

**WHITE PAPER**

**ON**

**INTERNATIONALLY COMPATIBLE BIOFUEL**

**STANDARDS**

**TRIPARTITE TASK FORCE**

**BRAZIL, EUROPEAN UNION &**

**UNITED STATES OF AMERICA**

**DECEMBER 31 2007**

## **DISCLAIMER**

The members of the Task Force are experts in the field of biofuels from each region and have been nominated by the regional standardization institutions and governmental bodies. Although in many cases already members of their regional fuels standardization activities, the experts do not in this case represent their parent standardization bodies and the remarks and conclusions contained in the report do not commit the regional standardization bodies to subsequent action.

The same applies for the representatives of the governmental bodies. The information in this White Paper should not be considered as the official position of the Government of Brazil, the European Commission or the Government of the United States of America.

Neither the Government of Brazil, nor the European Commission nor the Government of the United States of America, nor any person acting on their behalf may be held responsible for the use which might be made of the information contained herein.

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# **1 EXECUTIVE SUMMARY**

## **1.1 Background**

During trilateral discussions in 2006, the Government of Brazil, the European Commission (representing the European Union) and the Government of the United States of America affirmed their belief that the current market for biofuels is viable, the market will continue to grow within regions, and that international trade in biofuels would increase significantly by the end of the decade.

In February 2007, a conference was organized by the European Commission and the European Committee for Standardization (CEN), with the active participation of the U.S. National Institute of Standards and Technology (NIST) and the Brazil's National Institute of Metrology, Standardization, and Industrial Quality (INMETRO). This meeting, held in Brussels, convened a broad range of private-sector biofuels experts and government representatives from the EU, US and Brazil. The participants identified that differing standards for biofuels were a potential handicap to the free circulation of biofuels among the three regions.

To support the global trade of biofuels, representatives of Brazil, the EU and the U.S. agreed to promote, whenever possible, the compatibility of biofuels-related standards in their respective regions. Such compatibility would not only facilitate the increasing use of biofuels in each of the regional markets, but also would support both exporters and importers of biofuels by helping to avoid adverse trade implications in a global market.

Subsequently, the International Biofuels Forum – a governmental initiative among Brazil, China, the European Commission, India, South Africa, and the United States – was launched in March, 2007 to promote the sustained use and production of biofuels around the globe. The IBF also concluded that trade will play an increasing role in providing adequate supplies of biofuels to the markets where the energy demand for transport fuel is rising at an accelerated rate.

A Biofuels Standards Roadmap was developed in April 2007 that delineated the necessary steps that needed to be undertaken by the U.S., Brazil, and EU to achieve greater compatibility among existing biofuel standards (see Annex 1). This Roadmap was a key topic of discussion during the U.S.-EU Economic Summit held in the same timeframe. The Summit also acknowledged support of the goals of the IBF.

In June, a NIST and INMETRO-sponsored Biofuels Symposium in Washington, DC, convened representatives from Brazil, the EU and the U.S. to build on the work begun in Brussels. These representatives agreed to review existing documentary standards for biofuels and identify areas where greater compatibility could be achieved in the short and long term. According to the tripartite agreement, the standards to be considered were those produced by ABNT, ANP<sup>1</sup>, CEN and ASTM International and in effect before the end of 2007 (see Annex 2).

It was further agreed that only standards pertaining to the biofuels being currently traded – biodiesel and bioethanol – would be addressed; this was further limited to pure biofuels (as a blending component to diesel or petrol respectively) and not to ready-made blends.

Comprised of representatives from the private and public sectors, the Biodiesel Tripartite Task Force and the Bioethanol Tripartite Task Force each started their technical work in July. The immediate task was to classify the various specifications<sup>2</sup> into three categories:

- Category A: specifications that are already similar;
- Category B: specifications with significant differences between parameters and methods, but which might be aligned by work on documentary standards and measurement standards; and
- Category C: specifications with fundamental differences, perhaps due to emissions or environmental regulations within one or more regions, which are not deemed bridgeable in the foreseeable future.

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<sup>1</sup> Brazilian Petroleum, Natural Gas and Biofuels Agency

<sup>2</sup> “Specifications” refer to individual parameters, their values and related test methods.

The two groups were also to comment upon the extent and relative impact of the work that would be needed to bring closer alignment between the specifications, thus forming a preliminary basis for prioritization of next steps.

## **1.2 Discussion and Results**

Members of the two Tripartite Task Forces are to be congratulated for their extraordinary efforts. Both groups completed their tasks on schedule, using their own resources and conducting their work via teleconference or in conjunction with various related international meetings. There were commonalities with the approach and methodology used by both of the Task Forces. Each of the two groups assembled and translated existing standards from ABNT, ASTM International and CEN, and the units for specifications were converted to a common basis. Each Task Force first compared the standards as they presently exist. Since it was noted that many parameters were different, the Task force members entered into discussions and negotiations and were able to make specific recommendations to address these differences. They further agreed that these recommendations should be forwarded to standards bodies for consideration and possible implementation.

Summary results from each group are listed below in Tables 1 (biodiesel) and 2 (bioethanol). Note that these tables provide only a summary of the specifications in the three categories; detailed comments and further clarifications are given in Sections 2 and 3 of this report.

### **1.2.1 Summary and Analysis of Standards for Biodiesel<sup>3</sup>**

The Biodiesel Tripartite Task Force considered relevant standards and specifications, documents on the parameters and methods, and commentaries on the similarities or differences of the specifications.

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<sup>3</sup> For the purposes of the work of this task force, biodiesel is defined as mono-alkyl esters of long chain fatty acids derived from plant oils or animal fats and used, for example, as fuel for compression ignition, internal combustion piston engines.

### 1.2.1.1 General Observations for Biodiesel Standards

The current standards established to govern the quality of biodiesel on the market are based on a variety of factors which vary from region to region, including characteristics of the existing diesel fuel standards, the predominance of the types of diesel engines most common in the region, and the emissions regulations governing those engines. Europe, for example, has a much larger diesel passenger car fleet, while United States and Brazilian markets are mainly comprised of heavier duty diesel engines. It is therefore not surprising that there are some significant differences among the three sets of standards.

**Table 1: Classification of the Various Biodiesel Specifications**

<b>Category A</b> <i>similar</i>	<b>Category B</b> <i>significant differences</i>	<b>Category C</b> <i>fundamental differences</i>
sulfated ash	total glycerol content	sulfur content
alkali and alkaline earth metal content	phosphorus content	cold climate operability
free glycerol content	carbon residue	cetane number
copper strip corrosion	ester content	oxidation stability
methanol & ethanol content	distillation temperature	mono, di-, tri-acylglycerides
acid number	flash point	density
	total contamination	kinematic viscosity
	water content & sediment	iodine number
		linolenic acid content
		polyunsaturated methyl ester

Other sources of regional differences in biodiesel standards arise from the following factors. The biodiesel standards in Brazil and the U.S. are applicable for both fatty acid methyl esters (FAME) and fatty acid ethyl esters (FAEE), whereas the current European biodiesel standard is only applicable for fatty acid methyl esters (FAME). Also, the standards for biodiesel in Brazil and the U.S. are used to describe a product that represents a blending component in conventional



hydrocarbon based diesel fuel, while the European biodiesel standard describes a product that can be used either as a stand-alone diesel fuel or as a blending component in conventional hydrocarbon based diesel fuel.

It should also be noted that some specifications for biodiesel are feedstock neutral and some have been formulated around the locally available feedstocks. The diversity in these technical specifications is primarily related to the origin of the feedstock and the characteristics of the local markets. Though this currently translates into some significant divergence in specifications and properties of the derived fuels – which could be perceived as an impediment to trade – in most cases it is possible to meet the various regional specifications by blending the various types of biodiesel to the desired quality and specifications.

## **1.2.2 Summary and Analysis of Standards for Bioethanol**

The Bioethanol Tripartite Task Force considered relevant standards and specifications, documents on the parameters and methods, and commentaries on the similarities or differences of the specifications. It is important to note that in addition to conversion to common units, the U.S. denatured ethanol standard was converted to an undenatured basis so comparison could be made with the undenatured standards of the EU and Brazil.

### **1.2.2.1 General Observations for Bioethanol Standards**

The three current specifications have many similarities, largely due to the fact they all originate from a single (Brazilian) specification. Differences have been introduced as a result of the market developments, climatic conditions in each country and region, and feedstock variances. A significant difference among the three sets of standards is water content<sup>4</sup>, which is set at different

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<sup>4</sup> 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 unfavorable circumstances, such as in cold climatic conditions, a separation of the ethanol and water will occur (phase separation) 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. Since the EU has a wet logistics infrastructure the oil industry and vehicle constructors, in order to minimize the risk of such problems occurring, agreed with the ethanol producers to set a specification limit at a precautionary level. However, Brazil has been producing, processing, handling, distributing and blending fuel ethanol with gasoline at different levels for the past thirty years. During this period, the industry has not experienced any problem related to phase separation, considering different blending levels and climatic conditions under which ethanol is distributed in the country. Likewise, in the state of California USA, no problems have been associated with blending 5.7% vol ethanol with a 1 vol% water limit. While the requirement of a lower level of water does not prevent ethanol from being

levels primarily due to the varying ethanol concentrations permitted in gasoline and the gasoline distribution differences:

- The EU currently utilizes up to E5 and has the lowest limit of 0.24 vol %.
- The U.S. has the highest limit of 1.0 vol %.
- Brazil does not have maximum water content in its specifications, but levels are calculated to be a maximum of 0.4 vol% based on a minimum total alcohol content of 99.6 vol%.

For bioethanol, the Task Force concluded that there is no technical specification that constitutes an impediment to trade given the current situation. However, it is recognized that additional drying and testing will be required by Brazil and U.S. exporters wishing to supply the EU market. The impact and cost associated with these additional processes has not been evaluated by the Task Force.

**Table 2: Classification of the Various Bioethanol Specifications**

<b>Category similar<sup>5</sup></b>	<b>A Category significant differences</b>	<b>B Category fundamental differences</b>	<b>C</b>
color	ethanol content	water content	
appearance	acidity		
density	phosphorus content		
sulfate content	pHe		
sulfur content	gum / evaporation residue		
copper content	chloride content		
iron content			
sodium content			

exported, there are important economic implications for ethanol producers and exporters. The additional drying required increases the cost of production and can reduce productivity at the mill by up to 7%.

<sup>5</sup> This category also includes items that may not be similar at present but which the task force believes could be easily agreed upon. As an example: the U.S. does not currently have a limit on iron, sodium or electrical conductivity but does not believe adopting standards similar to those of the other regions would be problematic.

electrolytic conductivity		
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### **1.3 Benefits and Impact of Task Force Activities**

The Task Force members have collaboratively assembled a definitive and widely vetted list of Brazilian, EU and US standard specifications that are similar. In addition, they have identified a list of specifications that have significant, but alignable differences. Perhaps even more importantly, some indirect benefits have been derived. There is widespread agreement amongst the participating experts that the discussions and commitment to cross-border cooperation have been a major accomplishment that will support the increase in global trade of biofuels. The experts now have a better understanding of reasons why regional differences exist, and a new atmosphere of collegiality has been created – not only between countries but also between the private and public sector representatives. These positive outcomes foster a working environment that will support ongoing movement towards enhanced compatibility among the biofuels standards.

This report completes the commitment of work defined for the two Tripartite Task Forces in their charter (see Annex 2).

### **1.4 Recommendations**

The Task Forces Recommend that this report be disseminated to:

- standardization bodies of the tripartite agreement (i.e., ABNT, ANP, CEN and ASTM International) as a basis for ongoing discussions and cooperation that will promote alignment and mitigate divergence among evolving standards and specifications.

The Government Tripartite Leaders support this recommendation and further recommend that the report be disseminated to:

- other members of the International Biofuels Forum as a basis for ongoing discussions on more closely aligning their respective specifications and prioritizing future efforts for maximum impact.

The Government Tripartite Leaders request that Governments:

- affirm their support for the tripartite work as well as the work of the International Biofuels Forum;
- request that the standardization bodies of the Tripartite Agreement (re)nominate appropriate Tripartite Task Force members to progress the work being defined in the update of the Biofuels Standards Roadmap;
- request the standardisation bodies of the Tripartite Agreement to analyse the Task Forces reports and to consider adapting existing national standards wherever appropriate. Furthermore the standardisation bodies should attempt where possible, when developing and updating their standards on biodiesel and bioethanol from now on to consider the opportunity to align with the other standards in question;
- support efforts to initiate an analysis of the categorized specifications to study trade implications and appropriate next steps for harmonization;
- provide the necessary financial and other resources to support Task Force members and others identified to carry out the work proposed; and,
- support the development of internationally-accepted reference methods and certified reference materials for improving the accuracy of measurement results that underpin assessment of product quality, and help facilitate trade.

## **1.5 Plans for Future Work on Biofuels Standards in a Global Market**

### **1.5.1 Update of the Biofuels Standards Roadmap**

The continued development of the Biofuels Standards Roadmap is proposed, and work will begin in the first quarter of 2008. Due to the very limited timeframe in 2007, it was not possible to either recommend proposed specification values or complete the analyses of specifications as cited in the original Biofuels Standards Roadmap (see Annex 1). Therefore, it is recommended that this effort continue in 2008, and the Roadmap be further refined and detailed. These efforts will be focused primarily on the specifications in Category B – specifications that could be aligned – and should include such considerations as cost and impact of alignment /non-alignment

of specifications in a global market, and identification of financial and other resources required to accomplish this task.

### **1.5.2 Expanded engagement of the IBF**

At the quarterly IBF meeting on March 3, 2008 hosted at the Brazilian Embassy in Washington DC, the government leaders of the Tripartite Agreement will:

- provide a briefing to other members of the IBF on the completed work of the Tripartite Task Forces
- organize a special session with appropriate representation of experts to discuss trade and cost implications of further alignment of specifications
- recommend continued commitment from the IBF countries to further support the standards work begun in 2007 by Brazil, EU and U.S.

### **1.5.3 Continued engagement of technical experts in biofuels standards**

The government leaders of the Tripartite Agreement recommend that an update to the Biofuels Standards Roadmap be developed by technical experts to:

- identify those specifications that should be addressed by December 2008;
- identify those specifications for which a longer period of time may be needed to carry out additional testing to develop required performance data to justify differences or changes in specifications; and
- specify the financial resources necessary to support the ongoing work of the Task Forces

### **1.5.4 Timeline for Immediate Next Steps**

- A special session will be held at the IBF meeting in March 2008.

- The Biofuels Standards Roadmap Update is expected to be finalized by April 2008.
- The Trade Implication Analysis is expected to begin in 2008 and continue in 2009.

## 1.6 Conclusions

At present, bioethanol specifications are more closely aligned amongst the three regions than biodiesel specifications. Key factors influencing issues of alignment include the following:

- Bioethanol is a single chemical compound, whereas biodiesel is not a single chemical entity, but is derived from several types of feedstocks that can translate to variations in the performance characteristics of the finished fuel.
- Biodiesel, both FAME and FAEE, are two chemically different mixtures making it a challenge to develop a common standard that can address the complex fuel and engine requirements.

In spite of these challenges the biofuels industry on both sides of the Atlantic has found tools to enable the international trade global of biofuel products, however a full analysis of the costs was not in the scope of the work reported here. Further alignment of existing standards and specifications where necessary from both a technical and trade perspective will help establish *Internationally Compatible Standards for Biofuels* that should facilitate trade, improve efficiency of biofuels production, and promote innovative energy resources, and associated economic security.

**Antonio Simoes and Claudia Vieira Santos**, Ministry of Foreign Affairs of Brazil

**Kyriakos Maniatis**, Directorate General for Energy and Transport, European Commission

**Willie May and Ellyn Beary**, National Institute of Standards and Technology, USA

The tripartite partners are indebted to Dr Hrach Semerjian, who began this work on behalf of the US government at the meeting in Brussels, and was a key contributor to crafting the Tripartite Agreement in Washington DC.

31 December 2007

## Annex 1

### **Proposal outline for a road-map to develop commonly agreed international standards for biofuels**

#### **1. Background**

The International Conference on biofuel standards was attended also by missions from the USA, Brazil and several other countries. There was also strong industrial participation from the car and oil industries. On the first day, 28.02.07, the agenda covered presentations and extensive discussions of general policies on biofuels with main emphasis the EU and the US. On the second day there were two parallel workshops each on Bioethanol and Biodiesel.

#### **2. Main Conclusions from the Conference**

The conclusions of the Conference identified the following points:

1. It was agreed that International Standards are needed to facilitate and promote biofuels trade.
2. Any International Standard on biofuels should focus on the pure biofuel component and not blends with either petrol or diesel; these are based on national legislation and are out of the objectives of the Conference.
3. The work shall consider only biodiesel and bioethanol; second generation biomass to liquid (BTL) biofuels are still in the very early stages of development and should not be dealt with at present.
4. The existing process of an ISO procedure for International Standards is too lengthy and sometimes inconclusive. We can not rely on this.
5. The resulting International Standards from an accelerated process will need to be adopted by the various agencies (CEN, ASTM, NIST, ISO, Brazilian standardisation bodies) at a later stage via normal procedures.
6. The existing national standards have several differences when compared to each other, however, after careful examination it was concluded that these differences were not insurmountable and a lot of common ground already exists.
7. In the process for developing the International Standards for biofuels it will be critical to involve Brazil from the inception stage and others (Japan, India, etc) at a later stage. This is to ensure that the working group will be relative small and could work efficiently.

#### **3. Proposal for a Plan for Action**

3.1 It is recommended to establish a working group at two levels, a policy one with representatives from the European Commission (DG TREN), US Government (DoS & DoC) and the government of Brazil; and a technical one with representatives from CEN and ASTM + NIST and the Brazilian standardisation bodies. Each level has a



representative from the EU, US and Brazil to lead the working group. The management of the working group is carried out by this team of 6 persons and will be called the Management Team (MT).

The technical working group shall consist of two subgroups, each for bioethanol and biodiesel and with 5 experts representing the standardisation institutes. Therefore each technical group shall have 15 experts.

3.2 The mandate of the working group shall be to commonly develop a proposal for two compatible International Standards; one for Bioethanol and the other for Biodiesel.

3.3 The proposals for the compatible International Standards for Bioethanol and Biodiesel should be ready by the end of 2007.

3.4 It is recommended that in parallel, either one or both standardisation bodies request ISO to start the procedure for establishing the appropriate ISO committees and/or working groups and advises ISO that the proposal for International Standards for Bioethanol and Biodiesel will be submitted to ISO by the end of 2007.

3.5 Irrespectively of the ISO process, it is recommended that also CEN and ASTM should adopt the International Standards for biofuels.

#### **4. Road Map**

<b>Action</b>	<b>Time</b>
Nomination of the Management Team	March 15
The two technical persons from the MT draft a list of problems that need to be addressed for both biofuels	March 30
Nomination of the Working group	April 15
1 <sup>st</sup> meeting of the Working Group, Ongoing Conference in the US	June 27
2 <sup>nd</sup> meeting of the Working Group, Ongoing Conference on Biofuels in Brussels	July 31
3 <sup>rd</sup> meeting of the Working Group, 2 <sup>nd</sup> Conference on Standards, to be announced in the US	Fall 07
Final proposal for international standards	December 07

Brussels, 28 March 2007

## **Annex 2**

### **Task Forces on Bioethanol and Biodiesel**

The aim of the Bioethanol and Biodiesel Task Forces is to review existing documentary standards and identify areas where greater compatibility can be achieved in the short and long term.

