25th International Colloquium Tribology - Bridging Science and Industry – Driving a Sustainable Future with Tribology

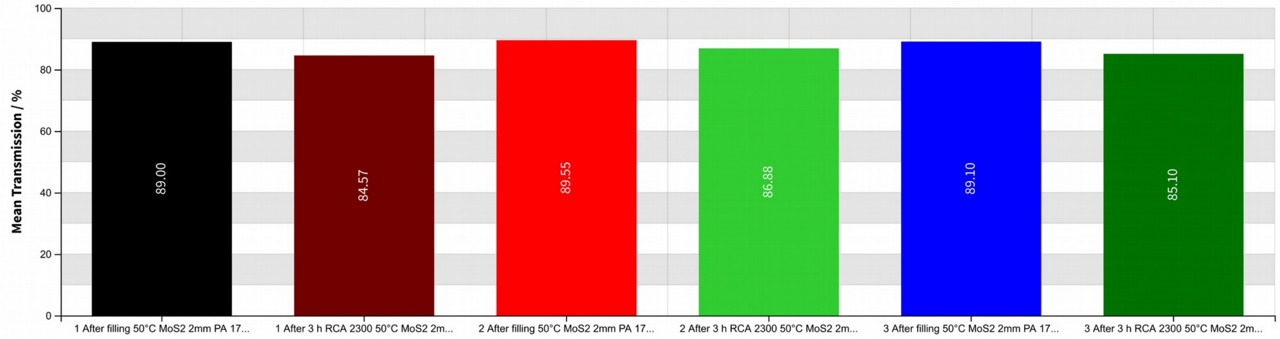
Topic: Testing and measurement techniques
Type: Oral presentation
Abstract no.: A-158
Status: submitted

Multistep analysis of lubricants – high analytical performance, low environmental impact

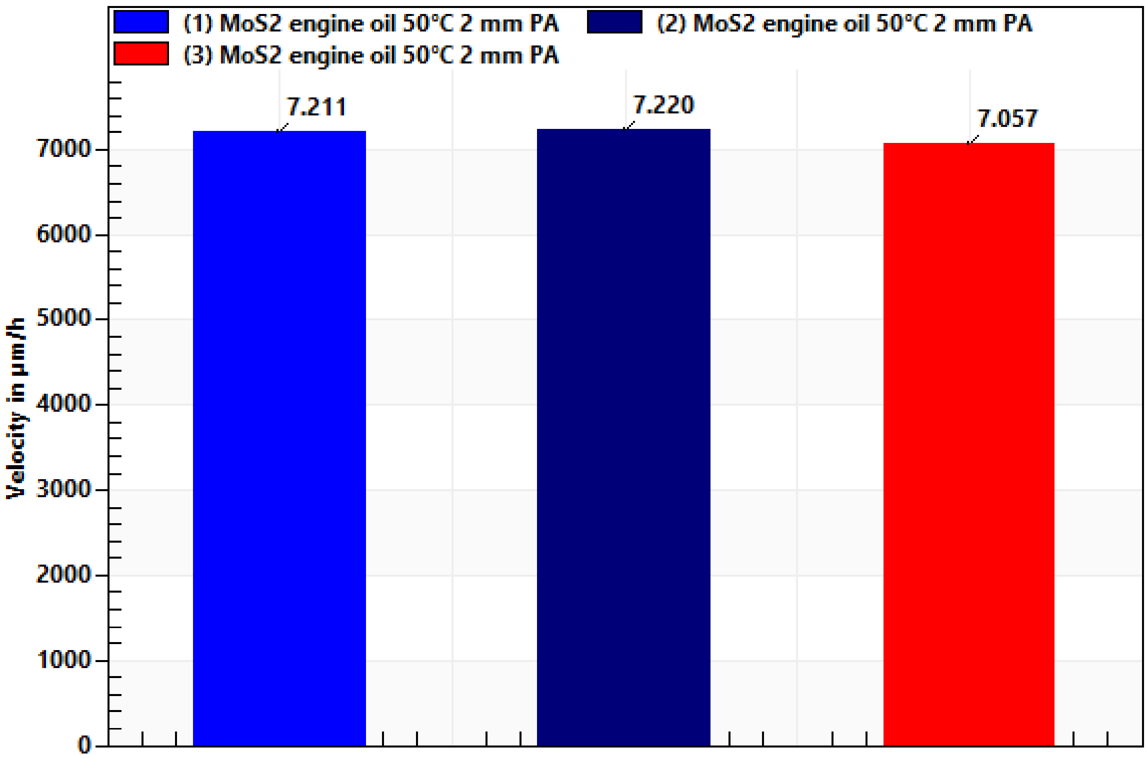
A. Uhl, V. D. Nguyen, A. Zierau
LUM GmbH, Berlin, Germany

