

Identify extraneous chemicals that contributed towards the failure of actuating mechanism in inner vial

By

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Objective: The second aging study of the Clicker assemblies had exhibited a failure rate of 2.5%. In the failed two samples, actuating mechanism did not activate during first try. This study was undertaken to identify if there is any chemical compound that was present on the gears that could have contributed to the failure of the actuating mechanism.

Background: It was hypothesized that there was a waxy substance that came from PE bags and/or HDPE components such as HDPE cap, and after aging study caused gears to become gummy. To test this hypothesis, a chemical analysis was done using hexane wash and FTIR analysis. These techniques were chosen for the following reasons:

- A. Various polymer additives can be extracted with high recovery efficiency using hexane from polymer surfaces by using room temperature extraction surface wash technique. In addition, Hexane is also inert towards ABS, PC and PE compounds as well as anticipated additives such as waxes and silicone oils, and therefore would not attack the components.
- B. Thereafter, FTIR (Fourier Transform Infrared) spectroscopy was applied to analyze and identify the different types of organic as well as many inorganic compounds that were present in the hexane wash. FTIR was chosen since it is one of the most powerful analytical chemistry instruments that is used to identify and quantitatively analyze additives that are present in plastics. Pure organic molecules give identical IR spectra. Hence, the IR spectrum of a material can be used as a "*fingerprint*" for identification purposes by comparing spectra of known database references to the sample spectrum. By comparing standard IR spectra of the known additive with unknown, it was hypothesized that the presence of additive could be identified that may have contributed towards the gummy behavior of actuating mechanism.

Collection of samples and analysis: Reference gear samples (virgin molded part), gears from the failed samples in the second aging study and HDPE cap (also from aging study) were collected and subjected to the following analysis to determine the root cause of failure.

Analytical test: All samples were washed using Hexane and collected hexane was subsequently subjected to IR analysis.

Result of FTIR Spectra: Analytical tests conducted by Seal Laboratories involved washing reference gear samples, gear samples from aging study and HDPE cap with hexane, collecting the solution and analyzing it using Fourier Transform Infrared (FTIR) technique.

FTIR results indicated that both gears from the aging study as well as the reference gears had amide wax on them. Also, silicone was present on the reference gears. However, amide wax was not present on the inside of the HDPE cap. For details, please refer to appendix.

Conclusions:

- A. Amide wax was definitely a contributing factor that caused actuating mechanism to become sticky. The mechanism that caused the amide wax to become a contributing factor was due to the fact that amide wax¹ melted during the aging study (higher temperatures) and became softer and then migrated onto the gears, thereby contributing to gumminess of the actuating mechanism.
- B. Hexane wash and subsequent FTIR analysis of HDPE cap proved that amid wax was not present and/or did not come from the cap.