#### **Objectives**

The objective of the Task Forces is to collect and analyse existing standards on pure bioethanol and biodiesel developed by ABNT, ASTM, and CEN and prepare a White Paper that identifies:

- a) a list of specifications<sup>1</sup> that are very similar or for which an agreement can be reached easily, and recommend proposed values;
- b) a list of specifications<sup>1</sup> that have differences in their values but are considered to be bridgeable after extensive discussions and exchange of information amongst the experts; the need for additional performance data and testing that may be required to justify differences or changes in specifications. The task force will either recommend proposed values for those specifications for which an agreement was reached, or recommend an action plan needed to reach an agreement.
- c) a list of specifications<sup>1</sup> that are either covered by law or other regulations, or those specifications that are too different to be deemed bridgeable in the foreseeable future.

#### **Membership**

The members of the Task Forces are nominated by ABNT, ASTM, and CEN but they act on their own personal capacity.

Representatives from China, India and South Africa, members of the International Biofuels Forum (IBF), will be invited to contribute through the IBF.

#### **Follow up**

The Task Force plans to submit the White Paper to the Brazilian, EC and US authorities by 31 December 2007. The White Paper will also be transmitted to the International Biofuels Forum.

After the submission of the White Paper, the members of the Task Force, subject to the approval of the US, Brazil and EC authorities, will decide whether it serves any purpose to continue the operation of the Task Force.

<sup>1</sup> Specifications refer to individual parameters, their values and related test methods

## Issues addressed

1. Nomination of Brazil, EC and US representation.  
**These are Mr. Simões, Mr. Maniatis and Mr. Semerjian,**
2. Nomination of Brazil, EU and US Task Force Leaders for Ethanol Task Force (ETF) and Biodiesel Task Force (BTF).  
**US and Brazil Task Force leaders and their experts (max 5) will be nominated by July 15<sup>th</sup> 2007 through their representatives; EU has already submitted their nominations.**
3. Nomination of Brazil, EU and US Task Force members for ETF & BTF.  
**See (2) above**
4. Need to decide who holds the pen per Task force.  
**Each Task Force will decide on the provision of the secretariat. It may be provided by the standardisation organisations involved.**
5. Principle of cooperation will be “consensus” by all parties.  
**Agreed.**
6. Each TF should address the issue of how to present the result/outcome to the standardisation organisations. This has to be done simultaneously.  
**Agreed.**
7. Each government representative should address the issue of how to present the result/outcome to its administration.  
**Agreed**
8. The ASTM paper, which compares existing standards, which was shared with other organizations will be used as a starting point for the discussions.

Washington DC, 2007.06.29

## **2 BIODIESEL TASK FORCE REPORT**

This report has been prepared by the designated Biodiesel Taskforce Leaders from Brazil, the European Union and the United States of America, namely

Mr. M Cabral – Brazil  
Mr. G F Cahill – European Union  
Mr. S Howell – United States of America

and assisted by their regional expert members of the Taskforce (Annex 1).

21 December 2007

## 2.1 **Summary for the Biodiesel Task Force**

This report sets out the findings of the task force that studied the case of biodiesel.

The purpose of this document is to advise the International Biofuels Forum on the status of alignment of the standards being used in Brazil, the European Union and the United States of America for biodiesel. The document informs the Governments of those parameters in the standards that are already similar, of those where significant differences between parameters and methods exist but which might be aligned by work on the products standards and methods, and of those where there are fundamental differences of the standards which are not deemed bridgeable in the foreseeable future. The report attempts to identify the extent of the work that would be needed to bring closer alignment between the standards,

The biodiesel task force, began the study by the circulation of standards, documents on the parameters and methods, and commentaries on the similarities or otherwise of the specifications. Estimates of the difficulties or ease of alignment were made. Two face-to-face meetings between the members of the teams took place in November and December 2007 to discuss in detail the information circulated as part of the study. The findings of the teams are:

Some existing specifications for biodiesel have been formulated mainly around the locally available feedstocks, and the diversity of feedstocks is translated into some significant divergences in specification properties of the derived fuels

Some existing specifications are based on use as a blend stock or extender for fossil based diesel fuel, rather than use as a 100% fuel for engines. This has resulted in some significant divergences in the specification properties of the pure biodiesel.

Measurement methods, while using similar techniques in many cases, employ procedures that are not aligned and so complicate the comparison of limit values of the three standards.

The categories A,B,C in the table below are described in the report. They indicate the estimated ease or difficulty of achieving a technical alignment of the parameter, method and limit value in question (A= easily done, B= feasible with effort, C= not feasible at present). The 'Misalignment Impact' (identified as 'none', 'minor', 'medium', or 'major') is an estimate of importance of the differences in the specifications as they relate to the exchange of biodiesel between the regions.

This table is a summary of Task Force comparisons on existing alignments (categories A, B, C) and the impact of misalignment. Parameter allocations are BR / EU / USA respectively.

<b>Category A Parameters Misalignment Impact (MI)</b>	<b>Category B Parameters Misalignment Impact (MI)</b>	<b>Category C Parameters Misalignment Impact (MI)</b>
Sulfated ash (A / A / A) MI: very minor	Total glycerol (A / A / A) for limit value (B / B / B) for method MI: minor	Sulfur content (C / C / C) MI: medium to major
Alkali & alkaline earth metals (A / A / A) MI: very minor	Phosphorus content (A / B / A) MI: medium	Cold climate operability (C / C / C) MI: very minor
Free glycerol (A / B / A) MI: minor	Carbon residue (B / B / B) MI: very minor	Cetane number (C / C / C) MI: major
Copper strip corrosion (A / A / B) MI: none	Ester content (B / B / B) MI: very minor	Oxidation stability (B / C / C) MI: medium
Methanol & ethanol content (A / A / A) MI: medium	Distillation temperature (B / B / B) MI: very minor	Mono, di-, tri-acylglycerides (B / B / C) MI: minor
Acid number (A / B / A) MI: very minor	Flash point (B / B / B) MI: minor	Density (C / C / C) MI: very minor
	Total contamination (B / B / B) MI: minor	Kinematic viscosity (C / C / C) MI: very minor
	Water content & sediment (B / B / B) MI: medium/major	Iodine number (A / C / A) MI: major
		Linolenic acid (A / C / A) MI: major
		Polyunsaturated methyl ester (C / C / C) MI: major

## **2.2 Recent, Current and Planned Activity of each Region**

### **2.2.1 Brazil**

The present Brazilian biodiesel specification (Resolution ANP n° 42/04), released to support the preliminary activities of the National Biodiesel Programme, was elaborated taking into account the wide variety of feedstocks expected to be used in Brazil, as well as the existing international experience and specifications (ASTM D6751 and EN 14214). Several properties listed in the provisional Brazilian specification still do not have established limits, but must have values reported. Others have more flexible limits, to accommodate feedstock diversity.

The Biodiesel Programme started with a mandatory blend of 2% biodiesel, applicable from 01 January 2008, and to be increased to 5% in 2013. In the last two years, some tests have been developed to evaluate the suitability of B5 and B20 blends for use in diesel vehicles. The aim is to validate the gradual increase in biodiesel proportion in diesel blending through engine bench tests and durability.

Moreover, discussions have been underway along this year and a new biodiesel specification proposal is under public inquiry. This new specification establishes limits for many properties, which previously only had to have values reported. A new specification must be issued by ANP by March 2008.

### **2.2.2 European Union**

The European standard for Fatty Acid Methyl Esters (FAME) to be used as automotive fuel was set in 2003 by the Comité Européen de Normalisation (CEN) and is known under the standard number EN 14214. This standard sets limits and measurement methods for FAME, generally known as biodiesel that may be used either as a stand-alone fuel or as a blending component in European diesel fuel. The CEN standard for diesel fuel, EN 590, requires that all biodiesel blended in the fuel must conform to the standard EN 14214.

At present, the European diesel fuel allows biodiesel to be blended at up to and including 5% by volume. Some national standards in EU countries allow biodiesel to be distributed as a stand-alone fuel, notably in Germany, for specially adapted vehicles.

The CEN is presently studying a revised EN 590 specification for diesel fuel that will permit up to and including 7% of biodiesel blend. Simultaneously CEN is studying a revision of the biodiesel standard EN 14214 with a view to widening the range of feedstock oils that may be used, without compromising the security of vehicles using this product either in blends or as a stand-alone fuel. At the same time the European Commission has mandated CEN to revise the EN 590 specification for diesel fuel up to 10% of biodiesel blend.

### **2.2.3 United States of America**

The United States of America (US) has chosen to use the specifications developed by ASTM International (ASTM) for both conventional diesel fuel and biodiesel. Specification efforts for biodiesel in the US began in 1993 in Committee D02 on

Petroleum Products and Lubricants. While the initial proposal for the biodiesel specifications at ASTM was for B100 as a stand alone fuel, experience of the fuel in-use with blends above B20 was insufficient to provide the technical data needed to secure approval from the ASTM members. Based on this, after 1994 biodiesel efforts within ASTM were focused on defining the properties for pure biodiesel which would provide a ‘fit for purpose’ fuel for use in existing diesel engines at the B20 level (20% biodiesel with 80% conventional diesel) or lower.

A provisional specification for B100 as a blend stock was approved by ASTM in 1999, and the first full specification was approved in 2001 and released for use in 2002 as “ASTM D6751 Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels”. The philosophy used to approve D6751 was the same as that used for the No. 1 and No. 2 grades of fuels within the conventional specification, ASTM D975: If the parent fuels meet their respective specifications then the two can be blended in any percentage and used in conventional diesel engines. No separate set of properties was needed for the finished blends of No. 1 and No. 2, if the parent fuels met their respective specifications. These same conditions hold true for biodiesel; if biodiesel meets D6751 and conventional diesel meets D975 the two can be blended and used in conventional engines with the restriction of the upper limit of 20% biodiesel content in the finished fuel.

While this mode of operation has served the US market well, there has been substantial effort since 2003 to develop and formally approve specifications for the finished blend of biodiesel and conventional diesel fuel. In addition, several improvements and changes to D6751 were also undertaken, some as a result of changes needed to secure approval of the finished blended biodiesel specifications. At the time of this report ballots to allow the formal acceptance of up to 5% biodiesel (B5) into the conventional diesel specifications for on/off road diesel fuel (ASTM D975) and fuel oil burning equipment (ASTM D396) and a new stand alone specification covering biodiesel blends between 6% and 20% have been approved through the Subcommittee level of Committee D02. A main committee D02 vote is expected in June of 2008. In addition, a ballot to implement a new parameter in D6751 to control the potential for filter clogging above the cloud point in B20 blends and lower has also passed the subcommittee and is on track for a June 2008 vote. Efforts to approve B100 and B99 as stand alone fuels have been discussed at ASTM, but have been put on hold in order to focus on the B5 and B6 to B20 blended fuel specification efforts.

## **2.3 Definitions and Technical Notes**

### **2.3.1 Definitions:**

For the purposes of the work of this task force, biodiesel is defined as mono-alkyl esters of long chain fatty acids derived from plant oils or animal fats and used, for example, as fuel for compression ignition, internal combustion piston engines. This definition is consistent with the explanatory note to heading 38.24 of the World Customs Organization Harmonized System.



The comparisons of Brazilian, EU and USA are made on the standards in place at the end of the year 2007. The standards are frequently reviewed and updated due to the rapidly evolving knowledge of these fuels and care should be taken to ensure the correct up to date standards are consulted past the date of this report.

The current standards for biodiesel in Brazil and the United States of America are applicable for both fatty acid methyl esters (FAME) and fatty acid ethyl esters (FAEE), whereas the current European Union biodiesel standard is only applicable for fatty acid methyl esters (FAME).

The standards of biodiesel in Brazil and the United States of America are used to describe a product that represents a blending component in conventional hydrocarbon based diesel fuel, whereas the European Union biodiesel standard describes a product that can be used either as a stand-alone diesel fuel or as a blending component in conventional hydrocarbon based diesel fuel.

### **2.3.2 Effort and Resources to Align**

The work to align the measurement methods and limit values of the parameters will require an investment in time and effort by specialists in laboratories, test facilities, and private companies. Assessment of these costs and who shall bear them remains to be established, but the task force leaders and members have assigned initial appraisals for the efforts which must be undertaken by their respective standard bodies in order to become aligned under the terms “minor”, “medium” or “major”. For some parameters, additional detail is provided on the efforts needed for alignment.

In addition to the time and effort by specialists, some efforts to align may also require investment in testing and research that must be contracted to outside parties or organizations. The task force leaders and members have assigned initial appraisals for of the outside resources which must be made available in order to accomplish alignment under the terms “none”, “minor”, “medium” or “major”.

### **2.3.3 Impact of non-alignment**

While some methods, test parameters, or parameter limit values are not currently aligned, their non-alignment may not have much of an impact if biodiesel made in one region is destined for use in another region. A preliminary assessment of the impact of the differences in the standard is also provided under the terms “none”, “very minor”, “minor”, “medium”, or “major”.

### **2.3.4 Status of comparisons:**

The comparisons of Brazilian, EU and USA are made on the standards in place at the end of the year 2007. The standards are frequently reviewed and updated due to the rapidly evolving knowledge of these fuels and care should be taken to ensure the correct up to date standards are consulted past the date of this report.

### **2.3.5 Use of biodiesel standards:**

The standards of biodiesel in Brazil and the United States of America are used to describe a product that represents a blending component in conventional hydrocarbon based diesel fuel, whereas the European Union biodiesel standard describes a product that can be used either as a stand-alone diesel fuel or as a blending component in conventional hydrocarbon based diesel fuel.

#### **2.4 Description of Procedures**

The initial technical step for this work was taken by the task force experts from the United States of America in a document circulated in June 2007. It listed the parameters appearing in the national specifications of Brazil, of the EU and of the USA and gave the notations 1, 2 and 3 similar to the A) B) and C) above.

The Brazilian and EU Taskforce experts then distributed documents that expanded on the basis of the initial US document, and the combined opinions are shown below in the commentaries for each parameter. Subsequent face-to-face meetings resulted in the opinions and commentaries presented below. A summary table of these parameters and their categories as decided by the task force members may be found in Annex 2.

#### **2.5 Parameters, Test methods & Limit Values Used in the Standardisation of Biodiesel**

This section of the report describes the parameters, measurement methods and limit values of the specifications used in the standards of the three regions participating in this phase of the tri-partite activity. The reason for each parameter is briefly explained, the methods used to measure it are noted, and the limit value retained by each region is given where appropriate. The commentaries made by each team of experts during the comparison exercise are included, and as a result of these commentaries a category of the potential for alignment is assigned.

## 2.5.1 Specifications for which an agreement may be easily achievable

### 2.5.1.1 Sulfated Ash

Ash content describes the amount of inorganic contaminants such as abrasive solids and catalyst residues, and the concentration of soluble metal soaps contained in the fuel. These compounds are oxidised during the combustion process to form ash, which is connected with engine deposits and filter plugging (Mittelbach, 1996). For these reasons sulfated ash is limited in the fuel specifications.

#### Limits and Methods:

Brazil	Limit: 0.02 % m/m max	Method: ABNT NBR 984/EN ISO 3987/ D874*
EU	Limit: 0.02 % m/m max	Method: EN ISO 3987
USA	Limit: 0.020 % m/m max	Method: ASTM D874

#### Commentary:

**Brazil cat. A)** The limit values for Brazil, the EU and the USA are aligned, except for the number of decimal places, which should be discussed. \* When more than one test method is mentioned the Brazilian specification, any one of such methods can be used alternatively.

**EU cat. A)** The limit values of the three regions are aligned.

Some difficulty may occur later if the CEN standard reduces the limit value at the request of vehicle constructors to diminish risks of diesel particulate filter clogging.

**USA cat. A)** Agree with Brazil. Regarding Test Methods, D874 and EN ISO 3987 are equivalent according to the Guide to ASTM Test Methods for the Analysis of Petroleum Products and Lubricants, 2<sup>nd</sup> Edition but a separate comparison of the procedures was not completed. However, both are for lubes and lube additives, so not optimized for FAME. Both report to 0.001 % mass. R and r are so close that the differences are immaterial. Similar discussion with Free Glycerol regarding significant digits.

During discussions, the second significant digit on the limit value appears to be the only obstacle to alignment. (US will refer to cross-check programme for data). BR and EU will consider modifying the limit value to incorporate the additional significant digit.

**Effort, Resources to Align:** Medium, Medium

#### Actions needed for harmonization:

Brazil to adjust limit value to second significant number

EU to adjust limit value to second significant number

More detailed review of the specific test methods used by each region

**Impact of Non-Alignment:** Very minor. It may be possible for a batch to measure 0.023% and be considered 'in spec' in Europe and Brazil and out of spec in the USA but

most commercial values are well below 0.02 so this occurrence is not expected to happen often.

### 2.5.1.2 Alkali and Alkaline Earth Metals

Metal ions are introduced into the biodiesel fuel during the production process. Whereas alkali metals stem from catalyst residues, alkaline-earth metals may originate from hard washing water. Sodium and potassium are associated with the formation of ash within the engine, calcium soaps are responsible for injection pump sticking (Mittelbach 2000). These compounds are partially limited by the sulphated ash, however tighter controls are needed for vehicles with particulate traps. For this reason these substances are limited in the fuel specifications.

#### Limits and Methods:

##### Group I Metals (Na + K)

Brazil	Limit: 10 mg/kg max	Method: EN 14108/14109
EU	Limit: 5 mg/kg max	Method: EN 14108-14109
USA	Limit: 5 mg/kg max	Method: EN 14538

##### Group II Metals (Ca + Mg)

Brazil	Limit: Report	Method: EN 14538
EU	Limit: 5 mg/kg max	Method: EN 14538
USA	Limit: 5 mg/kg max	Method: EN 14538

#### Commentary:

Brazil cat. A) The adoption of the 5 mg/kg for both limits is being discussed in Brazil. We will probably be able to reach an alignment in this parameter.

EU cat. A) The USA and EU limits are aligned; it remains to be seen if Brazil could agree to align on these values.

USA cat. A) In discussion, some methods changes in the EU are being studied for the Group I metals, thought these changes will not be applicable at the time of this report. Brazil asked for data on the Group II metals to help decide a Brazilian limit aligned with the EU and USA.

During discussions, the Brazilian experts considered that they could probably align their specification with the EU / USA values. An ICP method is being developed in Brazil, and such a method is presently being balloted in the EU.

**Effort, Resources to align:** Minor, None

#### **Actions needed for harmonization:**

More detailed review of the specific test methods used by each region.

**Impact of Non-Alignment:** Minor

Values should be below 5 ppm most of the time.

### 2.5.1.3 Free glycerol

The content of free glycerol in fatty acid methyl ester (biodiesel) is dependent on the production process, and high values may stem from insufficient separation or washing of the ester product. The glycerol may separate in storage once its solvent methanol has evaporated. Free glycerol separates from the biodiesel and falls to the bottom of the storage or vehicle fuel tank, attracting other polar components such as water, monoglycerides and soaps. These can lodge in the vehicle fuel filter and can result in damage to the vehicle fuel injection system (Mittelbach 1996). High free glycerol levels can also cause injector coking. For these reasons free glycerol is limited in the specifications.

