Development of Reference Fuels and their Importance for Lubricant and Additive Development

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Summary ............................................................................................................... 1
1. Historic Review ............................................................................................. 1
2. The Effect of Varying Fuel Quality on the Performance of Engine Lubricants and Additives ......................................................................................................... 3
3. Development, Production, Storage and Delivery of Reference Fuels ............... 6
4. Requirements versus Future Reference Fuels .............................................. 7

Summary

The authorities in the U.S.A. specified the use of reference fuels in emission testing as early as the 1960s. Europe followed soon after that and the automotive industry introduced reference fuels not only for emission testing, but also in the development of lubricants and additives. Today, engine and operating fluids development without such fuels would be inconceivable. The number of “Legislative Fuels” specified by the authorities and of “Experimentation Fuels” demanded by the automotive industry is constantly growing. The production of reference fuels within narrowly defined tolerances requires experience and considerable technical resources.

This paper highlights the performance requirements and the particular methods used in the production of reference fuels.

1. Historic Review

Haltermann’s fuel activities can be traced back to the year 1910. At that time the reduction of exhaust emissions played practically no role; - “the best and most economic fuel for automobiles and engines of any kind”, as it was

Fig. 1: Haltermann poster from the 1920s
then claimed in advertisements, happened to be benzene which was produced from coal.

The fuels market and its various quality grades have since seen profound changes and today the fuel business activities of Haltermann comprise a wide spectrum of reference fuels and development fuels.

This includes the production of both Diesel- and gasoline grades for the following applications:

<table>
<thead>
<tr>
<th>Engine development</th>
<th>Durability testing</th>
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<tr>
<td>Lubricant development</td>
<td>Emission testing</td>
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<tr>
<td>Additive development</td>
<td>Certification</td>
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<td></td>
<td>Production monitoring</td>
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In 1968 Haltermann had to face the problems of test fuels for the first time. Only the U.S.A. had at that time legislation according to which engine manufacturers had to meet specific emission limits in order to obtain certification for their cars. Until then VOLKSWAGEN had imported an American test fuel into Germany which was used to optimize the ignition timing of their export models with respect to CO-emissions. Test activities increased to such an extent that VOLKSWAGEN had to look for a European supplier of the test fuel. Haltermann therefore investigated the requirements of this tightly specified fuel called “Indolene 30” and was subsequently able to provide VW with the appropriate quality product.

Dr. Klinksiek, who was at the time responsible for this subject had pointed out during discussions about “Indolene 30” that VOLKSWAGEN expected a revision of emission legislation in Europe, similar to that of the U.S.A. In order to stay abreast with these developments Haltermann’s management decided in 1969 to join the DKA (Deutscher Koordinierungs-Ausschuss), the German national organization within the CEC (Coordinating European Council for the Development of Performance Tests for Transportation Fuels, Lubricants and other Fluids). It was expected that the American specification for emission tests would initially be taken over in Europe, but it soon became apparent that the adaptation to European climatic conditions and fuel qualities as well as the desire for independence would necessitate the development of a second, parallel specification. As a result the first “Informal CEC-sponsored Meeting” took place at the Paris Airport on January 22nd, 1969.

The subject of the meeting was: “Reference Gasolines for Emissions Type Approval Tests”. From this followed the development of the first CEC specifications for reference fuels with the designations:

- **ERF / G30**, also called “Indolen” (according to US-specs)
- **ERF / G1**, (specification adapted to European conditions)

These fuels were so-called “certification fuels”.

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*Trademark of The Dow Chemical Company*
Soon after, the first reference Diesel fuel was defined. The designation was:

**ERF / D1 , (for Diesel exhaust smoke measurement)**

The testing activities which then followed provided first insights into the effects of varying fuel quality on exhaust emissions. Before long the experience gained was utilized in the development of additional and increasingly sophisticated engine tests. Soon tightly specified fuels were also demanded by the automotive industry for “non-emission testing”, for example for lubricant or additive development or for approval testing. Today reference fuels with certain tightly specified properties are an essential element of engine development. The CEC Reference Fuel Manual alone currently defines some forty fuels for a variety of applications.

2. The Effect of Varying Fuel Quality on the Performance of Engine Lubricants and Additives

Today’s European standards which define the quality of commercial fuels sold at filling stations, i.e. EN 228 for gasoline and EN 590 for Diesel fuel, partly contain rather wide tolerances for a number of performance parameters. This is to account for variable crude oil quality and for the circumstances of large scale production processes. Fuels meeting the relevant specifications guarantee trouble-free operation, particularly since modern electronic engine management is able to compensate certain variations, for example by the use of knock-sensors.

