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The Importance of Measurement Uncertainty Arising from the Sampling Process in Conformity Assessment: The Case of Fuel Quality

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Abstract: There is growing concern regarding the environmental and operational safety aspects of fuel. The result of a physicochemical measurement is the outcome of a series of steps that begin with the sampling process. The information obtained from this step and the contribution from the analytical process define the measurement uncertainty, although most laboratories consider only the analytical contribution as a quality parameter. On the other hand, this variability can be used as vital information to evaluate conformity to a specification. This study aimed to use uncertainty information considering only the analytical uncertainty and, next, the analytical and sampling uncertainties in compliance assessment, taking physicochemical measurements of fuel as case studies. The first scenario, which is traditional and focused solely on analytical uncertainty, showed to be less rigorous than the second scenario, which combined sampling uncertainty with analytical uncertainty. The results indicated that for the flash point in jet fuel, the sulfur mass fraction in gasoline-ethanol blends, and the kinematic viscosity in diesel, the risks to consumers—first considering only analytical uncertainty and then combining analytical uncertainty with sampling uncertainty—were the following: 2.6% and 5.6%; 4.4% and 7.1%; and 1.6% and 18.9%, respectively. Since the initial result of each pair was below 5%, compliance with the specification is suggested. However, when accounting for sampling uncertainty, there is an indication of potential non-compliance with the specification. Therefore, it is concluded that the contribution of uncertainty arising from sampling must be considered in a conformity assessment.

Keywords: two-way ANOVA with interaction; ASTM D93; ASTM D7039; ASTM D445; fuel oils; guard bands

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1. Introduction

When burned, fuels are chemical substances that release energy in the form of heat, which can be used to generate mechanical work or heat environments and processes. They are widely used in a variety of applications, including automobiles, industries, building heating, and electricity generation. A fuel's efficiency and effectiveness depend heavily on its physicochemical properties, which determine how it behaves during combustion and its interaction with the systems that use it [1].

Such physicochemical properties are essential to understanding the following. (i) Performance: The way a fuel burns, the amount of energy it releases, and its efficiency can vary widely based on its physical and chemical characteristics. (ii) Emissions and environmental impact [2]: The chemical properties of a fuel influence the types and amounts of pollutants generated during combustion. For example, fuels with a high sulfur content can release sulfur dioxide, a pollutant that contributes to acid rain. (iii) Safety [3]: Knowing a fuel's flash point and flammability characteristics is essential to ensuring its safe handling and storage. More volatile fuels can pose greater risks of fire and explosion. (iv) Energy efficiency [4]: The calorific value of a fuel is a direct measure of its ability to generate energy. Fuels with higher calorific value are preferred in applications where efficiency is a priority. (v) System compatibility: Different engines and combustion systems have specific requirements for the fuels they use. For example, diesel engines require fuels with high cetane ratings to ensure efficient combustion. (vi) Regulation and standards: The physicochemical properties of fuels are often regulated by regulations aimed at reducing emissions and improving air quality. Understanding these properties is therefore crucial to complying with environmental legislation. (vii) Innovation and development of new fuels: As the world seeks more sustainable alternatives, physicochemical properties are becoming fundamental in the research and development of biofuels, synthetic fuels, and other renewable energy sources [5].

In summary, fuels' physicochemical properties define their performance and efficiency and have significant implications for safety, the environment, and technological innovation. An in-depth understanding of these properties is essential for energy industry professionals, engineers, and researchers working on energy-efficient and sustainable solutions.

The flash point temperature indicates how likely a test sample is to create a flammable mixture with air in a controlled lab environment. While it is essential, it is just one of several characteristics to evaluate when determining a material's overall flammability risk. Additionally, flash point values are utilized in shipping and safety guidelines to categorize materials as flammable or combustible. For exact definitions of these categories, one should refer to the specific applicable regulations [6,7].

Certain catalysts utilized in petroleum and chemical refining processes can become ineffective if even small quantities of sulfur-containing substances are present in the feedstocks. The test method for determining total sulfur in fuel is applicable for measuring sulfur levels in both process feeds and finished products, and it can also serve regulatory compliance purposes [8–13].

Numerous petroleum products serve as lubricants, and the efficient functioning of machinery, rely on using liquids with the right viscosity. Furthermore, the viscosity of various petroleum fuels plays a crucial role in assessing storage, handling, and operational conditions. Therefore, precisely measuring viscosity is vital for meeting several product specifications [14–16].

