A Technical Brief from Savant Labs:

Grease Oxidation Measurement by an Advanced Technique

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EXECUTIVE SUMMARY

Oxidation resistance is one of the most desirable properties of grease, particularly because of the stresses imposed by continuous operation often at high temperature. For many years the batch-to-batch oxidation resistance of grease has routinely been measured using ASTM Test Method D942[1].

Recently, this method has been implemented on the Tannas *Quantum*® instrument, which is an isothermal reactor eliminating the care and concern of a liquid bath. In conjunction with this improvement of D942, a further advancement in grease oxidation measurement has been developed by combining the ASTM D942 Test Method with infrared (IR) analysis. This technique considerably amplifies the very limited information provided by the D942 test alone.

INTRODUCTION

Grease is subject to many forms of stress in providing lubrication. One of the most critical of these stresses is oxidation. As a consequence, measurement of the oxidation stability of grease has been important. Increasingly advanced bench test techniques have been developed over the years to improve understanding of grease oxidation in applications. Even more important has been to learn how to improve oxidation resistance by formulation. In an early effort to develop a quality control tool for measurement of grease oxidation resistance, ASTM introduced the D942 Test Method in 1947. This method continues to be the most common test of grease oxidation stability.

The method is used routinely to measure comparative quality of batches of the same grease in the manufacturing process. According to this method, 20g of a grease sample is tested in five Petri-type dishes which provide a total exposed surface area of 125 cm^2. The combined stack of the five grease-filled dishes is inserted into a cylindrical pressure chamber at room temperature which is then raised to 99±0.5°C. With the increase in temperature, initial oxygen pressure increases and is then maintained at 110±2 PSI.

The test is continued for a chosen period of time, usually between 100 to 500 hours. During the chosen time interval, any decrease in oxygen pressure in PSI (from the initial value of 110 PSI) that results from grease oxidation, is reported as the test result.

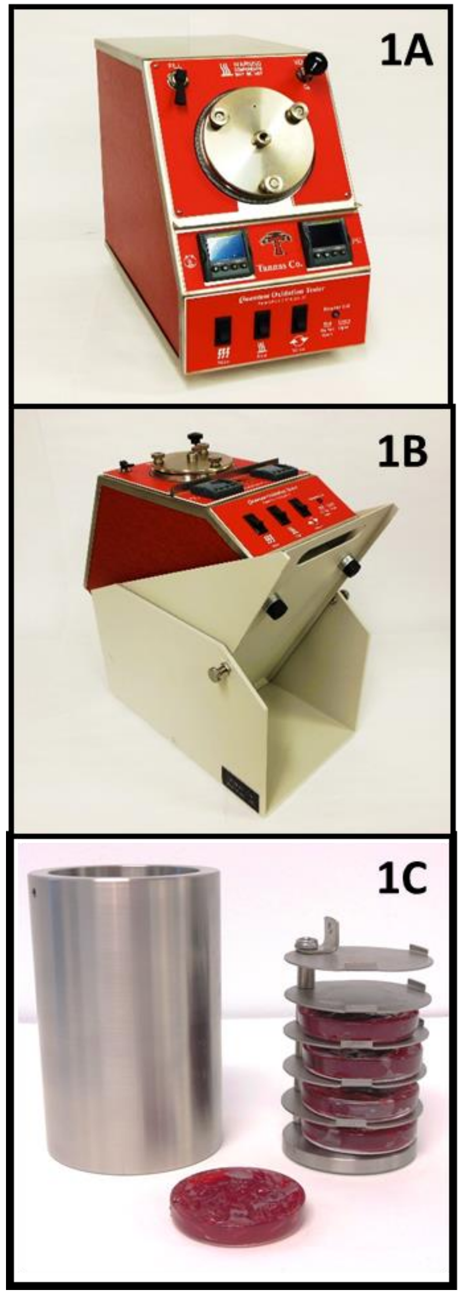

Fig. 1 -- *Quantum*® before (A) and after (B) Modification and Accessories (C) for ASTM D942 Test Method

