CEN/TC 19 2011 Conference

➔European biodiesel and bio-ethanol quality specification history

Barry Cahill, Autofuel Services

CEN, Avenue Marnix 17

1000 Brussels, Belgium

info@cen.eu / www.cen.eu

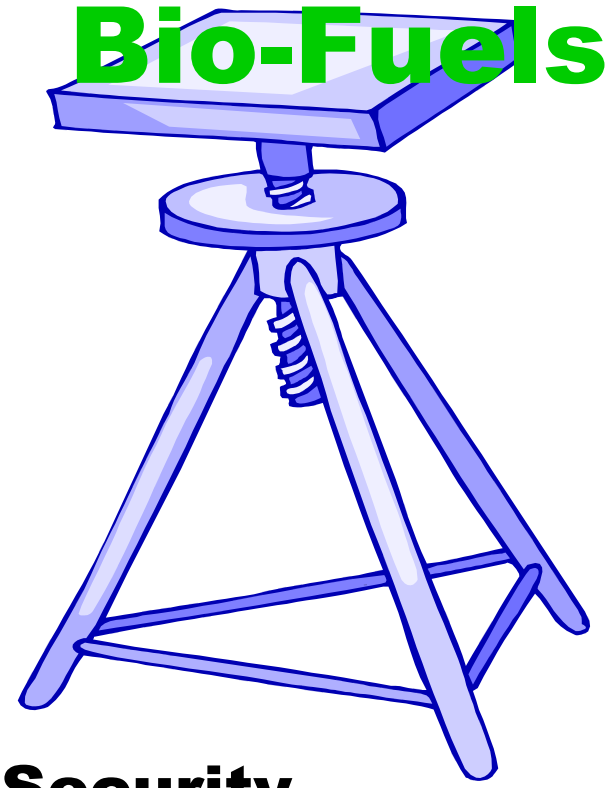
CEN/TC 19, Vlinderweg 6

2623 AX Delft, the Netherlands

ienergy@nen.nl

+31-152690330

Biofuels Sustained by Three Policy Legs



Agriculture

Energy Security

Environment

CEN BIOFUEL STANDARDS: INTRODUCTION

- European fuel standards essential to allow homogeneous market for distribution and use. Motorists may travel across borders without fear of fuels incompatible with their vehicles.
- Petrol and diesel fuels have been standardized in Europe for some years, and CEN standing committees WG21 (petrol) and WG24 (diesel fuel) work continuously to ensure fuel standards are compatible with vehicle requirements and EU environmental constraints such as the Fuel Quality Directive and Renewable Fuels Directive.