Recent studies have been conducted regarding the importance of controlling these physicochemical properties. They include the implementation of multivariate acceptance thresholds to minimize the overall risk of incorrect conformity decisions in the analysis of automotive fuels [17]. This leads to the utilization of data reconciliation techniques to address the inconsistencies between measurements taken by producers and consumers, facilitating informed decisions about conformity or non-conformity assessments [18]. Finally, experimental design can be applied to assess the impact of modifications in the testing procedure for the kinematic viscosity of opaque oils [19].

Fuel compliance assessment involves analyzing their physical and chemical properties to ensure they meet regulatory agency requirements, environmental standards, and specific characteristics for efficient and safe performance. The state of the art in measuring these properties has evolved with advances in technology. The most common techniques include spectroscopy, chromatography, viscosity, density, flash point, distillation, sulfur mass fraction, among others. These methodologies, combined, have allowed a more reliable assessment of fuel properties, contributing to the assessment of compliance with regulatory standards and to the optimization of performance in practical applications.

To optimize this production chain, producing fuels close to the specification limit refers to the process of manufacturing fuels that meet exactly or are very close to the standards established for their physical and chemical properties. This is essential to ensuring that fuels are safe, efficient, and compatible with the engines and systems for which they are intended.

Measurement uncertainty is a crucial tool in monitoring the physicochemical properties of fuels, especially when measurement results are close to specification limits. However, measurement uncertainty is currently considered only analytical uncertainty without considering the contribution from the sampling process.

The oil and gas industry has very strict procedures in place to ensure the homogeneity of products stored in tanks that are ready to be dispatched, based on the reproducibility parameter. However, this practice typically assesses only density and, in a few cases, viscosity at just three specific levels within these tanks, which does not always guarantee complete product homogeneity. This is because these commercial transactions can involve different recipients, potentially compromising compliance with specifications and leading to conflicts between the supplier and the consumers.

Sampling uncertainty is caused by the heterogeneity of a product within a storage tank and can be reduced by the following actions: (i) regular mixing to minimize phase separation; (ii) temperature control to prevent the solidification of components and ensure that the products remain in a fluid state; (iii) cleaning and maintenance to remove residues and contaminants; and (iv) use of additives to improve the stability and homogeneity of the stored derivatives.

It is important to emphasize the influence of the sampling procedure on measurement uncertainty related to the physicochemical properties of diesel [20]. This includes the findings from three empirical statistical methods that utilized data obtained from a balanced experimental design featuring duplicate samples analyzed twice across 104 petroleum retail stations [21], the role of the sampling process in the overall uncertainty assessment for high-resolution gamma-ray spectrometry of environmental soil, tap water, and aerosol filter samples [22], and a robust metrological assessment of trends and relationships among physicochemical parameters across extensive oceanic regions [23].

Although, there are not many studies related to sampling uncertainty in the petroleum industry and/or other similar fluid industries, we can highlight an investigation on the uncertainties in sampling related to short-duration measurements of varying frequencies for methane emissions from several sites in the Barnett Shale region, concluding that sampling error rises as the duration of high-emission events decreases, highlighting the significance of the temporal persistence of these events in the development of measurement protocols [24]. In a different study, the variability introduced by sampling with limited sample sizes of metocean data was investigated, focusing on the uncertainty in environmental contours utilized in the design of marine structures for the joint distribution of significant wave height and wave period across various sample sizes [25]. Finally, Finnish researchers employed two approaches to evaluate the measurement uncertainty associated with the sampling and analysis of soil contaminated by petroleum hydrocarbons [26].

Recently, the incorporation of uncertainty data into conformity assessment has become widespread across various fields, including environmental aspects related to the concentrations of multiple pollutants [17,27], logistical transactions involving fuels [18,28], denatured alcohols [29], services provided to the Brazilian regulatory agency [30], drug and medicine products [31], pharmaceutical products [32], microbiology [33], and radiopharmaceutical activities [34].

Therefore, this study aimed to evaluate two different scenarios. The first scenario, which is traditional, considers only analytical uncertainty, and the second one considers measurement uncertainty, such as sampling uncertainty, in addition to analytical uncertainty. This study evaluated the importance of uncertainty information arising from the sampling process in conformity assessment applied to the physicochemical properties of fuels.

