

'Providing Tribological Solutions'

TECHNICAL NOTE

FOURIER TRANSFORM INFRARED SPECTROPHOTOMETRY (FTIR) FOR MOV LONG LIFE GREASE

1. Background

The infrared region of the electromagnetic spectrum includes radiation at wavelengths between 0.7 and 500 microns, or in wave numbers between 14,000 and 20 cm^{-1} . The y-axis of the FTIR is the relative concentration of the component in absorbance values. Molecules have specific frequencies that are associated with their rotational and vibrational motions. This can be useful for product verification and condition monitoring.

2. Variation

Variability depends on whether the FTIR is done on a sample between pressed salt tablets or a reflectance cell. The salt tablets reportedly do not make for decent quantitative analysis because the thickness is uncontrollable. While the reflectance cell is designed for quantitative analysis because the beam strikes a perfectly flat surface it apparently really only works well for liquids that can flow across the surface. Good for liquids but not necessarily greases.

3. Interpretation

The FTIR is useful in qualitative identification of carbonate types in sulfonate thickened greases where the presence of a peak at $\sim 880 \text{ cm}^{-1}$ and 712 cm^{-1} confirm the presence of calcite. Calcite is one of the main components of the thickener and is one of the forms of the calcium carbonate that provides the extreme pressure (EP) characteristics. In addition, on each batch of MOV Long Life the supplier verifies the EP characteristics with a Timken OK load test.

Given the trigonal nature of calcite and the existence of hexagonal vaterite and orthorhombic aragonite it is to be expected that slight batch-to-batch differences in shape of carbonate peak will be seen. Naturally all these materials have the same chemistry (CaCO_3), just different symmetry and crystal shape calcite carbonate.

Factors that affect the shape and position of calcite peak in FTIR include soap (sulfonate), complex soap (carboxylate, acetate/borate salts) and residual promoters (water/alcohol). Batch yield (more or less oil) further affects peak position. Hydration of calcite (X, 2X, 3X etc. moles $\text{H}_2\text{O}/\text{mol}$ carbonate) is an additional factor that can produce changes in shape and intensity of calcite peaks in FTIR scans.

4. Limits

A characteristic of these greases is a calcite peaks between 878-882 cm^{-1} and a second one around 712 cm^{-1} . One problem with putting a wide range on the calcite peak is that amorphous calcium carbonate has a very broad peak at 860 and vaterite is around 874 cm^{-1} . There can be confusion.

The following were being used by one station for commercial dedication;

715 $\pm 10 \text{ cm}^{-1}$
880 $\pm 20 \text{ cm}^{-1}$
1430 $\pm 30 \text{ cm}^{-1}$
2890 $\pm 50 \text{ cm}^{-1}$.

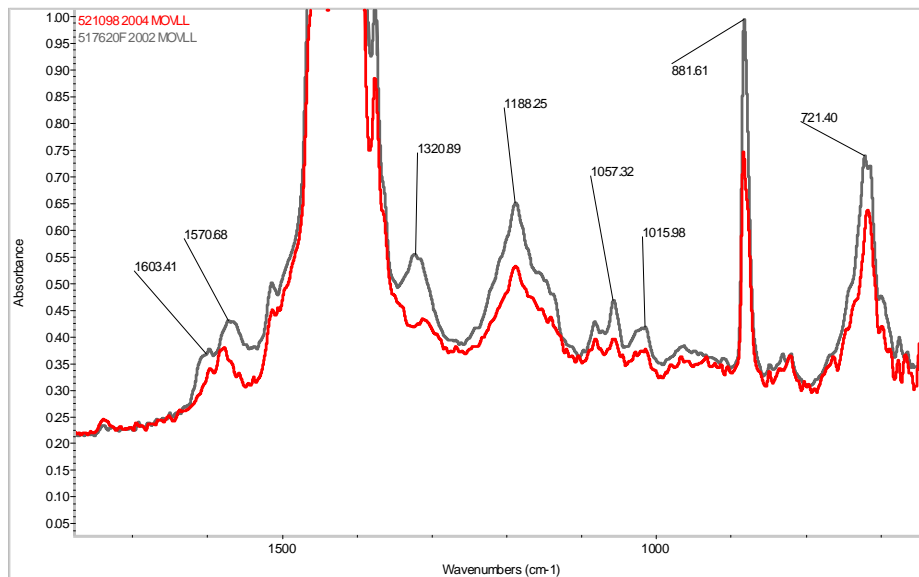
Note that in the EPRI work Exxon Nebula EP also showed absorbances at around 2890 and 1430 cm^{-1} so their presence will not confirm that the grease is MOV Long Life.