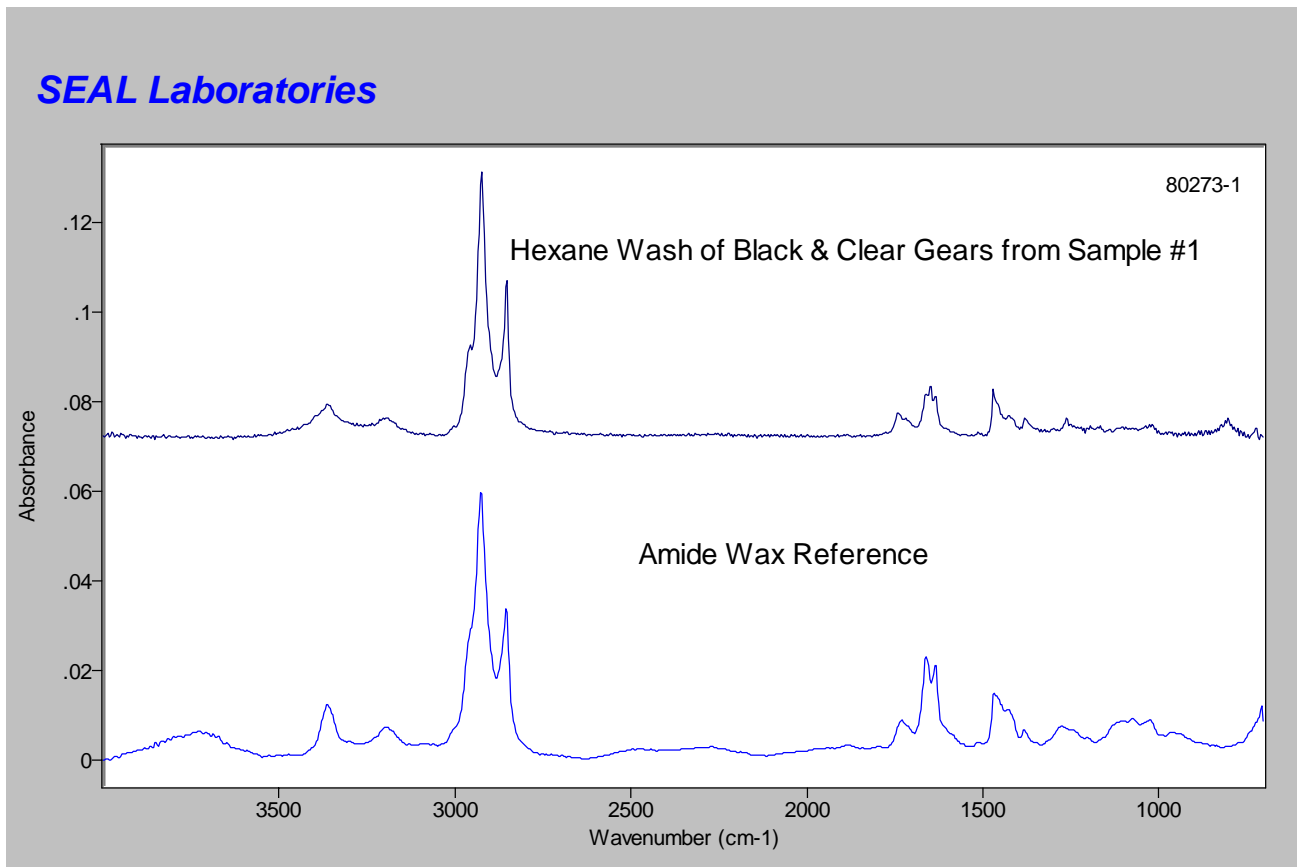
Recommendations:

- A. Further investigation should be carried out to see whether or not there is any amide wax that is present on the implant (from the aging study) and the clinical implications of having wax on the implant. There is a possibility of secondary migration of amide wax on to the implant due to higher aging temperatures and/or due to higher temperature packaging is exposed to during storage or transport.
- B. Molder should be instructed not to use silicone mold release agent to eject the parts from the mold.
- C. FDA approved PE bags should be used for transportation of parts.

¹ Amid wax is used as an external lubricant in manufacture of PE film and bags and therefore, it could only come from Polyethylene bags that were used to package and ship molded parts.

Appendix: Raw results of FTIR Analysis

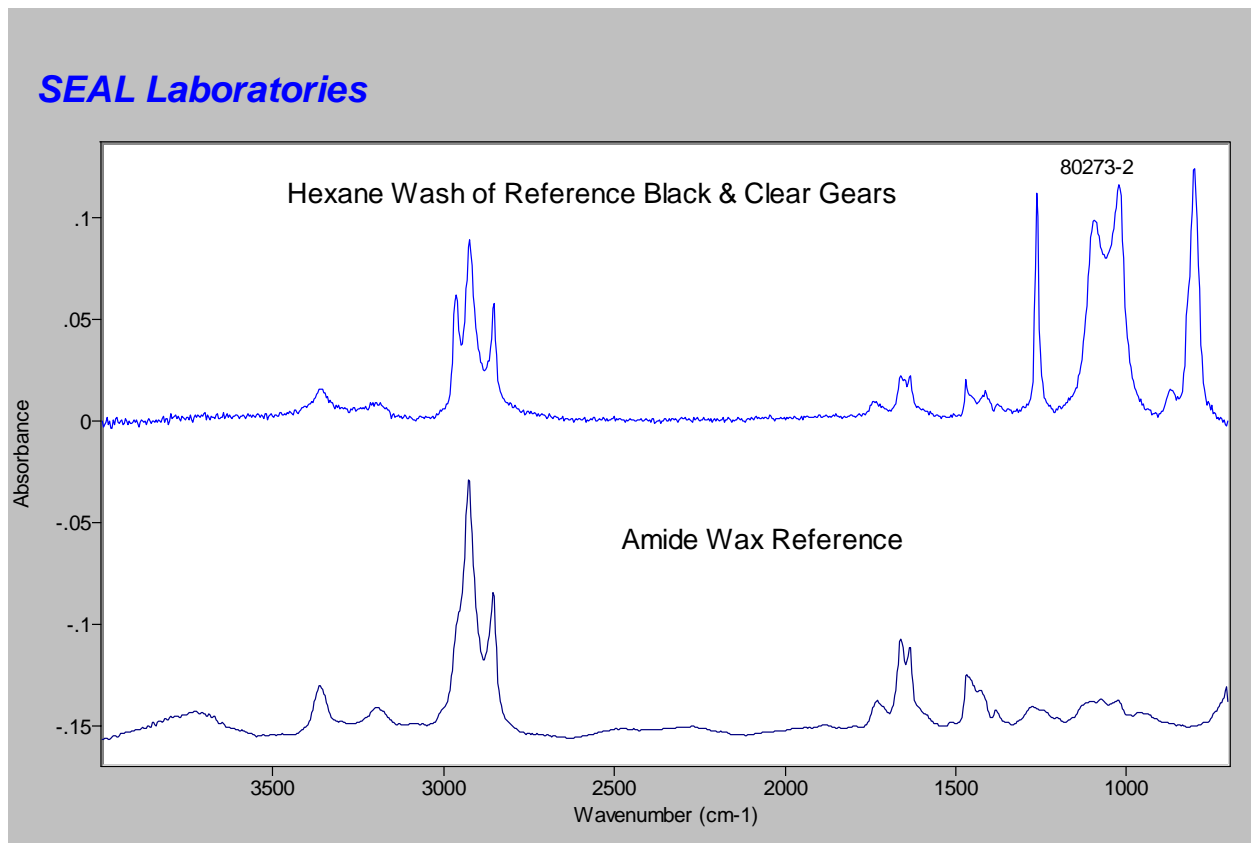
Sample gear:



