Identification of Lubricants – a New Approach

Lubricants become more and more parts of the construction in tribological systems. But in case of an oil change: How the performance of the fresh lubricant can be guaranteed? A novel approach from Fuchs for the online-identification of engine oils while filling fresh oil will be presented.

1 The Current Situation

Today's high-performance engines with

- High-pressure direct injection, e.g. Common-Rail or Unit Injector System
- Long oil change intervals, ASSYST technology, Condition Based Service, LongLife Service
- Exhaust gas treatment systems, e.g. three-way catalysts, SCR Technology or diesel particulate filters

all require high-performance engine oils which fulfil OEM requirements. Unsuitable or specification-deficient engine oil leads to a progressive drop in performance in the form of increasing fuel consumption or worsening emission values and possibly even serious engine failure.

Moreover, there can be measurable differences in quality among specified oils – engine oils can influence oil change intervals or the varying life of engine components. In addition, the engine oil influences fuel consumption, the effectiveness of exhaust treatment systems and emissions.

Complex bench tests, field trials and practical experience concerning the influence of specified engine oils on the performance and life of engine components have led to reliable oils. Specification-deficient oils generate risks and cause uncertainty. In future, the use of defined engine oils will become more important, e.g. regarding increasingly stringent emission standards.

In recent years, engine oils have become more and more of a constructional element in vehicles and are now an integral component of engine design. As a result, it should also have an identification number like other components but this may appear to be a far-fetched idea. Engine oil is an "unknown" engine component at present. Other parts can be subsequently identified along with the manufacturer; every cheap plastic part in today's vehicles has an identification number – but not engine oil. It is fluid and it changes its characteristic features rapidly during use, in some cases, as soon as it is mixed with other (used) oils when topping-up. Subsequent identification is either analytically impossible or extraordinarily complicated and expensive and even a plausible identification is far from having evidentiary value (guarantee, good will, etc).

Considering this situation, it may seem a good idea to give the engine oil containers an identity code. This would be a simple and immediately available method. But such indirect identification would be highly vulnerable to errors and falsification and if the oil came from a large tank, also technically difficult. The direct coding of the engine oil would be a much better option.

2 Possible Solutions

The following solution proposes marking the engine oil with a fluorescent substance for online identification when oil is added [1]. Alternatively, similar tasks would most certainly be much more complicated. In the past, for example, coding proposals were made but the marking substances could only be deciphered "offline" in complex laboratory procedures. Such methods were aimed at copy-protection and not for the real-time recognition when oil is added. The new ideas build on these foundations. Identification systems should display the following features:

- Should offer a characteristic, clear and copy-proof marking of the specified, approved oils
- Systems should (at least optionally) differentiate between different specifications and not just oils
- Number of available codes must be sufficiently large
- The marking should have no detrimental effect on oil characteristics and the concentration of marking substances should be as low as possible
- The on-board sensor location with which the coded oil is identified when oil is added should be simple, cost-effective, robust, reliable and durable
- The system must be simple to use and be ecologically and toxicologically harmless.

An advanced study by the lubricant manufacturer FUCHS to determine possible suitable sensor principles showed that within the framework of the above-mentioned parameters, the spectrofluorometric online-determination was particularly suitable but only when fresh oil is added and when the complex matrix of "used oil" does not compromise accuracy, **Figure 1**.

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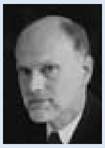

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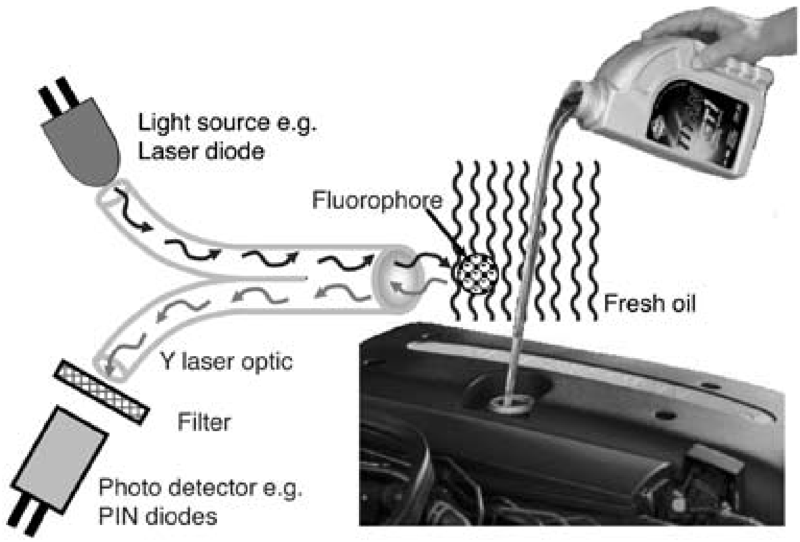