2. Materials and Methods

In this section, we present the metrological approaches used in this study: the uncertainty in measurements due to sampling processes, the application of uncertainty data in compliance evaluations, and the test methods used.

2.1. Evaluation of the Uncertainty Arising from the Sampling Process

The approach employed for estimating uncertainty in this study was the duplicate method [35]. To determine measurement uncertainty, it is essential to stratify both sampling and analytical sources. The variance among targets, the total amount of material that needs to be characterized, was established at 10% for the entire survey [36], with a sample size range of 8 to 12, with two samples collected from each target that expressed the between-sample variance. Finally, for each of the samples (S1 and S2), two separate measurements (A1 and A2) were conducted, of between-analysis variance, Figure 1.

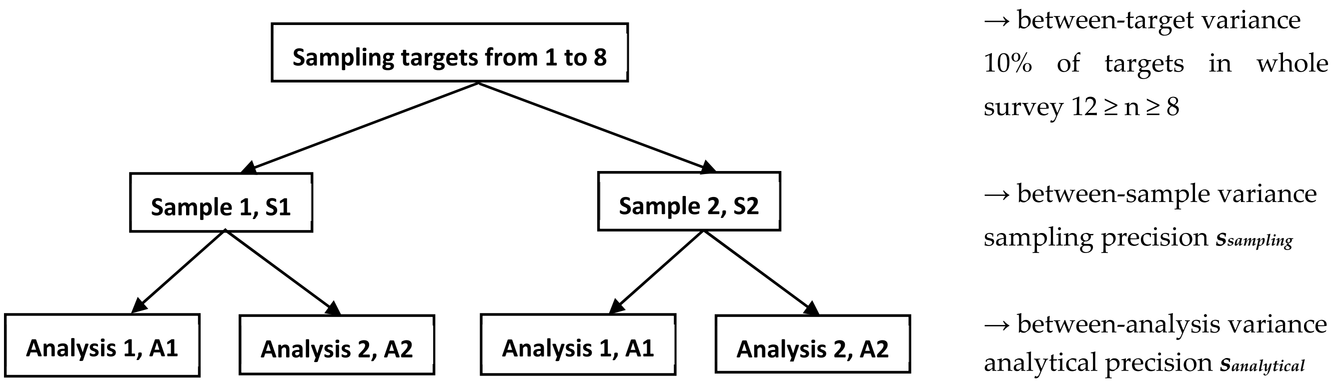

Figure 1. A balanced design.

In cases where the data followed a normal distribution, exhibited independence of residues, demonstrated homogeneity of variances (homoscedasticity), and were free from outliers, a traditional two-way analysis of variance (ANOVA) using a two-stage nested design was the statistical method applied [37], Table 1, considering *n* replicates of measurement, x_{ij}, *r* rows (Factor A), and *c* columns (Factor B).

Table 1. Algorithms for two-way ANOVA with interaction.

Source of Variation	Sum of Squares (*SS*)	Degrees of Freedom (ν)	Mean Square (*MS*)	*F*
Between S1S2 (A)	$\sum \overline{x}_{i.}^2/nc - \left(\overline{x}^2/nrc\right)$	$r-1$	SS_A/ν_A	MS_A/MS_R
Between-target (B)	$\sum \overline{x}_{.j}^2/nr - \left(\overline{x}^2/nrc\right)$	$c-1$	SS_B/ν_B	MS_B/MS_R
Interaction (I)	$SS_T - SS_A - SS_B - SS_R$	$(r-1)(c-1)$	SS_I/ν_I	MS_I/MS_R
Within or residual (R)	$\sum x_{ijk}^2 - \sum T_{ij}^2/n$	$rc(n-1)$	SS_R/ν_R	
Total (T)	$\sum x_{ijk}^2 - \left(\overline{x}^2/nrc\right)$	$rcn-1$		

From this ANOVA, estimates of the standard deviation for analytical and sampling uncertainty were determined using Equations (1) and (2), respectively:

$$\boldsymbol{s}_{analytical} = \sqrt{MS_{residual}} \tag{1}$$

$$s_{sampling} = \sqrt{\frac{(MS_{Between\ \mathrm{S1S2}} + MS_{Interaction}) - MS_{Within}}{2}} \tag{2}$$

where $s^2_{sampling}$ is the estimate of the sampling variance and $s^2_{analytical}$ is the estimate of the analytical variance.