Engine test procedures designed to evaluate the performance of lubricants and of additives require not only standardized test engines, test procedures and reference oils, but also fuels which have the least possible variations of their chemo-physical data.

Test fuels have to meet two basic requirements:

- **To relate to the performance parameter desired in the test**
- **To guarantee reproducibility of fuel quality / test results**

The second point is equally important as the first one and it often presents the greater challenge. Constant fuel quality is ensured during batch wise production in order to minimize variations of fuel data which would affect the repeatability and the reproducibility and hence the significance of a test result. Some examples illustrating this are shown below.

The most important parameters, particularly those affecting the test result in question, are kept constant for many consecutive batches.
The tolerance limits of the CEC specification, which are already very narrow, are not utilized by far.

The fact that merely meeting current commercial fuel standards (for example EN 228) is insufficient to satisfy today’s requirements relative to repeatability and reproducibility of engine tests can be demonstrated by the following example:

CEC Working Group PF-020 is developing an engine bench test to quantify the tendency of gasoline to produce inlet valve deposits (IVD). The test is designed to discriminate between fuels of different quality in order to allow the development of additives to reduce the formation of these deposits. A non-additized gasoline is used for reference purposes. If typical fuel according to EN 228 with the normal quality variations of the market was used, the performance improvement provided by an effective additive could be masked by test result variations due entirely to the variability of the basic test fuel.

Test results are quoted as average quantity of deposit (in mg) formed per cylinder. Fig. 5 shows test results from 3 fuels of different origin, each meeting EN 228 requirements. All results shown here were obtained on the same test bench with the same engine.
The extremely large deviation of test results would practically prevent the development of additives since a significant effect of the additive on the test result may not be recognized. *Base fuel response to additive treatment varies and hence test results would not be repeatable, let alone reproducible, if different base fuels were used for different tests on the same additive.*

The aromatics content of engine fuels is a parameter which has been studied extensively with respect to its effect on the combustion process and on exhaust emissions. Specially formulated model fuels were used for this purpose. Currently ongoing development work comprises more differentiation by investigating the effect of aromatics type and even of single substances.

The desire to reduce fuel consumption and concurrent efforts to reduce exhaust emissions resulted in increased demand for fuels with a wide variety of different oxygen contents and components.

Fuel sulphur was originally identified as primary cause of “acid rain”, which resulted in efforts to reduce its content, leading to extensive testing activities with fuels of different sulphur levels. In connection with further reductions, particularly with Diesel fuel, a new problem appeared: lack of lubricity. Additional reductions of sulphur content resulted from the fact that sulphur acts as catalyst poison with new exhaust aftertreatment devices.
3. Development, Production, Storage and Delivery of Reference Fuels

Fuels are extremely complex multi-component mixtures. Each component itself consists of a mixture of different components. The selection of suitable components is a principal issue which represents a large part of the “know how” in fuel development. Components are often available which theoretically or even in a real laboratory sample meet the requirements, but are not suitable in the end for a number of different reasons.

The main aspects which have to be observed when selecting the appropriate components are:

- **Function in the final product**
- **Availability**
- **Repeatability**
- **Chemo-physical properties**
- **Interaction with other components / parameters (alcohol/RON)**
- **Stability and shelf life**

For the production of a reference fuel it is first necessary to identify components which meet all the above requirements and at the same time are not untypical for the petroleum industry.

Once all the requirements have formally been met with respect to chemo-physical data, by model calculations and by experimental laboratory blends the fuel has to demonstrate its test-specific properties. For this purpose it is usually necessary to run full-scale engine tests or laboratory tests. If these tests show that fuel performance is in the desired range, large-scale production can begin.

For fuel production the selected components first have to be produced in sufficient quantities and stored separately. As a consequence it is possible, as in the case of the sludge fuel RF-86 that up to eight storage tanks have to be set aside for a period of 6 months. A “pilot batch” is then blended from these components which again has to prove its ability to produce sludge, for instance, in further engine tests. If these tests are successful, blending of the first full-scale batch can take place. In agreement with reference fuel users this batch may then have to be subjected to an additional bench test before final approval is given.

