CENTRUM VAN NORMALISATIE

Worldwide Fuels Standards

Overview of specifications and regulations on (bio)fuels

Prepared for IEA TASK 39 – Liquid biofuels

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1 Background of the task

Automotive use of (bio)fuels dictates requirements to these fuels. Especially the biological origin of the biofuels emphasizes the need for special biofuel standards. NEN, the Dutch Standardization institute has been requested to provide a short overview of 'the world of standardization' regarding automotive biofuels. The focus of the project is to specify backgrounds of biofuel standardization in different regions of the world, to indicate differences and to link those, if possible, to local or regional regulations.

This overview is commissioned by the IEA Task39 – Liquid biofuels and will be distributed among the task members. This document will be used as a point of origin by (international) policymakers that want to have insight in standardization issues regarding development of biofuels.

2 Introduction to the work

2.1 General

Alternative fuels and related issues, like the supply of raw materials, production technology, distribution and engine technology have been analysed and discussed for almost four decades. During this time period several production and engine technology tests have been performed with a broad range of alternative fuels. Different alternative fuels have also been used in fleet tests, with vehicle fleets ranging from a couple up to several hundreds of vehicles.

During the period of exploration, the need for specifications has been highlighted. Engine and vehicle manufacturers have more and more emphasised the importance of specifications, or rather standards. To some extent specifications from industry organizations have been set up and occasionally these have also been brought forward to national standards. Some work has also been done on international standards.

The work undertaken was to identify:

- existing standards for specifications, more specifically their requirements, limits and related test methods on international and national levels,
- ongoing work regarding specification of alternative fuels on international and national levels, and
- work that is considered or already planned on standardization of alternative fuels, on international and national levels, either initiated by industry or governmental regulations.

To limit the area of research the following areas were considered:

- ISO/IEC
- EU and EEFTA countries (i.e. CEN and national)
- North America (ASTM)
- Brazil, Argentina and other South American countries
- India
- Japan
- Australia

Both literature and standardization databases were used for the research. Besides that enquiries have been made at standardization institutes, fuel authorities and industry representatives.

2.2 Fuels addressed

The majority of the attention was aimed at liquid fuels, but also enquiries on gaseous or liquefied gaseous fuels were made. Several types can be identified, which can be structured as follows.

1) Liquid diesel engine fuels
   a) Fatty acid methyl esters (FAME) used as 100% fuel (B100)
   b) Fatty acid methyl esters blended in diesel at levels of 5, 10, 15, 20 or 30 % (*V/V*)
   c) Fatty acid ethyl esters (FAEE) used as 100% fuel
   d) Fatty acid ethyl esters blended at levels of 5, 10, 15, 20 or 30 % (*V/V*)
   e) Pure plant oil (PPO), also known as straight vegetable oil (SVO) or pure vegetable oil
   f) Pyrolysis oil
   g) Alcohol and alcohol derivatives in diesel fuels (E-diesel)
   h) Water in diesel emulsion fuels
   i) Synthetic (Fisher-Tropsch) diesel on the basis of natural gas (GTL)
   j) Synthetic (Fisher-Tropsch) diesel on the basis of biogas (BTL)
2) Liquid spark ignition engine fuels
   a) Hydrated bio-ethanol (E95) as a straight fuel
   b) Anhydrous bio-ethanol (E100) as a straight fuel
   c) Bio-ethanol blended at levels of 5, 10, 15 or 22 % (*V/V*)
   d) Ethyl-*tert*-butyl-ether (Bio-ETBE) from biological source, for blending into gasoline
   e) E85: mixture of 70 – 95% of ethanol with gasoline for flexible fuel vehicles
   f) Bio-methanol blended at levels of 5 % (*V/V*) or higher volume percentages
   g) Methyl-*tert*-butyl-ether (Bio-MTBE) from biological source, for blending into gasoline
   h) M85: mixture of ± 85% of methanol with gasoline for flexible fuel vehicles
3) Liquefied petroleum gas (LPG)
4) Di-methyl ether (DME), from industrial or biological sources
5) Gas
   a) Compressed natural gas (CNG)
   b) Liquefied natural gas (LNG)
   c) Biogas, methane from fermentation processes (CBG)
6) Hydrogen or bio-hydrogen

3 Results

Regions that have been addressed are EU-25 (CEN), North America (ASTM), South America, Australia, India and Japan. Information from South-Africa and Russia was found, but the exact data were hard to obtain, whereas from China only indicative data have been obtained during the short period of execution of the research work.

3.1 B100 and FAME-diesel blends' requirements

3.1.1 The majority of the B100 specifications are for both direct and blending purposes. Major examples of specific blend specifications are USA (ASTM) and Japan, where specific fuel requirements (such as cold flow properties and stability) are not incorporated in the standards. This immediately indicates the difference between the two leading biodiesel fuel specifications EN 14214 and ASTM D 6751. When looking at the rest of the world, and especially the test methods used, all countries have mimicked these two specifications and changed them to local needs.

Comparison of the ASTM and JIS blend specifications with other fuel specifications is sometimes difficult. When the actual diesel fuel specification is back-up, limits on flash point, density or corrosion can be less stringent or even non-existent. On one hand it makes setting and accepting specifications easier, on the other hand the full market potential for specifying the biodiesel blend quality takes longer and is more complicated.

3.1.2 In terms of the feedstock the requirements or indications are different. Most countries have explicitly mentioned that the FAME can be of vegetable or animal origin. But effectively, requirements such as oxidation stability, viscosity and iodine value limit full use of all oils. In practice, main feedstock due to the large production in Europe is rapeseed oil with more than 80 % of the total oil demand. Another is soybean oil which is the predominant feedstock in the USA. There the increasing biodiesel industry uses about 10 – 15 %, recycled frying oils, animal fats, sunflower oil, palm oil, and castor oil with about 5 % quota of biodiesel production.

In the early 1990s, Canadian canola production increased in response to higher market prices relative to cereal grains and increasing grain handling and transportation costs. Most of Canada's exportable canola supplies are purchased by Japan. The remainder of the crop is crushed for domestic consumption or export, primarily to the United States. Although canola is the more expensive biodiesel feedstock, recent bumper crops have depressed the price of canola, making it a more attractive feedstock for biodiesel. Tallow continues to be used as a biodiesel feedstock in Canada.

The Czech Republic has set a specification for rapeseed methyl ester (RME) for both B5 (3 - 5% RME) and B30 (31% RME at minimum). Density and sulphur content requirements are recalculated from EN 14214 limits, but water content is set at lower levels.

3.1.3 Looking at the technical differences (see Tables 1 and 2), only ASTM doesn't require a minimum ester content. If not used as a B100 product this minimum to prevent fraud of mixing with non-biological products, as in Europe, is unnecessary. In the USA the questions of the precision of the test method (EN 14103) and the political reason of allowing used kitchen oils, prevent an ester content requirement. Both ASTM and CEN work on improving the determination via an HPLC method.

Viscosity ranges are different in almost every country. Viscosity increases with higher contents of non-reacted glycerines and polymers. Indications are that some countries have accustomed this limit to the feedstock or chosen a broad range for their B100 product. Some countries have incorporated specific advices for animal fats' use. This accommodation is also known of requirements such as contents of glycerides or glycerol. Whereas Japan has already some doubts on the glycerides level (see later on), Brazil has a less strict limit for glycerol and no limits for the glycerides. Reason is that coconut and palm oil cannot be measured by gas chromatography due to interferences in the spectrum. This is also the reason that the method is under further development at CEN and that ASTM has not yet chosen to limit mono-, di- and triglycerides.

Although regulated sulphur content in normal diesel differs widely throughout the world, all countries strive to have ultra-low sulphur biodiesel. Biodiesels are virtually sulphur-free, especially those of fresh products, so this is also a marketing instrument for the biofuel industry.

Based on recent developments in terms of test methods, all alkali metals are now phased in into the requirements. ICP-OES becomes the regularly used method over AAS. It has been originally standardized in DIN and later on in CEN. Some countries have incorporated the requirement, but not yet set the limit, because they were awaiting the resulting CEN Standard.