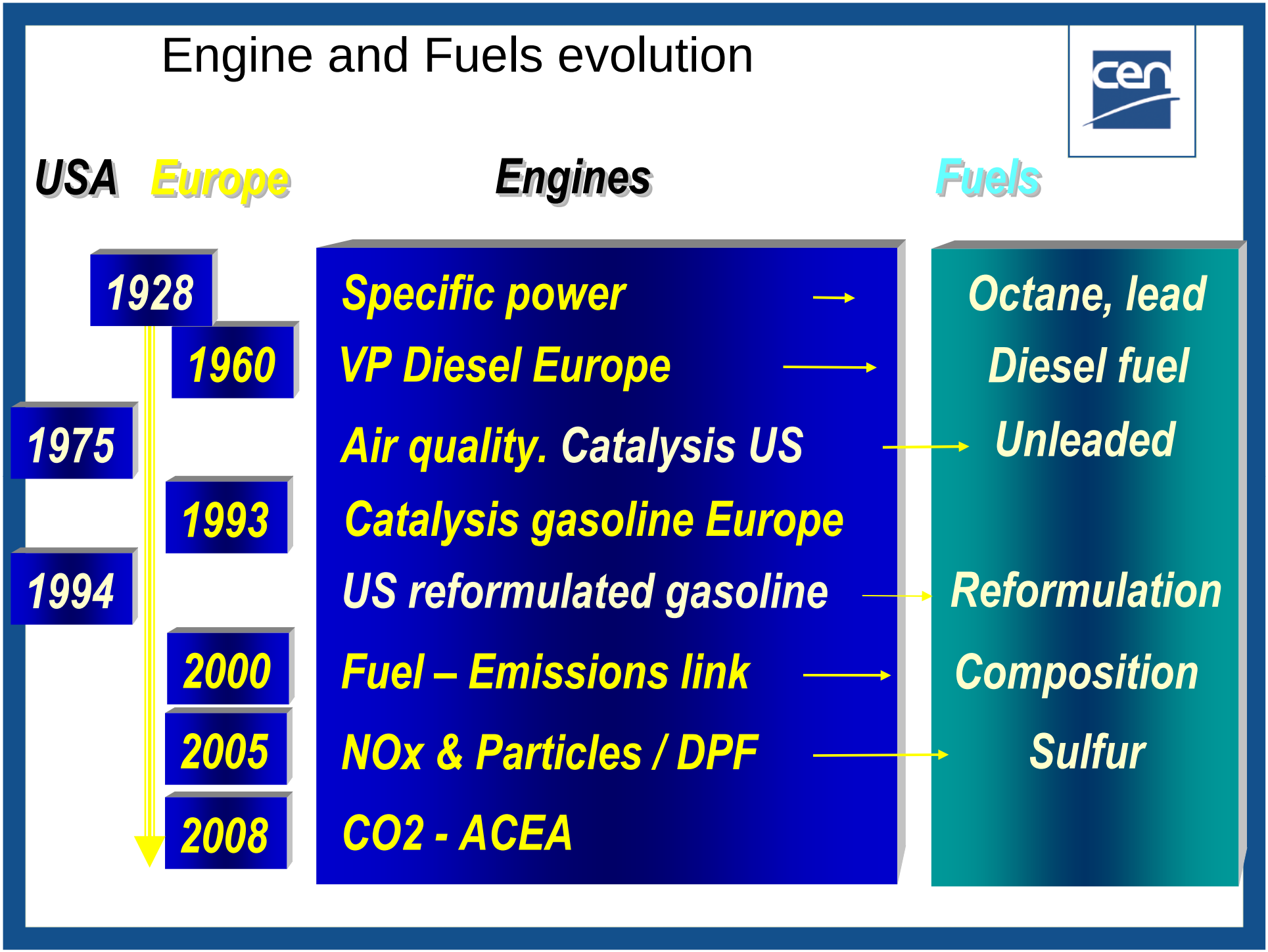
Renewable bio-fuels

- EU has turned to CEN to translate its policy on renewable fuels into quality standards to be adopted by Member States
- Biofuels identified as biologically sourced ethanol and ethers for petrol engines, and Fatty Acid Methyl Esters (FAME) for diesel engines. Ethanol and ethers both used as extenders in petrol vehicles, whereas FAME can also be a stand-alone pure diesel fuel as well as an extender for petroleum-based diesel.
- Task Forces set up to develop standards, working under guidance of WG21 (petrol) and WG24 (diesel fuel)
- EN 14214 FAME first published 2003, with later revisions
- EN 15376 Bio-Ethanol published 2007, with later revision

Development of the biofuel standards

- Traditionally standardization partners are oil refiners and automobile industry. The agricultural industry experts are now new actors in the CEN forum. It took time to inform them of the engines' fuel requirements.
- Engines have been developed over nearly 100 years on petroleum-based fuels, so new biofuel parameters have made the standardization work more complicated.
- The contribution of the agricultural industry experts in CEN fuel standardization has allowed the biofuel market to expand in Europe.

Engine and Fuels evolution



Fatty Acid Methyl Esters (FAME) for Diesel

- Pure vegetable and animal oils are generally unsuitable as diesel fuel, so FAMEs are the products closest to petroleum diesel fuel. Many different sources possible.
- FAME may be used as a stand-alone fuel (B100) or in blends with petroleum diesel fuel (B5 B7 B10 B30 etc.). The same FAME standard applies to each application.
- By far, B5 B7 and B10 are the most common applications as invisible to the end user and covered by vehicle warrantee. B100 is subject to constructor's approval, generally specially adapted engines for captive fleet use.
- Naturally diesel fuel EN 590 is examined and adapted according to its FAME content. Even small changes in fuel characteristics can have important engine consequences.