2.2. *Measurement Uncertainty as a Parameter in Conformity Assessment*

Measurement uncertainty allows for reducing risks faced by producers (such as rejecting a conforming item) and risks encountered by consumers (like accepting a non-conforming item).

The acceptance zone was created by narrowing the tolerance interval on both ends using guard bands, denoted as g. This zone is bounded by the upper and lower specification limits (USLs and LSLs, respectively) and their corresponding acceptance limits. At a significance level of 5%, each guard band was determined by multiplying the standard uncertainty, u, by 1.64. The acceptance range can be defined as USL minus $1.64 \times u$ and LSL plus $1.64 \times u$, emphasizing the risk of false acceptance (consumer risk). Conversely, the acceptance range can also be described as USL plus $1.64 \times u$ and LSL minus $1.64 \times u$, highlighting the risk of false rejection (producer risk) [37]. The Eurachem guide edition [38] considers ILAC G8 [39] and JCGM 106 [40], which use ISO 14253-1 [41] assumptions, decision rules based on guard bands.

Histograms display the most probable value, associated uncertainties (both analytical uncertainty alone and the combination of analytical and sampling uncertainties), guard bands, and lower and upper acceptance limits. In this research, Monte Carlo simulations (MCSs) incorporating 100,000 pseudorandom values for the physicochemical properties of fuels, based on the mean experimental values and their standard uncertainties, were employed to evaluate the risk to consumers. The consumer risk was calculated using MCSs, carried out in an MS-Excel spreadsheet. A random generator with a Gaussian distribution was created using the MS-Excel function "=NORM.INV(RAND();$\boldsymbol{y_i}$;$\boldsymbol{u_{y_i}}$)", where $\boldsymbol{y_i}$ is the mean of measured values and $\boldsymbol{u_{y_i}}$ is its standard uncertainty for the $\boldsymbol{i^{th}}$ parameter.

2.3. *Test Methods*

A brass test cup, designed to specific measurements, was filled to the marked level with the test sample and sealed with a cover that met the required dimensions. The cup was then heated, and the specimen was agitated at predetermined speeds according to one of the three established methods (A, B, or C). At set intervals, an ignition source was introduced into the cup while stirring was paused, continuing until a flash was observed [6].

A monochromatic X-ray beam, possessing a wavelength that effectively stimulated the K-shell electrons of sulfur, was directed onto the test sample housed within a sample cell. The Kα fluorescence emitted by sulfur at a wavelength of 0.5373 nm was captured by a stationary monochromator (analyzer). The sulfur X-ray intensity, quantified in counts per second, was recorded using an appropriate detector and then translated into sulfur concentration (mg/kg) in the test sample through an analytical calibration curve [8].

The duration for a specific volume of liquid to pass through the capillary of a calibrated viscometer by the force of gravity was recorded, maintaining a consistent driving head and a carefully regulated temperature. The kinematic viscosity was calculated by multiplying the recorded flow time by the viscometer's calibration constant. Two separate measurements were required to arrive at a reliable kinematic viscosity result, and the final value is the average of these two acceptable results [14].

3. Results and Discussion

This section describes the experimental results, calculations used, and analytical and sampling uncertainties, as well as the use of this information in compliance assessment.

3.1. Experimental Results

Samples were collected at eight equidistant sampling points in the storage tanks using side-mounted samplers, Figure 2.

Figure 2. A tank equipped with side samplers.

Analytical and sampling uncertainties were calculated for the flash point in jet fuel using Pensalab equipment (model OptiFlash PM, Manufacturer: Herzog, City: Osnabrück, Country: Germany.), following a procedure based on ASTM D93 [6]. For the sulfur mass fraction in a gasoline-ethanol blend, a monochromatic wavelength-dispersive X-ray fluorescence spectrometer (MWDXRF), brand Shimadzu (model SINDIE-7039-GEN-3, Manufacturer: XOS, City: New York, Country: United States of America.), was used following a procedure based on ASTM D7039 [8]. For the kinematic viscosity in the diesel, Cannon-Fenske-type viscometer tubes for clear products were used based on ASTM D445 [14], Tables 2–4, respectively. These physicochemical parameters were gathered and assessed in laboratories within the Brazilian oil industry in 2024. All equipment was calibrated.

Table 2. Flash point in jet fuel, °C.