3.2 B100 and FAME-diesel blends' perspectives

3.2.1 United States interest in biodiesel was stimulated by the Clean Air Act of 1990 combined with regulations requiring reduced sulphur content in diesel fuel and reduced diesel exhaust emissions. The Energy Policy Act of 1992 established a goal of replacing 10 percent of motor fuels with non-petroleum alternatives by the 2000 and increasing to 30 percent by the year 2010. By 1995, 10 percent of all federal vehicles were to be using alternative fuels to set an example for the private automotive and fuel industries.

The production of biodiesel in the U.S. tripled in 2005 to 75 million gallons from 25 million gallons in 2004. This was spurred on in large part by the Blenders Credit provision in the Energy Bill. The Engine Manufacturers Association (EMA) just released a B20 draft specification as an industry specification. The borderline is that the B20 blend should fulfil normal diesel fuel specification (D 975). The biodiesel blend stock standard (ASTM D 6751) is a applicable for blending up to 20%, but a B20 fuel specification is not published. A first draft is under discussion in ASTM, but for the moment finalisation of a B5 specification has prime attention. Next, short storage times are recommended and cautions for cold weather properties, tank cleanliness and fuel filter and delivery system check are given.

Last year , the National Biodiesel Board released a guidance for blends above 20%. Most auto, engine and fuel injector equipment manufacturers in the US strongly discourage use of blends over B20. These require significant additional precautions, handling and maintenance considerations, as well as potential fuel system and engine modification. Effectively, B20 is still not a common use, fully guaranteed fuel.

American industry has identified the necessity of a stability requirement for all biodiesel. Use of an extra acid number to determine storage stability is discussed. The general feeling is that if the B100 is stable, the B5 and B20 are also stable, which is supported by small investigations. The actual ASTM D 6751 does not contain an oxidation stability requirement. The original ASTM test D 2274 was proposed but not accepted and the Rancimat test (EN 14112) will be moved forward. Because it concerns low level blends (and not a B!00 fuel) ASTM accepts a 3 hours IP limit. Studies showed that a Rancimat of 3 hours IP ensures low deposits and long induction time for the blend. Oxidation stability is mostly uniform 6 hours IP world-wide. The allowance of 5 % (*V/V*) of biodiesel in diesel according to ASTM D 975, awaits the inclusion of an effective stability test in ASTM D 6751.

Flash point has been chosen in the past by ASTM as a requirement to limit the level of non-reacted alcohol. It is also connected to legal requirements concerning safety of handling and storage. 130 °C is chosen as the upper test limit to deliver a true flash point of 100 °C, due to several volatile components (residual alcohols). CEN chose to measure the alcohol and methanol content as such. ASTM discusses the addition of a maximum methanol and ethanol requirement, awaiting results of the applicability of EN 14110 to ethanol. As ASTM D 6751 is an alkyl ester specification, applicability to ethanol is essential. Future incorporation of a limit and EN 14110 is anticipated, while at the moment the methodology is incorporated in the specification (UOP 193).

Next discussion issue originating from the EMA is the identification of cold flow improvers. Similar possible problems of additives interference between EN 14214 and EN 590 have been identified in Europe and CEN has agreed to an intermediate solution of excluding the CFPP requirement for FAME for blending.

In addition, the FAME determination method (EN 14078) is felt as a necessity by US industry. Some States have already drawn up own regulations with detection methods. This to check for the bio-content of the fuel that they have alternative excise duties for.

3.2.2 The European Commission supports a study to improve the actual biodiesel standard. This project, called BIOScopes, tries to improve the test methods on ester content (EN 14103), di-, triglyceride and free glycerol content (EN 14105), poly unsaturated ester content (for which today no standardized test exists) and the FAME content determination test (EN 14078).The BIOScopes project will also produce a literature study to identify possibilities to increase the quantities of biodiesel by using alternative feedstock, to identify the need for further research and engine tests and the need for adaptations of the existing specifications EN 14214 and EN 590. Iodine value - seen by some parties on the market as a non-technical limitation for biodiesel feedstock, but chosen as the best alternative at that time - is a major topic in this investigation.

In parallel, a Mandate has been given to CEN to revise the existing specifications to allow for use of B10, if emissions and vehicle endurance could allow this. CEN will start this standardization work in two working groups who have planned their initial meeting in September 2006. This to allow Member States future compliance with the EC Biofuels Directive target of 5,75% energy content in 2010. For the moment the targets are indicative, but there seems to grow a majority in the Parliament for more restrictive indications.

3.2.3 India has a national specification (IS 15607:2005) for biodiesel (B100) as a blending component in diesel fuel. This biodiesel is a fatty acid alkyl (methyl or ethyl) ester and meant for usage as a blending component (up to 20 % (*V/V*)) with automotive diesel fuel meeting the specification of IS 1460:2005. However, the present “automotive diesel fuel” specification permits usage of biodiesel up to only 5 % (*V/V*) as a blending component.

The government of India is keenly promoting use of bio-diesel in diesel fuel and took major policy initiatives in this regard primarily to:

- Generate employment and improve economic conditions in rural areas
- Bring the waste lands (semi-arid lands) under cultivation of non-edible oil plants and the last but not the least
- Energy security by supplementing the nation's energy requirement through renewable energy sources

As per our knowledge, no oil company is marketing diesel fuel with biodiesel, primarily because of availability of biodiesel on a commercial scale. Lot of experimental projects are being carried out by oil companies and vehicle manufacturers. It takes about 3 to 5 years before oil companies start using biodiesel as a blending component on a commercial scale in India.

3.2.4 Japan has just finalised the specification of diesel fuel considering FAME 5% (*m/m*) and its FAME specification. The mandatory Diesel Fuel Standard established under the "Law for Quality Control of Gasoline" will be expanded to include biodiesel fuel and will continue to be applied to diesel fuel producers and distributors. Simultaneously, a specialized standard will be introduced for 'B100' having properties appropriate to blending in diesel fuel at a 5% content. The latter will be a non-mandatory standard under JIS. The Japanese FAME specification is almost the same as EN 14214 of Europe.

The Japanese diesel fuel specification allows 5% mass and is a little bit severe compared to the EU diesel specification. This in terms of requiring both sludge and acid oxidation stability. Also methanol, triglycerides and total acidity limits are set for diesel fuel, because studies have found those to give problems in the engines. The Japanese Motor Association conducts several long time storage and fuel tank equipment use test. Currently, the Japanese government (METI) is conducting the conformity tests on FAME. The results of on-going study suggests tentatively that current FAME specifications (EN 14214 and ASTM D6751) might not be sufficient.

3.2.5 The governmental Brazilian Biodiesel Program has set the ambitious target of introducing biodiesel - a domestic source of renewable energy - into the market, in order to decrease dependency on foreign diesel fuel, promote social development by creating job opportunities (in the countryside) and increase regional earnings and development. The Government established an initial priority for the Program of automotive fuels with 2% blending of biodiesel optional and will be compulsory in 2008, increasing up to 5% until 2013, taking into account the production capacity and the results of other aspects of the Program.

A Brazilian commercial specification for biodiesel fuel was established in 2004 (Regulamento Técnico Nº 4/2004 under RESOLUÇÃO ANP Nº 42). Test methods to evaluate the biodiesel obtained from different sources are based mainly on the international methods (ASTM, ISO). However, a conflict was identified concerning the analytical methodology when used for castor. So a method was developed by CENPES (Research Center of Petrobras) for mono-, di-, triglycerides, which were also standardized by ABNT. At the moment the limit has not yet been determined.

Other limits (see Table 2) have not been set by regulation, as field and engine experience with local feedstock is still limited. Brazil is in advance with the development of others oils like jatropha, mamona, etc. and they are using them in making biodiesel. The Brazilian government has approached the European Commission and the American Department of Energy on possibilities for international fuel specifications.

3.2.6 Argentina has just approved a new version of biodiesel 100% pure, for blending with diesel. In Argentina there is the possibility of using methanol or ethanol as raw material for making biodiesel. The latest issue of IRAM 6515-1 (the Argentinean Standard) is of 2006, but is still a voluntary standard. The Government regulation it is not so complete. Like Brazil, Argentina is developing jatropha type of plants in some places of the country, far from Buenos Aires and the Pampa area, the latter being used for soy, sunflower, etc. Argentina government is highly interested in developing deserts areas and using others oils than those used for food.