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The Tannas *Quantum*® instrument that has been qualified as a liquid-bath free approach for ASTM Test Method D2272, has been modified by adding a simple steel shelving to allow the instrument to be used alternatively in either D2272 (Figure 1A) or D942 (Figure 1B) test configuration. Since the *Quantum*® instrument is very simply adapted for running the ASTM D942 Test Method with the required geometry, heating capability and availability of oxygen pressurization for conduct of ASTM D942. The strong advantage of using the *Quantum*® for the D942 test is that it eliminates the need for a liquid bath and the safety concerns associated with handling such a liquid.

Accessories, including a sample rack, pans and a stainless steel volume reducing insert, were designed to comply with the method (Figure 1C). Application of the *Quantum*® for the ASTM D942 Test Method was validated by testing multiple grease samples at 100 and 200 hours for both repeatability and similarity to results on liquid-bath heated equipment. All results were within the D942 method's precision.

ADVANCED TECHNIQUE

Presently, the application of ASTM Test Method D942 is strictly limited to appraising manufacturing consistency and the test cannot be used to compare different types of greases. In addition, the test states that it should not be used to compare different greases in terms of oxidation resistance. Moreover, Test Method D942 states that it should not be used to predict the stability of a given grease stored in containers or the oxidation stability of grease used in bearings and motors since only controlled oxidation of a physically stable surface of grease is used in the D942 test. Savant Laboratories concluded that the oxidation conditions generated by D942 provided further meaningful data and have improved the test by applying special infrared analysis techniques. For clarity this will be called an Advanced Technique.[2, 3]

Infrared spectroscopy has, of course, often been used in the past to study grease oxidation and, in general, the level of oxidation in petroleum-based lubricants is measured in the carbonyl region (wavenumbers of 1670 to 1800 cm^{-1}) which includes the oxidation formation of various carbonyl compounds including, ketones, esters, carboxylic acids, carbonates, aldehydes, anhydrides and amides. The IR peak around 1715 cm^{-1}, is generally known as the "oxidation peak", and indicates breakdown of the lubricant and formation of oxidized by-products. Combining ASTM D942 and the IR analysis appeared to be a solid approach to measure grease oxidation response to the D942 test.

The Advanced Technique for oxidation measurement further analyzes the sample oxidized in ASTM Test Method D942 by advantageously using Fourier Transform Infrared (FTIR) technique. Thus, limitations of the D942 test alone have been overcome. To obtain the FTIR information, Attenuated Total Reflection (ATR) was used to gather the infrared data. Subsequent analyses of the spectra of the fresh grease compared to the grease after oxidation show that infrared information add much understanding to the severely limited information of ASTM D942 alone.

A test protocol has been developed for the Advanced Technique FTIR oxidation measurements. In procedural order:

1) Obtain the oxygen pressure loss of selected grease to the conditions of test applied through ASTM D942.
2) On disassembly, carefully sample the grease from a pan.
3) Compare the oxidation resistance of the greases using ATR-FTIR spectra using the Advanced Technique.