#### Limits and Methods:

Brazil	Limit: 0.02 % m/m max	Method: ABNT NBR 15341/EN 14105/14106
EU	Limit: 0.02 % m/m max	Method: EN 14105/14106
USA	Limit: 0.020 % m/m max	Method: ASTM D6584

#### Commentary:

**Brazil cat. A)** The limit values for Brazil, the EU and the USA are aligned, except for the number of decimal places, which should be discussed.

**EU cat. B)** The limit values for Brazil, the EU and the USA are practically aligned. The terminology difference between Brazil/USA and EU (glycerin and glycerol) refers in fact to the same substance.

**USA cat. A)** Agree with Brazil. The significant digits does impact the meaning of the specification, (i.e. 0.02 limit allows 0.024 where 0.020 limit would not). USA changing to 0.02 would essentially expand the limit, and significant data would be needed in order to justify this within ASTM.

In discussion, Brazil indicated it was working on a new method that will accommodate castor oil, with precision to match the ASTM method. The EU could examine the possibility of moving to the second significant decimal place but will require work to ensure the precision of the method is sufficient.

**Effort, Resources to align:** Minor, Minor

#### Actions needed for harmonization:

Brazilian method to be developed

EU limit value to be adjusted

More detailed review of the specific test methods used by each region

**Impact of Non-Alignment:** Minor

#### 2.5.1.4 Copper strip corrosion

This parameter characterizes the tendency of a fuel to cause corrosion to copper, zinc and bronze parts of the engine and the storage tank. A copper strip is heated to 50°C in a fuel bath for three hours, and then compared to standard strips to determine the degree of corrosion. This corrosion resulting from biodiesel might be induced by some sulfur compounds and by acids, so this parameter is correlated with acid number.

Some experts consider that this parameter does not provide a useful description of the quality of the fuel, as the results are unlikely to give ratings higher than class 1.

##### Limits and Methods

Brazil	Limit: class 1	Method: ABNT NBR 14539/EN ISO 2160/ASTM D130
EU	Limit: class 1	Method: EN ISO 2160
USA	Limit: class3	Method: ASTM D130

##### Commentary:

**Brazil cat. A)** A harmonised standard should either adopt class 1 or eliminate this parameter, which is seen as unnecessary by some experts.

**EU cat. A)** The EU experts generally feel that this parameter does not give useful information on the quality of biodiesel, as the copper strip is usually cleaned by the fuel and the results are always class 1 (CEN method). The parameter could be removed, as vehicles no longer have copper elements in their fuel systems. A steel strip corrosion test would be more realistic for the present fuel systems.

However a concern was mentioned regarding copper being present in home heating systems, if biodiesel should be present there. To be discussed.

**USA cat B)** The US experts agree with Brazil and the EU in general. Most values are always in Class1. Heating oil systems do contain copper in many uses, however, and heating oil experts at ASTM disagree that the tarnish sometimes found in this test is cleaned by biodiesel or other solvents. Class 3 in D6751 is a read-across from D975, while Class 1 is a read-across from EN 590. ASTM could not change to Class 1 without data showing Class 1 is required for engines or for heating systems. Before ASTM dropped the requirement, more data may be needed to show that all FAME really does meet at least Class 2 and discussion with ASTM heating oil section would be needed.

In discussion, the US experts agreed to check with the heating oil experts, but could support elimination of the test as well as the Brazilian and EU experts. Replacing the parameter with steel strip corrosion is not a viable alternative. While the governmental representatives indicated that the IBF Tri-Partite efforts need not take into account harmonization for heating oils, this may still be a major consideration within ASTM for D 6751 use as D 6751 is used for biodiesel for all applications.

**Effort, Resources to align:** Medium, Medium

##### Actions needed for harmonisation:

Brazil: discuss option for removal of this parameter

EU: confirm orientation for removal of this parameter

USA: gather data to justify removal of this parameter  
More detailed review of the specific test methods used by each region  
Impact of Non-Alignment: None

### 2.5.1.5 Methanol or Ethanol content

Methanol (MeOH) or ethanol (EtOH) can cause fuel system corrosion, low lubricity, adverse affects on injectors due to its high volatility, and is harmful to some materials in fuel distribution and vehicle fuel systems. Both methanol and ethanol affect the flash point of esters. For these reasons, methanol and ethanol are controlled in the specification.

#### Limits and Methods:

Brazil	Limit: 0.50 % m/m	Method: ABNT NBR 15343/EN 14110
EU	Limit: 0.20 % m/m	Method: EN 14110
USA	Limit: 0.2 % m/m (MeOH)	Method EN 14110
	Limit: 130° C flash point (EtOH or if MeOH not measured by EN 14110)	

#### Commentary:

**Brazil** cat. A) We believe it will be possible to adopt the limit of 0.20% mass in the harmonised specification. Since we favour the development of this specification for both FAME and FAEE, we believe this limit should be applicable to methanol and ethanol content.

It should be noted, however, that there is limited experience with the production of FAEE, and therefore with the application of this limit to ethanol content. We would like to discuss this issue with our European and American colleagues.

**EU** cat. A) USA and EU are aligned, the Brazilian limit is higher. Methanol is aggressive to the fuel injection system materials, so should be limited. It is hoped that the Brazilian limit could be aligned with the specification of the other two regions.

**USA** cat. A) ASTM limits is either 0.2 alcohol or 130 C minimum flash. Current alcohol method only applicable to methanol, and significant digit may be an issue. Method needs to be updated for ethanol so flash point only existing option for ethanol in ASTM at present. Work on ethanol content test is underway. If Brazil can harmonize on 0.2, then this can be an A at least for methanol content. Ethanol content needs a new test method and should be a B as this will take some time.

In discussion, the US could consider adding a significant digit to the limit value to align with BR/EU. A new method needs to be developed for ethanol content.

**Effort, Resources to align:** Medium, Medium

**Impact of Non-Alignment:** Minor

### 2.5.1.6 Acid number

Acid number or neutralisation number is a measure of free fatty acids contained in a fresh fuel sample and of free fatty acids and acids from degradation in aged samples. If mineral acids are used in the production process, their presence as acids in the finished fuels is also measured with the acid number. It is expressed in mg KOH required to neutralise 1g of FAME. It is influenced on the one hand by the type of feedstock used for fuel production and its degree of refinement. Acidity can on the other hand be generated during the production process. The parameter characterises the degree of fuel ageing during storage, as it increases gradually due to degradation of biodiesel. High fuel acidity has been discussed in the context of corrosion and the formation of deposits within the engine which is why it is limited in the biodiesel specifications of the three regions. It has been shown that free fatty acids as weak carboxylic acids pose far lower risks than strong mineral acids (Cvengros 1998)

#### Limits and Methods:

Brazil	Limit: 0.80 mg KOH/g max	Method: ABNT NBR 14448/EN 14104
EU	Limit: 0.50 mg KOH/g max	Method: EN 14104
USA	Limit: 0.50 mg KOH/g max	Method: ASTM D664

#### Commentary:

**Brazil cat. A)** We believe it will be possible to adopt the limit of 0.50 mg KOH/g in the harmonised specification.

**EU cat.B)** The EU experts consider this parameter of high importance to protect the materials of injection systems. Discussion is needed to see if our Brazilian colleagues can agree to the EU/USA limit value.

**USA cat. A for limit value, B for method)** The US experts believe this is in Category B as the test methods are not equivalent.

	ASTM D664	EN 14104
Scope	Petroleum products and lube	FAME
Method	Dissolve sample in a mixture of toluene, propan-2-ol and water. Titrate with alcoholic KOH to the end point of a glass indicating electrode.	Dissolve sample in diethyl ether and ethanol. Titrate with KOH in ethanol to a phenolphthalein end point.
Report	To 3 significant figures	To 3 significant figures
r	0.07 at 0.5	0.02
R	0.21 at 0.5	0.06



The AOCS Biodiesel Expert Panel is working on an Acid Number method comparison study, which may be useful.

**Effort, Resources to align:** Medium, Medium to Major

**Actions needed to harmonize:**

A round robin with the analytical method should be designed that covers the range of product from various feedstocks and processes available around the world. Product should meet all other pertinent specifications.

Once method harmonized, a fuel survey should be conducted in each region using the agreed method to determine status of current product in the market vs. the specification

If no issues are apparent, the new method may be balloted in each region's specification.

**Impact of Non-Alignment:** minor

## 2.5.2 Specifications for which an agreement may be eventually achieved after additional common work

### 2.5.2.1 Total Glycerol

Total glycerol is the sum of the concentrations of free glycerol and glycerol bound in the form of mono-, di- and triglycerides. The concentration depends on the production process. Fuels out of specifications with respect to these parameters are prone to coking and may thus cause the formation of deposits on injector nozzles, pistons and valves (Mittelbach et al. 1983). For this reason total glycerol is limited in the specifications of the three regions.

#### Limits and Methods:

Brazil Limit: 0.38 % m/m Method: ABNT NBR 15344/EN 14105/ASTM D6584

EU Limit: 0.25 % m/m Method: EN 14105

USA Limit: 0.24 % m/m Method: ASTM D6584

#### Commentary:

**Brazil cat. A) (B for methods)** The limit values for Brazil, the EU and the USA could be aligned, considering the proposal that is under discussion in Brazil. The standard methods defined by EN 14105 and ASTM D 6584 are gas chromatographic methods. In order to transform glycerol as well as mono- and diacylglycerols into more volatile compounds, the free hydroxyl groups of the sample are derivatized prior to the analysis. This procedure is not suitable for castor oil due to the presence of hydroxyl groups in the main carbon chain of the ester. Therefore, a new method for this purpose has been developed in Brazil (ABNT NBR 15344).

**EU cat. A) (B for methods)** The EU and USA are essentially aligned, and do not understand the Brazilian limit value as we feel good biodiesel production technologies can meet the EU/USA limit comfortably. It may be possible to remove this parameter if agreement is reached on mono-, di- and triglycerides. Discussion is needed with our Brazilian colleagues on this point.

**USA cat. A) (B for methods)** We consider this in category A for limit values, B for methods. The ASTM and EN methods both use different calculations and give different results. The methods need to be harmonized in order to compare results and this will take some time. More discussion is needed on this.

**Effort, Resources to align:** Medium, Medium

#### Actions needed to harmonise:

Work is needed on method alignment before discussing limit values.  
More detailed review of the specific test methods used by each region

**Impact of Non-Alignment:** Minor

### 2.5.2.2 Phosphorus content

Phosphorus in FAME stems from phospholipids (animal and vegetable material) and inorganic salts (used frying oil) contained in the feedstock. Phosphorus has a strongly negative impact on the long term activity of exhaust emission catalytic systems and for this reason its presence in biodiesel is limited by specification.

#### Limits and Methods:

Brazil	Limit: 10 mg/kg (Report)	Method: EN 14107/ASTM D4951
EU	Limit: 10.0 mg/kg max.	Method: EN 14107
USA	Limit: 10 mg/kg max	Method: ASTM D4951

#### Commentary:

**Brazil cat. A)** EU and US specified the maximum content of phosphorus in biodiesel samples to 10 mg/kg, whereas in Brazil this property is defined as “to report”. The adoption of the limit of 10 mg/kg is being discussed and could be aligned to other regions.

**EU cat. B)** The CEN specification will shortly be changed to limit phosphorus to 4 mg/kg. This is the result of a request from vehicle manufacturers who fear the long term negative effects of phosphorus on the emissions treatment systems. The EU producers of biodiesel can agree to this new limit, and the test method has been validated to measure this value. It remains to be seen if our American and Brazilian colleagues can follow this lead, as the increasingly stringent vehicle emission requirements require lower fuel phosphorus content levels.

**USA cat. A)** If Brazil can align with 10, then at present we appear aligned except for the significant digits. Similar discussion as other parameters for significant digits, only in this case going to more significant digits may be problematic in ASTM as data would be needed to justify the tightening of the requirement from 10 to 4 ppm max. The analytical method may not allow this level of precision in a specification according to ASTM guidelines. If the limit is considered for lowering to 4 ppm (EU position) significantly more data would be needed from OEM’s justifying the need and from samples in the field to determine the level of product that could meet this tighter specification. Also, if it is needs to be lower, the level may depend on the blend level used. 10 ppm with ASTM D 6751 already gives 2 ppm in the final blend with a B20 blend, assuming there is no phosphorous in petrodiesel. Note that the EU method will not support a limit of 2 because R runs from 1 to 2.5 ppm. The level of 4 ppm was chosen in part due to this method limitation.

**Effort, Resources to align:** Medium, Medium

**Impact of Non-Alignment:** Medium

Brazilian and US biodiesel could not be used in Europe without ensuring compliance with an eventual EU lower limit.

### 2.5.2.3 Carbon residue

Carbon residue is defined as the amount of carbonaceous matter left after evaporation and pyrolysis of a fuel sample under specific conditions. Although this residue is not solely composed of carbon, the term “carbon residue” is found in all three standards because it has long been commonly used. The parameter serves as a measure for the tendency of a fuel sample to produce deposits on injector tips and inside the combustion chamber when used as automotive fuel. It is considered as one of the most important biodiesel quality criteria, as it is linked with many other parameters. So for FAME, carbon residue correlates with the respective amounts of glycerides, free fatty acids, soaps and remaining catalyst or contaminants (Mittelbach 1996). Moreover, the parameter is influenced by high concentrations of polyunsaturated FAME and polymers (Mittelbach and Enzelsberger 1999). For these reasons, carbon residue is limited in the biodiesel specifications.

#### Limits and Methods

Brazil Limit: 0.10 % m/m max (100 % sample) Method: EN ISO 10370/ASTM D4530

EU Limit: 0.30 % m/m max (10 % sample) Method : EN ISO 10370

USA Limit: 0.050 % m/m max(100% sample) Method: ASTM D4530

#### Commentary

**Brazil cat. B)** In the EU, the limit for biodiesel carbon residue (determined on the 10% distillation residue) is set to a maximum value of 0.30 % mass, applying the same method used for fossil diesel. The respective limit in Brazil and the USA is lower, because the determination is conducted on the full sample.

For biodiesel, the recovery of a 10% distillation residue poses considerable problems due to the nearly identical boiling points of different esters which make the use of the full sample more reasonable.

**EU cat. B)** The EU chose to measure this parameter on a 10% sample in order to benefit from higher measurement precision. This may not be strictly necessary, and alignment on the 100% method appears feasible. However, it may be necessary to check the precision of the 100% method in view of the low value being measured before a decision is taken.

**USA cat. B)** Agree with Brazil. ASTM test methods group indicated scope already covers biodiesel; however neither the 100% or 10% test methods have been through a round robin for FAME that we are aware of.

In discussions, it was agreed that round robin testing data are needed. Preferably the 100% and 10% methods should both be evaluated simultaneously. The method used needs to be resolved before discussion can take place on the harmonized limit value. The issue of whether the biodiesel is only a blendstock component or also a stand-alone fuel is also pertinent to this discussion.

During discussions, the USA say they are willing to engage a round robin test with the EU on a method treating samples of 100%, and the limit value and significant digits can be considered once the methods are aligned. The question of use of biodiesel came up,

blendstock or stand-alone fuel. The US and Brazil are orientated specifically towards biodiesel as a blend component and not a stand-alone fuel.

**Effort, Resources to align:** Medium, Medium to Major

**Actions needed to harmonize:**

A round robin with analytical method should be designed that covers the range of product from various feedstocks and processes available around the world. Product should meet all other pertinent specifications.

Once method harmonised, a fuel survey should be conducted in each region using the agreed method to determine status of current product in the market vs. the specification

If no issues are apparent, the new method and limit can be balloted in each region's specification

If issues exist, bench testing or further evaluation of field and in-use data may be needed. This will likely be dependent on the blend level used.

Based on this, ballots could then be agreed upon for a new method and/or test limit

More detailed review of the specific test methods used by each region

Impact of Non-Alignment: Minor.

Carbon Residue is usually met if all other specification parameters are met, with the possible exception of biodiesel based on used frying oils.

#### 2.5.2.4 Ester content

This parameter is an important tool, like distillation temperature, for determining the presence of other substances and in some cases meeting the legal definition of biodiesel (i.e. mono-alkyl esters). Low values of pure biodiesel samples may originate from inappropriate reaction conditions or from various minor components within the original fat or oil source. A high concentration of unsaponifiable matter such as sterols, residual alcohols, partial glycerides and unseparated glycerol can lead to values below the limit. As most of these compounds are removed during distillation of the final product, distilled methyl esters generally display higher ester content than undistilled ones. (Mittelbach and Enelsberger, 1999)

**Limits and Methods:**

Brazil Limit: Report	Method: ABNT NBR 15342/EN 14103
EU Limit : 96.5 % m/m min.	Method: EN 14103
USA Limit: None	Method: Not Applicable

**Commentary:**

**Brazil cat. B)** We believe it will be possible to adopt a minimum limit of 96.5% for ester content in the harmonised specification, as suggested by the European colleagues.

**EU cat. B)** This parameter is intended to exclude poor quality feedstocks containing higher amounts of unsaponifiable material and with high content of polymers due to

treatment by poor process technology. It is also a requirement of the Italian Customs authorities. The test method should be upgraded. Discussion may be possible on removing this parameter if other precautions can be agreed, such as content of unsaponifiable material or similar. The parameter correlates with distillation range. If we keep this parameter, we need to adapt the method in order to cover also lauric oils such as coconut or palm kernel oil.

**USA cat. B)** Issue with the test method accuracy. Our understanding is the European test method group is considering dropping the specification because the method is un-able to measure accurately to 96.5%. This was the reason it was not adopted in D6751 originally, as ASTM members would not allow a method to be used in a specification without acceptable accuracy. If accurate test method was developed, USA would consider it in place of D1160 per above.

In discussion, the EU signalled an imminent round robin test, but it would not take lauric oils into consideration, so this would need to be taken into account by a new method to accommodate the wishes of the Brazilian experts. The latter are reviewing a possible method for this. The US could consider an alignment with BR/EU.

**Effort, Resources to align:** Medium, Medium to Major

**Impact of Non-Alignment:** Minor

Ester content is usually met if other parameters are met.

#### 2.5.2.5 Distillation temperature, 90% recovered, maximum

This parameter is an important tool, like ester content, for determining the presence of other substances and in some cases meeting the legal definition of biodiesel (i.e. mono-alkyl esters).

##### **Limits and Methods:**

Brazil	Limit: 360° C	Method: ASTM D1160
EU	Limit: none	Method: Not Applicable
USA	Limit: 360° C	Method: ASTM D1160

##### **Commentary:**

**Brazil cat.B)** The elimination of this specification is under discussion in Brazil.

**EU cat.B)** The EU experts have some doubts regarding the usefulness of this parameter. Perhaps it is intended to identify polymers from used frying oil or any other higher boiling components? If that is the case, other properties in the specification may be sufficient. This distillation property correlates with ester content. The precision of measurement may also be in doubt. The EU experts would like our Brazilian and American colleagues to explain the rationale for this parameter.

