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Application Note #AA3005

Determination of Wear Metals and Additives (Soaps) in Lubricating Oils by Atomic Absorption

Lubricating & hydraulic fluids are crucial to the proper functioning of many types of machines: automobiles, farm equipment, heavy-duty motors, airplanes, and military hardware. Most “lubes” are simple hydro-carbon based oils or greases that have certain additives in them to prevent wear and abrasion of components (piston rings, bearings, etc.) or oxidation degradation of the oil. These additives are typically “soaps” made from an organic acid and a metal (usually barium, zinc, calcium, or magnesium). As the oil ages, these materials are continually lost and need to be monitored so that they can be replaced to provide the proper level of protection.

Wear metals are formed in lube oils under the harsh conditions of temperature and pressure that occur in heavy machinery. Under continuous heat and pressure, the surface of the metal piece becomes slightly oxidized, forms salts with the degradation products of the oil, and becomes soluble in the oil. The friction of motion in machinery also causes micro-fine particles to actually shear off the surface and become suspended in the oil. Determining what these “wear metals” are and their respective levels is very important to the proper maintenance and operation of the equipment. Copper & tin can come from bearing wear, lead & antimony come from Babbit metal bushings, chromium & molybdenum from piston rings and seals, nickel & iron from crankshaft wear and engine body damage, aluminum & silicon from catalyst residues, and sodium & silicon from bad air filters and from water / coolant leaks. Careful monitoring of these materials can be used in preventative maintenance of large, expensive engines, minimizing potential repair costs.

One of the best technologies for this type of determination is Atomic Absorption Spectrometry. There are several techniques for the analysis of metals in petroleum matrices by AAS. The simplest is dilution of the oil with a solvent like MIBK or kerosene, and analysis using organo-metallic standards. This solution burns well in an air / acetylene flame and gives very good detection limits. Another calls for the dry ashing of the sample, where the organics are burned off to leave an inorganic residue (ash). The ash is dissolved in acids and analyzed as an aqueous solution. However, significant errors can occur in this method because many metals are easily volatilized (sublimation, distillation), giving low assays. Recently, the advent of microwave digestion technology enables the chemist to decompose the oil with acids under pressure. This results in a clear, aqueous solution that can be analyzed using simple water-based standards. For any of these methods, the Buck 210VGP system and an optional MEGA-1200 Microwave Digestion system are ideally suited for rapid and economical determinations of wear metals and additives in all types of hydrocarbons and lubricant media.

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Analysis of Wear Metals and Additives in Lubricating Oils

Sample: 30W Engine Oil; abused & oxidized, 0.1 – 10 micron particulates.

Preparation: (1) 10 grams diluted with 3:1 MIBK / Xylene solvent to 100ml
(2) 10 grams dry ashed @ 650°C, dissolved in acids to 100ml
(3) 10 grams microwave digested in HNO_3 / $HClO_4$ / H_2O_2 to 100ml

Calibration: Buck Certified AA Standards; Aqueous and Organo-Metallic, at 0.5 ppm and 5 ppm levels in appropriate matrices.

Instrument: Buck 210VGP Atomic Absorption Spectrophotometer.

Conditions: Air or N_2O / Acetylene flame, normal parameters.

Values are mg / kg (ppm) in the original sample unless otherwise noted.
AVG / sd = Average & deviation of 3 preps; D.L. = 3-sigma detectability.

		Method (1)		Method (2)		Method (3)	
Element	**Wavelength (nm)**	**AVG / sd**	**D.L.**	**AVG / sd**	**D.L.**	**AVG / sd**	**D.L.**
Ag	328	2.7 / 0.4	0.25	1.9 / 0.8	0.08	2.5 / 0.1	0.01
Ca	240	115 / 8.5	0.65	103 / 23	0.25	122 / 6.5	0.20
Cr	357	32 / 2.1	0.50	24 / 4.7	0.12	30 / 1.6	0.03
Cu	324	12 / 0.5	0.22	11 / 1.3	0.03	12 / 0.1	0.01
Fe	248	47 / 0.9	0.43	44 / 2.4	0.05	45 / 1.1	0.02
Na	589	4.9 / 0.8	0.46	3.1 / 1.1	0.31	5.5 / 0.4	0.17
Ni	232	8.4 / 0.7	0.55	7.9 / 1.4	0.17	8.1 / 0.5	0.12
Pb	283	18 / 0.9	0.65	11 / 3.7	0.27	16 / 0.4	0.15
Si	251	5.4 / 1.1	0.77	3.6 / 1.5	0.45	5.8 / 0.7	0.31
Sn	224	28 / 2.1	0.75	19 / 5.8	0.34	26 / 1.0	0.25
Zn	214	205 / 4.2	0.51	188 / 6.8	0.22	196 / 3.1	0.13

The above data shows the powerful flexibility and stability of the Buck 210VGP system for the wide-ranging requirements of the "wear metals" industry. The high precision of the major and trace data exemplifies the accuracy of the instrument. The overall high sensitivity of the various trace metals supports the interference-free quality of the data. Together, the combination provides for an unmatched pair in economy and performance.

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