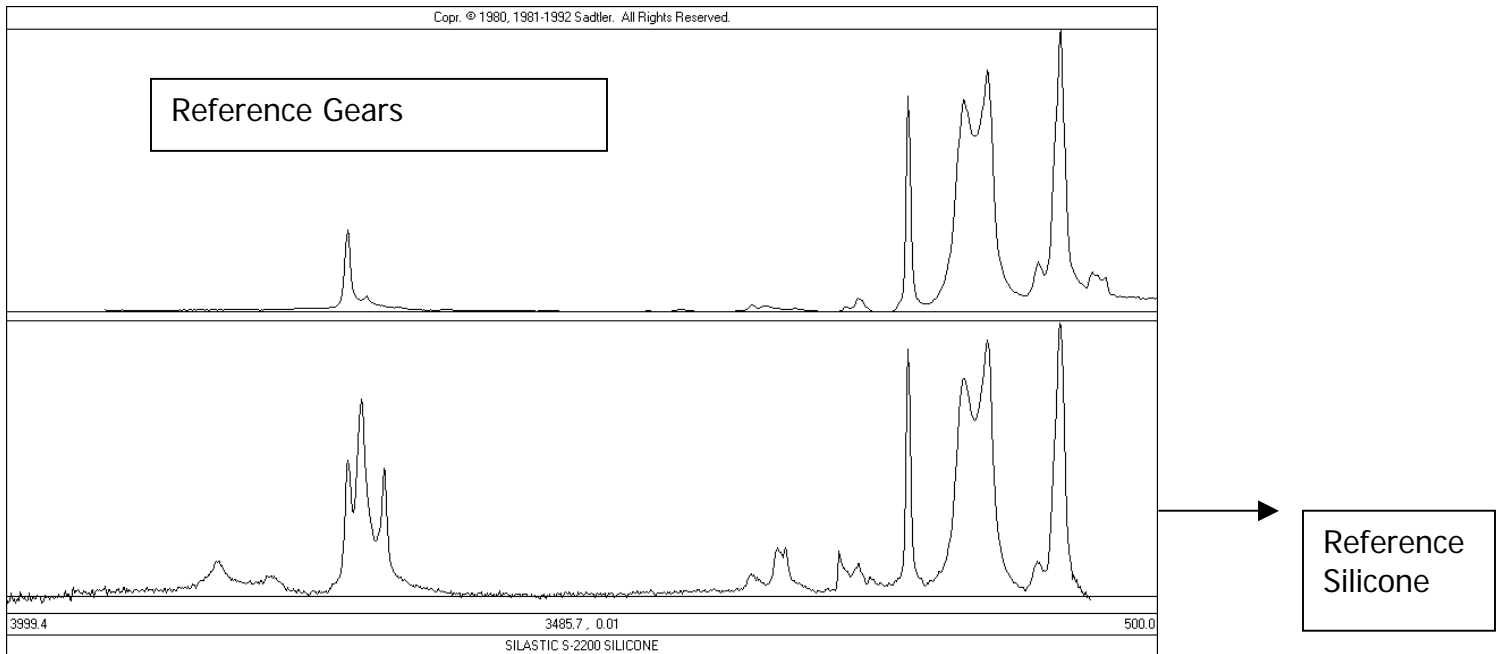
In this figure, the spectrum of the hexane wash from the Sample #1 gears is compared to a reference amide wax spectrum. The spectra are identical with identical peak positions (wavenumber positions) and peak shapes and heights.