Content

Graphite and molybdenum disulfide (MoS_2) are frequently used as solid lubricants, as are finely dispersed non-ferrous metals, ceramic particles (titanium nitride), or plastics such as PTFE (Teflon). Suitable soft metals include aluminum, copper, lead, indium, tin, and, at higher temperatures, gold, platinum, and silver [1].
On the example of MoS_2 in an engine oil, this paper discusses how to overcome challenges in particle and dispersion characterization of lubricants by applying an analytical multistep procedure, considering also the environmental impact.
Sustainable testing and less environmental impact are realized first by the small sample volume needed, typically 0.4 ml for turbid samples, like MoS_2-based engine oil or graphite-based products. Second, the use of a closed disposable sample cell (like the used, made of polyamide, PA, with optical pathlength of 2 mm) avoids any large volumes of solvents, e.g. for cleaning of instruments and glassware in “open” sample testing methods.
In 2025, new achievements in X-ray based Space and Time resolved Extinction Profiles (STEP-Technology) have significantly extended the analytical access to particle and dispersion characterization in lubricants, whenever inorganic components play a role, like those mentioned in the beginning. Now, lubricants also can be characterized by X-ray in a large temperature range, resultingly the particle size distribution can precisely be determined. Same for wear metals in lubricant oil, when optical detection wavelengths do not see changes. The particle volume concentration in sediments can now easily be calculated.
To benefit from the high analytical performance, this three-step procedure with multiple energy sources is suggested for lubricants:
1. Characterization of the dispersibility of particles like MoS_2, non-ferrous particles, ceramics and soft metals into liquid phases and of the resulting sample homogeneity by X-radiation.
2. Accelerated stability testing monitored by optical wavelengths and/or real-time separation testing monitored by X-ray with optional determination of the particle size distribution. Different kind of acceleration can be achieved by higher temperature at gravity or at higher gravity.
3. Determination of the sediment characteristics, including the volume concentration of the sedimented particles by X-radiation.



Multistep analysis: Steps 1 and 3. MoS2 in engine oil in triplicate. Mean integral X-ray transmission after filling (each left) and after accelerated separation at RCA 2000, 50 C for 3 h (each right). Sediment region. X-ray transmission decrease corresponds to the particle concentration increase.



Multistep analysis: Step 2. Sedimentation kinetics of MoS2 in triplicate. Determined in 30 min at RCA 2000, 50 C. Detection wavelength 870 nm. Threshold 5 % transmission, focus on fast particles.

Sample Name	Velocity in µm/h	StdDev in µm/h	Mean RCA in g	Corr. coeff.
(1) MoS2 engine oil 50°C 2 mm PA	7.211	165,6	1.990	0,9982
(2) MoS2 engine oil 50°C 2 mm PA	7.220	155,5	1.983	0,9984
(3) MoS2 engine oil 50°C 2 mm PA	7.057	168,3	1.986	0,9985

Sedimentation velocity with standard deviation at the mean relative centrifugal acceleration (RCA). Correlation coefficient confirms the applicability of the linear regression to determine the sedimentation velocity.

The formulation in original concentration is filled into one sample cell and undergoes three independent subsequent measurements, after filling, during accelerated separation or during real-time-settling and after the separation process. With the available observation by naked eye a matrix of four different information is obtained, which allows for a comprehensive understanding of particle properties and of the physical separation behaviour of formulations.

[1] https://de.wikipedia.org/wiki/Festschmierstoff, 30.4.2025

Authors

First author: Arnold Uhl
Presented by: Arnold Uhl
Submitted by: Arnold Uhl