

CHEMICAL & ENERGY ANALYSIS

LUBRICANT ANALYSIS USING GPC/SEC



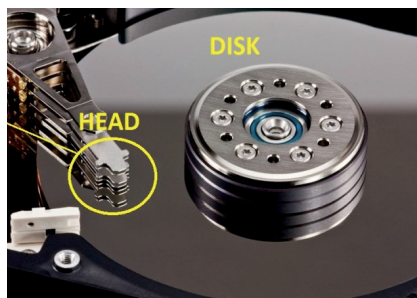
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Solution Note

Chemical & Energy

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Gel permeation chromatography (GPC) analysis is employed in lubricant molecular weight determination for the purpose of quality control in the hard disk manufacturing process.

Introduction

Lubrication is an essential and crucial process in the manufacturing operation and performance of hard disks. A layer of lubricant coated on the disk surface can effectively reduce wear and friction when disk drives operate at high speed. Figure 1 illustrates the layer by layer structure of a hard disk. This thin layer of lubricant also provides protection against corrosion of the underlying magnetic alloy. Reliability of hard disks largely depends on the durability of this thin film media and therefore many hard disk manufacturers develop their own proprietary lubricant.

Different methodologies and instrumentation are employed to confirm and control the properties and quality of the lubricant such as NMR, FTIR and GPC/SEC. Gel permeation chromatography / size exclusion chromatography (GPC/SEC) separates molecules based on their size in solution and is widely used in the quality control of lubricants. If a certain batch of lubricant's molecular size falls out of the desired range, it will fail specification and be rejected for the coating process.

Agilent's GPC/SEC software is dedicated standalone software for GPC/SEC analysis. It incorporates direct instrument control of Agilent's 1200 Infinity series HPLC modules. Characterisation of polymers is easy with the simplified workflow of the software and the comprehensive data reviewer makes visual comparison of multiple sample data files straightforward.

This application note describes how the Agilent 1260 Infinity GPC/SEC system and Agilent GPC/SEC software can successfully perform GPC analysis of lubricants for quality control.



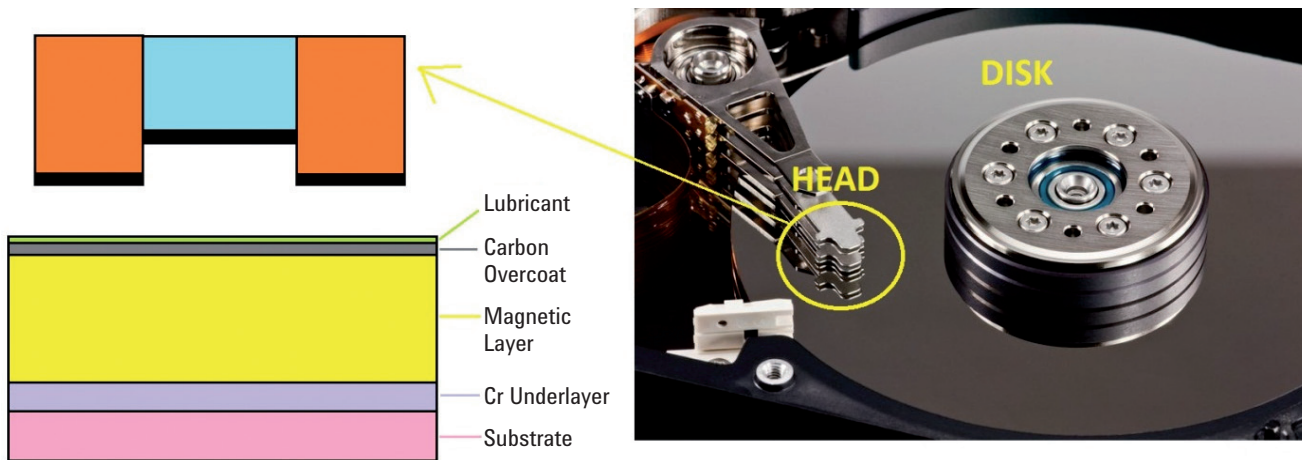


Figure 1: Diagram of hard disk and head. Lubricant is coated on the top layer of disk.

