APPLICATION NOTE

ICP-Optical Emission Spectroscopy

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OilPrep™ 8 Oil Diluter: High-Throughput Sample Preparation for Wear-Metals Analysis

OilPrep 8 Oil Diluter

Introduction

Used lubricant wear-metals analysis provides significant cost-savings for large scale industries such as oil and gas exploration, heavy equipment operations, mining and lumber industries, aerospace and environmental testing agencies. Within these industries, lubricant testing focuses on three key areas: *wear metals* (indicating deterioration of mechanical parts), *additive metals* (metals added for enhanced lubrication), and *contaminant metals* (foreign metals associated with dirt or coolant). When successfully applied, wear-metals analysis extends the life of replaceable fluids, minimizes unexpected operational down time by identification of damaged parts or impending failures, and also reduces environmental waste generation.

To meet the need for increased throughput in wear-metals analysis programs, PerkinElmer now offers a new high-throughput OilPrep 8 oil diluter equipped with ultrasonic liquid level detection (patent pending) and an 8-tip Varispan™ pipetting arm option for rapid "on-the-fly" reformatting and diluting of samples in various-sized vessels. The OilPrep 8 is a dedicated oil diluter that provides fully automated sample preparation for analyses such as ICP (inductively coupled plasma). Automated sample preparation is compatible with oil screening programs such as Caterpillar S•O•S℠ (scheduled oil services) program, military JOAP (Joint Oil Analysis Program), and ASTM® (American Society for Testing and Materials®) methods.

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This application note presents liquid handling performance capabilities of the OilPrep 8 oil diluter. Analytical results were determined for used oil samples and certified double-blind reference standards.

Case Study 1: Pipetting Performance Testing

Materials and Methods: Kerosene solvent (reagent-grade, Sigma-Aldrich®) and two commercially available 30W and 90W oils were selected to represent the range of viscosities present in motor oil testing laboratories. Relative densities for these fluids were determined using manually prepared standard curves (weight vs. volume). Oil sample volumes ranging from 300 uL – 900 uL were dispensed into tared 1.5 mL microfuge tubes (Axygen Biosciences®) using an OilPrep 8 oil diluter with an 8-tip Varispan pipetting arm, VersaTip™ option, and 1 mL disposable tips (PerkinElmer). Kerosene is a common oil-sample solvent used in ICP wear-metals analysis. Kerosene samples were pipetted into tared 17 mm vials (VWR®) using PerkinElmer Teflon®-coated VersaTips. Post-dispense weights were determined for each volume. Resulting weights and volumes were validated against previously hand-diluted density curves. Data were plotted as dispensed volume versus requested volume.

Results and Discussion: Pipetting oil presents several challenges for automated sample preparation. The ability to accurately deliver lubricant samples directly impacts downstream interpretation and ultimate effectiveness of any wear-metals screening program. Previous liquid sensing methods have been limited by the sample non-polarity, viscosity, color, density, or ambient room lighting. Liquid level sensing minimizes pipette tip submersion depths to reduce sample adhesion to the tip exterior, thereby enhancing accuracy of delivered samples, and eliminating sample-to-sample carryover. The use of non-contact ultrasonic liquid level detection (patent pending) in the OilPrep 8 oil diluter overcomes these challenges.

WinPREP® software's liquid handling performance files customize pipetting parameters. WinPREP can accommodate multiple performance file libraries with tailored settings such as pipetting speeds, slopes, offsets, delay times, and air gaps for optimized performance. In the OilPrep 8 oil diluter, performance files address various ranges of viscosities, particles sizes and densities. To demonstrate the combined use of ultrasonic liquid level sense and optimized performance file capabilities, oil and kerosene solvent were pipetted over a range of volumes. Oil and kerosene pipetting accuracy measurements fell within 4% or better of the requested volumes (Figure 1).