Sample	Target 1	Target 2	Target 3	Target 4	Target 5	Target 6	Target 7	Target 8
S1A1	46.0	50.0	47.0	52.0	44.0	46.5	50.5	45.0
S1A2	41.0	41.0	44.0	42.5	46.0	51.5	46.5	42.0
S2A1	42.0	46.5	40.5	43.5	40.0	42.5	40.5	45.0
S2A2	42.0	40.0	44.0	41.5	42.5	41.5	41.0	44.5

Table 3. Sulfur mass fraction in gasoline-ethanol blend, mg/kg.

Sample	Target 1	Target 2	Target 3	Target 4	Target 5	Target 6	Target 7	Target 8
S1A1	38.0	37.7	43.4	41.0	41.4	46.6	37.1	43.3
S1A2	43.3	42.8	39.9	41.8	40.0	40.8	43.2	43.8
S2A1	46.1	50.0	47.8	51.3	43.1	44.8	48.8	51.6
S2A2	46.6	43.2	38.7	45.6	45.1	47.9	39.7	45.1

Table 4. Kinematic viscosity at 40 °C in diesel, mm^2/s.

Sample	Target 1	Target 2	Target 3	Target 4	Target 5	Target 6	Target 7	Target 8
S1A1	4.483	4.373	4.605	4.297	4.430	4.572	4.412	4.528
S1A2	4.425	4.377	4.604	4.460	4.508	4.294	4.279	4.301
S2A1	4.016	3.866	4.089	4.368	4.079	4.267	4.273	4.157
S2A2	4.139	4.036	4.148	4.194	4.110	4.020	4.252	4.117

There were no outliers identified by the Grubbs test, the distribution was deemed Gaussian according to the Shapiro–Wilk test, the residuals demonstrated independence based on the Durbin–Watson test, and Bartlett's test indicated that the variances across different groups were homogeneous [42].

3.2. Evaluation of the Analytical and Sampling Uncertainties

The approach presented by Ref. [35] does not consider Type B evaluation of standard uncertainty. Since the aim of this study is to highlight the importance of the sampling contribution in comparison to the analytical contribution, the authors believe that using only Type A evaluation of standard uncertainty is not a significant issue, as Type B sources impact both approaches.

The analytical and sampling uncertainties were calculated using two-way ANOVA with interaction and Equations (1) and (2), Table 5.

Table 5. Analytical and sampling uncertainties.

	Analytical Uncertainty	Sampling Uncertainty	Measurement Uncertainty
Flash point (°C)	3.2	2.3	3.9
Sulfur mass fraction (mg/kg)	3.7	2.1	4.3
Kinematic viscosity (mm^2/s)	0.1007	0.2234	0.2445

The measurement uncertainty was calculated by the square root of the quadratic sum of the analytical and sampling uncertainties, considering only Type A standard uncertainties. All of them are expressed as standard deviations, that is, standard uncertainties.

Regarding the flash point in jet fuel, the analytical and sampling uncertainties are of the same order of magnitude. Concerning the sulfur mass fraction in the gasoline-ethanol blend, the contribution of the analytical uncertainty is greater than the contribution of the sampling uncertainty. On the other hand, the contribution of the sampling uncertainty is greater than the contribution of the analytical uncertainty for the kinematic viscosity in diesel.

3.3. Use of Analytical and Sampling Uncertainty Information in Compliance Assessment

The guard bands were calculated based on two scenarios: (i) considering only the analytical uncertainty and (ii) considering the analytical uncertainty plus the uncertainty arising from sampling.

Table 6 provides the mean measurement values, guard bands $1.64 \times u$, for a significance level of 5% and specification limits.

The acceptance range for the jet fuel's flash point was calculated by the LSL plus $1.64 \times \boldsymbol{u}$; however, for the sulfur mass fraction of the gasoline-ethanol blend and the kinematic viscosity of diesel, it was calculated by the USL minus $1.64 \times \boldsymbol{u}$. Since the sampling uncertainties were not negligible, different acceptance limits were reached, and consequently, distinct decisions regarding the compliance assessments were taken.

Table 6. Parameters used to assess compliance.

	Mean Value	Acceptance Limit Based on Analytical Uncertainty	Acceptance Limit Based on Analytical Uncertainty Plus Sampling Uncertainty	Specification Limit
Flash point (°C)	44.2	43.2	44.4	38 (TL)
Sulfur mass fraction (mg/kg)	43.7	43.9	42.9	50 (TU)
Kinematic viscosity (mm^2/s)	4.284	4.335	4.099	4.5 (TU)

TL = lower tolerance limit; TU = upper tolerance limit.