Figure 1: Online engine oil identification schematic

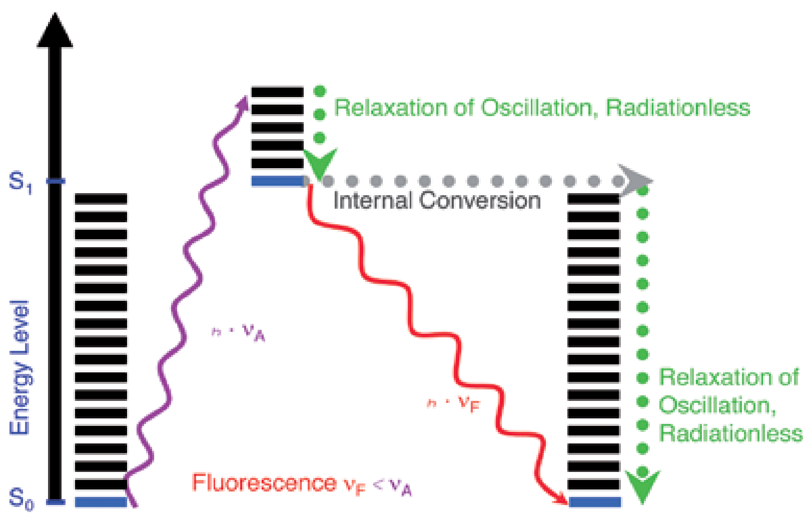

Figure 2: Jablonski Diagram for fluorescent radiation

3 Spectrofluorometry

Fluorescence is an optical phenomenon in which the molecular adsorption of a photon triggers the emission of another photon with a longer wavelength. The energy difference between the adsorbed and emitted photons ends up as molecular vibrations (or ultimately as heat). Usually the adsorbed photon is in the ultraviolet range and the emitted light (luminescence) is in the visible range. A schematic illustration is shown in **Figure 2**.

There are a number of natural and synthetic fluorescent dyestuffs. Generally known are, e.g. fluoresein and other phthaleins, coumarine-derivates, rhodamine and various proteins.

Examples of the use of fluorescence can be found in literature [2, 3, 4] .

4 Application of Fluorescent Marking of Engine Oils

This proposed development began with a feasibility study commissioned by Fuchs Petrolub AG and Robert Bosch GmbH and carried out at the University of Heidelberg's Institute of Physical Chemistry. Could uniform but not identical fluorescent markers be found for the enormous number of engine oils on the market? In principle, this depends on whether engine oils on the market had a systematic and sufficiently large "frequency gap" in their fluorescence spectrum in which suitable fluorophores at the lowest possible concentrations would be quantitively detectable. One of the initial tasks was to record the adsorption spectrum of a representative sample of engine oils on the market. This showed that the frequency range of 500 to 1,000 nm was well-suited for marking for a number of reasons – because there were no interfering adsorptions by base oils or additives – and this despite large variations in the additive chemistry used in the oils. Furthermore, suitable dyestuffs are available for this frequency range which proved to be suitable in principle after initial solubility tests. Relatively cost-effective and reliable detection systems are also available.