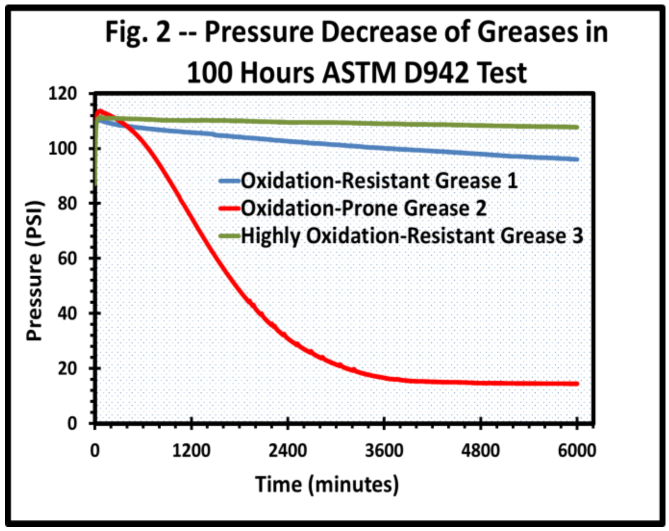
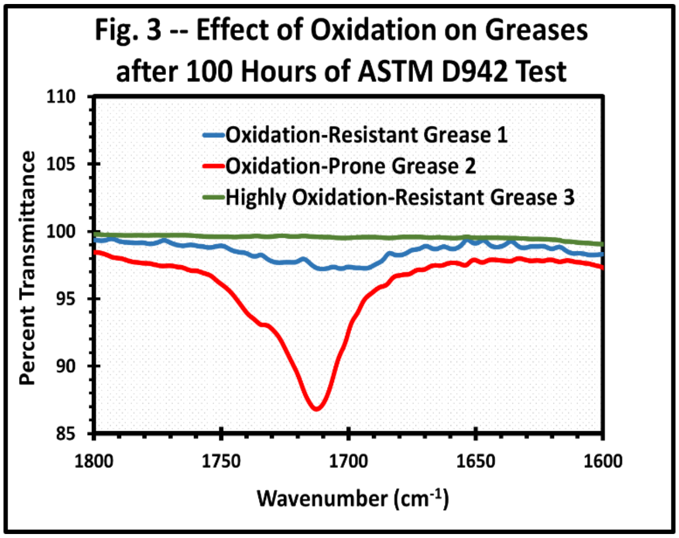


Figure 2 shows oxygen pressure-loss results from a set of three greases in 100-hour ASTM D942 tests. The three grease samples show considerably varying oxidation resistance. Among the three, Grease 1 is relatively resistant to oxidation as indicated by the decrease in oxygen pressure over 100 hours of exposure in the D942 test. Grease 2 is found to be by far the least stable of the three samples. In contrast to Greases 1 and 2, Grease 3 shows the most resistance to oxidation.

It is clearly evident in Figure 2, that the oxidation response of Grease 2 has virtually exhausted the available oxygen with the oxygen pressure dropping to 16 PSI (likely reflecting the pressure of the base oils in the grease composition or oxidized products). In contrast, oxygen pressure with Grease 3 never became less than 106 PSI indicating very little or no oxidation in 100 hours. Grease 1, on the other hand, shows some oxidation with an oxygen pressure decrease to 96 PSI in the 100-hour D942 test.



FTIR-ATR analyses of the three greases after 100 hours of exposure to oxygen in ASTM Test Method D942 test are shown in Figure 3 using only the 1700-1750 cm^{-1} wavenumber range associated with carbonyl oxidation products. It is evident that this limited section of the infrared spectra supports the oxidation pressure decrease plots of Figure 2 and in addition shows the level of oxidation associated with such pressure decrease of each grease. Thus the combination of ASTM D942 and the subsequent FTIR-ATR infrared spectral analysis of the original grease compared to the grease after oxidation showed that the Advanced Technique considerably enhances the information provided by the original D942 test. Multiple analyses of both pressure drop and infrared spectra of the three greases showed good replication.

With such information, ASTM Test Method D942 can be used to develop correlation with greases in service and permit selection of greases and additive package for specific applications.

PATH FORWARD

With this new ability to quickly convert the *Quantum*® instrument used in ASTM D2272 to D942 and back again, Savant Labs is now equipped to perform the standard ASTM D942 Test Method. In addition, as the developer of the Advanced Technique, Savant Labs has the expertise to test and compare greases and their formulary modifications.

Thus, customized projects and testing protocols can be developed easily to advance understanding of oxidation resistant of future grease formulations.

REFERENCES / ACKNOWLEDGEMENTS

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