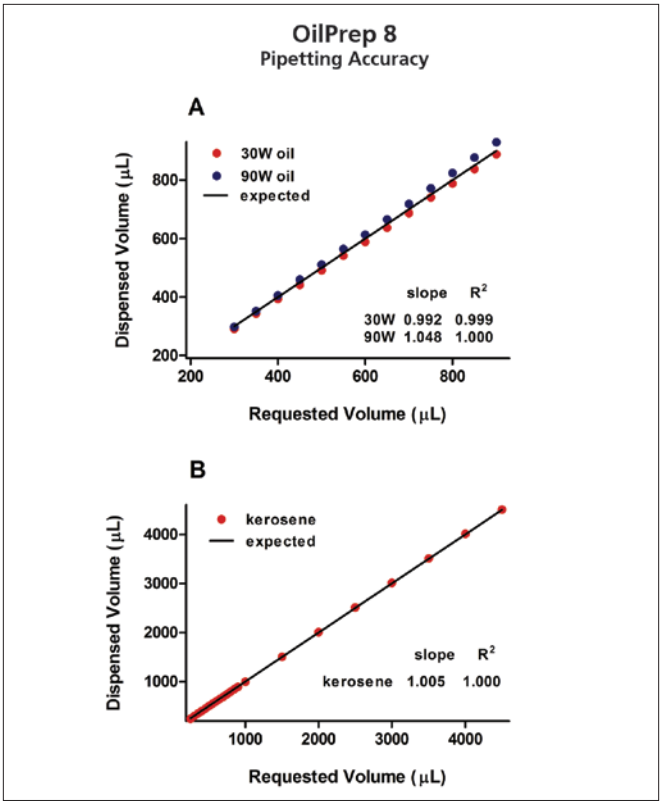

Figure 1. **OilPrep 8 oil diluter pipetting performance for oil (A) and kerosene solvent (B).** Data represent pipetting accuracies across a range of volumes, using either 1 mL disposable tips (oil samples), or fixed tips (kerosene, PerkinElmer VersaTip) with optimized WinPREP software performance files.

Case Study 2: ICP Wear-Metals Analysis – Double Blind Standards

Materials and Methods: Validation of completed wear-metals sample preparation (oil dispense and subsequent dilutions with solvent) was performed using commercially available Wear Metals Performance Testing Program (WM-PTP) double blind standards (VHG Labs®, Inc.). Standards were diluted ten-fold in triplicate using the OilPrep 8 oil diluter with optimized performance files and 1 mL disposable tips. Duplicate samples were diluted manually for comparison purposes.

Diluted samples were evaluated by inductively coupled plasma optical emission spectroscopy using a PerkinElmer® Optima™ 4300 V ICP-OES. Resulting data were plotted as parts per million (ppm) for individual metals types contained within the diluted standards. Data were provided courtesy of Caterpillar S•O•S℠ Services Laboratory, Peoria, IL.

Results and Discussion: The WM-PTP is designed to provide analytical laboratories with a method of monitoring their analytical performance as measured against Certified Reference Materials.[1] WM-PTP reference standards are oil samples containing known elemental constituents at precise concentrations. A series of these reference standards were prepared using the OilPrep 8 oil diluter.

ICP-OES analysis results fell within acceptable ranges for diluted VHG PTP reference standards: *±10 % at concentrations >20 ppm, and ±1 ppm for certified values <10 ppm.* Results demonstrated accuracies within 5% of manually diluted standards for samples greater than 10 ppm (Figure 2).

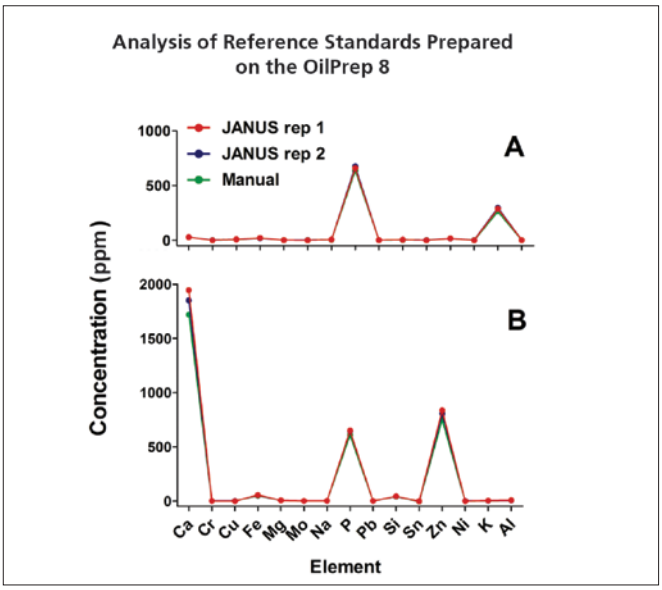

Figure 2. **ICP wear-metals analysis for samples prepared using the fully automated OilPrep 8 oil diluter.** VHG PTP double-blind standards were prepared manually and using the OilPrep 8 oil diluter, followed by analysis using a PerkinElmer Optima 4300 V ICP-OES. Two representative data sets (A and B) are shown.