4.1 Selection of Suitable Dyestuffs

In a second step, different fluorescent dyestuffs were tested with the objective of finding marking substances for two different frequency ranges which could serve to uniquely mark oil. As described above, these dyes have to fulfil a number of demands. Absolutely essential is that they are easily oil-soluble [5]. In addition, the corresponding dyestuff should display high absorption in the green or red spectrum to ensure the reliable coding in the identified frequency range ($\lambda >$ 500 nm). The dyestuff selection results can be summarized as follows:

- An initial selection of possible dyestuffs proved to be suitable: the fluorophores are soluble in engine oils and even at low concentrations, provide a clear signal against the background signal of the engine oil, **Figure 3**
- Within certain limitations, the fluorescent signal is linearly dependent on concentration, no errors are caused by masking or agglomeration; quantity-analogous coding is thus possible, **Figure 4**
- Using current components, it was determined that marking required a dyestuff concentration of 10^{-7} to 10^{-5} mol/l
- The selected fluorescent dyestuffs showed no tendency to chemical changes (decomposition, failure) within a time-frame of now two years (in fully-formulated engine oil).

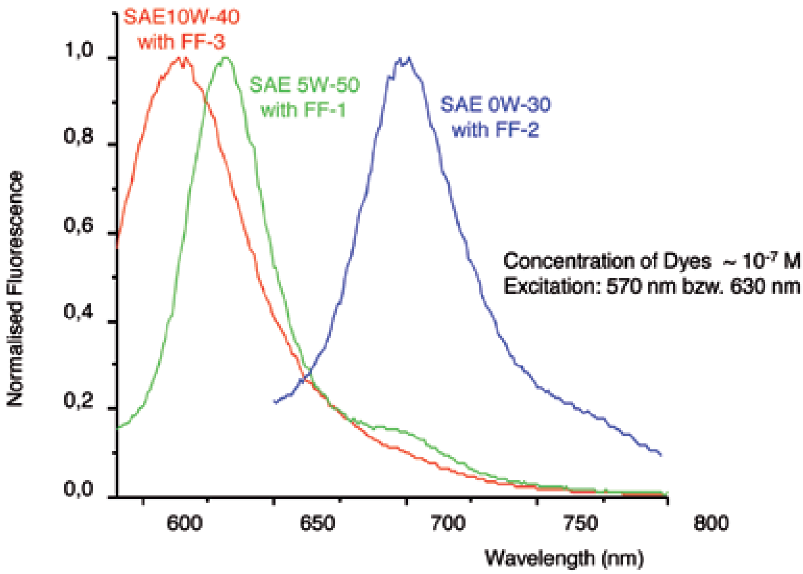

Figure 3: Engine oils with different dyes

4.2 Selection of Suitable Excitation Wavelengths and LEDs

There are two technical challenges involved in designing suitable detection equipment:

- Exciting the fluorescent dyestuff with LEDs of laser diodes
- Spectrally separate detection with photodiodes

With the aim of keeping future detecting hardware as cost-efficient as possible, one of the factors influencing the selection of the fluorescent dyestuff was that they could be exited by low-cost LEDs. A total of nine LEDs were tested.

Determining the excitation wavelengths also established the fluorescence detection windows. As part of the project, ten different engine oils were tested regarding a quantitative marking with dyestuffs.

Fluorescent dyes are extremely weak and are normally masked by the significantly greater intensity of the light in the illumination equipment. Because of shifts in frequency, it is possible to separate the excitation light and the weak fluorescence with a careful selection of optical filters. This ensures that only fluorescent light enters the detector.

Fluorescence is best observed at a right-angle to the excitation light source because this avoids interference between the excitation and fluorescent light, **Figure 5**. Excitation and emitted light could also be separated with optical filters.

To simplify the measurement process, ESE GmbH developed a novel fluorescence sensor. The heart of the sensor are the confocal optics. Excitation and emission light pass in parallel through one slit and are separated in the sensor by optical dividers and filters. This allows measurements to be performed irrespective of variance in distance and angle to the sample.

The complex optical filters used contain many layers which are specifically designed for a particular application. The compact optical detectors and very small filters significantly reduce the cost of filter manufacture.

Simple but high-performance LEDs are used to excite the fluorophores. The photodiodes and amplifiers used on the detector side generate an extremely low-noise signal.

This highly cost-effective sensor system offers outstanding resolution with maximum miniaturization. The measurement results are comparable with expensive laboratory equipment and 10,000 times more sensitive than previously-available hand-held apparatus.