3.3 Fatty Acid Ethyl Esters (FAEE)

No specific standards for FAEE were found. ASTM D 6751 and IS 15607 are standard specifications for alkyl esters, making explicitly no difference for methyl- or ethyl esters. The specifications in Argentina and Paraguay indicate that they are for both type of esters. Countries like Australia or Uruguay have not set a specific requirement for the type of alkyl esters. But the actual use of FAEE is limited.

French government has issued a study by the Institut France de Pétrole (IFP) on the impact on the fuel characteristics, engines operation and pollutant emissions of FAEE. This study incorporates both laboratory analysis and heavy and light duty engine tests. The aim is to study the possibility to fix a single standard (EN 14214) for the fatty acid esters (FAME and FAEE). This approach is supported by CEN and the EC. The earlier mentioned BIOScopes project also studies FAEE. In parallel, CEN has been given a Mandate to draft a Standard for FAEE.

Effectively, this type of product is still in a pre-market stage and some countries choose to not explicitly exclude any ester type at this moment. Tests in the market will have to show if more stringent requirements for FAEE are needed.

3.4 Pure vegetable oil

This non-esterified, straight version of natural oils, is often misleadingly marketed as biodiesel. Other indications are eco-diesel, green diesel or natural diesel. The US Department of Energy has stated that "Raw or refined vergetable oil, or recycled greases that have not been processed into biodiesel, are not biodiesel and should be avoided." Some European governments have made similar statements. For example, the higher viscosity and chemical composition of unprocessed fats and oils have been shown to cause problems in for example piston sticking and injection deposits. They also experience significant degradation compared to (bio)diesel.

European engine manufacturers do not support the use of pure vegetable oils like rape seed oil, palm oil and soy bean oil. ACEA will issue a position paper on this issue. Also burner producers do not allow the use of pure vegetable oils. Especially their poor (storage/oxidation/thermal) stability is considered as a main problem. Germany has developed a national pre-standard (DIN V 51605), which has been used by many PPO suppliers and local licensers as a quality designation. However, the development of the text to a full standard has experienced lots of troubles and raised many questions, partially due to doubts arising from a field trial with 100 tractors.

So more experience for pure vegetable oils is needed, even with adopted engines. Germany maintains the tax exemptions for PPO, but the availability is limited. At the moment PPO's are mainly used locally in furnaces, where the source of the oil and the burner application are close together, so that stability problems are limited. In Europe several companies sell retro-fit sets for diesel engines, but general public use is still far from developed. Several European national governments do not support the use of PPO, favouring FAME or second generation biofuels.

3.5 Pyrolysis oil

Apart from a draft document in Japan (TS Z 0025) of 2004, not many standards on this diesel fuel have been found. The application seems for the moment to be limited to stationary applications. Concerns on its toxicity exist.

Table 1: Comparison of biodiesel (blends) in Eurasian region

Parameter	CEN	ASTM	Germany	India	Japan	Australia
	EN 14214:2003 ***B100***	**D 6751** ***Blend*** [1]	**DIN V 51605** ***PPO***	**IS 15607** ***Blend***	**JASO/JIS** ***Blend***	**Regulation**
Ester content, min.	96,5 % (*m/m*) EN 14103			96,5 % (*m/m*) EN 14103	96,5 % (*m/m*) EN 14103	96,5 % (*m/m*) EN 14103
Density at 15 °C	(860 – 900) kg/m^3 EN ISO 3675 EN ISO 12185		(900 - 930) kg/m^3	(860 – 900) kg/m^3 ISO 3675 ISO 12185 ASTM D 4052	(0,86 – 0,90) g/ml JIS K 2249	(860 – 890) kg/m^3 EN ISO 3675 ASTM D 1298
Viscosity at 40 °C	(3,50 – 5,00) mm^2/s EN ISO 3104	(1,9 – 6,0) mm^2/s ASTM D 445	36 mm^2/s	(2,5 – 6,0) mm^2/s ISO 3104	(3,5 – 5,0) mm^2/s JIS K 2283	(3,5 – 5,0) mm^2/s ASTM D 445
Flash point, min.	120 °C EN ISO 3679	130 °C ASTM D 93	220 °C	120 °C	120 °C JIS K 2265	120,0 °C ASTM D 93
Sulphur content, max	10,0 mg/kg EN ISO 20846 EN ISO 20884	15 ppm ASTM D 5453	10,0 mg/kg	10,0 mg/kg ASTM D 5453	10 ppm JIS K 2541-1, -2, -6	10,0 mg/kg ASTM D 5453
Carbon residue (on xx % distillation residue), max	0,30 % (*m/m*) on 10% EN ISO 10370	0,050 % (*m/m*) on 100% ASTM D 4530 ASTM D 189 ASTM D 524		0,05 % (*m/m*) on 100% ASTM D 4530 ISO 10370	0,3 %(*m/m*) on 10% JIS K 2270	0,30 % (*m/m*) on 10% EN ISO 10370
Cetane number, min.	51,0 EN ISO 5165 ASTM D 613	47 ASTM D 613	39	51 ISO 5165	51 JIS K 2280	51,0 EN ISO 5165 ASTM D613
Sulfated ash content, max.	0,02 % (*m/m*) ISO 3987	0,020 % (*m/m*) ASTM D 874	0,01 % (*m/m*)	0,02 % (*m/m*) ISO 6245	0,02 % (*m/m*) JIS K 2272	0,020 % (*m/m*) ASTM D 874
Water content, max.	500 mg/kg EN ISO 12937			500 mg/kg ASTM D 2709 ISO 3733 ISO 6296	500 ppm JIS K 2275	

[1] if more test methods are mentioned the first one is the referee method

Parameter	CEN	ASTM	Germany	India	Japan	Australia
Water and sediment, max.		0,050 % (*V/V*) ASTM D 2709 ASTM D 1796				0,050 % (*V/V*) ASTM D 2709 ASTM D 1796
Total contamination max.	24 mg/kg EN 12662		24 mg/kg	24 mg/kg EN 12662	24 ppm EN 12662	24 mg/kg EN 12662 ASTM D 5452
Copper strip corrosion (3 h at 50 °C), max.	class 1 EN ISO 2160	class 3 ASTM D 130		class 1 ISO 2160	class 1 JIS K 2513	class 3 ASTM D 130
Oxidation stability at 110 °C, min.	6 hours EN 14112	3 hours EN 14112		6 hours EN 14112	to be agreed by producer and distributor	6 hours EN 14112 ASTM D 2274
Acid value, max.	0,5 mg KOH/g EN 14104	0,5 mg KOH/g ASTM D 664 ASTM D 3242 ASTM D 974	2,0 mg KOH/g	0,50 mg KOH/g	0,5 mg KOH/g JIS K 2501	0,80 mg KOH/g EN 14104
Iodine value, max.	120 g Iodine/100 g EN 14111			( ) [2] EN 14104	120 g Iodine/100 g JIS K 0070	
Linolenic acid methyl ester, max.	12,0 % (*m/m*) EN 14103			-	12,0 % (*m/m*) EN 14103	
Polyunsaturated methyl esters, max.	1 % (*m/m*)			-	1 % (*m/m*)	
Methanol content, max.	0,20 % (*m/m*) EN 14110			0,20 % (*m/m*) EN 14110	0,20 % (*m/m*) JIS K 2536 EN 14110	
Alcohol content, max.						0,20 % (*m/m*) EN 14110
Monoglyceride content, max.	0,80 % (*m/m*) EN 14105				0,80 % (*m/m*) EN 14105	
Diglyceride content, max.	0,20 % (*m/m*) EN 14105				0,20 % (*m/m*) EN 14105	