Case Study 3: ICP Wear-Metals Analysis – Used Oil Samples

Materials and Methods: Reproducibility of pipetting performance was determined using 24 unknown used oil samples. Identity and concentrations of 15 wear metals (Cr, Cu, Fe, K, Mg, Mo, Na, Ni, P, Pb, Si, Sn, Al, Ca, and Zn) were evaluated using the Optima 4300 V ICP-OES. Two independent runs were conducted and concentrations (ppm) plotted. Linear regression was performed to assess reproducibility. Data were provided courtesy of Caterpillar S•O•S Services Laboratory, Peoria, IL.

Results and Discussion: To demonstrate the utility of automated oil sample preparation, used oil samples submitted to Caterpillar S•O•S℠ Services Laboratory, Peoria, IL were processed on the OilPrep 8 oil diluter and analyzed for wear-metals elemental concentrations. Duplicate sample runs and subsequent analyses were evaluated for reproducibility. Concentrations of elements in each sample were plotted and linear regression reveals strong run-to-run reproducibility (slope = 1.024, R^2 = 0.997, Figure 3).

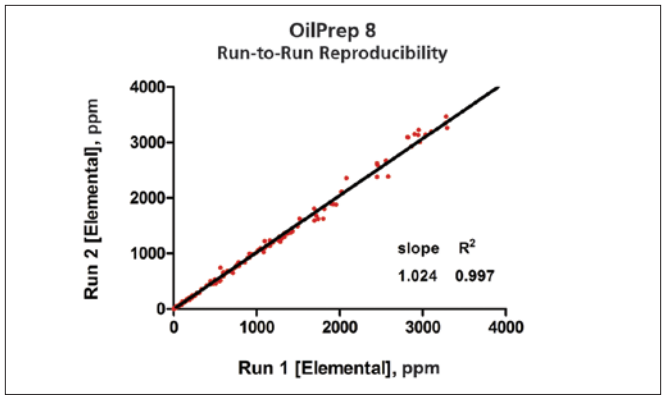

Figure 3. **Consecutive run-to-run reproducibility of fully automated used oil wear-metals ICP-OES sample analysis.** Data represents analysis of 15 wear-metal contaminants for 24 samples, plotted as measured concentration (ppm) across two consecutive runs.

Conclusions

Continued advancements in automated liquid handling, including the ability to integrate analytical instruments, have addressed increased demand for high-throughput lubricants screening programs. Effectiveness of these programs requires laboratories to quickly and accurately screen large numbers of samples with minimal turn-around time to customers. Automation of sample testing provides a highly robust and reproducible method for reducing screening time and labor costs.

- The OilPrep 8 oil diluter effectively prepared oil samples for analysis by inductively coupled plasma optical emission spectroscopy (ICP-OES). Results from these measurements are the same as those from oil samples prepared manually.
- Utilizing ultrasonic liquid level detection, the OilPrep 8 oil diluter has been shown to accurately dispense oil and solvent of various viscosities. When combined with pipetting functions, liquid level detection introduces a controlled-contact aspirate capability (pipetting at surface level) that adapts to variations in sample-source volumes.
- Demonstrating a high degree of liquid handling accuracy and precision with disposable pipette tip options that eliminate carryover and minimize tip wash time, the OilPrep 8 oil diluter executes oil sample dispensing with superior run-to-run reproducibility.

References

1. Instruction Manual: Wear Metals Performance Testing Program (WM-PTP) Featuring Real-Time On-Line Result Evaluation. VHG Labs, Inc. Manchester, NH.

Figure 4. **OilPrep 8 oil diluter with 8-tip Varispan pipetting arm and 1 mL disposable tips.** A chemically resistant fluid path can accommodate up to eight independent system liquids, allowing multiple assays to be performed on a single automated platform. Custom racks, flexible deck layouts, and optional deck sizes (Mini, Standard, and Expanded decks) provide modular adaptation to unique protocols and vessel formats. Shown here: wide mouth sample bottles and 17 mm ICP-OES destination vials.

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