4.3 Setting Up and Marking Matrix

As a reminder: The number of different codes should be sufficiently large to allow all of the different “Service Fill” oils on the market to be registered. Although only 20 codes and two colours were used in the first stage of the project, **Figure 6**, the second stage already can differentiate between 200 oils whereby the number of different dyestuffs should be strictly limited to four or five. The oils were therefore marked by a combination of dye chemistry and dye quantity although the necessary tolerances need to be respected to avoid so-called quenching processes and other sources of error.

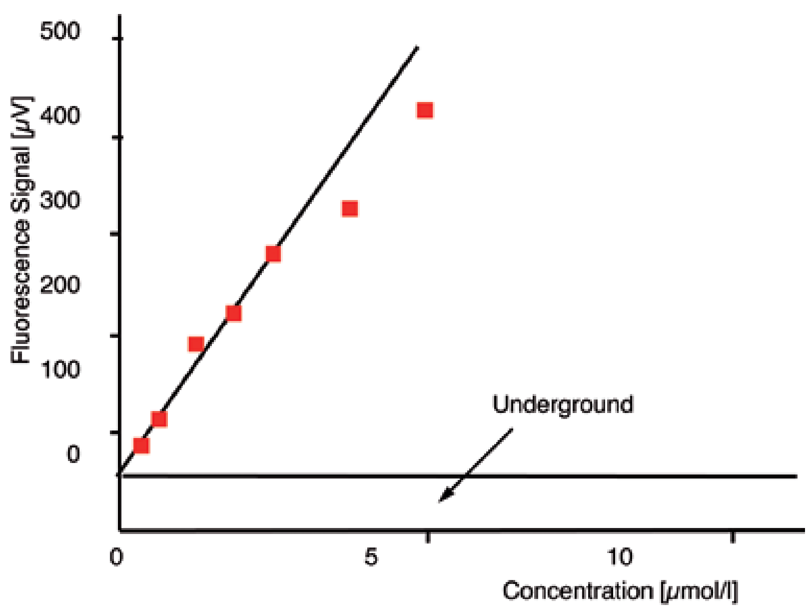

Figure 4: Intensity of a fluorescence dye – linearly dependent on the dye concentration, measured with an engine oil of Fuchs

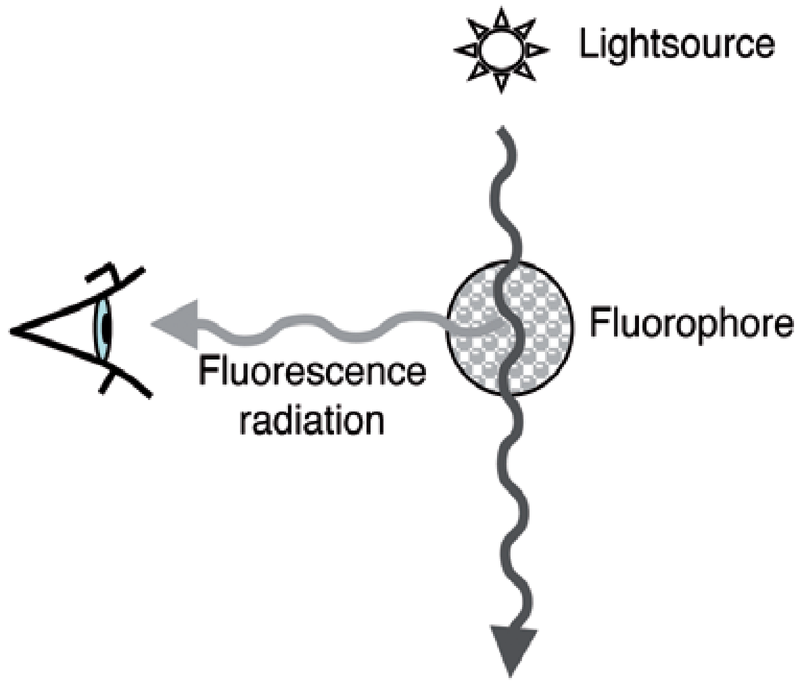

Figure 5: Observing fluorescence – ideally at right angles

Another project objective was that different engine oils with the same approval status should be recognized as being "equal" – the detector recognizes, so to speak, "specifications". Even "black-Box" lubricants, for example from competitors, can be marked with this system.