Figures 3–8 provide the histograms for the measurement values, the specification, and guard band limits, considering the p(AL)—probability density at the lower acceptance limit; p(AU)—probability density at the upper acceptance limit; AL—lower acceptance limit; AU—upper acceptance limit; TL—lower tolerance limit; and TU—upper tolerance limit.

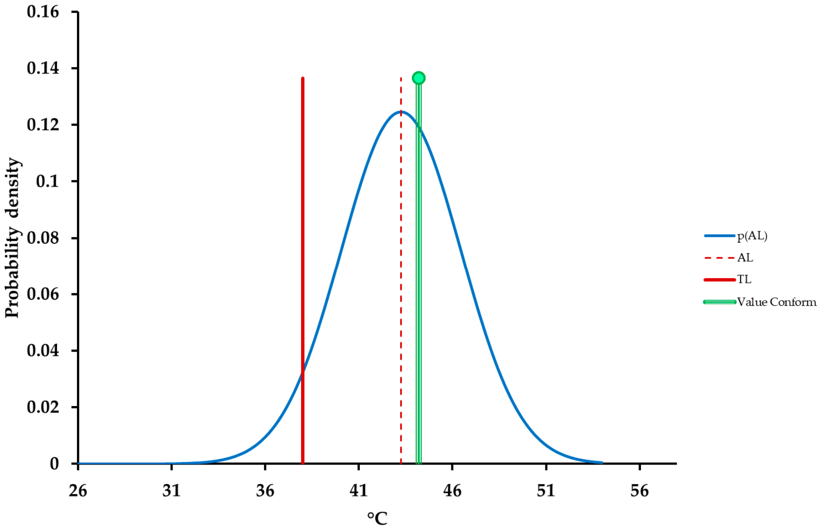

Figure 3. Histogram for the 5% significance level, flash point in jet fuel, considering only the analytical uncertainty.

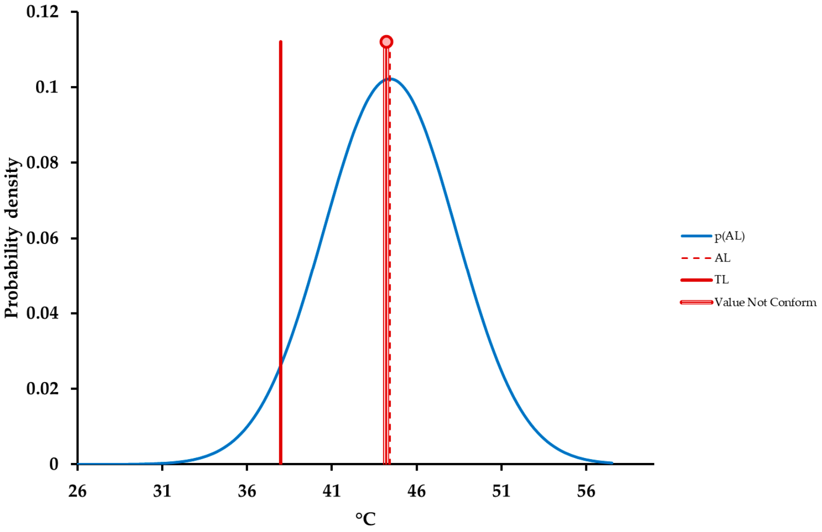

Figure 4. Histogram for the 5% significance level, flash point in jet fuel, considering the analytical uncertainty plus the uncertainty arising from sampling.

