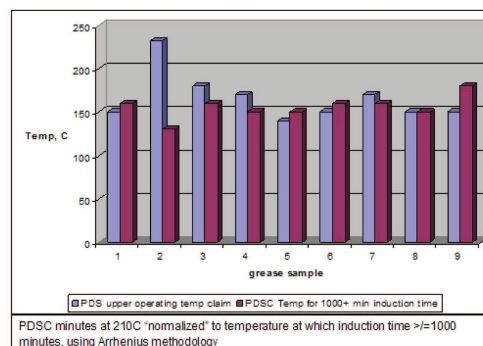
Dropping point versus Product Data sheet claims – Figure 1 shows graphically the relationship between dropping point and upper operating temperature claims. The most risky data sheet claim is only 59°C below its dropping point (grease 2). The most conservative claim is 160°C below the dropping point (grease 5). The average delta is about 120°C.

Figure 1 Claims versus Dropping Point



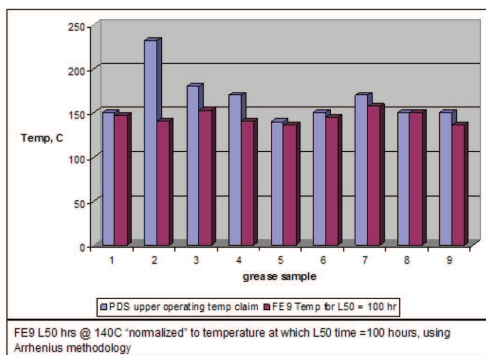
PDSC versus Product Data sheet claims – PDSC induction times at 210°C were normalised (using Arrhenius methodology) to the temperature at which the induction time would be greater than or equal to 1000 minutes in order to convert the data to temperature units for comparison to the data sheet claims. Figure 2 illustrates the relationship between the normalized PDSC data in °C (at 1000+ minutes) to the data sheet claims. The most risky data sheet claim is 102°C above the PDSC temperature (grease 2). The most conservative claim is 30°C below the PDSC temperature (grease 9). The average delta is 8°C above the PDSC temperature.

Figure 2 Claims versus PDSC



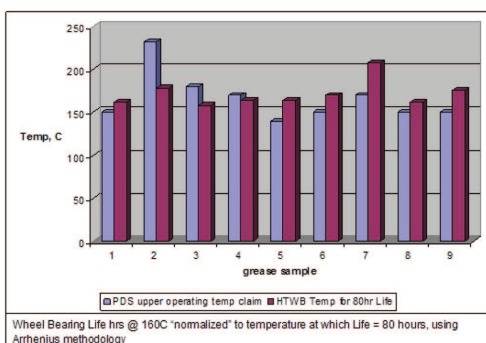
FE9 versus Product Data sheet claims – Similar to the PDSC, the FE9 L50 life at 140°C was converted (using Arrhenius methodology) to the temperature for a 100 hour L50 life. A 100 hour life was chosen based on the requirements of the DIN 51825 classification. Figure 3 shows compares the normalised FE9 data in °C (at 100 hours) to the data sheet claims. The most risky data sheet claim is 91°C above the FE9 temperature (grease 2). The most conservative data sheet claim is equal to the FE9 temperature (grease 8). The average delta is about 20°C above the FE9 temperature.

Figure 3 Claims versus FE9



High temperature wheel bearing life versus Product Data sheet claims – As with the FE9 life data, the HTWB life in hours needed to be converted to temperature units. A life of 80 hours was selected, based on the requirement in the NLGI GC certification mark (as described in ASTM D4950). The normalised life data, shown in °C (at 80 hours) is compared to data sheet claims in Figure 4. The most risky claim is 54°C above the HTWB temperature (grease 2). The most conservative claim is 38°C below the HTWB temperature (grease 7). The average delta is 20°C below the HTWB temperature.

Figure 4 Claims versus Wheel Bearing Life



Data summary comparison

The data from the four analyses was summarised into a single matrix, shown in Table 8. Ratings are arbitrarily determined and are as follows: -

Table 8 Summary PDS Comparison

Grease	PDS	DP	FE9	HTWB	PDSC
1	150	C	OK	OK	OK
2	232	R	VR	VR	VR
3	180	OK	R	R	R
4	170	C	R	OK	R
5	140	C	OK	C	OK
6	150	C	OK	C	OK
7	170	OK	OK	C	OK
8	150	R	OK	OK	OK
9	150	C	OK	C	C

KEY:

C	CONSERVATIVE
OK	REASONABLE
R	RISKY
VR	VERY RISKY

C = Conservative = more than 130°C below drop point, more than 20°C below PDSC temperature, more than 0°C below FE9 temperature, more than 20°C below HTWB life temperature.

OK = Safe = from 100 to 130°C below drop point, from 10°C above to 20°C below PDSC temperature, from 0 to 20°C above FE9 temperature, from 10°C above to 20°C below HTWB life temperature.

R = Risky = less than 100°C below drop point, from 20 to 50°C above PDSC temperature, from 20 to 30°C above FE9 temperature, from 10 to 30°C above HTWB life temperature.

VR = Very Risky = more than 50°C above PDSC temperature, more than 30°C above FE9 temperature, more than 30°C above HTWB life temperature.

It is interesting to observe that significant differences were found between these nine greases' high temperature claims and their relative rankings based on each of the four tests in the program. Some data sheet claims carry more risk to the end user than others. Other observations:

- Several of the greases' high temperature claims appeared to be risky or worse by more than one test (Greases 2, 3 and 4)
- One grease appeared to have a safe claim based on dynamic or PDSC testing, but seemed risky by dropping point (Grease 8)

A second data examination was performed, comparing how each of the four tests ranked the nine greases, shown in Table 9. Observations from that comparison: -

Table 9 Relative Ranking Comparison

	Dropping Point	PDSC	FE9 Life	HTWB Life
Relative Ranking	Grease			
1	1,9	9	7	7
2	6	1,3,6,7	3	2
3	4	4,5,8	8	9
4	3	2	1	6
5	5		6	4,5
6	2		2,4	8
7	7		5,9	3
8	8			1
9				

- While Grease 9 was ranked best by drop point and PDSC and near the top by HTWB life, it was one of the poorest by FE9 (blue highlights)
- While Grease 1 was also ranked best by drop point, it was last by HTWB life (blue highlights)
- While Grease 7 was ranked best by both dynamic tests, FE9 and HTWB life, it had the second lowest drop point (yellow highlights)
- While Grease 2 was ranked near best in HTWB life, it was ranked near worst by FE9 (tan highlights)
- While Grease 3 was ranked near best by FE9, it was ranked near worst by HTWB life (green highlights)

All this goes to illustrate that there appears to be no ranking correlation:

- Between drop point and either of the dynamic tests
- Between FE9 and HTWB life

Next Steps:

It still appears that an industry standard approach to making high temperature claims for greases would be vastly better than the current confusing and conflicting rule of thumb industry guidance. The claims made on several grease's data sheets compared to their performance in dynamic life tests illustrates the sometimes substantial end user risks existing today. However, given the lack of correlation between the dynamic life tests used in this test program, the mechanism of failure for various greases in these and other dynamic tests needs to be further investigated.

Acknowledgements:

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