Based on the promising results of the Heidelberg study as well as addition work performed by ESE GmbH, Atto-Tec GmbH and Fuchs, the system was further developed into demonstration apparatus which can handle the functional elements and oil coding of about 20 different oils, **Figure 7**. This realisation is oriented towards the well-known „oil-filler neck".

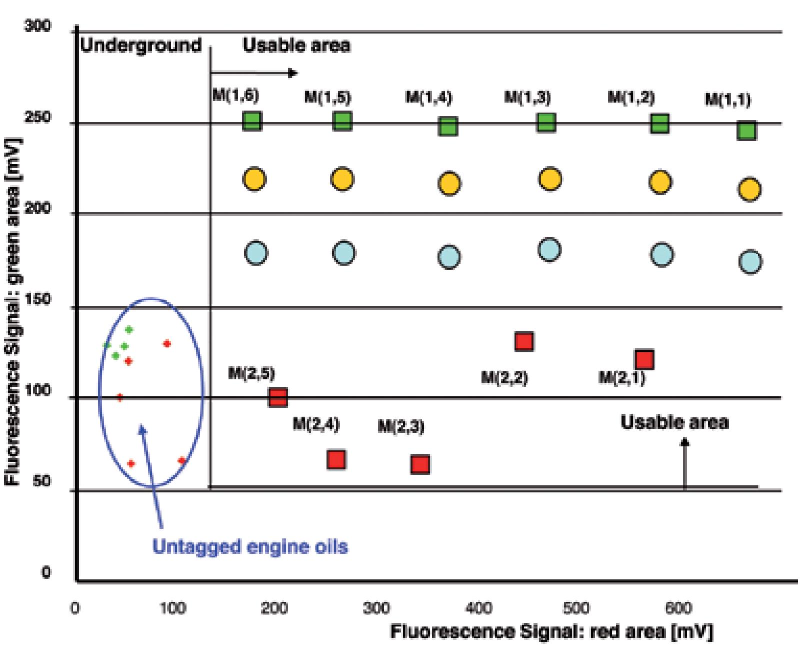

Figure 6: Sketch of a 4×5 array for marking of 20 oils with 2 dyes

5 Benefits for the Motorist

In the first section, the focus was on the OEM's viewpoint: the OEM has to guarantee the overall function of the vehicle and engine oil is increasingly being seen as an integral part of engine design – a trend in which ACEA specifications only reflect a part of all engine oil specifications. How does the end user benefit from the above system of identifying engine oil?

- The motorist has full control over which oil he gets when his car is serviced in a workshop – the incorrect use of a non-specified oil would be recorded
- The identification and storage of this information by the motor management module could avoid disputes in the case of warranty work
- The life of many engine components is largely influenced by the engine oil. For example, the life of particle filters. These expensive exhaust gas treatment systems only achieve their full working life if special, low-ash oils are used
- A non-conforming engine oil can lead to poorer performance, increased fuel consumption and worsening emission values, in other words, to changes which happen only very slowly and which are seldom attributed to the engine oil
- Finally, oil change intervals depend largely on the quality of the engine oil used. Ultimately, only engine concepts with optimally matched lubricants can achieve the objective of increasing oil change intervals – with the result that engine oils will become increasingly specific.

The increasingly common dynamic oil change interval scheduling with specific oil condition monitoring could also profit from oil identification. Current systems are only based on predictions concerning viscosity and other oil parameters because these systems cannot be calibrated to the actual oil in the engine.

For example, an advanced online-viscosity sensor can detect thinning of the engine oil but only as a relative change.

But to detect a fall in viscosity under a given threshold caused by, for example, fuel in the oil, the current factual viscosity must be known. Such absolute viscosity determination requires the calibration of the sensor to the engine oil in the sump and the oil must be recognized by the oil condition sensor. The oil condition sensor can be calibrated by the online identification of the oil used.