[2] ( ) Indicates that the value shall be reported, but a limit is not yet set

Parameter	CEN	ASTM	Germany	India	Japan	Australia
Triglyceride content, max.	0,20 % (*m/m*) EN 14105				0,20 % (*m/m*) EN 14105	
Free glycerol/glycerine, max.	0,02 % (*m/m*) EN 14105 EN 14106	0,02 % (*m/m*) ASTM D 6584		0,02 % (*m/m*) ASTM D 6584	0,02 % (*m/m*) EN 14105 EN 14106	0,020 % (*m/m*) ASTM D 6584
Total glycerol, max.	0,25 % (*m/m*) EN 14105	0,240 % (*m/m*) ASTM D 6584		0,25 % (*m/m*) ASTM D 6584	0,25 % (*m/m*) EN 14105	0,250 % (*m/m*) ASTM D 6584
Group I metals (Na+K) , max.	5,0 mg/kg EN 14108 EN 14109	5,0 mg/kg EN 14538	not specified	( ) EN 14108 EN 14109	5,0 mg/kg EN 14108 EN 14109	5 mg/kg EN 14108 EN 14109
Group II metals (Ca+Mg) , max.	5,0 mg/kg EN 14538		20 mg/kg …	( ) -	5,0 mg/kg EN 14538	5 mg/kg EN 14538
Phosphorus content, max.	10,0 mg/kg EN 14107	0,001 % (*m/m*) ASTM D 4951	10,0 mg/kg EN 14107	10,0 mg/kg ASTM D 4951	10,0 mg/kg EN 14107	10,0 mg/kg ASTM D 4951
Distillation, 90% recovered, max.		360 °C ASTM D1160				360 °C ASTM D 1160
CFPP, max.	climate dependent		not determinable	climate dependent	to be agreed by producer and distributor	
Pour point					to be agreed by producer and distributor	
Cloud point		( ) ASTM D 2500 ASTM D 3117				

Table 2: Comparison of biodiesel (blends) in American region

Parameter	CEN	ASTM B10	ASTM B20	Brazil	Argentina	Colombia	Paraguay	Uruguay
	EN 14214 ***B100***	**D 6751** ***Blend***	**D xxxx:2004** ***B20***	**ANP Nº 42** ***B100***	**IRAM 6515-1**[3] ***B100***	**DE 100-04** ***B100***	**PNA 16 018** ***B100***	**UNI 1100** ***B100***
Ester content, min.	96,5 % (*m/m*) EN 14103			( ) [4] EN 14103	96,5 % (*m/m*) EN 14103	96,5 % (*m/m*) EN 14103	96,5 % (*m/m*) EN 14103	96,5 % (*m/m*) EN 14103
Density at 15 °C	(860 – 900) kg/m^3 EN ISO 3675 EN ISO 12185			( ) ASTM D 1298 ASTM D 4052	(0,875 – 0,900) g/l EN ISO 3675 EN ISO 12185	(860 – 900) kg/m^3 ASTM D 4052 ISO 3675	(850 – 900) kg/m^3 ISO 3675 ISO 12185 ASTM D 7042	
Viscosity at 40 °C	(3,50 – 5,00) mm^2/s EN ISO 3104	(1,9 – 6,0) mm^2/s ASTM D 445	(1,3 – 4,1) mm^2/s ASTM D 445	( ) EN ISO 3104	(3,5 – 5,0) mm^2/s EN ISO 3104 ASTM D 445		(3 – 6,5) mm^2/s IRAM-IAP A 6597 ISO 3104 ASTM D 445	(1,9 – 6,0) mm^2/s ISO 3104 ASTM D 445
Flash point, min.	120 °C EN ISO 3679	130 °C ASTM D 93	38 °C ASTM D 93	100 °C EN ISO 3679 ASTM D 93	130 °C EN ISO 3679 ASTM D 93	120 °C ASTM D 93 ISO 2719	120 °C ISO 3679 ASTM D 93	100 °C ISO 3679 ASTM D 93
Sulphur content, max	10,0 mg/kg EN ISO 20846 EN ISO 20884	15 ppm ASTM D 5453	15 ppm ASTM D 5453	( ) ASTM D 4294 ASTM D 5453	10 mg/kg EN ISO 20846 EN ISO 20884 ASTM D 5453		10,0 mg/kg ASTM D 2622 ASTM D 5453	10,0 mg/kg ASTM D 2622 ASTM D 5453
Carbon residue (on xx % distillation residue), max	0,30 % (*m/m*) on 10% EN ISO 10370	0,050 % (*m/m*) on 100% ASTM D 4530 ASTM D 189 ASTM D 524	0,035 % (*m/m*) on 10% ASTM D 524	0,1 % (*m/m*) on 100% EN ISO 10370	0,05 g/100g on 100% ASTM D 4530 EN ISO 10370	0,3 % (*m/m*) on 10% ISO 10370 ASTM D 4530	0,3 g/100g on 10% ASTM D 4530 ISO 10370 ASTM D 189	0,10 %(*m/m*) on 10% ASTM D 4530 ISO 10370 ASTM D 189 ASTM D 4530
Cetane number, min.	51,0 EN ISO 5165	47 ASTM D 613	40 ASTM D 613	( ) EN ISO 5165 ASTM D 613		47 ASTM D 613 ISO 5165	45,0 ASTM D 613 ISO 5165	45 ASTM D 613 ISO 5165

[3] if more test methods are mentioned the first one is the referee method

[4] ( ) Indicates that the value shall be reported, but a limit is not yet set

Parameter	CEN	ASTM B10	ASTM B20	Brazil	Argentina	Colombia	Paraguay	Uruguay
Sulfated ash content, max.	0,02 % (*m/m*) ISO 3987	0,020 % (*m/m*) ASTM D 874	0,01 % (*m./m*) ASTM D482	0,02 % (*m/m*) ISO 3987	0,02 % (*m/m*) ISO 3987	0,02 % (*m/m*) ASTM D 874 ISO 3987	0,05 % (*m/m*) ISO 3987	0,02 % (*m/m*) ISO 3987 ASTM D 874
Water content, max.	500 mg/kg EN ISO 12937				0,050 g/100g ASTM D 4928 ISO 12937	500 mg/kg ASTM E 203 EN ISO 12937		500 mg/kg ISO 12937
Water and sediment, max.		0,050 % (*V/V*) ASTM D 2709 ASTM D 1796	0,050 % (*V/V*) ASTM D 2709	0,05 % (*V/V*) NBR 9842			0,05 % (*m/m*) ASTM D 4928 ISO 12937	0,05 % (*m/m*) ASTM D 2907
Total contamination max.	24 mg/kg EN 12662			( ) EN 12662	24 mg/kg EN ISO 12662[5]	24 mg/kg EN 12662		
Copper strip corrosion (3 h at 50 °C)	class 1 EN ISO 2160	class 3 ASTM D 130	class 3 ASTM D 130	class 1 EN ISO 2160 ASTM D 130	class 1 IRAM-IAP A 6553 ASTM D 130 ISO 2160	class 1 ISO 2160 ASTM D 130	class 1 IRAM-IAP A 6533 ASTM D 130 ISO 2160	class 3 ASTM D 130 ISO 2160
Oxidation stability at 110 °C, min.	6 hours EN 14112			6 hours EN 14112	6 hours EN 14112 ASTM D 2274	6 hours EN 14112	6 hours EN 14112 ASTM D 2274	6 hours EN 14112
Acid value, max.	0,5 mg KOH/g EN 14104	0,5 mg KOH/g ASTM D 664 ASTM D 3242 ASTM D 974	0,2 mg KOH/g ASTM D 664	0,8 mg KOH/g EN 14104 ASTM D 664	0,5 mg KOH/g IRAM 6558 EN 14104 ASTM D 664	0,5 mg KOH/g EN 14104 ASTM D 664	0,8 mg KOH/g IRAM 6558 EN 14104 ASTM D 664	0,8 mg KOH/g EN 14104 ASTM D 664
Iodine value, max.	120 g Iodine/100 g EN 14111			( ) EN 14111	150 g Iodine/100 g EN 14111	120 g Iodine/100 g EN 14111		
Linolenic acid methyl ester, max.	12,0 % (*m/m*) EN 14103				12 % (*m/m*) EN 14103	12 % (*m/m*) EN 14103		12,0 % (*m/m*) EN 14103
Polyunsaturated methyl esters, max.	1 % (*m/m*)							1 % (*m/m*) EN 14103
Methanol content, max.	0,20 % (*m/m*) EN 14110			0,5 % (*m/m*) EN 14110		0,20 % (*m/m*) EN 14110	0,5 % (*m/m*) EN 14110[6]	