Production of such fuels presents special challenges for a company. Not only is a large number of storage tanks required for the very many different components and fuel grades, but these tanks have to be of very different sizes. Thus Haltermann uses storage tanks with a capacity spectrum ranging from 10,000 liters to 5 million liters. Some of these tanks are pressurized in order to avoid changes of quality during storage, for example by loss of vapor pressure. If oxidation of relatively instable products needs to be excluded it is possible to provide storage under a nitrogen blanket.
With the production of such tightly specified fuels the requirements regarding the entire piping system differ from those of a large oil refinery. In this case it is not possible to use “mixed product piping” because the quantity of product required for flushing the system could exceed the actual batch size. Separate and dedicated piping of every single storage tank has to be provided right up to the point of blending. Since product volumes are usually quite small the quality can be affected already by traces of contaminants, for example by rust particles. This can often be avoided reliably only by the use of costly stainless steel fittings and valves.

A production facility such as ours has to support various kinds of distribution system according to the large range of product volumes which have to be handled. First of all a loading point is required for complete ships´ cargos, but beyond that, the entire spectrum of modern transportation equipment has to be catered for. Appropriate filling and handling equipment as well as administration have to be provided for rail and road tank cars, tank containers and barrels of various shapes and sizes right down to ½ liter stainless steel cans.

In all phases of “fuel life” the laboratory is of key importance. Already the very first conception of a fuel grade involves mathematical modeling which is checked by means of laboratory blends. The laboratory accompanies the product through all stages of development right up to delivery. Fuels which are stored over a longer period of time are subjected to a strict Quality Monitoring program in order to ensure constant product quality. A reliable and approved facility for analytical work is essential to ensure that customers have confidence in the laboratory. Haltermann’s laboratory in Hamburg-Wilhelmsburg has therefore been accredited according to DIN EN 45001 since 1998. The laboratory regularly invites auditors who supervise working procedures and test methods in order to confirm the high quality level of the work.

Additionally the entire company is certified by an environmental and quality management system according to ISO 14001 and ISO 9001.

4. Requirements versus Future Reference Fuels

Extremely rapid progress of the entire automotive industry leads to increasingly dynamic development in nearly all related areas. This affects not only the OEMs themselves, but also their suppliers including the oil industry as producers of fuels and lubricants. The safe operation of modern propulsion systems can be assured only by close cooperation between all partners in research and development.

**Suppliers**
- Injection Systems
- Tank Systems
- Catalysts

**Oil Industry**
- Fuels
- Additives
- Lubricants

**Auto Industry**
- Engines
- Transmissions

The high performance requirements of the latest engine generation versus components and operating fluids are likely to increase even further in future. Continually reduced exhaust emission limits of European legislation add to the
need for highly defined test fuels. Modern analytical instruments now permit the investigation of many fuel parameters and studies of their influence on engine operation.

In the past a whole bunch of development targets was often pursued by using a single fuel grade.

Today, in contrast, the influence of many fuel parameters is examined in a most discriminating way, entailing a demand for very many different fuels. A few typical fuel properties and currently studied effects on performance or on vehicle components can be quoted as follows:

<table>
<thead>
<tr>
<th>Property</th>
<th>Effect</th>
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<tbody>
<tr>
<td>Sulphur content</td>
<td>Effect on exhaust after-treatment systems</td>
</tr>
<tr>
<td>Oxygenate content</td>
<td>High / low temperature</td>
</tr>
<tr>
<td>Vapor pressure</td>
<td>Performance</td>
</tr>
<tr>
<td>Benzene content</td>
<td>Fuel consumption / Fuel efficiency</td>
</tr>
<tr>
<td>Aromatics content</td>
<td>Startability</td>
</tr>
<tr>
<td>aromatics distribution</td>
<td>Behavior in tank system</td>
</tr>
<tr>
<td>Distillation range / Final</td>
<td>Sludge &amp; deposits formation</td>
</tr>
<tr>
<td>boiling point</td>
<td>Lubricity</td>
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</tbody>
</table>

It may be expected that the trend towards more and more specialized fuel grades will continue into the future. The challenges facing the manufacturer of such fuels will thus also increase. A review of recent years shows a significant increase in the number of different fuel grades whilst volumetric demand per grade decreased. From the viewpoint of a fuel producer we may summarize the following future trends:

- The number of components requiring storage will increase
- More components means more storage tanks will be needed
- A larger number of blending vessels will be required

The increasing number of different fuel grades and increasingly sophisticated demands versus these fuels require appropriate growth of technical structure and personnel, as discussed in chapter 3 of this paper.