If the oil condition sensor detects a "dangerous" viscosity, the motor management could minimize or eliminate operating conditions which promote wear, i.e. by avoiding high loads at low engine speeds. As soon as the viscosity signal returns to normal, full engine performance is again available.

6 Summary and Outlook

The system presented here to mark and identify lubricants online is a response to current developments on the engine oil market. Increasing demands on engine lubrication require increasingly specialized oils. The accompanying logistical problems cannot be solved with such a marking system but can, at least, be made more manageable.

- The engine oil can be identified as an integral component in the operation of an engine
- The spectrofluorometrical online identification when oil is filled seems particularly well-suited
- Feasibility studies were positive and a test system was built and is available for demonstration purposes

Without doubt, there are a number of issues which need to be resolved before the presented system could go into general use. In particular, a harmonized oil-coding strategy needs to be developed by all manufacturers which also works in a competitive environment. The marking system was therefore developed as an open, "on-top" marking system. This new concept for the identification of engine oils when they are filled is now being discussed by specialists and the motoring world.

Naturally, the system could also be used to offer protection against product and brand piracy although this aspect was not in the forefront during its development. The combination of special fluorophore chemistry and the precise metering of dosages is probably protection enough against imitators.

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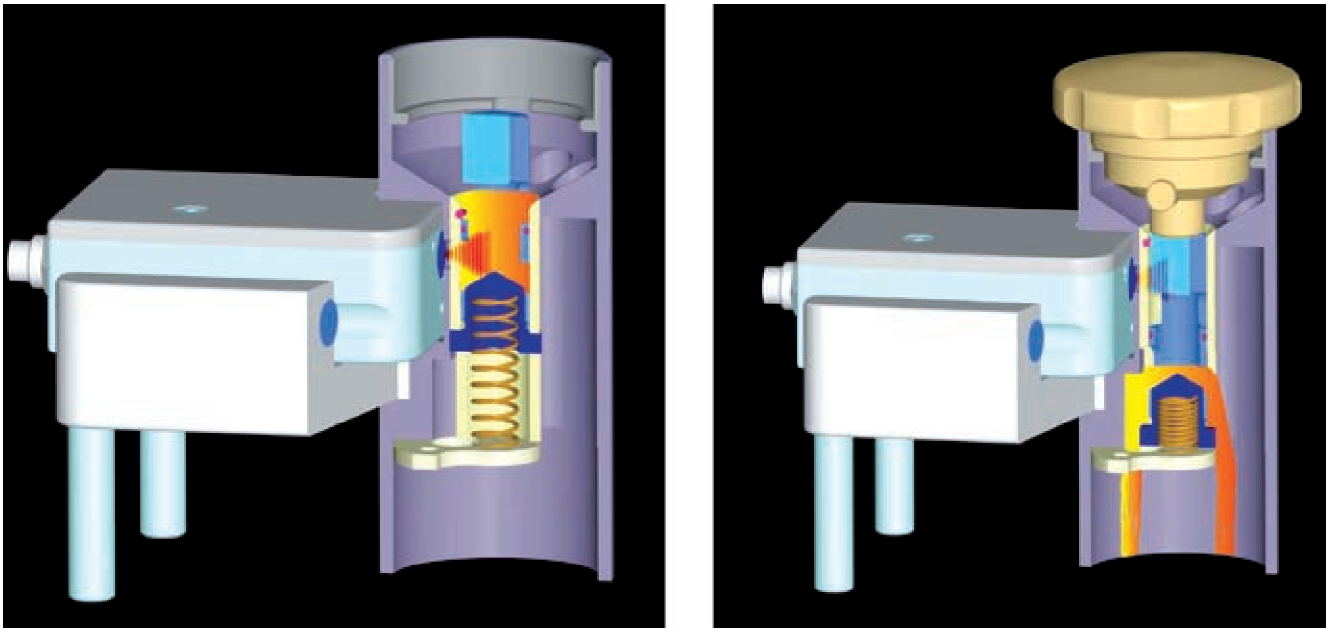

Figure 7: Oil-filler neck with detector unit, on the left while filling ("on-state"), on the right closed ("off-state")