TA Instruments

Thermal Analysis & Rheology

Thermal Analysis Application Brief

Oxidative Stability of Oils & Greases

Number TA-132

SUMMARY

The useful lifetime of lubricating oils and greases is determined largely by their ability to resist oxidation. Differential scanning calorimetry (DSC) provides a rapid method for predicting the oxidative stability of these materials.

INTRODUCTION

Many materials, ranging from plastics to oils and lubricants to foods, exhibit useful lifetimes that are dependent on their resistance to oxidation. As a result, antioxidants are usually added to these last materials to improve their lifetimes. Since there is typically a level of antioxidant beyond which no improvement in lifetime occurs even though additional cost is incurred, the ability to rapidly predict the long term effects of different levels of antioxidants (as well as different types of antioxidants) has high value. Differential scanning calorimetry (DSC), and particularly pressure DSC, provides a convenient method for predicting oxidative stability. DSC is a viable alternative to oven aging or other classical tests, because DSC requires small samples (<5mg) and evaluations are complete in several hours or less.

EXPERIMENTAL

Differential scanning calorimetry (DSC) measures the heat flow into or out of a material as its temperature is changed or held isothermal. Since the oxidative degradation of most materials is an exothermic process (heat is evolved), DSC can be used to determine oxidative stability. Usually this is accomplished by measuring the amount of time the material can be held at a specific temperature before the degradation exotherm occurs. The evaluation temperature for a specific material is chosen to allow evaluations to be completed in a reasonable timeframe, yet give reasonable resolution and repeatibility between results for slightly different antioxidant levels.

Since both temperature and concentration of the reactive gas (oxygen) affect the rate of degradation, increasing the reactive gas concentration by increasing pressure means that lower temperatures can be used while still obtaining reasonable analysis times. This ability to run the analysis at lower temperatures is important because lower analysis temperatures usually more closely approach the actual in-use temperatures.

In these experiments, a lubricating grease stabilized with different levels of a common antioxidant were evaluated using pressure DSC at 167°C and 3.4 MPa oxygen.

RESULTS

Figure 1 shows the PDSC curve for the grease with 1.5% antioxidant. Two different points on the DSC curve can be used to determine the beginning of oxidative degradation. They are the first deviation from baseline and the extrapolated onset. The preferred approach depends on the material being evaluated. However, whatever approach is ultimately chosen, it should be used for all samples in a specific comparison study. Figure 2 shows the results for the entire series of samples using both determination approaches. These results indicate that above 4% the benefits of additional antioxidant are minimal.

Temperature control is quite critical in this procedure. Using the rule-of-thumb that a chemical reaction rate doubles for each 10 degreee increase in temperature, a one degree variance in temperature can produce a 10% difference in results. The sample thermocouple in the DSC or PDSC cell permits the operator to measure the actual temperature of the sample and assure himself of its accuracy and control during the course of the experiment.

There are several variations of this same basic prcedure in common use. One of these is temperature programming of the sample from ambient temperature. The temperature at the onset of oxidation is taken as a measure of oxidative stability (1,2,3). Noel has been able to correlate this type of analysis with the ASTM Rotary Bomb Test (D-2272) (4).

Similar DSC and PDSC procedures can be used to determine the oxidative stability of a wide variety of products, including motor oils (1), brake fluid (2), lubricants (2), greases (3), polyolefins (5) and plastic pipe (6).

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OXIDATIVE STABILITY VS. ANTIOXIDANT LEVEL

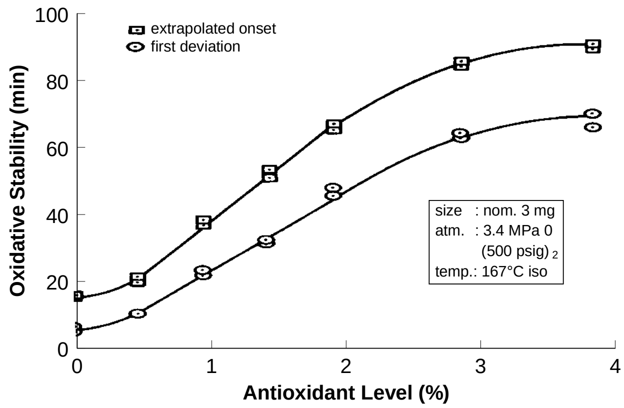

Figure 2

OXIDATIVE STABILITY 1.5% ANTIOXIDANT IN GREASE

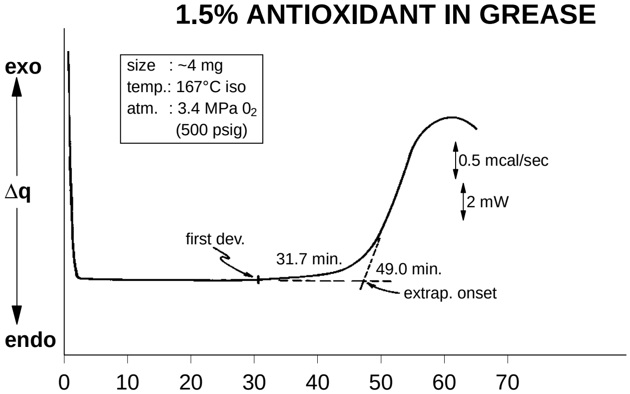

Figure 1

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