[5] which is probably a mistake and should read EN EN 12662
[6] Also including free ethanol

Parameter	CEN	ASTM B10	ASTM B20	Brazil	Argentina	Colombia	Paraguay	Uruguay
Alcohol content, max.					0,20 % (*m/m*) EN 14110			0,2 % (*m/m*) EN 14110
Monoglyceride content, max.	0,80 % (*m/m*) EN 14105			( ) EN 14105 EN 14106 ASTM D 6584	0,80 % (*m/m*) EN 14105	0,80 % (*m/m*) EN 14105 ASTM D 6584	0,80 % (*m/m*) EN 14105	0,80 % (*m/m*) EN 14105 ASTM D 6585
Diglyceride content, max.	0,20 % (*m/m*) EN 14105			( ) EN 14105 EN 14106 ASTM D 6584	0,20 % (*m/m*) EN 14105	0,20 % (*m/m*) EN 14105 ASTM D 6584	0,20 % (*m/m*) EN 14105	0,20 % (*m/m*) EN 14105 ASTM D 6585
Triglyceride content, max.	0,20 % (*m/m*) EN 14105			( ) EN 14105 EN 14106 ASTM D 6584	0,20 % (*m/m*) EN 14105	0,20 % (*m/m*) EN 14105 ASTM D 6584	0,20 % (*m/m*) EN 14105	0,20 % (*m/m*) EN 14105 ASTM D 6585
Free glycerol/glycerine, max.	0,02 % (*m/m*) EN 14105 EN 14106	0,02 % (*m/m*) ASTM D 6584		0,02 % (*m/m*) EN 14105 EN 14106 ASTM D 6584[7]	0,02 % (*m/m*) EN 14105 EN 14106 ASTM D 6584	0,02 % (*m/m*) EN 14105 EN 14106 ASTM D 6584	0,02 % (*m/m*) EN 14105 EN 14106 ASTM D 6584	0,02 % (*m/m*) EN 14105 EN 14106 ASTM D 6584
Total glycerol, max.	0,25 % (*m/m*) EN 14105	0,240 % (*m/m*) ASTM D 6584		0,38 % (*m/m*) EN 14105 EN 14106 ASTM D 6584	0,25 % (*m/m*) EN 14105 ASTM D 6584	0,25 % (*m/m*) EN 14105 ASTM D 6584	0,25 % (*m/m*) EN 14105 ASTM D 6584	0,25 % (*m/m*) EN 14105 ASTM D 6584
Group I metals (Na+K) , max.	5,0 mg/kg EN 14108 EN 14109	5,0 mg/kg EN 14538		10 mg/kg EN 14108 EN 14109	5,0 mg/kg EN 14108 EN 14109	5,0 mg/kg EN 14108 EN 14109 ASTM D 5863ASTM D 5863a	5,0 mg/kg EN 14108 EN 14109	10,0 mg/kg[8] EN 14108 EN 14109
Group II metals (Ca+Mg) , max.	5,0 mg/kg EN 14538			( ) EN 14538		5,0 mg/kg EN 14108 EN 14109 ASTM D 5863ASTM D 5863a		5,0 mg/kg[7] EN 14538

[7] All methods are not applicable to coconut or palm oil feedstock
[8] At an experimental level

Parameter	CEN	ASTM B10	ASTM B20	Brazil	Argentina	Colombia	Paraguay	Uruguay
Phosphorus content, max.	10,0 mg/kg EN 14107	0,001 % (*m/m*) ASTM D 4951	0,001 % (*m/m*) ASTM D 4951	( ) EN 14107	10,0 mg/kg EN 14107	10,0 mg/kg EN 14107 ASTM D 4951	10,0 mg/kg EN 14107	( ) EN 14107
Distillation, 90% recovered, max.		360 °C ASTM D1160	343 °C ASTM D1160	360 °C ASTM D1160		360 °C ASTM D1160	360 °C ASTM D1160	
CFPP, max.	climate dependent		climate dependent	- 7 °C ASTM D 6371	climate dependent	( ) EN 116 ASTM D 6731	0 °C IP 391 EN 116	( ) ASTM D 6731 EN 116
Pour point, max.						( ) ASTM D 97		
Cloud point, max.		( ) ASTM D 2500 ASTM D 3117				( ) ASTM D 2500 ISO 3015	3 °C ASTM D 2500 ASTM D 3117	( ) ASTM D 2500
Aromacity, max.			35 % (*V/V*) ASTM D1319					
Thermal Stability, 90 min, pad reflectance			70 min ASTM D 6468			70 min ASTM D 6468		
Lubricity, max.					250 µm ISO 12156-1		250 µm ISO 12156-1	

3.6 Water in diesel emulsion fuels

Emulsion fuel is a mixture of 5 % (*m/m*) up to 25 % (*m/m*) water, diesel and additives to allow use in internal combustion engines. This diesel fuel is typically used by specific heavy duty fleets, for example, urban busses, and helps to reduce the air pollution in big cities. The lack of a European specification on emulsion fuels was the incentive to develop the CWA 15145:2004 “Automotive fuels - Water in diesel fuel emulsions for use in internal combustion engines - Requirements and test methods”. A CWA is a separate not normative document. In general, diesel emulsions cannot be co-mingled with normal diesel. Their use is therefore restricted to captive fleets. According to the engine manufacturers, the use of diesel emulsions in light duty vehicles is not suitable.

CWA 15145 has been based on a French standard (NF M15-021) of 2002 and experience of Italian and UK industry. It is written for 5 % (*m/m*) to 15 % (*m/m*). Two grades of emulsion fuel are introduced, that differentiate for the amount of water and other related parameters (density, viscosity, stability). The higher water content emulsions (Grade A) provide the best environmental benefits, while the lower water content emulsions (Grade B) will provide less power loss, thus enabling applications where power loss is a critical issue.

In ASTM a small group of experts is drafting an emulsion fuel specification. It started of as a quite general effort, but the first draft (WK 6248) received a lot of negative reactions from the membership. The scope is now limited to off-road engines in the agricultural sector and the water sector. Another indication of limitation of the use of the fuel is the fact that a major producer, Lubrizol, has withdrawn from the US market, whilst maintaining activities in Europe.

Furthermore, a Czech publication of 2004 (CSN 65 6509) is known, which sets a water range of 9 %(*m/m*) to 15 %(*m/m*). Next, the requirements on ash content, lubricity and viscosity are less stringent, whereas sulphur content, cold flow and density follow the line of the CWA 15145.

The major differences between CWA 15145 and the ASTM draft, lay in the following requirements, apart from those originating from the local diesel requirements.

- Water content. ASTM has a range of 4 – 22 % (*m/m*), choosing the whole available range. Whereas CEN uses two grades (5 to 8 and 8 to 15), with specific applications.
- Sulphur content. ASTM chose a fixed range, whereas CEN chose a calculation on the basis of the actual sulphur requirement of the automotive diesel. Within Europe the non-road diesel requirements are nearing the regular diesel specifications. CEN studied the applicability of the sulphur test methods for emulsion fuels.
- Total nitrate is required by Italy as a cetane improver to be sure to retain combustion properties in the fuel. Hence this legal requirement is copied in CWA 15145, although for so-called micro-emulsion its use is arguable. CEN made a small check which gave no final conclusion; emulsions seem to have an effect on cetane number, no matter how small the droplets are.