Experimental

Instrumentation and software

Description	Model Number
Agilent 1260 Infinity Vacuum Degassing Unit	G1322A
Agilent 1260 Infinity Isocratic Pump	G1310B
Agilent 1260 Infinity Standard Autosampler	G1329B
Agilent 1260 Infinity Thermostatted Column Compartment	G1316A
Agilent 1260 Infinity Refractive Index Detector	G1362A
Agilent GPC/SEC software v.1.2	G7850AA & G7854AA

Chromatographic conditions

Parameter	Setting
Column	PLgel 3 μm 100 \AA , 300 x 7.5 mm (PL1110-6320)
Mobile phase	THF (stabilized with 0.025 % BHT)
Column Temperature	30°C
Flow rate	1.0 mL/min
Calibrants	Individual Polystyrene Standards (Mp 4910, 2940, 2170, 1370, 770 and 380 g/mol)

Samples/Calibrants

The lubricant sample in this experiment is a linear molecule with a polar end group at each end of the polymer chain. Each of the individual calibrants, and the lubricant samples were dissolved in THF at an approximate concentration of 1mg/mL. The concentration

accuracy is usually not critical for conventional GPC analysis as the calculation of the molecular weight distribution, and molecular weight averages are not dependent on the sample concentration.

Results and Discussion

The Mw of the certified lubricant sample is expected to be around 3100 g/mol \pm 5%, with a polydispersity of 1.0 to 1.2, against the Polystyrene narrow standards. For this reason, a PLgel 3 μ m 100 Å, 300 x 7.5 mm column was chosen due to its suitable linear

Mw operating range (up to 4,000 g/mol) and high efficiency (>100,000 p/m), which provides excellent resolving power for the sample.

Figure 2 shows the column calibration curve with linear correlation co-efficiency of 0.995 in the 380 to 4910 g/mol Mw range.

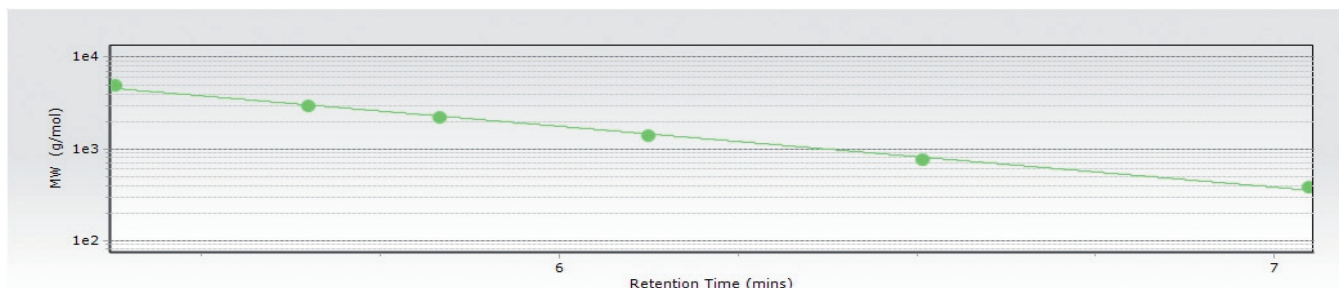


Figure 2. Column calibration curve of Polystyrene in THF generated by Agilent GPC/SEC software.

Samples from three different batches were analysed and are shown overlaid in Figure 3. Due to the concentration difference between the samples, the peak heights are different. However, this doesn't affect the molecular weight distribution calculation of the samples, as shown in Figure 4. The elution time of the peak, which is used to calculate molecular weight distribution is largely associated with the flow precision of the pump and this determines the reproducibility of the molecular weight distribution. The Agilent 1200 Infinity isocratic pump delivers ultrahigh precision flow, with triplicate runs of the same sample producing a retention time RSD of 0.05%.

