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### Fuel Smuggling Investigation: Simple Differentiation Between Brazilian and Venezuelan Fuels (Diesel Oil and Gasoline) Based on Their Biofuel Content by GC/MS and FTIR

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**Abstract.** Fuel smuggling across borders is a persistent criminal activity observed in numerous countries worldwide. In Brazil, particularly, illegal fuel imports, mainly diesel oil and gasoline, primarily originate from Venezuela. The main driver behind this criminal activity is the significant price gap, as Venezuela currently (October 2024) offers the cheapest diesel oil and the third cheapest gasoline worldwide. Nowadays, Brazilian diesel and gasoline are priced at 270 and 32 times higher, respectively, than Venezuela's prices, making this criminal enterprise highly lucrative. Therefore, this Technical Note aims to provide details of analyses by GC/MS (Gas Chromatography/Mass Spectrometry) and FTIR (Fourier Transform Infrared Spectroscopy) to differentiate between Venezuelan and Brazilian fuels (diesel oils and gasolines), in order to contribute to smuggling investigations. This is achieved by identifying the presence or absence of mandatory additives, such as biodiesel and ethanol, which are legally required in Brazilian fuels but absent in Venezuelan fuels. The methodology presented can be applied to the most common cases found in Brazil, which are those involving smuggling without subsequent adulteration. Considering that GC/MS and FTIR are widely available instrumental techniques found in many forensic laboratories around the world, this approach could be useful in other countries facing this type of smuggling at their borders, especially when fuels exhibit differences based on the biofuels added to their composition. Therefore, there is a strong interest in disseminating this simple methodology.

**Keywords:** Fuel smuggling; Gasoline; Diesel oil; GC/MS; FTIR.

## 1. Introduction

Fuel smuggling across borders is a recurring crime in various countries around the world<sup>1-4</sup>. Specifically in Brazil, fuels, primarily diesel oil and gasoline, are brought into the country illegally, mainly from Venezuela<sup>1</sup>.

The main motivating factor for this type of crime is the substantial price difference. According to a recent update from *globalpetrolprices.com* (October 2024), Venezuela, the primary source of fuel smuggling into Brazil (based on fuel analysis experience in smuggling cases handled by the National Institute of Criminalistics), offers the cheapest diesel oil (US\$ 0.004 L<sup>-1</sup>) and the third cheapest gasoline (US\$ 0.035 L<sup>-1</sup>) globally<sup>5</sup>. Meanwhile, in Brazil, according to the same source, the prices of both fuels, diesel (US\$ 1.082 L<sup>-1</sup>) and gasoline (US\$ 1.106 L<sup>-1</sup>), are much higher. Therefore, Brazilian diesel and gasoline are priced at 291 and 33 times higher, respectively, than Venezuela's prices, making this type of crime highly profitable.

Fuel smuggling presents a substantial threat not only to the economy but also to the environment and public health. Environmentally, the circulation of unregulated fuels can lead to significant ecological damage, with the potential for spills and contamination in sensitive areas, as the conditions of transportation and storage do not comply with necessary legal requirements due to the inherently clandestine nature of such operations. Furthermore, smuggled fuels often fail to meet established safety and quality standards, resulting in increased pollutant emissions and contributing to air and water degradation. Economically, fuel smuggling undermines the competitiveness of the legitimate market, impacts fiscal revenues, and jeopardizes national energy security. Socially, the practice of smuggling not only exposes individuals to the health risks associated with low-quality fuels but also fosters violence and organized crime, perpetuating a cycle of illegality and inefficiency in public policy enforcement<sup>6-9</sup>. Given these factors, it is imperative that robust strategies be implemented to combat fuel smuggling, including the application of analytical techniques such as those discussed in this study.