Table 3: Comparison of water emulsion diesel fuels

Property	unit	ASTM draft WK 6248		CWA 15145	
		minimum	maximum	Grade A	Grade B
Density	kg/m3	-	-	828 - 880	825 - 865
Water content	% (*m/m*)	4,0	22,0	≥ 8 - 15	5 - 8
Stability at production	% (*V/V*)	-	-	9	7
Free water	% (*V/V*)	absent	absent	absent	absent
Viscosity at 40°C	mm^2/sec	-	-	2,00 – 5,50	2,00 – 5,50
Sulfur content	% (*m/m*)	0,050	0,500	calculated	calculated

Property	unit	ASTM draft WK 6248		CWA 15145	
		minimum	maximum	Grade A	Grade B
Total contamination	mg/kg	-	-	24 max.	24 max.
Copper strip corrosion	index	no. 3		Class 1	Class 1
Flash point	°C	38	52	70	70
Total nitrate EHN	mg/kg	-	-	0,070 min.	0,050 min.
Lubricity	µm	-	-	400 max.	400 max.
CFPP	°C	According to local diesel spec		According to local diesel spec	
Distillation	°C	- / 300	307/357	-	-
Cetane number		40	-	-	-
Aromaticity	% mass	35	35	-	-

3.7 Methanol

In the US, some experience with methanol blends (70 to 85 volume % methanol and 30 to 14 volume % hydrocarbons) for use in ground vehicles with automotive spark-ignition engines is available (ASTM D 5797). However, the use is limited and the specification not updated since its original drafting in 1996. In Europe negative views exist on the use of methanol in fuels, which is associated with several issues (i.e. toxicity, material incompatibility). Even the current limit of 3 % methanol in EN 228 is considered by some as an undesirable parameter. Views of the CEN stakeholders have been requested on limiting methanol to 0,2 % max. The use of methanol as such and in blends for stationary applications is also considered to be very limited. A European specification on methanol is not foreseen.

3.8 Ethanol blends for regular cars

3.8.1 General perspectives

Ethanol (ethyl alcohol) is a clear, colourless liquid with a faint odour. It has a high latent heat of vaporisation and contains oxygen, characteristics that are relevant to its environmental performance in combustion as a motor fuel, and in its storage and distribution. Ethanol can be produced in two forms: hydrated and anhydrous. Hydrated ethanol, usually produced by distillation from biomass fermentation, contains 95% ethanol with the balance being water. It is suitable for use as a straight spark ignition fuel in warm climates or for blending as a 15% emulsion in diesel. A further process of dehydration is required to produce anhydrous ethanol (100% ethanol) for blending with petrol.

EU production in 2005 was close to 900 million litres of bio-ethanol, whereas consumption was 1200 million. Especially, Sweden, Germany and UK are large importers, with a tendency of going from grain to wine alcohol. Spain is the biggest exporter. Brazilian production in 2005 is estimated at 15½ billion litres and USA is worldwide leader with 16,2 billion. China is third in line.

As far as is known from the enquiry, only Brazil has regulations allowing the hydrated ethanol variant to be used as a fuel. This type is used in existing vehicles with no or minor modifications at levels of 20 % (*V/V*). Czech Republic has a specific ethanol specification, CS 65 6511, which effectively is setting the requirements for ethanol for ETBE. It demands a high purity (99,7 vol%) and further a limited set of requirements.

Argentina had an experience in the 80´s with the mixture of ethanol and gasoline, which lasted until 1989. Because of the international price of ethanol they switched to export. The use of ethanol in gasoline since that time was interrupted. Now, the Argentinean Standardization institute studies the specifications of ethanol for being used in mixture with gasoline as a fuel in automotives, again. This alcohol is the anhydrous variant for blending with gasoline.

India's national specification (IS 2796) originates of 2000, permitting usage of ethanol up to 5% in gasoline. However the actual usage of ethanol by oil companies is controlled by government through notifications based on supply and demand logistics. Presently government notification stipulates usage of ethanol at 5% vol in around 10 states producing ethanol from sugar cane subject to availability of ethanol. It is expected that as more and more ethanol is produced in the country, 5% usage is extended to remaining states also.

As suggested by the Indian government, some of the national oil companies and vehicle manufacturers are engaged in evaluating the suitability of gasoline containing 10% ethanol in the existing vehicles with no or minor modifications. Once this study establishes that there will not be any adverse affects with regard to usage, BIS is expected to consider for inclusion 10 % in national specifications. Once it forms part of national specifications, regulators may consider bringing out notification with regard to usage of gasoline containing 10% ethanol in a phased manner as being done in the case of gasoline containing 5% ethanol. While some of the oil companies and vehicle companies are carrying out studies with regard to usage of gasoline containing around 20% ethanol it is at this stage not possible to indicate the time frame for introduction of such a fuel in India.

China will probably decide to have a nation-wide requirement for blending ethanol, following their intention that in 2020 renewable energy will account for 10 percent of China's annual energy consumption. Currently, eight of its provinces have made E10 mandatory at local petrol pumps. But policy makers experience problems due to availability of supply.

The majority of the automobiles sold in North America are designed with full warranty protection to operate with ethanol-blended gasoline at a concentration of up to 10% ethanol, without engine modification. More than half of all US states have passed alternative fuel-related incentives. These vary from supportive executive orders and state agency requirements to more aggressive incentives that provide tax credits for installing fuelling equipment and procuring vehicles. The incentives tend to reflect the level of local support for alternative energy sources. The use of a tax rate based on a fuel’s energy content is an incentive that has been adopted by several states.

The provinces regulate gasoline quality in Canada. National specifications have been produced by the Canadian General Standards Board (CGSB) of the Government of Canada, and many provinces (including Ontario) require that all gasoline meets CGSB standards. All ethanol-blended gasoline sold in Canada meets specifications enforced by the provincial governments, including specifications regarding volatility levels. The data of the specification are presented in table 4, but not further discussed.

CEN in Europe began its work by examining the American standard ASTM D 4806-01al. This standard is intended for ethanol to be blended at up to 10% in gasoline, so exceeding the current legislative maximum of 5% permitted in European gasoline (EN 228). CEN concluded for a number of reasons that the American specification could not be applied per se in Europe.

- The only denaturant permitted in the ASTM specification is gasoline, whereas in the EU some member states require more than one denaturant for tax exemption reasons.
- Ethanol in the EU is traded in many cases as a pure blending product without denaturants, so a specification without denaturants is required for the determination of conformity with a minimum quality standard. The ASTM specification is written with gasoline already included.
- The water content of the ASTM specification was considered too high for some wet distribution systems in the EU.

Therefore, an examination of the existing ethanol specifications was undertaken, including the ASTM standard quoted above, and the specifications in force in Brazil, in Canada, in Poland, in Sweden and in the Ukraine. Each parameter contained in these specifications was examined for its pertinence in the context of a draft EU standard, for the limit value chosen and for the test method by which it is measured.

Advice was sought also from industry experts on the suitability of the test methods associated with the parameters chosen. The result of these activities is a table of parameters, limit values and test methods issued for ballot in March 2006 under the reference prEN 15376.

It cannot be foreseen how long further discussions on this method will take, but industry and the European Commission have an urge to produce this Standard. Major discussions points still are the denaturing of the ethanol and the acceptable water content. The draft specification has an indication that at each level above 5 %, it shall be reviewed, as the majority of the levels are set for that level. Tendency is to develop one future specification for ethanol as a blending component, just like with FAME, instead of having several specifications for E5, E10 and E85 in the future.

However, the Biofuels Directive of the EC (Directorate General Transport and Energy) sets a target of 5,75% (energy value) for diesel and gasoline automotive use, which cannot be accomplished by 5% ethanol blending. Hence, the 5% maximum ethanol in the Fuels Directive needs to be increased and a 10 KPa waiver for the vapour pressure should be allowed. On the other hand, indications from investigations done by the industry organizations CONCAWE and ACEA are that above 5 % (*m/m*) the evaporative emissions become too high. Even with attempts to have a constant volatility, the emission increase. This mainly due to permeation of and memory effects in the fuel distribution and engine fuelling systems. Therefore, directions of the EC (Directorate General Environment) are awaited.