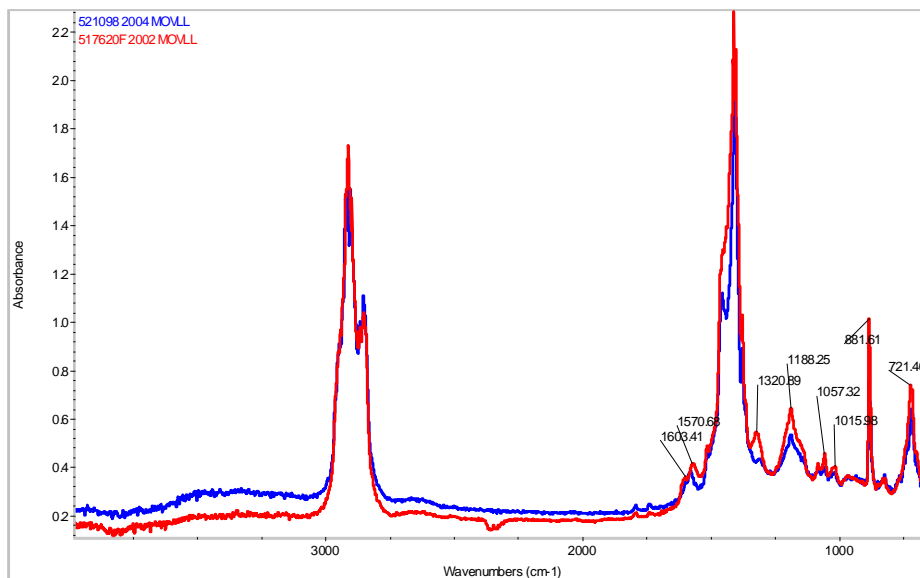
5. 'Typical' Spectrums

The following spectrums were done by Herguth Laboratories and show the variation in batches two years apart. Plus, in the EPRI report* on the qualification of MOV Long Life there are spectrums (scans) for aged, irradiated and EQ'd samples.

It was concluded in the EPRI work that there were no significant changes in the FTIR analyses between the various grease samples evaluated. This is good in that the grease had not been significantly degraded but it also suggests that FTIR may not be suitable for in-service testing unless the grease has been even more severely stressed.

* Comparative Analysis of Nebula and MOV Long Life Greases for Limitorque Main Gearbox Applications, EPRI, Palo Alto, CA: 2002, 1003483





Herguth Laboratories, Inc. are located at 101 Corporate Place, Vallejo, CA, 94590.
Phone: 800-645-5227 and website: www.Herguth.com

6. Summary

When testing by a lab for the first time there are not likely references that can be used to gauge what is 'acceptable'. However, guidance can be given on what is 'normal' or expected. This is because of differences between labs and the specific equipment and as more spectrums by that specific lab are taken the batch-to-batch variation can be better assessed.

7. Commercial Grade Dedication

MOV Long Life is manufactured at an ISO registered facility and each batch is tested for color, appearance, penetration, dropping point, Timken OK load, halogens and trace metals. Further, the only greases made at this facility are calcium sulphonate and packaging is directly from manufacturing kettles into final containers. MOV Long Life in the original the Forsythe, sealed totes, drums, kegs and pails has not been repackaged by others. Certificates of Analysis are available for each batch.

If site verification is required the tests typically include the following; appearance, penetration and dropping point. A few stations use FTIR.

Our thanks to W. Mackwood and R. Muir at Chemtura Co/Cie and B. Herguth at Herguth Laboratories, Inc. for input into the preparation of this Technical Note.