According to the Brazilian Federal Constitution, in the first paragraph of Article 144, the crime of smuggling is specifically listed as one of the responsibilities of the Brazilian Federal Police<sup>10</sup>. Therefore, investigations into fuel smuggling always involve collecting samples for laboratory analysis, aiming to contribute to the conclusion of the case. Thus, forensic chemistry is essential in this type of investigation.

The routine at the National Institute of Criminalistics of the Brazilian Federal Police shows that diesel oil and gasoline are the two types of fuels smuggled from Venezuela to Brazil. To understand the differences between the fuels of these two countries, it is interesting to observe the related Brazilian legislation. Brazilian gasoline, for over 80 years, has been blended with ethanol, starting in 1931 compulsorily with at least 5% anhydrous ethanol, currently standing at 27% ethanol (E27)<sup>11-13</sup>. Regarding Brazilian diesel oil, in accordance with the requirement for biodiesel blending, it began in 2008 at 2%, gradually increasing to the current 14% (B14)<sup>11,14,15</sup>. However, for the corresponding Venezuelan fuels, these additives are not added<sup>16</sup>, and this difference can be leveraged through chemical analyses to distinguish between them.

GC/MS (Gas Chromatography/Mass Spectrometry) and FTIR (Fourier Transform Infrared Spectroscopy) are highly effective and widely used techniques for fuel analysis<sup>17-24</sup>. GC/MS is a powerful tool for separating components in complex mixtures through gas chromatography and identifying them with mass spectrometry. This makes it ideal for analyzing fossil fuels, which consist of a wide range of components<sup>17-20</sup>. The analysis of their chromatographic profiles allows for the differentiation of various fuel types and the identification of additives such as ethanol and biodiesel, which are central to this study. FTIR identifies functional groups in molecules by analyzing their characteristic infrared absorption spectra, providing a rapid and reliable method for detecting components like ethanol and biodiesel. Additionally, its portability makes it particularly useful in field operations<sup>25-28</sup>. Several handheld FTIR devices are available, enabling efficient on-site screening in cases of suspected fuel smuggling. These preliminary analyses can subsequently be complemented by more detailed laboratory examinations. Both techniques are widely accessible in forensic laboratories worldwide and in Brazil, where they play a crucial role not only in fuel analysis but also in diverse forensic applications, including the investigation of drugs, pharmaceuticals, explosives, and pesticides.

Previous work presents a study related to gasoline adulteration with alcohol, including Venezuelan gasoline, through the assessment of standards samples and the use of chemometric tools, utilizing FTIR analysis<sup>1</sup>. This study is quite useful for cases of gasoline smuggling followed by adulteration with ethanol addition. However, based on the casework reviewed at the National Institute of Criminalistics, such

instances of adulteration aimed at bypassing inspection are exceedingly rare. Similarly, diesel oil adulteration with biodiesel after smuggling is virtually nonexistent.

Therefore, for the vast majority of cases, analyses identifying the absence of ethanol in gasoline and biodiesel in diesel oil are generally sufficient to provide the necessary response within the context of investigations into Venezuela-Brazil fuel smuggling, without the need for Venezuelan fuel standards or the use of more complex techniques, which is necessary for the application and updating of the methodology of the previously mentioned study<sup>1</sup>.

Hence, this Technical Note aims to provide details of analyses by GC/MS and FTIR to differentiate between Venezuelan and Brazilian diesel oil and gasoline, readily applicable for most cases, which are those involving smuggling without subsequent adulteration. Given that GC/MS and FTIR are highly common instrumental techniques found in many forensic laboratories around the world, this approach could be useful in other countries facing this type of smuggling at their borders, especially when fuels exhibit differences based on the biofuels added to their composition. Therefore, the dissemination of this simple methodology is of interest.

## **2. Materials and methods**

### **2.1 Reagents and materials**

In this study, Brazilian standard fuels obtained from the National Agency of Petroleum, Natural Gas and Biofuels (ANP) were used: Type C gasoline, Type B S10 diesel, and B100 Biodiesel. Additionally, Venezuelan fuels (gasoline and diesel oil) from seizures made by the Brazilian Federal Police were also utilized.