The data reviewer window of the software allows us to compare and overlay normalized molecular weight distributions of a number of different samples. Figure 4 shows the overlaid molecular weight distribution plots of the three batches of lubricants, together with their molecular weight averages tabulated below. The Agilent GPC/SEC software enables the entire workflow to be interactive and easily manageable. Based on the calculated results, the Mw for all 3 samples has met the required Mw specification for quality control and therefore they are considered as certified batches for use.

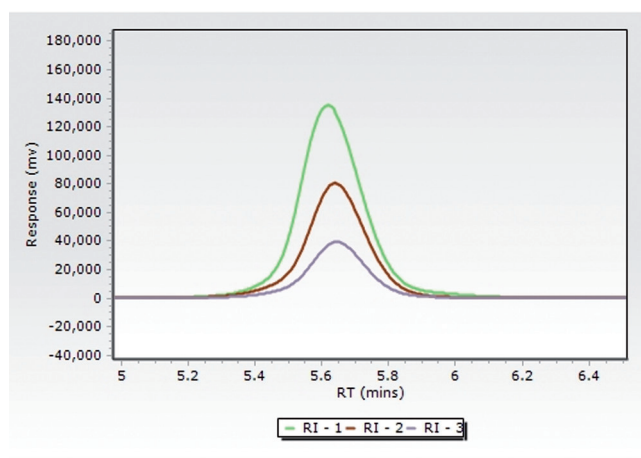
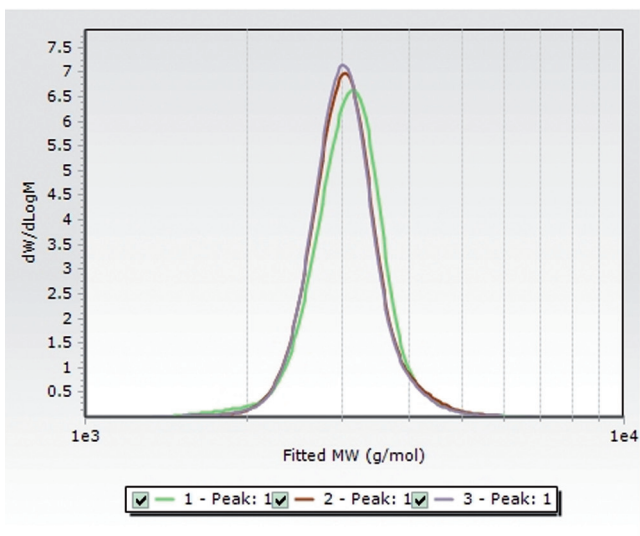


Figure 3. Overlay of chromatograms for 3 batches of lubricant.



Summary							
Sample Name	Mp	Mn	Mw	Mv	Mz	Mz + 1	PD
1	3135	3027	3107	3174	3186	3269	1.026
2	3034	2994	3065	3128	3140	3223	1.024
3	3017	2981	3049	3110	3121	3201	1.023

Figure 4. Overlay of molecular weight distribution plots for 3 batches of lubricant and summary table.

Conclusion

The Agilent 1260 Infinity GPC/SEC system delivers high precision flow to ensure data reliability and confidence in reproducible results. Agilent's GPC/SEC software is user-friendly, dedicated GPC software which integrates instrument control, data analysis and data review. Its result driven, simplified work flow allows even the inexperienced user to achieve excellent results for the most complex calculations. The data reviewer is the perfect solution for identifying subtle differences in the molecular weight distribution between samples.

This GPC method and instrumentation have been successfully adopted in lubricant molecular weight distribution determination, and provides a critical step in maintaining the quality control process of the hard disk manufacturing industry.



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© Agilent Technologies, Inc. 2014
Published in USA, March 31, 2014
5991-4312ENE

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