The residue from the hexane wash of the Sample #1 gears is an amide wax.

Reference gear:

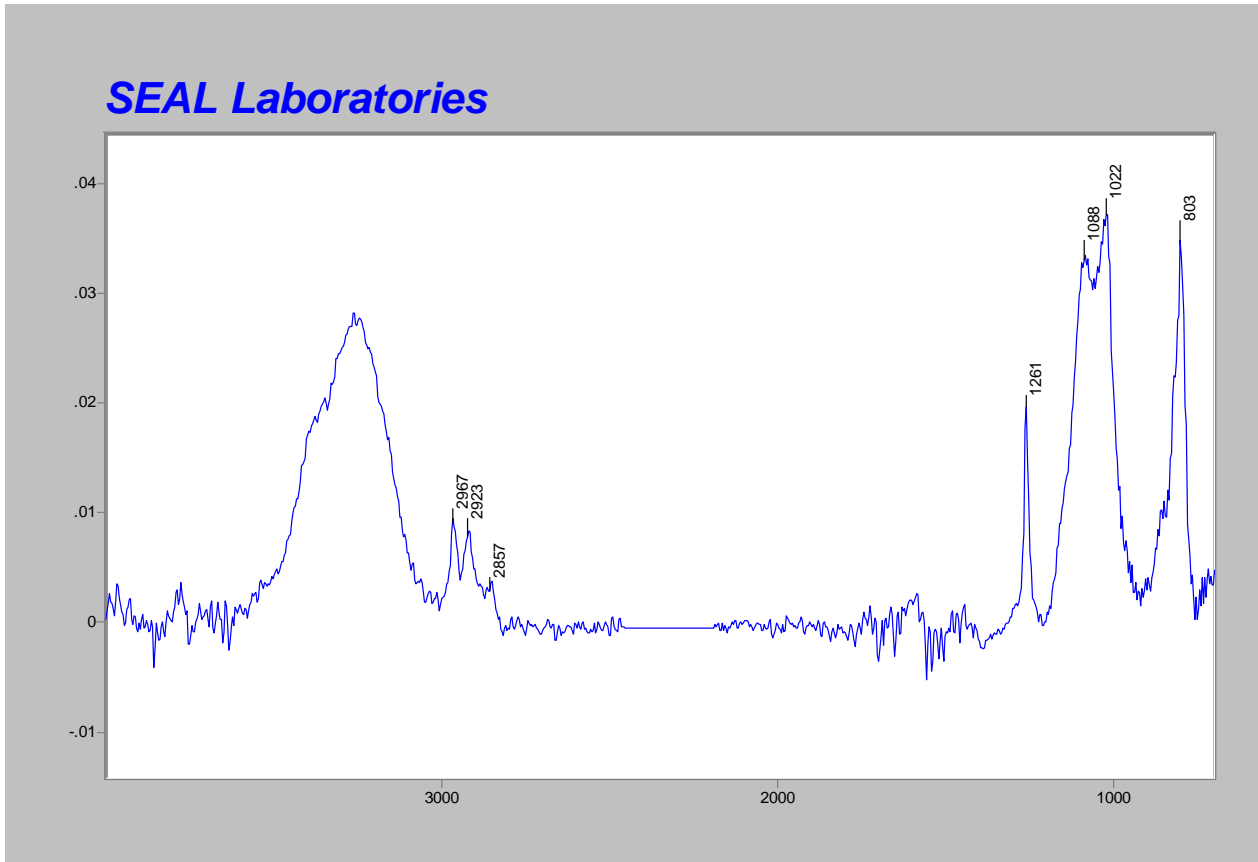


In this figure, the spectrum of the hexane wash from the reference gears is compared to a reference amide wax spectrum. This spectrum contains the amide wax spectrum but also contains peaks from presence of silicone.



In this figure, the spectrum of the hexane wash from the reference gears is compared to a reference silicone spectrum. The residue from the hexane wash from the reference gears is a mixture of amide wax and silicone.

HDPE cap:



Hexane wash of PE Cap shows absence of Amide Wax and presence of silicone.