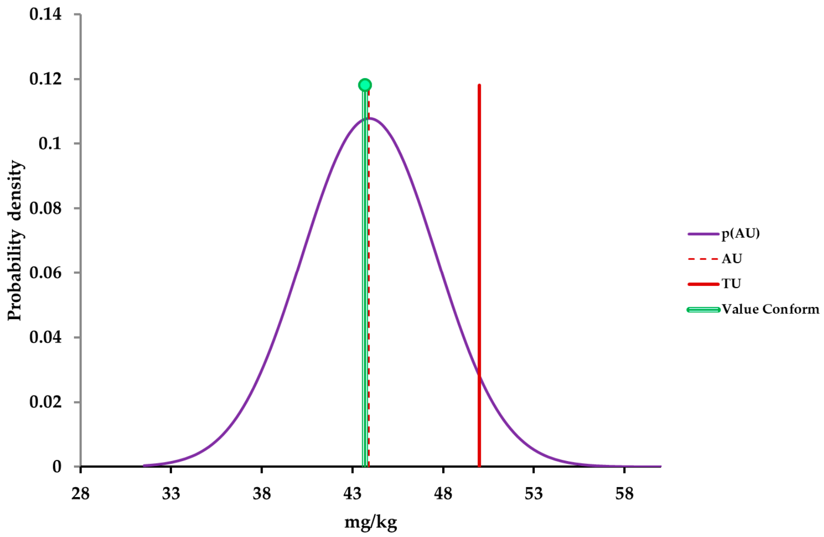

Figure 5. Histogram for the 5% significance level, sulfur mass fraction in the gasoline-ethanol blend, considering only the analytical uncertainty.

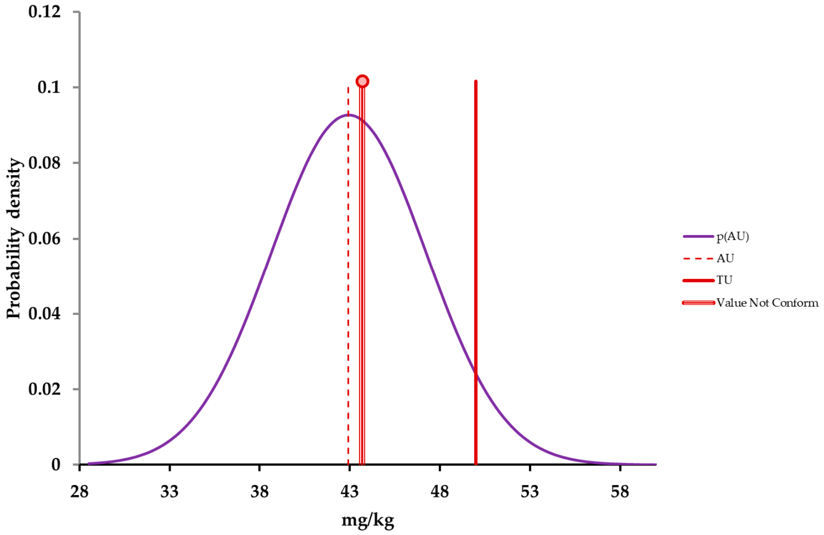

Figure 6. Histogram for the 5% significance level, sulfur mass fraction in the gasoline-ethanol blend, considering the analytical uncertainty plus the uncertainty arising from sampling.

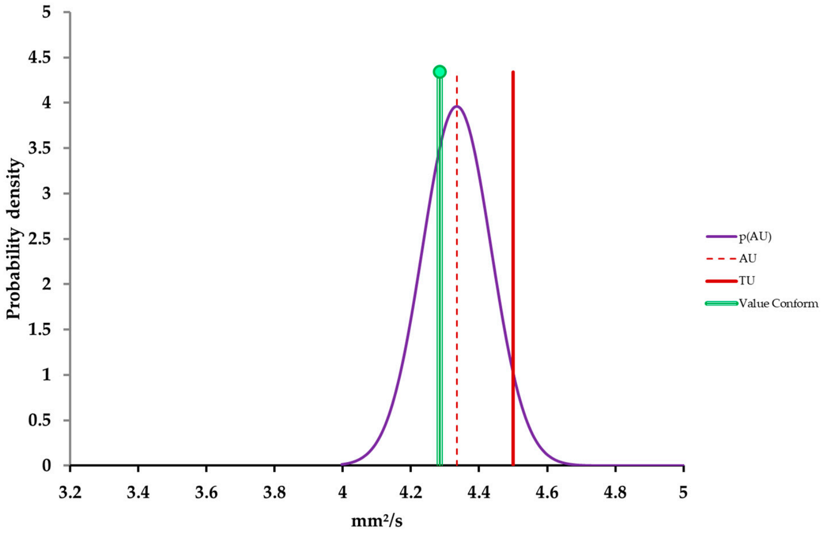

Figure 7. Histogram for the 5% significance level, kinematic viscosity in diesel, considering only the analytical uncertainty.