**USA cat B)** This parameter was added to make sure unscrupulous blenders did not adulterate B100 with heavy petroleum components; this contamination would not be detected by any other requirement in the specification. The EU addressed the same issue

with their ester content requirement. We are open to discussion about how best to control adulteration, as ASTM D1160 is a difficult and costly test and biodiesel companies would welcome a different method. Testing should be side-by-side comparison of several different FAMES that have been spiked with high boiling contaminants. See which approach detects contamination adequately with the least cost/sample prep. Perhaps look at un-saponifiables as an option? There is already an ongoing effort to investigate alternative test methods to ASTM D1160.

**Effort, Resources to align:** Medium, Medium

**Impact of Non-Alignment:** Minor.

### 2.5.2.6 Flash point

Flash point is a measure of flammability of fuels and thus an important safety criterion in transport and storage. The flash point of petrodiesel fuels are only about half the value of those for biodiesels, which therefore represents an important safety asset for biodiesel. The flash point of pure biodiesels is considerably higher than the prescribed limits, but can decrease rapidly with increasing amount of residual alcohol. As these two aspects are strictly correlated, the flash point can be used as an indicator of the presence of methanol in the biodiesel.

Flash point is used as a regulation for categorizing the transport and storage of fuels, with different thresholds from region to region, so aligning the standards would possibly require a corresponding alignment of regulations.

#### Limits and Methods

Brazil	Limit: 100°C	Method: ABNT NBR 14598/EN ISO 3679/ ASTM D93
EU	Limit: 120°C	Method: EN ISO 3679
USA	Limit: 93°C	Method: ASTM D93
	(130°C only if methanol not measured directly)	

#### Commentary:

**Brazil cat B)** Presently, the Brazilian specification defines a flash point minimum of 100° C, while CEN and ASTM are stricter, with minimums of 120 and 130° C, respectively. Nevertheless, it should be noted that the 100°C limit value is compatible with NFPA code to non-hazardous category. If there is a specific test for alcohol control, the flash point limit should not be necessary for this purpose.

**EU cat. B)** The flash point in the CEN specification was fixed to fall into the non-hazardous category, and a reduction from 120°C to 101°C has recently been agreed and will be balloted because of a more precise measurement method. The non-hazardous category does not change

**USA cat. B)** We would also put this in A or B, but some discussion could allow closer alignment. We agree with Brazil, as D 6751 recently changed to allow a lower flash if alcohol is measured and controlled directly (See Section 1.10). Work on improving flash

point accuracy with alcohol contaminated biodiesel is underway. USA uses D 93 for many regulations, so it would need to remain an option, if not the referee method.

In discussion, it appears that the measurement methods are compatible, and the issue for the USA would be to specify the flashpoint and methanol content separately. The regulatory aspect of the flashpoint limit alone, at 93°C for the USA, would have to be dealt with to obtain eventual alignment between the regions.

**Effort, Resources to align:** Medium, Minor to Medium

**Impact of Non-Alignment:** Minor

### 2.5.2.7 Total contamination (solids)

Total contamination is defined as the quota of insoluble material retained after filtration of a fuel sample under standardized conditions. It is limited to  $\leq 24$  mg/kg in the European specification for both biodiesel and fossil diesel fuels. The Brazilian and American biodiesel standards do not contain this parameter, as it is argued that fuels meeting the specifications regarding ash content will show sufficiently low values of total contamination as well. The total contamination has turned out to be an important quality criterion, as biodiesel with high concentration of insoluble impurities tend to cause blockage of fuel filters and injection pumps. High concentrations of soaps and sediments are mainly associated with these phenomena (Mittelbach 2000).

#### Limits and Methods:

Brazil	Limit: Report	Method: EN ISO 12662
EU	Limit : 24 mg/kg max	Method : EN ISO 12662
USA	Limit: None	Method: Not Applicable

#### Commentary:

**Brazil cat. B)** We believe it will be possible to adopt an upper limit of 24 mg/kg for contaminants, to be measured at the production site.

**EU cat. B)** The EU experts consider this to be a most important parameter. A too high level of contaminants can cause filter blocking at filling pumps and on vehicles. Fuel injection equipment suppliers are concerned for the premature wear of the injection system components. Some cases of high contaminants have already been seen in Europe. A lower limit than 24 mg/kg would be welcomed but the precision of the test method does not allow it at present. This point needs to be discussed with our American and Brazilian colleagues in the context of separating the water + sediment constraint.

**USA cat. B)** ASTM total contamination test for biodiesel currently being balloted at ASTM. Some changes compared to petrodiesel method are needed to get accurate results with biodiesel. ASTM considering adoption once TM is approved and Karl Fischer moisture vs. D 2709 and further field samples of B100/B99 in the field and at production. Some question about justification of the limits, why 24 vs. some other number?



**Effort, Resources to align:** Medium, Medium to Major

**Impact of Non-Alignment:** Minor

### 2.5.2.8 Water content and sediment

The Brazilian and American standards combine water content and sediment in a single parameter, whereas the European standard treats water as a separate parameter with the sediment being treated by the Total Contamination property. Water is introduced into biodiesel during the final washing step of the production process and has to be reduced by drying. However, even very low water contents achieved directly after production do not guarantee that biodiesel fuels will still meet the specifications during combustion. As biodiesel is hygroscopic, it can absorb water in a concentration of up to 1000 ppm during storage. Once the solubility limit is exceeded (at about 1500 ppm of water in fuels containing 0.2% of methanol), water separates inside the storage tank and collects at the bottom (Mittelbach 1996). Free water promotes biological growth, so that sludge and slime formation thus induced may cause blockage of fuel filters and fuel lines. Moreover, high water contents are also associated with hydrolysis reactions, partly converting biodiesel to free fatty acids, also linked to fuel filter blocking. Finally, corrosion of chromium and zinc parts within the engine and injection systems have been reported (Kossmehl and Heinrich, 1997). Lower water concentrations, which pose no difficulties in pure biodiesel fuels, may become problematic in blends with fossil diesel, as here phase separation is likely to occur. For these reasons, maximum water content is contained in the standard specifications.

#### **Limits and Methods: Water content and sediment**

Brazil	Limit: 0.050 % v max	Method: ASTM D2709
EU	Limit: None	Method: Not Applicable
USA	Limit: 0.050 % v max	Method: ASTM D2709

#### **Limits and Methods: Water content**

Brazil	Limit: None	Method: Not Applicable
EU	Limit: 500 mg/kg max	Method: EN ISO 12937
USA	Limit: None	Method: Not Applicable

#### **Commentary:**

**Brazil cat B)** We believe it will be possible to adopt separate limits for water content and total contamination, as suggested by the European colleagues. We believe it will be possible to adopt an upper limit of 500 mg/kg for water content, to be measured at the production site. The test method should be Karl-Fischer titration (ISO 12937), which is more appropriate to determine the amount of absorbed water.

**EU cat B)** The EU prefers to have separate limits for water and sediment (total contamination), but the EU water content maximum of 500 mg/kg is close to Brazil/USA combined limit value if the latter does not contain sediment. The EU experts feel it is best

to separate these contaminants for a better description of fuel quality, and would like to discuss this possibility with Brazilian and American colleagues.

**USA cat B)** Water and Sediment, Water Content and Total Contamination address the same basic properties. We agree in principle water and sediment should be separated and ASTM is considering this already for D6751. ASTM is working towards that goal for diesel fuel and a change in D975 will probably carry over to a change in D6751. We are open to discussion on this property with the understanding that we are really talking about test methods and less about limits. There is also a question about free water vs. soluble water in the fuel, with D 2709 measuring free water which is the main concern in fuel systems. EN 12937 is a Karl Fisher method for water. Its scope is petroleum products boiling at less than 390°C. After checking in *Guide to ASTM Test Methods for the Analysis of Petroleum Products and Lubricants, 2nd Edition* D1364 seems comparable, whose scope is volatile solvents and chemicals used in paints, etc. Some discussion at recent Biodiesel Technical Workshop on different water methods for biodiesel which would be of assistance. Both test methods report to 0.001 mass%. Some question where the spec would apply (i.e. production site or distribution tank). Further discussion warranted.

**Effort, Resources to align:** Medium, Medium

**Impact of Non-Alignment:** Medium to Major

## 2.5.3 Specifications for which an agreement may not be deemed reachable.

### 2.5.3.1 Sulfur content

Fuels with high sulfur contents have been associated with negative impacts on human health and on the environment, which is the reason for current tightening of national limits. Low sulfur fuels are an important enabler for the introduction of advanced emissions control systems. Engines operated on high sulfur fuels produce more sulfur dioxide and particulate matter, and their emissions are ascribed a higher mutagenic potential. Moreover, fuels rich in sulfur cause engine wear and reduce the efficiency and life-span of catalytic systems. Biodiesel fuels have traditionally been praised as virtually sulfur-free. The national standards for biodiesel reflect the regulatory requirements for maximum sulfur content in fossil diesel for the region in question.

#### Limits and Methods:

Brazil	Limit: Report	Method: EN ISO 20846/20884/ASTM D5453
EU	Limit: 10.0 mg/kg max	Method: EN ISO 20846/20884
USA	Limit: 15/500 mg/kg max	method: ASTM D5453/ASTM D4294

#### Commentary:

**Brazil cat. C)** The sulfur is limited to a maximum content of 10 mg/kg in the EU standard. The upper limit of sulfur content in the Brazilian review proposal is 50 mg/kg. In accordance with EU proposal, we support the regional requirements for sulphur levels in biodiesel fuel as a solution for difficulties of specification alignment.

**EU cat. C)** The EU biodiesel sulfur limit is based on legislative requirements for fossil diesel fuel, and the biodiesel producers agree its feasibility.

Regional requirements for sulphur levels in biodiesel fuel may be a solution for difficulties of specification alignment. In discussion, it was felt that alignment of the limit values was difficult due to the regulations for sulfur content on fossil fuels specific to each region. In practice, the sulfur level of a biodiesel for export might be the subject of contractual arrangements between buyer and seller as a function of the sulfur regulations in the region of use.

**USA cat. C)** Sulfur content in the US is regulated by the US Environmental Protection Agency (US EPA). At present, conventional diesel fuel has 3 available maximum sulfur levels, depending on the application: 5000 ppm, 500 ppm, and 15 ppm. These correspond in general to heavy fuel oils for industrial use (5000 ppm), off road fuel (500 ppm), and on road fuel (15 ppm) The biodiesel specification, D6751, has two sulfur maximum grades, 500 ppm and 15 ppm. While most biodiesel falls well within the 15 ppm maximum sulfur grade, some biodiesel from animal fats or used cooking oils and other oil sources such as mustard have been shown to have sulfur levels higher than 15 ppm. While levels higher than 15 ppm are rare, it is still acceptable to utilize biodiesel with sulfur values higher than 15 in the same way as conventional diesel fuel. There are

no known cases of un-adulterated biodiesel with sulfur levels higher than 500 ppm, so no need for a 5000 ppm maximum sulfur grade for biodiesel. Since this is a regulatory constraint it is not possible to change these levels to a higher value. There is no performance reason to change it to a lower level, especially since D6751 is as a blending stock not a pure fuel.

**Effort, Resources to align:** Major, Major

**Impact of Non-Alignment:** Medium to Major

### 2.5.3.2 Cold climate operability

The behaviour of automotive diesel fuel at low ambient temperatures is an important quality criterion, as partial or full solidification of the fuel may cause blockage of the fuel lines and filters, leading to fuel starvation and problems of starting, driving and engine damage due to inadequate lubrication. The melting point of biodiesel products depend on chain length and degrees of unsaturation, with long chain saturated fatty acid esters displaying particularly unfavourable cold temperature behaviour.

#### **Limits and Methods: Cloud point (°C):**

Brazil	Limit: None	Method: Not Applicable
EU	Limit: Based on National Specifications	Method: EN ISO 23015
USA	Limit: Report	Method: ASTM D2500

#### **Limits and Methods: Cold Filter Plugging Point (°C):**

Brazil	Limit: Based on National Specifications	Method: ABNTNBR 14747/ASTM D371
EU	Limit: Based on National Specifications	Method: EN 116
USA	Limit: None	Method: ASTM D6371

#### **Commentary:**

**Brazil cat. C)** The cold filter plugging point (CFPP) describes the fuel filterability at low ambient temperatures. CFPP is a limited parameter in the European biodiesel. This is reasonable due to the direct use of B100. In the ASTM standard the property defined as “to report” is the cloud point, which generally relates to the temperature at which crystals begin to precipitate from the fuel. Cloud point and cold filter plugging point should be defined in commercial agreements depending on the region importing the fuel and only reported in a harmonized standard.

**EU C)** For both of these cold flow properties, the possibilities of alignment of specifications are very uncertain, due to widely varying climatic conditions that require quite different cold flow performances. For this very reason, alignment of specification should not be attempted as it would create a large diversity of differing biodiesel specifications. This would be detrimental to the objective of international trade in these products. Furthermore, the EU specification EN 14214 will shortly be modified to allow exemption of biodiesel used as a blending stock from cold flow properties. This will avoid risks of incompatibility of biodiesel cold flow additives and fossil diesel fuel cold flow additives, so the final fuel blender/distributor will assume responsibility for the cold

flow performance of the finished fuel. Nevertheless, consideration should be given to setting regional standards for biodiesel, for example one winter grade, one summer grade, depending on the region importing the product. This approach could avoid a too great difference of cold flow properties between the biodiesel and fossil diesel, e.g. summer biodiesel blended into winter fossil diesel, resulting in a heavy additivation treatment. Such standards may not necessarily be formal, simple commercial agreements may be sufficient to cover the situation. If discussion shows that this approach is not practical, then a simple reporting of these parameters will be the minimum necessary. Discussion is needed on this point.

**USA cat. C)** Cold Flow addressed with additives or use as blend. Current CEN cold flow specs eliminate most animal fats, palm, coconut, other saturated fat based biodiesel. Some feedstocks (palm, beef fat) may not work in some climates. We agree with both Brazil and EU, although the EU statement is a bit unclear. This should be left at report for the reasons stated above. Regarding test methods, ASTM says the two CFPP methods are equivalent. Both report to 1°C. Precision below.

	ASTM D6371	EN 116
r	1.76°C	1°C
R	0.102(25-X) °C	0.103(25-X) °C

In discussion, it was indicated that the EU would not require cold flow property limits for the biodiesel as a blendstock, as the final fuel distributor would take responsibility for meeting market requirements. This approach avoids several incompatible additives arriving in the blend. While alignment of methods and limit values may be difficult, it may be possible to leave this constraint to commercial agreements.

**Effort, Resources to align:** Major (leave to regional requirements), Minor to Medium

**Impact of Non-Alignment:** Minor

### 2.5.3.3 Cetane number

The cetane number of a fuel describes its propensity to combust under certain conditions of pressure and temperature. High cetane number is associated with rapid engine starting and smooth combustion. Low cetane causes deterioration in this behaviour and causes higher exhaust gas emissions of hydrocarbons and particulate. In general, biodiesel has slightly higher cetane numbers than fossil diesel. Cetane number increases with increasing length of both fatty acid chain and ester groups, while it is inversely related to the number of double bonds.

The cetane number of diesel fuel in the EU is regulated at  $\geq 51$ . The cetane number of diesel fuel in the USA is specified at  $\geq 40$ . The cetane number of diesel fuel in Brazil is regulated and specified at  $\geq 42$ .

**Limits and Methods:**

Brazil	Limit: Report	Method: EN ISO 5165/ASTM D613
EU	Limit: 51.0 min	Method: EN ISO 5165
USA	Limit: 47 min	Method: ASTM D613

**Commentary:**

**Brazil cat. C)** Given the significant difference between the limits adopted in the different standards analysed, it will probably be difficult to reach agreement on a single value to be applied. Therefore, the harmonised standard should only require that this property be reported, and should leave the limit values to be defined in commercial agreements.

**EU cat. C)** The EU cetane number minimum limit is aligned with the EN 590 fossil diesel fuel specification requirement. Using low cetane biodiesel as a blending component requires the fossil fuel refiner to use higher cetane to obtain compliance for the finished fuel. Reducing the EU cetane limit would pose major difficulties for the vehicle constructors, whose vehicles are certified on fuel of 51 minimum cetane. Emissions compliance would be jeopardised by low cetane fuels in the EU. Cetane number at least should be a reportable item.

**USA cat. C)** The USA cetane number minimum for diesel is 40 minimum as described by ASTM D975. Biodiesel cetane number values should be the same as the performance limits in D975 or higher. For ASTM D6751, the cetane number has been set at 47 minimum which is a value that shows the true performance of biodiesel but does not eliminate any known biodiesel feedstock.

In discussion, it was made clear that the cetane number is feedstock dependent and is governed by national regulations for diesel fuel. An alignment of national regulations appears very difficult, so commercial agreements might be the best approach for this parameter.

**Effort, Resources to align:** Minor, None

If contract parameter only for both.

**Impact of Non-Alignment:** Major

Cetane value of 51 eliminates some feedstocks for biodiesel production.

#### 2.5.3.4 Oxidation stability

Due to their chemical composition, biodiesel fuels are more sensitive to oxidative degradation than fossil diesel fuel. This is especially true for fuels with a high content of di- and higher unsaturated esters, as the methylene groups adjacent to double bonds have turned out to be particularly susceptible to radical attack as the first step of fuel oxidation (Dijkstra et al. 1995). The hydroperoxides so formed may polymerise with other free radicals to form insoluble sediments and gums, which are associated with fuel filter

plugging and deposits within the injection system and the combustion chamber (Mittelbach and Gangl, 2001). Where the oxidative stability of biodiesel is considered insufficient, antioxidant additives might have to be added to ensure the fuel will still meet the specification.

**Limits and Methods:**

Brazil	Limit: 6 hours min	Method: EN ISO 14112
EU	Limit: 6 hours min	Method: EN ISO 14112
USA	Limit: 3.0 hours min	Method: EN ISO 14112

**Commentary:**

**Brazil cat. B)** The oxidation stability of biodiesel is a crucial parameter to ensure correct fuel performance in the vehicles, as well as in storage and distribution. We believe the limit of six hours should be adopted, to be measured at the production site. Oxidation stability additives are proven to be effective and should be allowed.

An adequate specification for oxidation stability shall eliminate the need for this latter parameter from the specification.

**EU cat. C)** The oxidation stability of biodiesel is a crucial parameter to ensure correct fuel behaviour in the vehicles, as well as in storage and distribution. Inadequate stability can lead to deposit formation in the fuel that can easily block filters and immobilise the vehicle. The EU experts consider that 6 hours is the lowest acceptable limit, and may consider increasing the value in the CEN specification review.

This parameter will be of even greater importance for the transport of biodiesel product from one region to another, as its oxidation resistance diminishes in time, leading to an out of specification situation when the product finishes its transport cycle.

**USA cat.C)** Oxidation stability specification eliminates the need for CEN iodine value. This property should be discussed with all data to properly place it in one of the categories. EU and Brazil should be made aware of the NREL study that was used to adopt 3 hour IP in D6751 for use with B20 blends and lower. Higher blends may need high levels of IP. Further this should be linked to other properties such as iodine content, linolenic ester content and polyunsaturated ester content. If oxidation stability is properly specified then those parameters should not be needed.

In discussions, whereas the three regions are aligned on the test method, the limit value for the USA product is lower than the current BR/EU limit. The major point of contention on the limit value is the stand alone fuel use in the EU compared to the blendstock component usage for BR and USA. A further difficult point may be where the standard will be required to be met, either at the point of production or at the point of delivery, as oxidation stability degrades with time. The EU standards committees are presently debating a possible increase in minimum requirements for oxidation stability.

