A General Method to Investigate Longer Term Interactions between Minor Components in Complex Lubricant Formulations. A Forensic Lubricant Formulation Test

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1. Introduction: Automotive lubricants are complex formulations of over twenty major and minor components, each of which has a range of molecular weights, reactivities and related structures. Whilst each component has a main function in the lifetime of the lubricant, they often have additional multifunctionality. Undesirable cross-additive interactions can occur during the lubricant's service lifetime, negating a desired additive action.

It is now common for service intervals of European vehicles to be over 20, 000 km, and increasing, or two years, Over this extended time or service interval, low intensity/slow interactions between minor additives now become increasingly important. Lubricant operating temperatures have increased as volumes have decreased. Additives are either acids or bases; increased temperature, longer time for reaction, increased circulation rates for smaller volumes will, taken together, accelerate reactions between minor components.

Counterfeiting of standard and premium lubricants is increasing in volume and also sophistication, stretching bench analytical methods to their limits and beyond.

But investigating interactions between minor components of a complex formulation is difficult. A simple, step-wise, univariate matrix approach for each component in turn is tedious, expensive, probably insensitive for its purpose and not cost effective.

2. Methodology and Analyses: An alternative approach to investigating the interactions between minor components is described which simultaneously varies several components of complex lubricant formulation in an experimentally designed matrix of tests to generate a data base. Multivariate data analysis can then identify contributions from each component.

The necessary sequential steps for this general method of investigation are:

- a suitably accelerated engine test at least two power levels, in this case a Reduced Oil Total Volume, ROTV, version of a standard test engine,
- a meaningful, experimentally designed, matrix of lubricant formulations for different structures of reactant additives, in this case, mono-, bis- and tris-succinimide dispersants, together with primary and secondary ZDDP's,
- multiple lubricant sampling during the course of the engine tests and subsequent suitable analyses,
- normalisation of test results and assembly into a data base,
- test of parameters for significance, rejecting parameters which do not affect the results,
- multivariate analysis of the significant test parameters using the **Partial Least Squares-Discriminant Analysis** method, **PLS-DA,**
- leading to discrimination between the actions and interactions of additives.

3. Results: These steps will be used to illustrate a clear differentiation between the performance of mono-, bis- and tris-succinimides in formulated lubricants and their interactions with primary and secondary ZDDP's. The balance of these interactions change for different power levels.

An example will also be given of the use of this general investigative method to the forensic detection of marginally different lubricant formulations using performance tests and subsequent analyses.

4. References: M.R Sutton, PhD thesis, De Montfort University, 2003; M.A Aruna, PhD Thesis, De Montfort University, 2007.