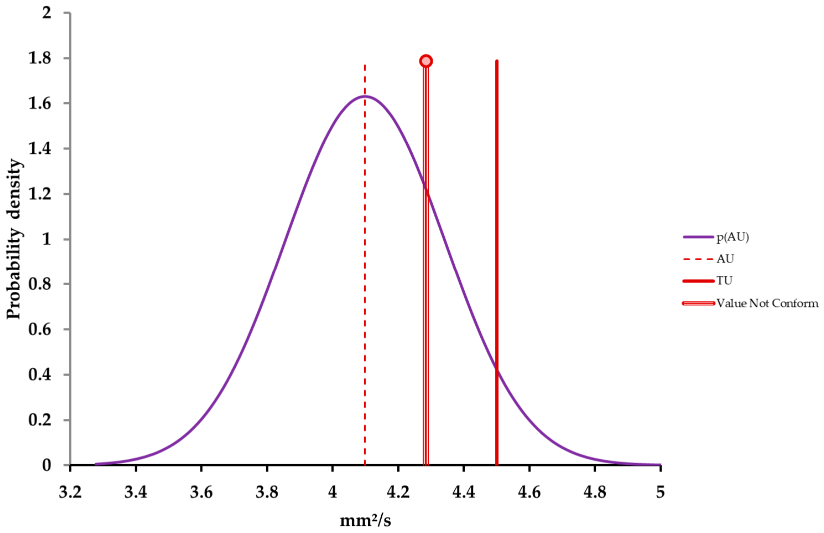

Figure 8. Histogram for the 5% significance level, kinematic viscosity in diesel, considering the analytical uncertainty plus the uncertainty arising from sampling.

The measured flash point value of the jet fuel was above the lower tolerance limit, suggesting compliance with the specification. Consequently, this presents an estimated consumer risk of 2.6% associated with this measurement value.

The measured flash point value of the jet fuel was below the lower tolerance limit, indicating non-compliance with the specification. Consequently, this presents an estimated consumer risk of 5.6% associated with this measurement value.

The measured sulfur mass fraction value of the gasoline-ethanol blend was below the upper tolerance limit, suggesting compliance with the specification. Consequently, this presents an estimated consumer risk of 4.4% associated with this measurement value.

The measured sulfur mass fraction in the gasoline-ethanol blend was above the upper tolerance limit, indicating non-compliance with the specification. Consequently, this measurement value associated with it presents an estimated consumer risk of 7.1%.

The measured kinematic viscosity in diesel was below the upper tolerance limit, suggesting compliance with the specification. Consequently, this presents an estimated consumer risk of 1.6% associated with this measurement value.

The measured kinematic viscosity in the diesel was above the upper tolerance limit, indicating non-compliance with the specification. Consequently, this presents an estimated consumer risk of 18.9% associated with this measurement value.

It was observed that the guard band values were significantly lower without the contribution of uncertainty arising from the sampling process. On the other hand, when the two uncertainty contributions were correctly considered, the guard bands assumed more realistic values, which caused the physicochemical properties of the fuels to be out of specification. Therefore, this study highlights the importance of connecting two approaches: the use of sampling uncertainty in addition to the traditional analytical uncertainty as information for conformity assessment. Nowadays, sampling uncertainty is rarely considered, which can underestimate measurement uncertainty and, consequently, mask the risk results for the consumer, as in the cases presented in this study.

4. Conclusions

This study successfully evaluated two distinct scenarios regarding the use of uncertainty information in compliance assessment. The first scenario, which is traditional and focused solely on analytical uncertainty, showed to be less rigorous than the second scenario, which incorporated measurement uncertainty, including sampling uncertainty alongside analytical uncertainty. In the three case studies highlighted, the evaluation of physicochemical properties of fuels that were out in the specification was enhanced by including sampling uncertainty, in contrast to the traditional method which only considers analytical uncertainty. This modification revealed that, although the properties appeared to be within specification when assessed solely on analytical uncertainty, they were, in fact, non-compliant.

The main drawback and obstacle to this practical implementation is that 32 trials must be performed to reliably assess the contribution of sampling, which can, at first glance, make the measurement process more expensive. However, when considering the supplier–customer relationship, this expense may be offset by the fact that there are more realistic predictions regarding the conformity assessment of the marketed product.

Future work can develop multivariate acceptance limits to minimize the risk of incorrect conformity decisions in the analysis of automotive fuels and consider the Type B evaluation of standard uncertainty, in addition to the Type A evaluation of standard uncertainty used in this study.

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