**Effort, Resources to align:** Major

**Impact of Non-Alignment:** Major

### 2.5.3.5 Mono-, di- and tri-acylglycerols

The EU standard specifies individual limit values for mono-, di- and tri-acylglycerides as well as a maximum value for total glycerol. The standards for Brazil and the USA do not provide explicit limits for the contents of partial acylglycerides. In common with the concentration of free glycerol, the amount of glycerides depends on the production process. Fuels out of specification with respect to these parameters are prone to deposit formation on injection nozzles, pistons and valves (Mittelbach et al. 1983).

#### **Limits and Methods: Monoacylglycerol, max. (% mass)**

Brazil	Limit: Report	Method: ABNT NBR 15342/EN ISO 14105
EU	Limit: 0.80 % m/m	Method: EN ISO 14105
USA	Limit: None	Method: Not Applicable

#### **Limits and Methods: Diacylglycerol, max. (% mass)**

Brazil	Limit: Report	Method: ABNT NBR 15342/EN ISO 14105
EU	Limit: 0.20 % m/m	Method : EN ISO 14105
USA	Limit: None	Method: Not Applicable

#### **Limits and Methods: Triacylglycerol, max. (% mass)**

Brazil	Limit: Report	Method: ABNT NBR 15342/EN ISO 14105
EU	Limit: 0.20 % m/m	Method: EN ISO 14105
USA	Limit: None	Method: Not Applicable

#### **Commentary:**

**Brazil cat. B)** A specific evaluation of each compound effect in the engine performance will be necessary before limits can be defined. The standard methods defined by EN 14105 and ASTM D 6584 are gas chromatographic methods. In order to transform glycerol as well as mono- and diacylglycerols into more volatile compounds, the free hydroxyl groups of the sample are derivatized prior to the analysis. This procedure is not suitable for castor oil due to the presence of hydroxyl groups in the main carbon chain of the ester, so new methods for this purpose have been developed in Brazil (ABNT NBR 15342 and 15344). Note that, contrary to what was stated in the EU commentaries, the Brazilian specification does not establish values for these parameters, but only requires that they be reported.

**EU cat. B)** These parameters are measured by the same method as for free and total glycerin, this latter is calculated from the individual glycerides. These parameters are important in describing the quality of biodiesel, not only from a hardware perspective but also to ensure that the fuels are operable over a wide range of conditions. They should be limited individually. The triglyceride content is a good indicator of pure vegetable oil in the biodiesel. Brazil and the EU are practically aligned; the USA measures these parameters already to calculate total glycerin, so discussions may allow an alignment of the three regions' specifications.

**USA cat. C)** No data exists for limiting the individual Mono-, Di- and Tri-glycerides to specific values, nor is the analytical accuracy been determined for each of the test method



values, at least within ASTM. Data needed to justify individual limits would be cost prohibitive without a corresponding benefit. If users are interested in the data, it is available from the test method for these purposes (i.e. if all the Total glycerin is from tri-glyceride then it may be a leaky valve rather than a production issue). Total glycerin value has been well established to be acceptable in use for fuels system fouling and injector coking needs and is sufficient for these purposes. There is discussion at ASTM on further limits for some monoglycerides based on cold flow impacts. More discussion is needed.

In discussion, the EU mentioned that mono-acylglycerides are under investigation regarding the formation of deposits in cold storage conditions. The USA is also interested in cold flow properties related to these parameters. BR requested that the other partners might consider their lately developed measurement methods.

**Effort, Resources to align:** Major, Major

**Impact of Non-Alignment:** None to Minor

### 2.5.3.6 Density

The densities of biodiesels are generally higher than those of fossil diesel fuel. The values depend on their fatty acid composition as well as on their purity. Density increases with decreasing chain length and increasing number of double bonds, or can be decreased by the presence of low density contaminants such as methanol.

#### Limits and Methods

Brazil Limit: Report at 20°C Method: ABNT NBR 7148/14065/ ASTM D1298/D4052

EU Limit: 860-900 kg/m<sup>3</sup> Method: EN ISO 3675/12185

USA Limit: Report Method: Not Applicable

#### Commentary:

**Brazil cat. C)** In Brazil, this property is defined as “to report”. In situations where biodiesel is used in relatively high dilution factors, there are reasonable arguments for a greater flexibility in the specification. The tendency is to establish a limited range, but more flexible than the EU one in order not to create an unnecessary restriction in the employment of different feedstocks.

**EU cat. C)** The EU experts consider that all variations of biodiesel will fall within the CEN and Brazilian specification, so this should not present a difficulty for the USA to align on these limits.

Brazil reports its density measurement at 20°C, the EU at 15°C.

**USA cat. C)** We agree that all the biodiesels meeting other specs fall well within the EU and Brazilian limits. This is outlined in D6751 footnote:

NOTE X1.3—The density of biodiesel meeting the specifications in Table 1 falls between 0.86 and 0.90, with typical values falling between 0.88 and 0.89. Since biodiesel density falls between 0.86 and 0.90, a separate specification is not needed. The density of raw oils and fats is similar to biodiesel; therefore use of density as an expedient check of

fuel quality may not be as useful for biodiesel as it is for petroleum based diesel fuel. This section has been added to provide users and engine interests with this information.

Adding a density specification for biodiesel in ASTM would first mean adding one for D975, which does not currently have a density specification. This would require significant effort and resources and the cost/benefit is highly questionable.

In discussion, it was questioned if this parameter is needed. The EU thinks it is needed for stand alone 100% biodiesel, but the Brazilian experts feel it would exclude castor oil based biodiesel. A reflection should be made on usefulness of this parameter. An alignment of specifications would be very difficult, but a removal of the density parameter for pure biodiesel might be feasible.

**Effort; Resources to align:** Minor if removed, major for alignment; Minor if removed, Minor to medium otherwise

**Impact of Non-Alignment:** None to Minor.

### 2.5.3.7 Kinematic viscosity

The kinematic viscosity of biodiesel is higher than that of fossil diesel, and in some cases at low temperatures becomes very viscous or even solid. High viscosity affects the volume flow and injection spray characteristics in the engine, and at low temperatures may compromise the mechanical integrity of injection pump drive systems (when used as stand alone B100 diesel fuel).

#### **Limits and methods:**

Brazil Limit: Report Method: ABNT NBR 10441/ EN ISO 3104/ ASTM D445

EU Limit: 3.5-5.0 mm<sup>2</sup>/s Method: EN ISO 3104

USA Limit: 1.9-6.0 mm<sup>2</sup>/s Method: ASTM D445

#### **Commentary:**

**Brazil cat. C)** Kinematic viscosity at 40°C is limited to the range 3.5 – 5 mm<sup>2</sup>/s in the EU biodiesel fuel standards. The ASTM specification allows a broader range of values (1.9 – 6 mm<sup>2</sup>/s). In Brazil, this property is included as “to report”. The tendency is to establish a range of 3.5-6.0 mm<sup>2</sup>/s, more limited than the ASTM specification, yet more flexible than the CEN one in order to accommodate a wider variety of feedstocks. In situations where biodiesel will be used with big dilution factors, there are reasonable arguments for a greater flexibility in the specification.

**EU cat. C)** There is not complete unanimity among the EU experts on classifying this parameter as b) or c). The USA limits are considered very wide from a European point of view. The lower limit of 1.9 appears to be related to fossil/bio blends rather than pure biodiesel, whereas the upper limit of 6.0 may pose risks for the integrity of injection pump drive trains in cold climate conditions. The CEN EN 14214 standard includes a note to control viscosity to a maximum of 48 mm<sup>2</sup>/s at -20°C in order to protect injection pump drive trains. Discussion is needed to see if American and Brazilian colleagues can agree with the EU specifications.

**USA cat. C)** This is a property that is appropriate for finished fuels but takes on a different meaning for blendstocks and the fact that D6751 is for blendstocks explains the difference between ASTM and CEN. Previous attempts to harmonize D 6751 on the upper side with CEN were defeated at ASTM due to lack of a technical reason for reducing the parameter and the likelihood the lower level may limit feedstocks without a solid technical reason.

In discussion, it became clear that the issue is between a parameter for pure biodiesel or biodiesel/fossil blends. An alignment appears to be difficult until this principle is resolved, and then a wider specification may be required for new feedstock material. The parameter might be left as a contractual requirement for commercial exchanges.

**Effort, Resources for alignment:** Major, Minor to Medium

**Impact of Non-Alignment:** Minor

### 2.5.3.8 Iodine number, linolenic acid methyl ester and polyunsaturated biodiesel

Iodine number is a measure of the total unsaturation within a mixture of fatty acids, and is expressed in grams of iodine which react with 100 grams of biodiesel. Engine manufacturers have argued that fuels with higher iodine number tend to polymerize and form deposits on injector nozzles, piston rings and piston ring grooves when heated (Kosmehl and Heinrich 1997). Moreover, unsaturated esters introduced into the engine oil are suspected of forming high-molecular compounds which negatively affect the lubricating quality, resulting in engine damage (Schaefer et al 1997). However, the results of various engine tests indicate that polymerization reactions appear to a significant extent only in fatty acid esters containing three or more double bonds (Worgetter et al. 1998, Prankl and Worgetter 1996, Prankl et al 1999). Three or more-fold unsaturated esters only constitute a minor share in the fatty acid pattern of various promising seed oils, which are excluded as feedstock according to some regional standards due to their high iodine value. Some biodiesel experts have suggested limiting the content of linolenic acid methyl esters and polyunsaturated biodiesel rather than the total degree of unsaturation as it is expressed by the iodine value.

#### **Limits and Methods: Iodine number**

Brazil	Limit: Report	Method: EN ISO 14111
EU	Limit: 120 g/100 g max	Method: EN ISO 14111
USA	Limit: None	Method: Not Applicable

#### **Limits and Methods: Linolenic acid**

Brazil	Limit: None	Method: Not Applicable
EU	Limit: 12.0 mg/kg max	Method: EN 14103
USA	Limit: None	Method: Not Applicable

#### **Limits and Methods: Polyunsaturated ( $\geq 4$ double bonds) methyl ester**

Brazil	Limit: None	Method: Not Applicable
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EU Limit: 1 mg/kg max  
USA Limit: None

Method: In Development  
Method: Not Applicable

**Commentary:**

**Brazil cat. C)** Iodine number is a measure of total unsaturation within a mixture of fatty material, regardless of the relative shares of mono or polyunsaturated compounds. Due to the low oxidative stability of fatty acid esters containing three or more double bonds, some standards also limit the content of linolenic acid methyl esters and polyunsaturated biodiesel with four or more double bonds. Three- or more-fold unsaturated esters, however, only constitute a minor share in the fatty acid pattern of several promising seed oils, which are excluded from serving as biodiesel feedstock, despite the use of stability additives which are proven to be effective. The use of these three parameters has been considered unnecessary once an oxidative stability specification is defined. Therefore, we believe that limits for iodine value, linolenic acid methyl ester, and polyunsaturated methyl esters with four or more double bonds should not be included in a harmonised specification.

**EU cat. C) (Iodine number)** The EU experts consider there is room for discussion concerning iodine value as a debate is under way in CEN concerning the raising of iodine value. While a total removal of iodine value would be considered imprudent for the European market, a higher iodine value combined with other precautions such as reinforced limits on linolenic acid, polyunsaturates and oxidation stability may be feasible. For this reason, a discussion among the experts from the three regions would be useful.

**EU cat. C) (Linolenic acid)** The EU experts consider the limit on linolenic acid methyl ester as a protection against extremely unstable and polymerising oils. Work may be required to see if products that cannot meet this EU specification can be stabilised sufficiently to behave correctly in distribution and in engines.

**EU cat. C) (Polyunsaturated methyl ester)** The EU experts consider the limit on polyunsaturated methyl esters with four or more double bonds as a protection against extremely unstable and polymerising oils. A limit on polyunsaturates with three double bonds may also be a useful guarantee of good stability.

CEN methods experts are developing a suitable test method.

**USA cat. C) (for iodine, linolenic acid, polyunsaturates)** Oxidation stability specification eliminates the need for CEN iodine value. Current CEN iodine value based on rapeseed oil based biodiesel. CEN eliminates soybean, sunflower, and other vegetable oils. CEN eliminates the use of stability additives which are proven effective.

**Effort, Resources to align:** Major, Major

**Impact of Non-Alignment:** Major

Iodine number eliminates soybean, sunflower and other unsaturated oils from meeting specifications regardless of the use of stability additives or use as a blend stock rather than a stand alone fuel.

## 2.5.4 Conclusions and Recommendations

The standards established to govern the quality of biodiesel on the market are based on a variety of factors which vary from region to region. Some biodiesel standards have been decided essentially on the basis of the feedstock available for obtaining the oil to manufacture the methyl and ethyl esters that are biodiesel. Biodiesel standards have also been developed based on the characteristics of the diesel fuel standards existing in each region, which are different. Biodiesel standards in each region have also been developed based on the predominance of the types of diesel engines most common in the region and the emissions regulations governing those engines. Europe has a much larger diesel passenger car market, while United States and Brazilian markets are mainly comprised of heavier duty diesel engines. It is therefore not surprising that there are some significant differences between the three standards.

The use of biodiesel also leads to different standards being set. In Brazil and the United States of America, biodiesel is perceived primarily as a blend stock for extending the volume of fossil diesel fuel, and so some parameters are set on the understanding of a certain percentage of biodiesel being present in the finished fuel. In the European Union, the standard for biodiesel describes a product that may be used as a stand alone fuel, therefore some EU limit values are set to different levels to those of Brazil and USA, and the specification is more extensive. This same quality of biodiesel is also used as a blendstock in the EU. This difference in usage of biodiesel represents in some cases a considerable difficulty in achieving the eventual establishment of a common specification.

A number of parameters, measurement methods and limit values are quite close in the three standards, and a modest amount of study and work in common among the standards experts could result in a closer alignment in the specification of these parameters.

A further set of parameters are significantly different either in their measurement methods or their limit values. These parameters would need a significant amount of work and time to establish common methods and agreement on limit values appropriate to an agreed biodiesel quality level.

A third set of parameters, not necessarily common to all the standards but perceived as necessary or related to regional regulations, are considerably different across the standards. They represent the technical choices taken to ensure the perceived necessary quality for biodiesel according to its use as blendstock, as fuel for heavy duty vehicles or for passenger cars.

It is recommended this report be circulated to the members of the International Biofuels Forum as a basis for discussion on more closely aligning the specifications and prioritizing future efforts in those areas that would serve to maximize the impact of such efforts.

It is also recommended that the report is circulated to the standardization bodies of the IBF members so that further evolution of the current standards may take into account closer alignment with other members' standards, rather than diverging from them.

## **2.5.5 Annexes**

### **2.5.5.1 Annex 1. Members of the Tri-Partite Biodiesel Taskforce**

### **2.5.5.2 Annex 2. Summary Table of Properties in Category A, B, C**

### **2.5.5.3 Annex 3. Table of Specifications of ASTM, ANP, and CEN**

#### 2.5.5.4 ANNEX 1 Members of the Tri-Partite Biodiesel Task Force

##### Brazilian members

**Mr. Marco Tulio S. Cabral**  
Ministry of External Relations  
Mission of Brazil to the E.U.  
(co-leader)

**Ms. Rosângela Moreira de Araujo**  
Brazilian Petroleum, Natural Gas and  
Biofuels Agency – ANP

**Mr. Romeu José Daroda**  
National Institute of Metrology – INMETRO

**Mr. Henry Joseph Junior**  
National Association of Automobile  
Manufacturers – ANFAVEA

**Mr. Francisco Emilio Baccaro Nigro**  
Sao Paulo State Government  
Institute of Technological Research

**Ms. Silmara Wolkan**  
PETROBRAS

##### European Union members

**Mr. Barry Cahill**  
PSA Peugeot Citroën  
France  
(co-leader)

**Mr. Jürgen Fischer**  
ADM Hamburg AG  
Germany

**Mr. Günter Kleinschek**  
Scania  
Sweden

**Mr. Pascal Manuelli**  
Total  
France

**Mr. Martin Mittelbach**  
Karl-Franzens-University Graz  
Austria

**Mr. Jacco Woldendorp**  
Shell Global Solutions  
Netherlands

##### United States of America members

**Mr. Steve Howell**  
MARC-IV Consulting  
  
(co-leader)

**Mr. Loren Beard**  
Chrysler

**Mr. Scott Fenwick**  
Archer Daniels Midland Company

**Mr. Roger Gault**  
Engine Manufacturers Association (EMA)

**Ms. Donna Hoel**  
ExxonMobil

**Mr. Steve Westbrook**  
Southwest Research Institute

### 2.5.5.5 ANNEX 2: Tri-partite Task Force on Biodiesel Standards

Comparison of Brazilian, European and United States biodiesel parameters					
Property	Property Comparison			Comments	Test Methods
	Easily aligned (A)	Alignment possible with discussion or work (B)	Very Different (C)		
Free glycerol	USA, BR	EU		Decimal place to be clarified. Work needed to overcome changing the significant digits.	BR needs new test method applicable to castor oil FAME and FAEE to achieve precision needed for redefined limit.
Sulfated ash	BR EU USA			Decimal place to be clarified, minor issue if more decimal places used as this changes the specification, major issue if not. EU and BR could consider modifying limit to 0.020.	ISO method to be checked for validity of precision for adjusted limit value
Group I metals (Na+K)	BR EU USA			Brazil discussing adoption of same limits as EU and USA, considered probable.	ICP method is being balloted in EU as acceptable test method. In BR, an ICP method is being defined.
Group II metals (Ca+ Mg)	BR EU USA			Brazil discussing adoption of same limits as EU and USA, considered probable.	In BR, an ICP method is being defined.



Property	Property Comparison			Comments	Test methods
	(A)	(B)	(C)		
Carbon residue		BR USA EU		Limit values for BR USA can be aligned. EU could consider a limit value on basis of changed test method. US could investigate significant digits.	EU could consider a test method based on 100% sample rather than 10% distillation residue
Flash point		BR EU USA		Discussions needed to align the limit value. Depends if method used for control of methanol & flash, or flash alone for safety and handling. Work needed to align methods, and regulations category may affect limit alignment possibilities.	Methods different in US and EU which could be major issue for US (D93 vs. D3828). EU will ballot both methods due to new precision data. BR adopts NBR 14598 based on D93, but considers D93 and EN ISO 3679.
Copper strip corrosion	BR EU	USA		Confirmation needed that USA could agree to the deletion of this parameter. Removal could be considered; need to confirm with heating oil group at ASTM. ASTM does not have separate biodiesel standard for heating oil. All regions will examine opportunity to delete this parameter	

Property	Property Comparison			Comments	Test methods
	(A)	(B)	(C)		
Phosphorus content	BR USA	EU (if current spec changed)		Limit value reduction now under ballot in EU. Present day limit values may be aligned if BR discussions conclude on this. Possible differences between B100 as a neat fuel and B100 as blend stock for Brazil and US. EU vehicle producers insisting low values needed for exhaust emissions reasons	
Total glycerol	BR EU USA (for limit value)	BR EU USA (for test method) (Medium Term)		BR considers new method to be reviewed there allowing not only castor oil but also other feedstocks will allow alignment of three regions	Method alignment discussion is necessary as calculations in the methods provide different results.
Methanol or Ethanol content	BR, EU, USA (methanol only)	(Ethanol methods in this category)		BR considering alignment on EU. USA could consider adding significant digit to align with Brazil and EU limits, and asks to include ethanol for the case of ethyl esters. For USA, parameter will be met if flash point used for methanol presence.	New method for measuring ethanol is being developed in Brazil.
Acid number	BR EU USA (for limit)	USA (for method)		EU and USA limits are aligned, BR considering alignment with them. USA could consider aligning with Brazil and EU method.	Methods are dramatically different.