Characteristics of CEN FAME standard

Table 1 — Generally applicable requirements and test methods

Property	Unit	Limits		Test method [a] (See Clause **Erreur ! Source du renvoi introuvable.**)
		minimum	maximum	
FAME content [a]	% (*m/m*)	96,5 [b]	–	prEN 14103
Density at 15 °C [c]	kg/m^3	860	900	EN ISO 3675 EN ISO 12185
Viscosity at 40 °C [d]	mm^2/s	3,50	5,00	EN ISO 3104
Flash point	°C	101	–	EN ISO 2719 [e] EN ISO 3679 [f]
Sulfur content [g]	mg/kg	–	10,0	prEN ISO/DIS 20846 prEN ISO/DIS 20884[l] prEN ISO/DIS 13032
Cetane number [i]	–	51,0	–	EN ISO 5165
Sulfated ash content [j]	% (*m/m*)	–	0,02	ISO 3987
Water content	mg/kg	–	500	EN ISO 12937
Total contamination	mg/kg	–	24	EN 12662 [k]
Copper strip corrosion (3 h at 50 °C)	Rating	class 1		EN ISO 2160
Oxidation stability	h. at, 110 °C	8,0	–	EN 15751 [l] EN 14112
Acid value	mg KOH/g	–	0,50	EN 14104
Iodine value	g iodine/100 g	–	120	EN 14111
Linolenic acid methyl ester	% (*m/m*)	–	12,0	EN 14103
Polyunsaturated (≥ 4 double bonds) methyl esters	% (*m/m*)	–	1,00	EN 15779
Methanol content	% (*m/m*)	–	0,20	EN 14110
Monoglyceride content	% (*m/m*)	–	0,80	EN 14105
Diglyceride content	% (m/m)	–	0,20	EN 14105
Triglyceride content [a]	% (m/m)	–	0,20	EN 14105
Free glycerol	% (m/m)	–	0,02	EN 14105 [l] EN 14106
Total glycerol	% (m/m)	–	0,25	EN 14105
Group I metals (Na+K)	mg/kg	–	5,0 [n]	EN 14108 [m] EN 14109 EN 14538
Group II metals (Ca+Mg)	mg/kg	–	5,0	EN 14538
Phosphorus content [g]	mg/kg	–	4,0 [n]	EN 14107

Characteristics of FAME standard (cont.d)

- FAME is different to diesel fuel, so the standard contains precautions against negative engine effects:
- Resistance to oxidation:
  - Test for oxidation stability=> “Rancimat” test EN 15751 initially required 6 hours resistance to oxidation for pure FAME, now 8 hours since B7 became widespread. (The finished diesel fuel containing 7% FAME requires 20 hours).
  - Iodine value fixed at 120 g iodine/100g., difficult for some feedstocks.
  - Limits on glycerol/glycerides: Limits were set on glycerol, mono-, di- and tri-glycerides to limit the risk of potentially unstable fuels forming solid deposits.
  - Use of stability enhancing additives is recommended. An effect equivalent to that of 1000 mg/kg of butylated hydrolyl-toluene (BHT) to FAME is proposed (except for arctic grades)

Characteristics of FAME standard (cont'd)

- Cold operability:
  - Same requirements for B100 as for EN 590 diesel fuel, with climatic grades dependent on national climatic requirements
  - Originally no special cold operability requirement for FAME blending component, but recent market problems had dictated special requirements to be set by CEN. The formation of solid deposits at ambient temperatures higher than CFPP with consequent filter blocking have caused difficulties for filling stations and vehicle users.
  - Saturated mono-glycerides strongly suspected as bad actors, but no standardized test method exists to characterize them.
  - Therefore an upcoming revision of EN 14214 FAME proposes new limits for cloud point, cold filter plugging point and total monoglycerides for FAME blending component in order to limit risk of deposit formation at low temperatures.

Characteristics of FAME standard (cont'd)

- Impurities:
  - Limits set for Group I (Na, K) and Group II (Ca, Mg) metallic salts to avoid exhaust catalyst poisoning. Recent discussions indicate Na may cause injector lacquering so possible lower limits may be set. This requires adequate test methods to measure low levels, and requires also industrial feasiblity by producers to reduce these impurities.
  - Phosphorus also degrades exhaust catalysts, so original limit of 10 mg/kg now lowered to 2 mg/kg

- The EN 14214 FAME standard is complicated, containing 19 pages and 9 tables of requirements, so careful reading is necessary by all users to ensure compliance.