Table 4: Comparison of bio-ethanol gasoline blends

Parameter	**CEN Anhydrous**	**ASTM Anhydrous denatured**	**Brazil Anhydrous**	**Brazil Hydrated**	**Canada Anhydrous**	**Poland**
	prEN 15376	**D4806**	**RESOLUÇÃO Nº36**	**RESOLUÇÃO Nº36**	**CGSB-3.511-93**	
Ethanol, min.			99,6 %(*V/V*)	95,1 %(*V/V*) ASTM D5501		
Ethanol and higher alcohols, min.	98,7 *%(m/m)* EC/2870/2000	92,1 %(*V/V*) ASTM D5501	99,3 %(*m/m*) NBR 5992	(92,6 – 93,8) %(*m/m*)) [9] ASTM D5501	98,75 %(*m/m*)	99,6 (V/V)
Higher alcohols C3-C5, max	2,0 *%(m/m)* EC/2870/2000 EN 13132 EN 1601		3,0 %(*V/V*) NBR 13993			
Methanol, max.	1,0 *%(m/m)* EC/2870/2000 EN 13132 EN 1601	0,5 %(*m/m)* ASTM D1152				0,1 (V/V)
Existent gum content (solvent washed), max.		5,0 mg/100mL ASTM D381		5 mg/100mL NBR 8664		
Water content, max.	0,3 *%(m/m)* prEN 15489	1 %(*V/V*) = 1,6 %(*m/m*) ASTM D1193			0,1 %*m/m*)	0,4 %(*m/m*)
Denaturant content	10	1,96 – 5,0 %(*V/V*) ASTM D5580			5 max %(*V/V*)	
Inorganic Chloride content, max.	20 mg/L prEN 15484 prEN 15492	40 ppm (32 mg/l) ASTM D512				40 mg/kg Chlorine

[9] (92,6 - 94,7) in distribution and importation (hydrocarbon contamination)

[10] The concentration of denaturant(s) is to be decided by national authorities and shall meet requirements of EN EN 228

Parameter	CEN Anhydrous	ASTM Anhydrous denatured	Brazil Anhydrous	Brazil Hydrated	Canada Anhydrous	Poland
Halogen, max.				1 ppm (chloride ion) NBR 10894 NBR 10895 ASTM D 512	10 (chlorine)	
Copper content, max.	0,1 mg/kg prEN 15488	0,1 mg/kg ASTM D1688	0,07 mg/kg NBR 10893		0,1 mg/l	0,1 ppm
Iron content, max				5 mg/kg NBR 11331		
Phosphorus, max.	0,5 mg/L prEN 15487					
Sulphur, max.	10,0 mg/kg prEN 15485 prEN 15486	30 ppm ASTM D5453				
Sulfate, max.		4 ppm *annexed*		4 mg/kg NBR 10894 NBR 12120		
Sodium content, max				2 mg/kg NBR 10422		
Acidity (as acetic acid CH_3 COOH), max.	0,007 *%(m/m)* prEN 15491	0,007 %(*m/m*) ASTM D1613	30 mg/l NBR 9866 ASTM D1613	30 mg/l NBR 9866 ASTM D1613	30	0,03 gr / l (30 mg / l)
pHe	6,5 – 9,0	6,5 – 9,0 ASTM D6423		6,0 – 8,0 NBR 10891		
Appearance	clear and bright	clear & bright	Clear and impurity free	Clear and impurity free		
Density at 20°C			791,5 kg/m^3 min. ASTM D4052	(807,6 – 811,0) kg/m^3 [11] ASTM D4052	789	
Electrical conductivity, max.			500 µS/m NBR 10547 ASTM D 1125	500 µS/m NBR 10547 ASTM D 1125		
Involatile material, max.	190 mg/100ml EC/2870/2000	–			30 mg/L (at 100°C)	20 mg/L (dry extract)
Aldehydes as acetaldehyde						0,2 g/L
Esters as ethyl acetate						0,2 g/L
Fusel oils as amyl alcohol						0,2 g/L

3.8.2 Comparison of requirements

3.8.2.1 The ethanol and higher alcohols content is specified as a minimum to ensure a correct degree of purity for its use in automobiles, and to qualify for Custom and Excise recognition. ASTM has a

[11] (805,0 - 811,0) in distribution and importation

fairly lower limit, mainly because a denaturant (gasoline) is allowed. The maximum value of higher saturated alcohols (C3 – C5) is limited to 2,0 % (*m/m*) at CEN and 3,0 % (*m/m*) in Brazil, this being a comfortable accommodation for the value normally seen from ethanol production. These are the only two specifications with a separate limit. Consideration in Europe was given to copy the Brazilian limit. But whereas these alcohols may be also be added to gasoline under the heading of oxygenates allowed by EN 228 as acceptable alcohols, it was decided that the higher saturated alcohols were sufficiently treated by the oxygenates directive EEC/85/536 and in the EN 228 gasoline specification.

Methanol is naturally present in industrially produced ethanol in small quantities, and can contribute to the combustion as an oxygenate. However, methanol is toxic, it has a very high heat of vaporisation, it is strongly hydroscopic and contributes strongly to the formation of azeotrope and thus high vapour pressure. It may require a co-solvent to prevent separation and can be aggressive towards certain metallic and non metallic materials in the on-board fuel delivery systems. Thus its volume should be limited.

The ASTM specification limits methanol content in ethanol to 0,5 % maximum. The European gasoline specification permits up to 3 % methanol in the gasoline, with the presence of co-solvents to prevent separation. This possibility has not been used in recent times. In view of the apparently successful situation of 10 % ethanol in the USA, CEN incorporated a value of 1 % maximum methanol in the ethanol specification. So giving the same maximum methanol concentration when ethanol is present at 5% (E5) as that allowed in the USA for E10.

3.8.2.2 Ethanol is hygroscopic, and can collect water both from its distribution system and from ambient air. Blends of fuel ethanol and gasoline have a limited solvency for water, depending on ethanol content, the temperature of the blend and the aromatic content of the base gasoline. In unfavourable circumstances, a separation of the ethanol and water will occur and form an aqueous lower phase in both the storage tank and the vehicle fuel tank that will cause serious operating problems for the engines. The oil industry and vehicle constructors, in order to minimise the risk of such problems occurring, ask the ethanol producers to limit the water content of their neat product to the lowest practical value.

CEN initially discussed a level of 0,2 % (*V/V*) water in neat ethanol, based on current Swedish production practice. The ASTM standard is set higher, at 1,0 % (*V/V*), though lower values are necessary at very low ambient temperatures. American ethanol distilleries operate a voluntary agreement with gasoline blenders to limit water content at the factory gate at 0,65%. After 25 years of distributing ethanol there is still a de facto 0,35% water pick-up limit for the distribution system beyond the factory gate. The Americans still believe that this is a necessary measure to avoid water pick-up disputes.

On the basis of not imposing constraints on ethanol that are greater than gasoline (in this case in the USA for E10, as the EN 228 does not have a maximum water content limit), the ethanol producers suggested adopting the ASTM value. However, the European maximum ethanol content of 5% being lower than that in the USA, can lead to easier water dropout. The ethanol producers proposed to set the water limit at 0.2 % (*V/V*) in neat ethanol, this being a satisfactory level both for refiners and constructors. This level is still under discussion as the Swedish industry has long-time experience with slightly higher levels. This also based on the import of Brazilian anhydrous ethanol, which specification sets no limit at all for water content.

3.8.2.3 Sulphur limits are in USA and Europe accustomed to the gasoline specifications and regulations. Other countries do not have this requirement copied in their ethanol specification, but the marketed gasoline should still fulfil the national limits.

Heavy deposits of sodium sulphates had been found in mechanisms of the fuel distribution chain and had also caused deposits in vehicle injectors during the years 2002 – 2003 in certain areas of the USA. Experts working in ASTM identified the deposits as sodium sulphates originating from the ethanol blended in the gasoline. The ASTM took action and this year a limit of 4 ppm of sodium sulphate in ethanol is accepted. The same limit is copied in the Brazilian specification. However the measurement method is still not fully accepted. The potentiometric lead titration determination, D4806, is now used, but serious objections for the use of lead in several labs in the States is noted. Alternative methods, like ion chromatography, and the lead titration itself are under improvement, but the results so far are not great. The method and limit value is expected to be introduced into the ASTM specification by the year 2008.

Introducing such a sulphate limit for the European ethanol may cause problems of incoherence with the 10 ppm limit of sulphur. The European ethanol producers agreed to survey the current levels of sodium sulphates in their industry and to report back at every meeting of CEN. No indications within the EU market of problems have been found and also in the USA the problems seems to be under control.