Property	Property Comparison			Comments	Test methods
	(A)	(B)	(C)		
Distillation temperature 90%		BR EU USA (Medium Term)		BR and USA are aligned, EU does not have a limit, but in Brazil the elimination of this specification is under discussion. Rationale for limit needs to be discussed to achieve three regions alignment. Limit used to detect fraud.	USA could consider removing T-90 if precision of ester content test method is improved. Efforts are ongoing in EU to do this.
Ester content		BR EU USA (Medium Term)		EU alone has a limit, BR may align with EU. USA could align with BR and EU if test method precision is improved.	BR method for Lauric oils being developed. Precision of the existing EU method under review. EU method under development to include other oils.
Water content and Sediment		BR EU USA (Medium Term)		BR and USA have aligned combined standard. EU has separate water and sediment (total contamination) standards. BR may align with EU at production site only and not downstream. USA could consider aligning with BR and EU.	
Water content		BR EU USA (Medium Term)		BR could align with EU at 500ppm, at production plant only and not downstream. US will consider alignment; eventual limit will depend on methods choice.	

Property	Property Comparison			Comments	Test methods
	(A)	(B)	(C)		
Total contamination (solids)		BR EU USA (Medium Term)		No limit for BR & USA, but BR and USA may align with EU limit. CEN considering a limit change further to a method precision improvement.	ASTM and EU efforts to develop and evaluate modified methods.
Oxidation stability	BR EU (short term)	BR EU USA (Long Term)	USA (short term)	Important performance parameter. EU and USA far apart on limit values EU discussion to modify limits. USA limits based on blend stock use only.	EU discussing methods covering blends as well as pure fuel.
Mono-, di- & tri-acylglycerols		BR EU	USA	USA does not have limits, BR report only, but BR has developed new methods for biodiesel based on castor. EU looking at mono- in relation to cold climate deposit formation. US and BR could consider individual limits if additional work completed.	Can BR methods be accepted by EU and USA? BR wants castor oil biodiesel to be taken into account in method.
Sulfur content			BR EU USA	Limits based on regional regulations. Lowest common denominator probably not possible. May be contractually decided level depending on region importing from elsewhere.	

Property	Property Comparison			Comments	Test methods
	(A)	(B)	(C)		
Cold climate operability (Cloud & CFPP)			BR EU USA	Limits based on regional, climatic conditions. May be contractually decided depending on region importing from elsewhere. Final fuel distributor will take local quality responsibility. "Report" is suggestion. Difference exists between pure fuel use and blendstock use.	
Density			BR EU USA	EU has upper/lower limits, BR & USA report only. EU may limit feedstock range. Value of parameter questioned, may hinder coconut or castor oil biodiesel.	
Kinematic viscosity			BR EU USA	EU has narrow limits; USA has wide limits, BR reports only. BR suggests compromise limits to allow wider feedstocks. Fundamental issue of blend component versus finished fuel requirements.	
Cetane number			BR EU USA	Wide divergence in limit values based on regional regulations. BR suggests report only, leaving limit values to be defined in commercial agreements. High values may hinder feedstock choice.	

Property	Property Comparison			Comments	Test methods
	(A)	(B)	(C)		
Iodine number	BR USA		EU	EU limit value seen as reducing feedstock choice. EU discussing a moderately higher limit value. BR and USA disagree with iodine number parameter, and rely on stability limit. Oxidation stability test would then be of prime importance. EU unwilling to delete the parameter as suggested by BR & US but willing to discuss limit values.	
Linolenic acid methyl ester	BR USA		EU	EU has limit value, BR & USA do not. BR considers it excludes some promising oils. BR & USA suggest relying on oxidation stability parameter.	
Polyunsaturated ( $\geq 4$ double bonds) methyl esters	BR USA		EU	EU has limit value, BR & USA do not. BR considers it excludes some promising oils. BR & USA suggest relying on oxidation stability parameter.	EU method needs to be verified and balloted.

### 2.5.5.6 Annex 3: Biodiesel Specification Requirements

Property	Test Methods			Limits			
	USA ASTM D6751	EU EN 14214	Brazil ANP 42	Units	USA ASTM D 6751	EU EN 14214	Brazil ANP 42
Sulfated Ash	D874	ISO 3987	ABNT NBR 6294/ ISO 3987/ ASTM D874	% mass	0.020 max	0.02 max	0.02 max
Group I Metals (Na + K)	UOP 391	EN 14108/ EN 14109	EN 14108/ EN 14109	mg/kg	5 max	5 max	10 max
Group II Metals (Ca + mg)	UOP 389	EN 14538	EN 14538	mg/kg	5 max	5 max	Report
Methanol or Ethanol Content	-	EN 14110	ABNT NBR 15343/ EN 14110	% mass		0.20 max	0.50 max
Acid Number	D664	EN 14104	ABNT NBR 14448/ EN 14104/ ASTM D664	mgKO H/g	0.50 max	0.50 max	0.80 max
Free Glycerol	D6584	EN 14105/ EN 14106	ABNT NBR 15341/ EN 14105/ EN 14106	% mass	0.02 max	0.02 max	0.02 max
Total Glycerol	D6584	EN 14105	ABNT NBR 15344/ EN 14105/ ASTM D6584	% mass	0.24 max	0.25 max	0.38 max
Copper Strip Corrosion	D130	EN 2160	ABNT NBR 14359/ EN 2160/ ASTM D130	Rating	Class 3	Class 1	Class 1

Property	Test Methods			Units	Limits		
Phosphorus Content	D4951	EN 14107	EN 14107/ ASTM D4951	% mass	0.001 max	0.0010 max	Report
Carbon Residue (on 100% Sample)	D4530	EN 10370	EN 10370/ ASTM D4530	% mass	0.050 max		0.10 max
Ester Content	-	EN 14103	ABNT NBR 15342/ EN 14103	% mass	-	96.5 min	Report
Distillation Temperature, 90% Recovered	D1160	-	D1160	°C	360 max	-	360 max
Flash Point	D93	EN 3679	ABNT NBR 14598/ EN 3679/ ASTM D93	°C	130.0 min	120 min	100 min
Total Contamination	-	EN 12662	EN 12662	mg/kg	-	24 max	Report
Water and Sediment	D2709	-	D2709	% volume	0.050 max		0.050 max
Water Content		EN 12937		mg/kg	-	500 max	
Oxidation Stability, 110°C	EN 14112	EN 14112	EN 14112	hours	3.0 min	6.0 min	6.0 min



Property	Test Methods		
Monoacylglycerol Content	-	EN 14105	ABNT NBR 15342/ EN 14105
Diacylglycerol Content	-	EN 14105	ABNT NBR 15342/ EN 14105
Triacylglycerol Content	-	EN 14105	ABNT NBR 15344/ EN 14105
Sulfur Content	D5453	EN 20846/ EN 20884	EN 20846/ EN 20884/ ASTM D5453
Cloud Point	D2500	EN 23015	
Cold Filter Plugging Point	D6371	EN 116	ABNT NBR14747/ ASTM D6371
Density at 15°C		EN 3675/ EN 12185	
Density at 20°C			ABNT NBR 7148/ ABNT NBR 14065/ ASTM D1298/ ASTM D4052
Linolenic Acid Methyl Ester	-	EN 14103	-

Units	Limits		
% mass		0.80 max	-
% mass		0.20 max	-
% mass		0.20 max	-
mg/kg	15/500	10	500 (note 3)
°C	Report		
°C		(5 max (Grade A) 0 max (Grade B) -5 max (Grade C) -10 max (Grade D) -15 max (Grade E) -20 max Grade F)	
kg/m <sup>3</sup>		860 - 900	
kg/m <sup>3</sup>			Report
% mass	-	12.0 max	-

Property	Test Methods			Units	Limits		
Polyunsaturated ( $\geq 4$ double bonds) Methyl Esters				% mass	-	1 max	-
Cetane Number	D 613	EN 5165	EN 5165 / D613		47 min	51.0 min	Report
Iodine Value	-	EN 14111	EN ISO14111	g iodine/ 100 g	-	120 max	-

### **3 BIOETHANOL TASK FORCE REPORT**

This report has been prepared by the designated Ethanol Task Force Leaders from Brazil, the European Union and the United States of America, namely

Mr. E. Kloss – Brazil

Mr. R. Saunders – European Union (EU)

Dr. B. Bonazza – United States of America (US)

and assisted by their regional expert task force members.

21 December 2007

### **3.1 Summary for the Bioethanol Task Force**

The US denatured ethanol standard was converted to an undenatured basis so comparison could be made with the undenatured standards of the EU and Brazil. In addition, units in the three standards were converted to a common basis.

Comparison of the standards was made in two ways. First, the standards were compared as they presently exist. This resulted in many parameters in the standards being slightly or significantly different. However, a second comparison was made after negotiations among the three regions as to what they could accept for the future. This negotiation resulted in the following conclusions:

1. Ethanol purity would be defined by ethanol and water content, the remainder being left for methanol and higher (C3-C5) alcohols.
2. Brazil and the US would specify a minimum ethanol content of 98.0 vol %; whereas, the EU would specify the lower minimum of 96.8 vol %. It is hoped that after further negotiations the EU can harmonize at the 98 vol % minimum.
3. Water content is the most difficult parameter to agree upon because it is based upon the ethanol content of the gasoline-ethanol blend that the individual countries use. The lower water limit in Europe is to protect the motorist from the negative impact on vehicles due to the higher risk of phase separation at their lower ethanol levels. The amount of ethanol used in gasoline is tied to each country's regulatory framework, making negotiated changes to this parameter difficult. Both the US and Brazil believe the European water content is set at a conservative level. The level should be reviewed once the distribution system has been dried following introduction in a market. The US provides the example of California, where ethanol is blended at 5.7% with a water content specification at 1 vol % max.
4. There are differences in the inorganic chloride specification, but the US and EU will review the specification with the aim of lowering the limit closer to the Brazil limit. Inorganic chlorides contribute to the corrosiveness of the fuel.
5. Only the EU has a phosphorus limit on ethanol, and it is based on data from the ethanol producers. The US and Brazil do not have limits, but have agreed to collect data in order to determine the phosphorous levels in their products. With this information, a decision can be made as to whether a phosphorus specification should be adopted. Phosphorus may be an issue if ethanol is produced from non-traditional feedstocks and processes.
6. There are three parameters that cannot be compared because different test methods are used in their measurement. These include residue by evaporation (gum), acidity and pHe. Efforts by the three regions in standardizing test methods for these parameters should lead to agreement. This is an area recommended for further work during 2008.
7. Brazil's ethanol standard has a specification for electrical conductivity, but the US and EU standards do not. Brazil feels strongly that this specification is necessary, because it is a simple and cheap way to measure ethanol contamination. The US and EU will consider adding a conductivity specification similar to Brazil's.

8. Negotiations either brought all other parameters into harmony among the three regions or it was deemed that the parameters were unimportant and would be dropped.
9. The work-to-date is based on a cursory review of test methods. A more in depth comparison is required.

Although the paragraphs above highlight areas of difference, when practical considerations, such as current trade flows, are taken into account most of the parameters appear to be in harmony. The only parameter where there is a pertinent difference is water content. Therefore, if Brazil or the US wishes to export ethanol into the EU, there would be a requirement for additional drying of the ethanol. It should be recognized that there is a cost and a potential loss of productivity involved in this drying process which have not been evaluated by the Task Force. Ethanol exports to the EU would also require confirmatory testing of the phosphorus content.

The findings of the teams are:

The three specifications have many similarities, and this is largely due to the fact they originated from the Brazilian specification. However, regional differences exist as a result of the market developments, climatic conditions in each region, and feedstock.

There is no technical specification that constitutes an impediment to trade given the current situation. However, it is recognised that additional drying will be required by Brazil and US exporters wishing to supply Europe. There is a cost associated with the drying process and there may be a loss of productivity. This has not been evaluated by the Task Force. There may also be some minor additional testing required to fully comply with the EU specification.

When the EU permits ethanol to be blended with gasoline at 10 vol % (E10), many of the specification differences may be resolved. However, this updated version of the EU specification is unlikely to occur before 2010.

Ethanol trade may benefit from the application of similar specifications. Given that some parameters have quite similar values in the three standards, it is recommended that an international standard for trade in ethanol be developed in accordance with the values summarized in the tables below.

**a) List of specifications that are very similar or for which an agreement may be reached**

<b>Parameter</b>	<b>Recommended Value</b>
Color	Dyes permitted. Do not use for export
Appearance	Clear and bright, visibly free of suspended or precipitated contaminants.
Density	Measure and report density and temperature.
Sulfate	4 mg/kg maximum
Sulfur content	10 mg/kg maximum
Copper	0.1 mg/kg maximum
Iron	No need to specify if conductivity adopted
Sodium	No need to specify if conductivity adopted
Electrical Conductivity	500 uS/m maximum <sup>1</sup>

<sup>1</sup> Work to develop common test method

**b) List of specifications that have differences in their values but are considered to be bridgeable**

<b>Parameter</b>	<b>Recommended Value</b>
Ethanol Content	No consensus (96.8 to 98 vol %) minimum
Acidity	0.0038 to 0.007 mass % maximum <sup>1</sup>
Phosphorus	0.5 mg/kg maximum <sup>2</sup>
pHe	6.5 – 9.0 <sup>3</sup>
Gum/ Evap. Residue.	5 mg/100ml maximum <sup>3</sup>
Chloride	1 mg/kg maximum

<sup>1</sup> Keep current limits until a common pHe test method and limits are developed

<sup>2</sup> Survey field to determine phosphorus levels present

<sup>3</sup> Work to develop a common test method

Some of these differences may be resolved once the EU permits 10 vol % ethanol to be blended into gasoline. However, this will take a revision of EN 15376 which is not likely to occur until 2010.

**c) List of specifications that are too different to be deemed bridgeable in the foreseeable future.**

The only parameter falling into this category is “Water Content” which is set at different levels due to the varying ethanol concentrations permitted in gasoline in the regions and the gasoline distribution differences. The EU has the lowest limit of 0.24 vol %; whereas, the US has the highest limit of 1.0 vol %. Brazil does not have a maximum water content, but levels are calculated to be a maximum of 0.4 vol% based on a minimum total alcohol content of 99.6 vol%.

It is recommended that Europe reviews the water content specification at the earliest opportunity. Since there is very little European market experience of ethanol at present it is difficult to make a judgement. Therefore, it is proposed that following introduction in a regional market, possibly Sweden, a study should evaluate the impact of higher water content. This should then be used by CEN to influence the setting of the water content in EN15376.

In addition, there are a number of test method issues and potential adjustments in specification limits once certain studies are completed. If the EU allows E10 and develops more experience with ethanol blending, their minimum water content limit could be increased, but not in the short term.

### **3.2 Recent, Current and Planned Activity in each Region**

#### **3.2.1 Brazil**

The most recent Brazilian standard for hydrous and anhydrous ethanol is Resolução ANP no. 36/2005. The use of ethanol as a blending component with gasoline at high levels (20-25 vol %) or as pure fuel (E100) in the domestic market for more than thirty years has led to the development of materials compatible with their characteristics, but has also determined the need for additional controls in the

specification, particularly on pH, ions and metals. Those needs are reflected in the current specifications.

The values and parameters defined in Resolução ANP no. 36/2005 are verified by the test methods developed by Brazilian National Association for Technical Standards (ABNT) and are listed in the Resolução.

No changes in the Brazilian specification for hydrous or anhydrous ethanol are expected in the foreseeable future.

### **3.2.2 European Union**

The Ethanol Task Force (ETF) of experts was assembled under CEN TC19/WG 21 to develop an EN specification for anhydrous ethanol as a blending component in gasoline. This activity was undertaken in response to a mandate to CEN from the European Commission in support of its policy to promote renewable fuels. It should be noted that the standard was developed for a 5 vol % blend of ethanol in gasoline, and the limit values of the properties were chosen for this percentage. The draft standard EN 15376 has been accepted by all member bodies and will be formally published early in 2008.

In January 2007, the European Commission indicated its intention to create a gasoline grade containing 10 vol % maximum ethanol content in EN 228 gasoline. This will require an amendment of the new ethanol standard. The ETF have already begun to examine EN 15376 with a view to adapting it to this new fuel grade, in anticipation of a mandate from the Commission to CEN. The approach of the ETF is to define a single grade of ethanol that may be used safely as a blend component at all percentage levels with gasoline.

The future work programmes in CEN on ethanol related issues are described below;

- Evolution of the ethanol standard EN 15376 largely following the conclusions and recommendations of this report.
- A new specification is being developed for E 85 and this work programme is likely to take two to three years.
- Evaluate the gasoline specification EN 228 to consider increasing the permitted ethanol limit to 10 vol %. This specification is unlikely to be published before 2010.

### **3.2.3 United States of America**

The US ethanol standard (ASTM D4806) was recently updated with the inclusion of two new ion chromatography test methods for determination of inorganic chlorides and sulfates. This will increase precision of the methods and allow determinations at lower levels. Within ASTM, the US is reviewing many of the test methods in the standards for ethanol, E85 (D5798) and gasoline (D4814) to ensure that the test methods are applicable to the required ethanol concentrations and that the scopes and precision statements cover ethanol products. Further, the US is investigating lowering the maximum limit for inorganic chlorides in D4806 and is discussing changes to volatility parameters for ethanol-gasoline blends in D4814.

On the legislative front, the US has just passed new energy legislation (H.R. 6, The Energy Independence and Security Act of 2007) which would increase the mandated use of renewable fuels from 4.7 billion gallons in 2007 to 9 billion gallons in 2008 and increasing to 36 billion gallons in 2022. In addition, this legislation makes a historic commitment to develop cellulosic ethanol by requiring that by 2022 the US produce 21 billion gallons of advanced biofuels, such as cellulosic ethanol.

### **3.3 Definitions and Technical Notes**

The process started with the circulation of the individual standard organization's fuel grade ethanol standards to the nominated team leaders. Each task force compared and identified areas of difference between the standards and circulated initial comments. The key outcome of this initial review was an assessment that the standards are on different basis. The ASTM standard is for denatured ethanol; whereas, the ANP and CEN standards are for undenatured ethanol. Since the purpose of the work was to compare the properties and their test methods in the standards and assess their compatibility it was agreed that only undenatured fuel grade ethanol would be evaluated. The ASTM specification was therefore adjusted to take account of the denaturant putting the properties on an undenatured basis. The EN specification differed fundamentally from the others in that it was by mass content rather than by volumetric content. Therefore, the EN specification was re-calculated on a volumetric basis so the three specifications could be compared directly. These property limits on a similar basis are shown in Annex II.

