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

by J. Albrecht Spangenberg and Dipl.-Ing Lutz Reichenbächer, Haltermann Products
A subsidiary of The Dow Chemical Company

Summary
Authorities in the United States 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, the development of engine and operating fluid 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. Historical 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 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 Products comprise of a wide spectrum of reference fuels and development fuels. This includes the production of both diesel and gasoline grades for the following applications:

- Engine development
- Lubricant development
- Additive development
- Durability testing
- Emission testing
- Certification
- Production monitoring

In 1968 Haltermann had to face the problems of test fuels for the first time. At that time only the United States had 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 on 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 this test fuel. Haltermann therefore investigated the requirements of this tightly specified fuel called Indolene 30 and was subsequently able to provide Volkswagen 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 United States. In order to stay abreast of 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 Paris Airport on 22 January 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’. Soon after, the first reference diesel fuel was defined. The designation was:
- ERF / D1 (for diesel exhaust smoke measurement).

The testing activities that 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 parameters:
  - To relate to the performance parameter desired in the test
  - To guarantee reproducibility of fuel quality / test results

The second point is of equal importance to the first and 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:

[Figure 2: Range of sulphur content for RF-93 and other specs
Figure 3: Range of density for RF-90 and other specs
Figure 4: Very little variation of distillation range for 6 batches of RF-89]

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 in sufficient 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). It 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. Figure 5 shows test results from three fuels of different origin, each meeting EN 228 requirements. All results shown here were obtained on the same test bench with the same engine.

[Figure 5: IVD test results with three base fuels according to EN 228]

The extremely large deviation of test results would practically prevent the development of additives since a significant effect of the additive on the test results may not be recognized. Base fuel response to additive treatment varies and hence 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 effects on the combustion process and on exhaust emissions. Specially formulated model fuels were use 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 a 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 after-treatment 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 that 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 six 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 many then have to be subjected to additional bench tests before final approval is given.

Production of such fuels present special challenges for a company. Not only are 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 unstable 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 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 ship cargoes, 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 half-liter stainless steel cans.

In all phases of ‘fuel life’ the laboratory is of key importance. 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 that are stored over a long 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 customer confidence in the laboratory. To this end, Haltermann’s laboratory in Hamburg has been accredited with DIN EN 45001 since 1998. The laboratory regularly invites auditors to supervise working procedures and test methods to confirm the high quality of the work.

The entire company is also certified by an environment and quality management system according to ISO 14001 and ISO 9001 standards.

4. Requirements versus Future Reference Fuels

Extremely rapid progress in the entire automotive industry has led 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.
The high performance requirements of the latest engine generation versus components and operating fluids are likely to increase even further in the 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 operations.

In the past a whole bunch of development targets were often pursued 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>
</tr>
</thead>
<tbody>
<tr>
<td>Sulphur content</td>
<td>Effect on exhaust after-treatment systems</td>
</tr>
<tr>
<td>Oxygenate content</td>
<td>High / low temperature performance</td>
</tr>
<tr>
<td>Vapor pressure</td>
<td>Fuel consumption / fuel efficiency</td>
</tr>
<tr>
<td>Benzene content</td>
<td>Startability</td>
</tr>
<tr>
<td>Aromatics content /distribution</td>
<td>Behavior in tank system</td>
</tr>
<tr>
<td>Distillation range / final boiling point</td>
<td>Sludge and deposits formation</td>
</tr>
<tr>
<td></td>
<td>Lubricity</td>
</tr>
</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. Form 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 above.

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