FAME: outstanding items

- This presentation has not covered all the characteristics of FAME, only the most outstanding ones.
- The standard will evolve again in the future:
  - Measurement of cetane number of FAME
  - Review of Na content limit
  - Integration of Fatty Acid Ethyl Esters
  - New and updated test methods, ongoing
  - Modifications related to any future market fuel problems

Bio-ethanol standard EN 15376

- Once again, the agricultural experts were new actors in the definition of CEN bio-ethanol standard EN15376 for blending with petrol.
- An American ethanol fuel standard existed when CEN work began in 2003. Nevertheless it took longer than expected to develop a standard adapted to European requirements.
- Ethanol is used in blends with petrol to provide finished fuel (E5 E10 E85…) but CEN focused originally on ethanol for E5 with latest evolution to take the E10 situation into account. This aligns with the EU policies on renewable fuels and the CEN petrol specification EN 228 is also being adapted to E10.
- A separate CEN Taskforce defines the specification of E85.

Characteristics of bioethanol standard EN 15376

Property	Unit	Limits		Test methods
		minimum	maximum	
Ethanol content and higher saturated alcohols	*% m/m*	98,7		EN 15721
Higher saturated (C3-C5) mono-alcohols content	*% m/m*		2.0	EN 15721
Methanol content	*% m/m*		1,0	EN 15721
Water content	*% m/m*		0,300	EN 15489 EN 15692
Total acidity (expressed as acetic acid)	*% m/m*		0,007	EN 15491
Electrical conductivity	*µS/cm*		2,5	prEN 15938
Appearance		Clear and colourless		EN 15769
Inorganic chloride content	mg/kg		6,0	EN 15484 EN 15492
Sulfate content	mg/kg		4,0	EN 15492
Copper content	mg/kg		0,100	EN 15488 prEN 15837
Phosphorus content	mg/l		0,15	EN 15487 prEN 15837
Involatile material content	mg/100ml		10	EN 15691
Sulphur content	mg/kg		10,0	EN 15485 EN 15486 prEN 15837

Characteristics of bio-ethanol standard EN 15376

- Ethanol content:
  - Set at 98.7% to satisfy Customs and Excise recognition
  - Higher alcohols C3-C5 at 2% max. included in ethanol content (can be by-product in process, and not harmful)
- Methanol content:
  - Set at 3% max. due to EU Fuel Quality Directive. Methanol not desirable, harms health, engine components damage. USA sets 0.5%. Polish experts proposed CEN limit of 0.1%. Fortunately not used by the industrial actors today.
- Water content:
  - Ethanol is hygroscopic, and suspended water can fall out of fuel in certain circumstances. Harmful for engines. First proposal was 1% max., disapproved by Auto/Oil. Finally 0.3% max. chosen. Most difficult case is E5.

Characteristics of bio-ethanol standard EN 15376

- pHe/conductivity:
  - Desirable to set between pHe 6.5-9.0 to limit component corrosion. Methods difficulties did not permit limits to be set.
  - Eventually electrical conductivity used to characterize acidity with limit of 2.5µS/cm being set.
- Inorganic chlorides:
  - Corrosive to metals. Limit originally 20mg/l for ethanol in E5, later set at 6 mg/kg for ethanol in E10 (thanks to ethanol producers' cooperation).
- Sulfate content:
  - USA found sodium sulfate deposits in filling station filters. CEN took precautions to set a 4 mg/kg max. limit similar to ASTM (maybe too high for E85)

Characteristics of bio-ethanol standard EN15376

- Denaturants:
  - Ethanol is a basis for many agreeable alcoholic beverages, so Customs and Excise are very interested.
  - Industrial ethanol must be denatured to prevent human consumption.
  - Wide range of denaturing products used by C&E, some harmful to engines and humans (notably methanol).
  - CEN not permitted to contradict C&E, but does recommend non-harmful denaturants:
    - Petrol / ETBE / MTBE /Isobutanol / Isopropanol / Tertiary Butyl Alcohol (TBE)
- Biological source:
  - EU wants renewable ethanol for tax exemption
  - C14 scintillation method distinguishes biological from fossil feedstock
  - Otherwise “Audit Trail” may be used.

Further evolution of bio-ethanol standard EN 15376

- The bio-ethanol standard is secure for use in E5 / E10 but may require further examination for E85
- A single ethanol standard for all uses is desirable for cost reduction from rationalized logistics.
- Some ethanol impurities may need to be reduced again for use in E85
- CEN is combining the Ethanol Taskforce and the E85 Taskforce in a single group to treat the further evolution of ethanol in all circumstances.

European biodiesel and bio-ethanol quality specification history

Thank you for your attention!