It was recognised that the test methods used in different regions may disguise the fact that specifications appear similar, but are in fact very different. This is why the statement of work required the evaluation of specifications and test methods. The US team compared the specifications and test methods used in the three standards and produced a spread sheet classifying the individual property standards according to the categories a), b) and c), listed below. The comparison of test methods was only to the level felt necessary to say the results would be similar. It was assumed future efforts would include a detailed evaluation of the analytical methods. The document was further modified with input from Europe and Brazil at the ASTM meeting in Phoenix, Arizona on December 5, 2007. This resulted in a document agreed upon by all and which classified properties of the ethanol standards as they exist today. Further, discussions at the Phoenix meeting resulted in additions to the spread sheet indicating where agreement could easily be reached toward formulating a harmonized document. The spread sheet is presented in Annex III, and it was used to develop the property discussion under "FINDINGS" below.

Since ethanol is a pure substance, the specifications are largely about controlling the contaminants. There are some variations among the specifications on the contaminants due to the differing ethanol levels in blended gasoline. These are discussed in detail in the following sections.

### **3.4 Parameters, Test methods & Limit Values Used in the Standardisation of Bioethanol**



This section of the report describes the parameters, measurement methods and limit values of the specifications used in the standards of the three regions participating in this phase of the tri-partite activity. The reason for each parameter is briefly explained, the methods used to measure it are noted, and the limit value retained by each region is given where appropriate. The commentaries made by each team of experts during the comparison exercise are included, and as a result of these commentaries the potential for harmonisation is assessed and the needed resources for such an action are estimated.

### 3.4.1 Specifications for which an agreement may be easily achievable

#### 3.4.1.1 Color

**Limits and methods:**

This parameter is not listed in the EN or ASTM specifications but there is a requirement to add an orange dye in the ANP specification for anhydrous ethanol. ANP does not require the use of dyes for exported ethanol. The CEN and ASTM specifications permit the addition of dyes and chemical markers, see CEN 15376 section 4.1, and ASTM D4806 section 4.2.

**Commentaries:**

Ethanol may have a natural yellow color which derives from the presence of proteins. It is not anticipated that this will cause the petroleum or automotive industry problems, but a color scale is being investigated in CEN to quantify the issue.

**Potential action for harmonization:**

It was agreed that dyes would be permitted, but their use would not be mandated.

**Resources needed:**

None.

#### 3.4.1.2 Ethanol Content and Higher Alcohol Content

**Limits and methods:**

All three specifications describe these parameters differently, which at first sight creates a significant problem. ASTM specifies a minimum ethanol content and ANP specifies one only for ethanol that is not produced by the fermentation of sugarcane. The ASTM limit is 93.9 vol % minimum and the ANP minimum limit is 99.6 vol %. CEN specifies a minimum ethanol plus higher alcohol (C3-C5) content at 98.8 vol %. ANP specifies a total alcohol content minimum limit at 99.6 vol %.

**Commentaries:**

The real issue is related to controlling the contaminants, such as water, methanol, and higher alcohols.

**Potential action for harmonization:**

It was decided this limit was not necessary when a high minimum ethanol content is specified along with a maximum water content.

**Resources needed:**

None.

### 3.4.1.3 Ethanol + Heavier (C3-C5) Alcohols

**Limits and methods:**

Only the EU specifies a limit for ethanol + heavier (C3-C5) alcohols. Brazil specifies a minimum level for total alcohol content. The US only specifies minimum ethanol content. The EU uses a density method similar to ASTM D4052 to determine these properties.

**Commentaries:**

Density methods do not work if significant amounts of methanol or C3-C5 alcohols are present.

**Potential action for harmonization:**

It was decided this limit was not necessary when a high minimum ethanol content is specified along with a maximum water content.

**Resources needed:**

None.

### 3.4.1.4 Total Alcohol

**Limits and methods:**

Only Brazil specifies a minimum limit for total alcohol (ethanol + methanol + heavier alcohols) at 99.6 vol %. A density method similar to ASTM D4052 is used to determine these properties.

**Commentaries:**

Since Brazil's alcohol from the fermentation of sugarcane contains very little methanol and heavier alcohols, their limit is essentially a minimum limit on ethanol content.

**Potential action for harmonization:**

It was decided to only specify a minimum ethanol content.

**Resources needed:**

None.

### 3.4.1.5 Heavier (C3-C5) Alcohols

**Limits and methods:**

Only the EU specifically limits the amount of C3-C5 alcohols at 2 vol % maximum. The US limits these heavier alcohols to a 4.5 vol % maximum based on the limits on ethanol, water and methanol. Brazil reports that very little heavier alcohol is in their ethanol. The EU uses a GC procedure to determine this property.

**Commentaries:**

CEN justifies setting a limit on the higher alcohols as a means of controlling the purity of ethanol.

**Potential action for harmonization:**

It was decided this limit was not necessary when a high minimum ethanol limit is specified along with a maximum water content.

**Resources needed:**

None.

### 3.4.1.6 Density

**Limits and methods:**

Only Brazil has a specification for density. This is the test method used by Brazil to determine purity. It also allows for a quick check for water content in the field. It is only accurate if the fuel ethanol does not contain significant quantities of methanol and/or C3-C5 alcohols which are denser than ethanol.

**Commentaries:**

Although not specified in all areas, actual product density values would be expected to be very close for all three regions if water, methanol, and higher alcohol contents are negligible. However, a density measurement is needed to correct volume to a reference temperature and to use in calculating vol % results from reported mass % results.

**Potential action for harmonization:**

It was agreed it should be reported along with the measurement temperature.

**Resources needed:**

None.

### 3.4.1.7 Methanol Content

**Limits and methods:**

Both the ASTM and CEN specifications have precise limits on methanol. Brazil has no specification for methanol. The CEN specification (1.0 vol%, max) is double that of the ASTM limit (0.53 vol%, max), but this is due to the maximum quantity of ethanol permitted in the finished blend with gasoline, 5 vol % (E5) for the EU and 10 vol % (E10), for the US.

In the US, methanol content is determined using ASTM D5501, a GC test method. In the EU, EC/2870/2000 Method III, a GC test method, is used.

**Commentaries:**

CEN is currently preparing for an E10 blend specification and it is anticipated that at this point the limit will be reduced to be compatible with the ASTM limit. It is reported that the methanol content of ethanol from the fermentation process is typically below 0.1 vol%.

It should be noted that in some EU countries, Customs requires that methanol be used as a denaturant at a minimum level of 2 vol %. This would give a 0.2 vol % concentration in the finished 10 vol % ethanol/gasoline blend, and it would have no effect on a common undenatured ethanol specification.

**Potential action for harmonization:**

With a high minimum ethanol content limit, no limit is needed on methanol content.

**Resources needed:**

None.

### 3.4.1.8 Hydrocarbons

Only the ANP specification controls the hydrocarbon limit in handling and distribution systems.

This is not an issue in the EU and US as hydrocarbons are often used as a denaturant. It could be an issue for some Customs authorities where the tax incentive is only given on the ethanol portion, so essentially it is zero. In the United States, a tax incentive is allowed for up to 5 vol % denaturant.

**Potential action for harmonization:**

Such a property limit is not needed for a common denatured fuel ethanol standard.

**Resources needed:**

None.

### 3.4.1.9 Sulfate

**Limits and methods:**

Both the ANP and ASTM specifications have a limit of 4 mg/kg maximum sulfates; however, the Brazil specification is for hydrous ethanol. Anhydrous ethanol is made from hydrous ethanol, so the specification would also apply to anhydrous ethanol. CEN has no limit. However, problems of sulfate deposits were identified too late to be included in the development of the CEN specification.

Ion chromatography (IC) test methods are used in the US (ASTM D7319 and D7328), Brazil (NBR 10894) and EU (draft method prEN 15492), and would be expected to give comparable results. A potentiometric titration method, ASTM D7318, is also used in the US.

**Commentaries:**

It is CEN's intention to include a 4 mg/kg limit at the next revision. All responsible blenders within the EU are expected in the interim to set a company limit of 4 mg/kg to protect consumers.

**Potential action for harmonization:**

With the adoption of a 4 mg/kg limit by CEN, the three specifications are aligned.

**Resources needed:**

None.

### 3.4.1.10 Copper

**Limits and methods:**

The limits set by all the standards bodies are very close. US and EU specification limits are 0.1 mg/kg, maximum and Brazil is 0.07 mg/kg, maximum, which is very similar. The test methods used by US (ASTM D1688), EU (EN 15488) and Brazil (NBR 10893) should give comparable results.

**Commentaries:**

Brazil raised a question whether this parameter is still required, as it was intended to prevent copper contamination from production facilities with copper tubes and stills. Copper is an oxidation catalyst and will increase the oxidation rate of fuels. However, CEN believes it is necessary as ethanol may enter the fuel chain from whiskey distilleries when it is unfit for human consumption. Copper stills are commonly used in liquor manufacture. It is proposed that ICP be used to measure Cu, Na, Fe and P in one test.

**Potential action for harmonization:**

The three countries have similar maximum limits on copper and are willing to accept a common 0.1 mg/kg maximum limit.

**Resources needed:**

None.

### 3.4.1.11 Sodium and Iron

**Limits and methods:**

Sodium (2 mg/kg, max.) and iron (5 mg/kg, max.) are only specified in the ANP specification for hydrous ethanol.

**Potential action for harmonization:**

Brazil agreed limits on these materials were not needed if ethanol had limits on conductivity and chloride content.

**Resources needed:**

None.

### 3.4.1.12 Sulfur Content

**Limits and methods:**

The ANP specification does not include a limit for sulfur; whereas, both CEN and ASTM have limits. The limit for the EU is 10 mg/kg maximum. The US has a federal

specification maximum limit of 30 mg/kg and a California maximum limit of 10 mg/kg for denatured ethanol.

The US and EU test procedures are basically equivalent. The US has three test methods for sulfur, but two test methods (ASTM D2622 and D5453) are used more frequently. D2622 is considered to be equivalent to EU EN 15485 (Wavelength Dispersive X-ray Fluorescence Spectrometry) and D5453 is considered to be equivalent to EU EN 15486 (Ultraviolet Fluorescence Method).

**Commentaries:**

The practice in the US is to target for 5 mg/kg for undenatured ethanol and 10 mg/kg for denatured ethanol. The sulfur content of ethanol is naturally very low typically 1 or 2 mg/kg. Brazil is expected to establish a sulfur specification for ethanol in the future.

**Potential action for harmonization:**

It was agreed a common limit of 10 mg/kg maximum was acceptable.

**Resources needed:**

None.

### 3.4.1.13 Appearance

**Limits and methods:**

All three specifications have exactly the same description “Clear and bright and no impurities”. Although “no impurity” does not appear in the ASTM or CEN standards, it is implied.

**Commentaries:**

The ASTM specification includes a workmanship statement that requires the product to be free of any adulterant or contaminant that may render the material unacceptable for its commonly used applications.

**Potential action for harmonization:**

All three specifications are aligned.

**Resources needed:**

None.

### 3.4.1.14 Electrical Conductivity

**Limits and methods:**

This parameter only exists in the ANP specification. It was not deemed necessary in the CEN specification due to the ethanol content being below 5 vol % in gasoline.

**Commentaries:**

The test is essentially included as a measure of corrosivity and ionic contaminants. The main advantage of this test is that it can easily be used in the field. These contaminants can cause corrosion and failure of vehicle fuel system components.

Brazil feels this specification is very important because conductivity can easily and quickly determine if ethanol is contaminated. This contamination can come from ions, including those that are not specified. In the US, ASTM is discussing a conductivity specification for ethanol.

**Potential action for harmonization:**

It was decided to work on test method development between the three countries.

**Resources needed:**

The three regions should commit resources to work on test method development.



### **3.4.2 b) Specifications for which an agreement is eventually achievable after additional common work**

#### **3.4.2.1 Ethanol Content**

##### **Limits and methods:**

A minimum ethanol limit can be calculated by subtracting the major contaminants such as water, methanol, and higher alcohols limits from 100. The minimum limits thus are 93.9 vol % for the US; 99.6 vol % for Brazil, which contains very little other alcohols; and 96.8 vol % for the EU.

Both Brazil and the US specify ASTM D5501 GC method to determine ethanol content, but Brazil only applies it to ethanol not produced by the fermentation of sugarcane. For sugarcane based ethanol, Brazil uses density since the product contains very little other alcohols. EU does not directly specify ethanol, but instead specifies Ethanol + C3-C5 alcohols and methanol limits.

##### **Commentaries:**

While ethanol is a pure chemical, practical processing results in a number of contaminants, largely higher alcohols, at low levels that are not considered harmful.

Brazil agreed that a minimum limit of 98 vol % was an acceptable limit. Brazil advised that they needed this high of a level to provide proper calibration for their engines. The US could accept a minimum limit of 98 vol % for trade purposes. This limit was believed sufficient to provide room for water, methanol, and heavier alcohols. The EU advised they would need a minimum of 96.8 vol % to allow for possible levels of methanol and C3-C4 alcohols. A gas chromatographic method is required to determine ethanol and other alcohols contents.

##### **Potential action for harmonization:**

EU should consider adopting a minimum limit of 98% for ethanol content.

##### **Resources needed:**

None.

#### **3.4.2.2 Gum/Residue by Evaporation**

##### **Limits and methods:**

Although the procedures are different in the three specifications, they are essentially trying to define the same parameter, which is the residue remaining after the ethanol is evaporated. The different procedures make it very difficult to compare the specification maximum values of 5.3 mg/100 ml for the US, 10 mg/100 ml for the EU and 5.0 mg/100 ml for Brazil. The US (ASTM D381) and Brazil (NBR 8644) procedures are similar except that the US measures heptane washed gum and Brazil measures unwashed gum. EU uses a procedure from Annex II of ECD/2870/2000, which measures an unwashed residue, similar to Brazil, but under different conditions.

**Commentaries:**

The US has discussed eliminating the heptane wash and reporting unwashed gum, which would be similar to the Brazil procedure. Alternatively, the US may use an ethanol wash.

**Potential action for harmonization:**

It was agreed that a study should be undertaken to determine how results from the three methods compare and to work toward a common test method.

**Resources needed:**

Further work is required to the development of a common test method. This would allow the regions to set a common limit for the parameter.

### 3.4.2.3 Chloride

**Limits and methods:**

The US and EU chloride specification limits for undenatured ethanol are 42 mg/kg, maximum and 25 mg/kg, maximum. The Brazil specification limit is 1 mg/kg, maximum for hydrous ethanol. Anhydrous ethanol is made from hydrous ethanol, so the specification would apply to both products.

An ion chromatography (IC) test method is used in Brazil (NBR 10894). US currently use a potentiometric titration method and two newly developed IC methods (ASTM D7319 and D7328). The EU currently uses a potentiometric titration method and is developing an IC method (prEN 15492), which provides similar results.

**Commentaries:**

Chloride is a most aggressive contaminant relating to corrosion and Brazil does not want the limit raised. The auto companies would like to see a 1 mg/kg maximum limit. The EU would like to see their limit reduced, especially if they move to E10 and E85. The US limit for E85 is 1 mg/kg maximum. A question was raised about the ability to measure 1 mg/kg chloride, but it was reported that the new ASTM IC methods lower detection limit is 0.2 mg/kg.

It appears that the US and EU specification limits are too high. The US will discuss a lower specification for chloride once the new IC methods have been adopted.

**Potential action for harmonization:**

The three regions should continue to exchange information on an eventual harmonization of the three specifications for chloride as the US and EU move to lower limits.

**Resources needed:**

None.

#### 3.4.2.4 Acidity

##### **Limits and methods:**

The maximum specification for acidity in the US (0.0074 mass %) is slightly higher than that for the EU (0.007 mass %). Brazil's specification is roughly half of this value (0.0038 mass %), due to the higher concentration of ethanol in their gasoline blends.

Test methods for measuring acidity for the US (D 1613), EU (EN 15491) and Brazil (NBR 9866) are all quite similar, involving a water-ethanol titration, and should produce quite similar results. The ABNT method may have a slight bias as a different indicator is used in the titration.

##### **Commentaries:**

In Europe, acidity is a concern for ethanol manufactured from wine alcohol because of the complex acids which may be produced. The US doesn't believe that acidity correlates with any field problems. This is why the pHe test method was developed. The acidity titration correlates with buffering strength rather than acetic acid content. The EU and Brazil believe a limit is needed to protect against long-term corrosion problems.

##### **Potential action for harmonization:**

It was decided to keep the current limits while additional work is conducted on the pHe test method.

##### **Resources needed:**

The three regions should commit resources for the additional work required on the pHe test method.

#### 3.4.2.5 pHe

##### **Limits and methods:**

US (6.5-9.0) and Brazil (6.0-8.0) specification limits for pHe are very similar in appearance however, the Brazil specification is only on hydrous ethanol. It is doubtful if the two procedures would give equivalent results because of the equipment employed. The US test method is ASTM D6423 and the Brazil test method is NBR 10891. pHe was excluded from the CEN specification due to problems associated with repeatability of the ASTM procedure when ASTM specified equipment was not used.

##### **Commentaries:**

The EU believes that more water and denaturant were need to make the test method workable. Once this is resolved it may be included. The EU has just issued a new test method for pHe. The US reported that experience has shown the ASTM test method to work with undenatured ethanol, denatured ethanol, E10, and E85 with very low water levels. It is believed that the problem likely is due to the meters and electrodes being used. The ASTM test method requires the use of specific meters. Because of the test equipment differences, it is not possible to correlate the test methods.

##### **Potential action for harmonization:**

It was agreed for the US to work with the EU and Brazil to come up with a common workable test method for pHe.

**Resources needed:**

The three regions should commit resources for the additional work required on the pHe test method.

### 3.4.2.6 Phosphorus

**Limits and methods:**

This parameter only exists in the CEN specification to protect the exhaust catalyst from phosphorus, which is a powerful poison. EU has a specification of 0.50 mg/L, maximum as measured by test method EN 15487 (Ammonium molybdate spectrometric method).

**Commentaries:**

The source of phosphorus may be the fertilizers and nutrients used in the fermentation process or from the feedstock. The EU limit is based on what the ethanol producers can manufacture, but the EU would like to set a lower limit. Brazil sees no need for such a limit.

**Potential action for harmonization:**

Data needs to be collected to determine the level of phosphorus currently present. A longer term consideration is what the phosphorus level might be when ethanol is made from non-traditional feed stocks.

**Resources needed:**

Resources are required for collecting data to determine the presence of phosphorous in ethanol, considering the different feed stocks.

### 3.4.3 c) Specifications for which an agreement is not deemed reachable

#### 3.4.3.1 Water Content

**Limits and methods:**

All countries are different, but utilize the same test procedure (Karl Fischer). The US has a 1.0 vol %, maximum; but actual water levels are more commonly 0.6-0.7 vol %. The EU has a 0.24 vol %, maximum. Brazil has no water specification, but by calculation water is in the range of 0.4 vol %.

**Commentaries:**

Brazil advised they were willing to set a maximum limit of 0.5 vol %. The low water content for EU is due to the lower concentration of ethanol blended into gasoline (~5 vol %) and a wet gasoline distribution system. The US blends 5.7-10 vol% ethanol into gasoline successfully allowing up to 1.0 vol % water in ethanol and believes the low level required by the EU is set conservatively. Brazil blends between 20-25 vol % ethanol in gasoline. At these high ethanol levels, the fuel can hold much water, and phase separation is not an issue.