3.8.2.4 A debate occurred in Europe on which of the two parameters, solvent washed gums or involatile material, should be specified in the automotive ethanol draft standard. The former is required in general gasoline and ASTM ethanol standards, whereas the latter was preferred by Swedish and Canadian standards.

Both parameters are associated with impurities in the ethanol that are associated with fuel filter blocking and engine deposit formation. Tests for solvent washed gums are associated with the removal of performance additives and their carrier oils, but it is unlikely that these will be incorporated into a neat ethanol before blending with gasoline. European work showed that involatile materials in ethanol may exceed solvent washed gums by a factor of two. Therefore the involatile material parameter was chosen by CEN and a compromise value of 10 mg/100ml was accepted as a limit value.

3.9 Ethanol for use in so-called flexible fuel cars

Today the use of E85, a fuel which is composed out of approximately 85% ethanol, is still a niche market but growing progressively. Already in 1999 a ASTM specification has been established, which has since not been updated. Mainly because the fuel itself has in the first years been hardly available.

In some EU countries – e.g. Sweden and Spain - adapted cars use this type of gasoline, too. Several other countries, like Germany, the Netherlands or Switzerland, have small pilot projects for fleets. Initiated by Ford, a CEN Workshop Agreement (CWA 15293:2005) has been established, which lays down the technical specifications for this fuel. The specification is based on the ASTM work and on the developments within CEN on the E5 specification (prEN 15376, see above). This CWA is a product that is not ratified or adopted by the Member States, but can freely be used for trade or regulatory (licences) purposes. Effectively it has been used in Germany and the Netherlands. French government has recently established a stakeholder group to write this CWA into a national standard.

When looking to the differences, these are merely historical. Acidity, pHe and inorganic chloride content are the same, whereas the content of gum, water and phosphorus is set at limits related to regional gasoline specifications or test method detection boundaries at the time of writing of the specifications. The ASTM (E75-E85) specification is of an early period (1996 – 1999) when the ethanol specification has just been developed. Sulphur levels in ASTM are still high (210 to 300 ppm) and calculated from the regular gasoline values. The CEN document choose to keep track of the upcoming 10 ppm requirement. The worst case of the blend limit is 10 ppm in ethanol and 50 ppm in 25% of gasoline, makes for E85 20 ppm at this moment and 10 ppm in future.

The major difference is in terms of volatility. Volatile hydrocarbons are needed for adequate cold startability. Ford requires 34 kPa. To meet hot and cold vehicle driveability requirements under the seasonal and geographical conditions, different classes can be chosen. Three classes are chosen by ASTM, with alcohol minima of 79, 74 and 70 % (*V/V*). The hydrocarbon and ether content is always 17 % (*V/V*) at minimum, making the product effectively an E83.

The European CWA limits the producer to two classes. The writers wanted to limit the amount of classes; the majority of applications around the time of writing were in Sweden and Spain. Next, the blenders need to use the available petrol in the market. The problem was that in Spain the producers were restricted to vapour pressure of petrol complying to EN 228. It was decided to set a minimum of 75 % ethanol content which just conforms with a minimum 35 kPa for the final fuel in Spanish summer.

For other discussion points and ideas towards future general ethanol specifications see paragraph 3.8.

3.10 Ethanol in diesel

Present national specifications of automotive diesel do not permit usage of ethanol as a blending component. Considering the reservations of fuel pump and vehicle manufacturers with regard to safety aspects, inadequate lubricity of diesel blended with ethanol especially in present day high pressure fuel injection systems and availability of sufficient ethanol for blending in diesel (for instance, India consumes large quantity of diesel, which is around six times of gasoline), various experts consider it may not happen at least in the foreseeable future.

In all countries, gasoline gets first priority compared to diesel with regard to blending with ethanol considering the present intentions of various stakeholders and associated techno-commercial issues. In the USA the Department of Defence requests use of alternative fuels and the military is currently using biodiesel and E-Diesel. The general guideline is to handle it like gasoline and limit the use to centrally-fuelled fleets. The major use is based on 7,7% (*V/V*) ethanol and 1% diesel improver, but no general standards have been applied. In Europe some work has been done in the past in Sweden, which is now again assessed for future standardization possibilities by the already mentioned BIOScopes project.

In Europe, the use of ethanol or similar low-flashpoint materials in diesel fuel is not generally accepted. The members of CEN from oil and motor industries are unanimous in rejecting the use of these materials on safety grounds (i.e. generation of vapour-air mixture, which falls within the flammability range). Only professionals should handle ethanol in diesel blends. The development of a CWA is recommended for this category. The recommendation for using ethanol in the diesel context will be to encourage ethyl esters as a diesel fuel component if the biodiesel taskforce recommends these products.

3.11 Di-Methyl Ether (DME)

This product is mainly investigated and used in Japan and South-Korea. For most oil industries it is still a development product. On initiation of the Japanese industry, ISO has accepted to include DME in its scope. Proposals for starting a working group on a fuel specification (under ISO/TC 28/SC 4) and some measuring standards (ISO/TC 28/SC 5) are anticipated to come from either Japan or France for this year.

3.12 Liquefied Petroleum Gas (LPG)

ISO 8216-3 *Petroleum products; Fuels (class F); Classification; Part 3 : Family L (Liquefied petroleum gases)* gives the basic ideas for classification. ISO 9162 *Petroleum products; fuels (class F); liquefied petroleum gases; specifications,* originates from 1989 and is an international trade quality designation. It has no specific automotive requirements.

As the major use is in cylinders, the automotive application has not been standardized much. Originating from the industries' request to market LPG as an alternative fuel back in the nineties when sulphur became an issue in Europe, CEN published EN 589 *Automotive fuels - LPG - Requirements and test methods* in 1999 and a revision in 2004. Some updating of test methods has been undertaken, but pending discussions and investigations on sulphur-free odorants no future revisions of the limit are to be expected.

ASTM D 1835, *Standard Specification for Liquefied Petroleum (LP) Gases*, gives four qualities of LPG, of which one (special duty) is meant for automotive use. Maximum sulphur level is set higher, but maximum vapour pressure lower than with CEN. Next to this, Australia, another big user of LPG drafted a specification more in line with the European requirements:

3.13 Natural gas (CNG / LNG)

Compressed natural gas (CNG) has been used to some extent as a fuel for internal combustion engines in compressor stations, co-generation systems, and vehicles of various types for many years now. With the natural gas industry well established and the need for alternative, low emission fuels, the situation is improving. During the past decade natural gas vehicles (NGV's) have become a viable option with some one million units now in use around the world. In traditional natural gas countries, like Italy and the Netherlands, government is preparing (additional) legislation or tax incentives for CNG vehicles and NGV fuelling stations.

In majority, the equipment and vehicles are standardized all over the world, whereas the gas used is that what is locally available. In general, the international specification ISO 15403 *Natural gas - Designation of the quality of natural gas for use as a compressed fuel for vehicles*, is used as the basis. This method is adopted by CEN. The originator of the document, ISO/TC 193, will publish a revision of this quality designation in two parts in 2006. Part 1 *Designation of quality*, basically the old 2000 Standard, stipulates the international requirements placed on natural gas used as a motor fuel. It gives warnings and general requirements for safe operation, corrosion prevention, fuel system poisoning and optimal climatic performance. It gives details on calculation of calorific value (Wobbe Index) and octane number. The requirements are meant for the constructors, to develop high-performance equipment for CNG.

A new Part 2 of ISO 15403 is an informative Technical Report, which incorporates a detailed gas composition and quality specification. It addresses content of water, sulphur compounds, particulates, higher hydrocarbons, CO2, free oxygen, glycol, methanol, oil content and corrosive components. It is a quality designation at deliverance to the car, allowing reformulation at the pump of the natural base gas. Due to limited experience and worldwide consensus, an informative character for this specification was chosen as an intermediate.

Liquefied natural gas (LNG) may also be stored in the fuel tanks of natural gas vehicles. This, however, will be the subject of a future separate ISO Standard.

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