Phase separation due to water occurs more readily at lower levels of ethanol in gasoline.

This is probably the most difficult parameter on which to obtain agreement and could be the greatest deterrent to trade. At this time, it would not be possible for Europe to raise the water limit due to the operational difficulties it would create. This does create problems for imports of ethanol from Brazil and the US into Europe. However, this could be resolved by additional drying at the production facilities in the US, or Brazil prior to export to Europe.

### **3.5 Ethanol trade flows and implications**

Among the three regions, ethanol trade for the foreseeable future will consist of Brazil exporting to the US and the EU and the US exporting to the EU. It is unlikely that the EU will export ethanol or that Brazil will import large amounts of ethanol on a regular basis. When this reality is considered it allows many of the apparent technical differences to be viewed differently, since many of the parameters in the Brazilian specification are tighter than in either the EU or US specifications. Therefore, the requirement to harmonize the specifications disappears and it allowed the task forces to prioritize their efforts.

With these guidelines and the negotiated parameters for future ethanol specifications, it appears that ethanol water content will be the main technical issue. However, this technical issue can be solved. Ethanol exported by the US or Brazil would have to be dried before it would be acceptable in the EU, since the water content required by the EU is the lowest of the three regions. This additional drying will have an impact on the cost of production and may decrease productivity (Note: see also footnote 11 in page 7).

Another parameter that could impact trade is chloride ion content. Since the EU chloride specification is lower than that of the US, exports from the US to the EU would require confirmatory testing for chloride. This is not expected to be a significant issue, because the actual level of chlorides found in ethanol in the US is significantly below the EU requirement. Phosphorus content is another parameter that would have to be certified for exports to the EU from Brazil or the US. Presently, neither the US or Brazil have a phosphorus specification, but the US would consider adding a phosphorus specification. None of the other ethanol property limits would be expected to impact trade. This assumes that test methods will be harmonized for those parameters that presently utilize different methods and that agreement will be reached on the value of these parameters. In conclusion, there is no specification that represents a technical barrier that cannot be overcome by additional processing or testing.

### **3.5.1 Annexes**

#### **3.5.1.1 Annex 1: Ethanol Task Force Technical Membership**

#### **3.5.1.2 Annex 2: Ethanol Specifications for US, Brazil and EU**

#### **3.5.1.3 Annex 3: Comparison of US, EU and Brazil undenatured ethanol standards**

**3.5.1.1 Annex 1: Ethanol Task Force Technical Membership**

**US Task Force on Ethanol**

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### 3.5.1.2 Annex 2: Ethanol Specifications for US, Brazil and EU

#### Ethanol Specifications for US, Brazil and EU

PROPERTY	US		Brazil		EU prEN 15376
	D 4806	D 4806 Undenatured	Anhydrous	Hydrous	
Color	Dye Allowed, but not mandated	Dye Allowed, but not mandated	Dye mandated for in country, but not for export.	Dye prohibited for in country	Dye Allowed, but not mandated
Ethanol Content, vol %, min.	92.1	93.9	99.6 <sup>(3)</sup>	--	[96.8]
Ethanol + C3-C5 sat. alcohols, vol %, min	--	[98.4] <sup>(2)</sup>	--	--	98.8
Total Alcohol, vol %, min.	--	[98.95]	99.6	95.1	[99.76]
C3-C5 sat. alcohols, vol %, max	-- <sup>(1)</sup>	[4.5]	--	--	2.0
Water content, vol %, max	1.0	1.05	[0.4]	[4.9]	0.24
Density at 20C, kg/m3, max	--	--	791.5	807.6	--
Methanol, vol %, max	0.5	0.53	--	--	1.0
Denaturant, vol %, min/max	1.96 / 5.0	No Denaturant	No Denaturant	No Denaturant	Set By Country 0/1.3
Hydrocarbons, vol %, max	--	--	3 <sup>(4)</sup>	3 <sup>(4)</sup>	--
Solvent-washed gum, mg/100 mL, max	5.0	5.3	--	--	--
Gum or Resid by Evap, mg/100ml, max	5 (washed gum)	5.3 (washed gum)	--	5 (unwashed) <sup>(5)</sup>	10 (unwashed) <sup>(5)</sup>
Electrical Conductivity, uS/m, max	--	--	500	500	--
Sulfate, mg/kg, max*	4	4.2	--	4	Working
Inorganic Chloride, mg/kg, max	40.	42.1	--	1	25
Copper, mg/kg, max	0.1	0.105	0.07	--	0.1
Sodium, mg/kg, max	--	--	--	2	--
Iron, mg/kg, max	--	--	--	5	--
Acidity, mass % (mg/L), max	0.007 (56)	0.0074 (58.9)	0.0038 (30)	0.0038 (30)	0.007
pHe	6.5 – 9.0	6.5 – 9.0	--	6.0 – 8.0	Dropped
Phosphorus, mg/L, max	--	--	--	--	0.5
Sulfur, mg/kg, max.	30.	5	--	--	10
Appearance	Clear & Bright	Clear & Bright	Clear & No Impurities	Clear & No Impurities	Clear & Bright

(1) Not specified by can be calculated for US. (Heavy alcohol content = 100 - ethanol content - methanol content - water content)

(2) Numbers in [ ] are calculated estimates and not specified limits

(3) Limit only applies to ethanol not produced by fermentation from sugarcane or ethanol contaminated by other types of alcohol

(4) Applies only to imported ethanol

(5) Procedures are likely different.

### 3.5.1.3 Annex 3: Comparison of US, EU and Brazil undenatured ethanol standards

#### Annex III

#### COMPARISON OF US, EU AND BRAZIL UNDENATURED ETHANOL STANDARDS – page 1

Property	Specification Status	Specification Property Limit Comparison <sup>(1,2)</sup>			Comments
		Same	Close /Same	Very Different	
Color	Present	US, EU, BR <sup>(4,5-EU&amp;US)</sup>			The US and EU allow the use of dyes, but they are not mandated. BR mandates the use of dyes only for anhydrous ethanol sold within the country. Dyes are not mandated for export product. Since, we are discussing what will ultimately become an export specification, all countries are the same.
	Future	US,EU,BR			All in agreement.
	Trade Impact				None
Ethanol	Present			US, BR, EU <sup>(5-EU only)</sup>	Only US and BR specify ethanol content, but their values are different. The US spec is 93.9 vol %, min. The BR spec is 99.6 vol %, min. but applies only to ethanol not produced by fermentation of sugar cane . US and BR specify the ASTM D5501 GC method. BR specifies total alcohol content (below) by density for ethanol made with sugar cane. EU does not specify ethanol, but instead specifies Ethanol + C3-C5 alcohols (below). EU ethanol content can be calculated to be 96.8 vol%.
	Future	US,BR	EU		Measure ethanol by GC and specify minimum ethanol content. US & BR could go with a minimum of 98% by volume, but the EU would need a min of 96.8% by volume.
	Trade Impact				EU would not be able to export to the US or BR, but the US and BR would be able to export to EU.
Ethanol + C3-C5 alcohols	Present	EU		BR, US <sup>(5)</sup>	The EU has a specification for this parameter and uses a density method (EC/2870/2000 Method I, Appendix II, Merthod B), which is similar to ASTM D4052. Density methods do not work if significant amounts of methanol or C3-C5 alcohols are present. This property is not in the US or BR specification.
	Future	US,EU,BR			Not necessary.
	Trade Impact				None
Total Alcohol	Present	US, EU, BR <sup>(5-EU&amp;US)</sup>			Only BR specifies total alcohols (BR=99.6 vol%). However, calculated values for the US and EU are very close to the BR specification. Calculated minimum requirements for EU and US are EU=99.8 vol% and US=99.0 vol%, respectively.
	Future	US,EU,BR			Not Necessary
	Trade Impact				None
C3-C5 alcohols	Present	US, BR <sup>(5)</sup>		EU	EU has a max of 2 vol % as determined by the GC procedure described in EC/2870/2000 Method III. US & BR have no specification, but by calculation, US could have up to 4.5 vol % C3-C5 alcohols. In actual product, the amount of C3-C5 appears to be very small for all three countries. The US is looking for field data to confirm this point. BR has data that indicates C3-C5 alcohols are very low (range from 1.0 to 1.2% vol including components not quantified or specified). This implies that the parameter above (Ethanol + C3-C5 alcohols) actually closely represents the ethanol content.
	Future	US,EU,BR			Not necessary.
	Trade Impact				None
Water Content	Present			US, EU, BR	All countries are different, but utilize the same procedure (Karl Fisher). US=1 vol%, max;, but actual water levels are more commonly 0.6-0.7 vol%. EU=0.24 vol%, max. BR has no water specification, but by calculation water is in the range of 0.4 vol%. The low water content for EU is believed to be due to the lower amount of ethanol blended into gasoline (~5 vol%). US blends 5.7-10 vol% ethanol into gasoline, and BR blends between 20-25 vol%. Phase separation due to water occurs more readily at lower levels of ethanol in gasoline.
	Future			US,EU,BR	It is not possible to come together on the actual value. This could be the greatest barrier to trade. BR feels that 0.5 vol% is what they would like. This could be a problem with export of ethanol to the EU. If EU goes to E10, their water minimum could be raised, but not in the short term. Further work is required.
	Trade Impact				BR can export to the US, but additional drying would be required for US or BR export to EU.

**Annex III (continued)**  
**COMPARISON OF US, EU AND BRAZIL UNDENATURED ETHANOL STANDARDS - page 2**

Property	Specification Status	Specification Property Limit Comparison <sup>(1,2)</sup>			Comments
		Same	Close /Same	Very Different	
Density	Present	US, EU <sup>(5)</sup>		BR	US & EU do not specify density. Brazil has a specification that is used to determine the water content (purity) of the ethanol by NBR 5992. Although specifications differ, actual product density values would be expected to be very close for all 3 countries if water, methanol and higher alcohol contents were negligible.
	Future	US,EU,BR			Measure and report density and temperature.
	Trade Impact		None		
Methanol Content	Present		US, EU	BR <sup>(5)</sup>	US (0.5 vol% max) is similar to EU (1.0 vol% max), both being determined by comparable GC procedures. BR has no specification for methanol. BR has found that methanol content is very low. They assume there is none, and this may be the case for most products.
	Future	US,EU,BR			Not necessary.
	Trade Impact				None
Hydrocarbons	Present	US, EU <sup>(5)</sup>		BR	Only BR has a specifications for hydrocarbons (3.0 vol%, max). This limit is only required for imported product to insure it has not been contaminated with hydrocarbons. Test method is NBR 13993, which utilizes a salt water separation procedure.
	Future	US,EU,BR			Not necessary.
	Trade Impact				None
Gum / Residue by Evaporation	Present			US, EU, BR	All three procedures are different. The US (ASTM D381) and BR (NBR 8644) procedures are similar in that the US measures heptane washed gum and BR measures unwashed gum. EU uses a procedure from Annex II of ECD/2870/2000, which measures an unwashed residue, similar to BR, but obtained under different conditions. NOTE: The US may discuss eliminating the heptane wash and report unwashed gum, which would be basically like the BR procedure.
	Future			US,EU,BR	Work on test method harmonization is required.
	Trade Impact				Additional testing would be required on exported product.
Electrical Conductivity	Present	US, EU <sup>(5)</sup>		BR	Only BR has a conductivity spec. of 500 uS/m, max, as measured by NBR 10547. BR feels this specification is very important because conductivity can easily and quickly determine if a product is contaminated. This contamination can come from ions, including those that are not specified. A cheap commercial meter can be used to measure conductivity. The US is discussing a conductivity specification for ethanol.
	Future	BR	US,EU		US and EU will consider adding a conductivity specification. The equipment is inexpensive and the test is easy to perform. This property provides good overall quality of the ethanol. Test method development between the 3 countries is needed.
	Trade Impact				No effect for exports from BR to US or EU. Additional testing required for export to BR.
Sulfate	Present		US, BR	EU	US & BR both have a sulfate spec of 4 mg/kg; however, BR's spec is for hydrous ethanol. Anhydrous ethanol is made from hydrous ethanol, so the specification would also apply to anhydrous ethanol. EU is considering a sulfate specification, but does not presently have one. Ion chromatography (IC) test methods are used in the US (ASTM D7319 & D7328) and BR (NBR 10894) and EU (draft method prEN 15492) would be expected to give comparable results.
	Future	BR, US, EU			EU is planning on including a specification for sulfate that would mirror the US and BR.
	Trade Impact				None
Chloride	Present			US, EU, BR	Current or developing test methods for Cl for the three countries should give similar results; however, the specifications are not the same. The US spec for undenatured ethanol is 42 mg/kg, max; whereas, the EU spec is 25 mg/L, max and the BR spec is 1 mg/kg, max. for hydrous ethanol. Anhydrous ethanol is made from hydrous ethanol, so the specification would apply to both products. Ion chromatography (IC) test method is used in BR (NBR 10894). US currently uses an ion selcetive electrode and EU currently uses a potentiometric titration method. The US has developed two IC methods (ASTM D7319 and D7328) and will be incorporating in this into the US standard. EU is also developing an IC method (prEN 15492). It appears that the US and EU specification are too high. The US will discuss a lower specification for Cl once the new IC method has been adopted.
	Future			BR, US, EU	US and EU are examining going to a lower limit. US and EU auto companies would like to see a limit of 1 mg/kg. BR's limit is 1 mg/kg and they feel that this limit is very important and should not be raised. EU and US ethanol companies will have to be consulted and my resist this very low limit.
	Trade Impact				No impact for export from the BR to US or EU. Export from US to EU would require limit confirmation.

**Annex III (continued)**

**COMPARISON OF US, EU AND BRAZIL UNDENATURED ETHANOL STANDARDS - page 3**

Property	Specification Status	Specification Property Limit Comparison <sup>(1,2)</sup>			Comments
		Same	Close to Same	Very Different	
Copper	Present		US, EU, BR		US & EU specifications are 0.1 mg/kg, max. and BR is 0.07 mg/kg, max., which are very similar. The test methods used by US (ASTM D1688), EU (EN 15488) and BR (NBR 10893) should give comparable results. Brazil raised a question whether this parameter is still required, as it was intended to prevent Cu contamination from production facilities with copper tubes and stills. Cu is an oxidation catalyst and will increase the oxidation rate of fuels. The US will push to utilize ICP to measure Cu, Na, Fe and P in one test.
	Future	BR, US, EU			This is basically the same number for all three countries and they are in agreement. All appear to be willing to agree to 0.1 mg/kg.
	Trade Impact		None		
Sodium	Present	US, EU <sup>(5)</sup>		BR	US & EU do not specify sodium. BR has a spec of 2 mg/kg on hydrous ethanol as measured by NBR 10422 (flame photometry).
	Future	BR, US, EU			BR can eliminate this specification if conductivity and chloride specifications are maintained. This brings all countries into agreement.
	Trade Impact				None
Iron	Present	US, EU <sup>(5)</sup>		BR	US & EU do not specify iron. BR has a spec of 5 mg/kg on hydrous ethanol as measured by NBR 11331 (atomic absorption).
	Future	BR, US, EU			BR can eliminate this specification if conductivity and chloride specifications are maintained. This brings all countries into agreement.
	Trade Impact				None
Acidity	Present	US, EU	BR		Test methods for measuring acidity for the US (D 1613), EU (EN 15491) and BR (NBR 9866) are all quite similar, involving a water-ethanol titration, and should produce quite similar results. The ABNT method may have a slight bias as a different indicator is used in the titration. The standards are slightly different. The US specification (0.0074 mass%) is slightly higher than the EU spec (0.007 mass%). BR's spec is roughly half of this value (0.0038 mass%).
	Future	US, EU	BR		US feels acidity does not correlate with corrosion in ethanol and is not a useful test. The EU and BR feel that this parameter is useful in protecting against long term problems in vehicles. It is recommended that the US and EU consider going to the BR limit. Poor precision with the method is an issue.
	Trade Impact				Exports from BR or US to the EU will have no impact.
pHe	Present			US, BR, EU	US (6.5-9.0) and BR (6.0-8.0) specs are very similar; however, the BR specification is only on hydrous ethanol. It is doubtful if the two procedures would give equivalent results because of the equipment employed. The US test method is ASTM D6423 and the BR test method is NBR 10891. EU has no pHe spec. EU considered pHe, but they had problems with repeatability of the procedure. The EU has just issued a new test method for pHe in October 2007. It has not yet been incorporated into the ethanol specification. The new European test method is similar to the Brazilian standard NBR 10891 and will probably give a result similar to the Brazilian method. It is not expected to give a result that is similar to ASTM D6423.
	Future			BR, US, EU	Further work is required to come up with a common test method in the three regions. Our members will recommend this work be carried out as a joint project.
	Trade Impact				BR export to EU is not affected, but export to the US would require confirmation with the US test procedure.
Phosphorus	Present	US, BR <sup>(5)</sup>		EU	US & BR have no phosphorus spec. EU has a spec of 0.50 mg/L, max. as measured by test method EN 15487 (Ammonium molybdate spectrometric method). It will be determined why the EU is concerned about P. US and BR do not feel this spec is required.
	Future	US, EU		BR	The EU feels it is necessary to provide P control. Their limit is based on data from the ethanol producers. The US and BR do not have data at present, but will try to collect data. P could be an issue when producing ethanol with non-traditional feedstocks and processes. US would like P control. BR believes that P levels are very low and do not need to be controlled.
	Trade Impact				Additional testing would be required on exported product to the EU.

### Annex III (continued)

#### COMPARISON OF US, EU AND BRAZIL UNDENATURED ETHANOL STANDARDS - page 4

Property	Specification Status	Specification Property Limit Comparison <sup>(1,2)</sup>			Comments
		Same	Close to Same	Very Different	
Sulfur	Present		US, EU	BR <sup>(5)</sup>	US has a spec of 30 mg/kg for denatured ethanol; however, without a hydrocarbon denaturant, a spec of 5 mg/kg would be appropriate for undenatured ethanol. EU has a spec of 10 mg/kg for undenatured ethanol, and BR has no current specification for sulfur in ethanol. NBR 9867 is listed as a method for determining levels of sulfur in ethanol. US and EU test procedures are basically equivalent. A US spec for undenatured ethanol is estimated to be 5 mg/kg. Therefore, US and EU have similar specifications and BR would be significantly different. US has three test methods for sulfur, but two test methods are used more frequently. (ASTM D2622 and D5453). D2622 is considered to be equivalent to EU EN 15485 (Wavelength Dispersive X-ray Fluorescence Spectrometry) and D5453 is considered to be equivalent to EU EN 15486 (Ultraviolet Fluorescence Method). Brazil is expected to establish a sulfur specification for ethanol in the future.
	Future	BR, US, EU			There is agreement that S could be set at 10 mg/kg.
	Trade Impact				None
Appearance	Present	US, EU, BR			All three countries require the product be clear and bright with no impurities. This is determined by visual examination.
	Future	BR, US, EU			All in agreement.
	Trade Impact				None

**Footnotes**

- (1) See tab "Ethanol Specs for US, Brazil and EU"
- (2) See tab "D 4806-07 Properties On An Undenatured Basis" for Estimated Values for Undenatured Ethanol
- (3) Assignments made during 10/1/07 conference call.
- (4) US=United States; EU=European Union; BR=Brazil
- (5) Property is not specified.