For comparisons with the obtained results, a reference standard of n-alkanes (C<sub>7</sub> - C<sub>40</sub>), 1000 mg L<sup>-1</sup>, was purchased from Sigma-Aldrich® (UNSPSC code: 41116107). From this, a 20 mg L<sup>-1</sup> solution was prepared for use. Ethanol 99.8% was acquired from Dinâmica® Química Contemporânea Ltd. Ultrapure water (18,2 MΩ cm at 25 °C) obtained from a Millipore Direct-Q5 purification system. Grade 5 Helium was supplied by White Martins Gases Industriais Ltd.

### **2.2. Sample preparation**

Except for the procedures described in the next paragraph for two specific samples, the analyses conducted in this study did not require any sample preparation for either

the GC/MS and FTIR techniques. For GC/MS, the samples were directly transferred to appropriate vials for analysis, while for FTIR, fuel samples were continuously placed onto the ATR (attenuated total reflectance) crystal using a Pasteur pipette until the spectrum was acquired.

For the FTIR analysis of Brazilian Type C gasoline without ethanol, a simple preparation was conducted. The ethanol present in this gasoline was extracted by adding water according to the procedure outlined in NBR 13992 standard (Brazilian Standard 13992:1997, titled "Analysis of Fuels - Determination of Ethanol Content in Gasoline")<sup>29</sup>, with the organic phase then separated and directly subjected to FTIR analysis. For GC/MS analysis, the B100 biodiesel sample was diluted in ethanol to achieve a suitable concentration for the method.

## 2.3. Instrumentation

### 2.3.1. Gas chromatography/mass spectrometry (GC/MS)

For the GC/MS analysis, each fuel sample was poured directly into the GC/MS vials. They were then analyzed using two different methods, one for diesel oil-related analyses and the other for gasoline-related analyses.

The GC/MS analysis were performed on an Agilent 6890N gas chromatograph coupled to an Agilent 5973 mass selective detector and Agilent 7683B autosampler. Table 1 presents the chromatographic parameters used in the GC/MS analysis of the fuel oil and gasoline samples, with the column: RXi-5MS methyl siloxane, 30 m × 250 μm (i.d.) × 0.25 μm film thickness for both.

**Table 1.** Chromatographic conditions used in the GC/MS analysis of the fuel samples.

Parameters	Diesel	Gasoline
Injection volume	0.2 μL	0.2 μL
Injection port temperature	250 °C	230 °C
Injection mode	Split 140:1	Split 100:1
Initial temperature	50 °C	50 °C
Initial time	0.00 min	0.00 min
Rate	15 °C min <sup>-1</sup>	15 °C min <sup>-1</sup>
Final temperature	300 °C	300 °C
Final time	11.00 min	0.00 min
Carrier gas	Helium (0.9 mL min <sup>-1</sup> )	Helium (0.9 mL min <sup>-1</sup> )

Data were analyzed using Agilent GC/MSD ChemStation version 17 software for Enhanced Data Analysis, and all the mass spectra of the analytes were compared with spectra in the NIST 17 MS Database (Agilent Technologies), using MS Search Program v.2.3 (Agilent Technologies).

### **2.3.2 Fourier transform infrared (FTIR)**

Fuel samples were analyzed using a Thermo Scientific Nicolet iS10 FTIR Spectrometer equipped with an ATR (attenuated total reflectance) accessory and a DTGS detector operating at room temperature. The samples were directly analyzed on the ATR accessory. Spectra were obtained from 16 co-added scans measured with a resolution of 4  $\text{cm}^{-1}$  in the 4000-650  $\text{cm}^{-1}$  range. Spectra collection and analysis were performed using OMNIC 8.1.0.10 software (Thermo Fisher Scientific, Waltham, MA, USA).

## **3. Results and discussion**

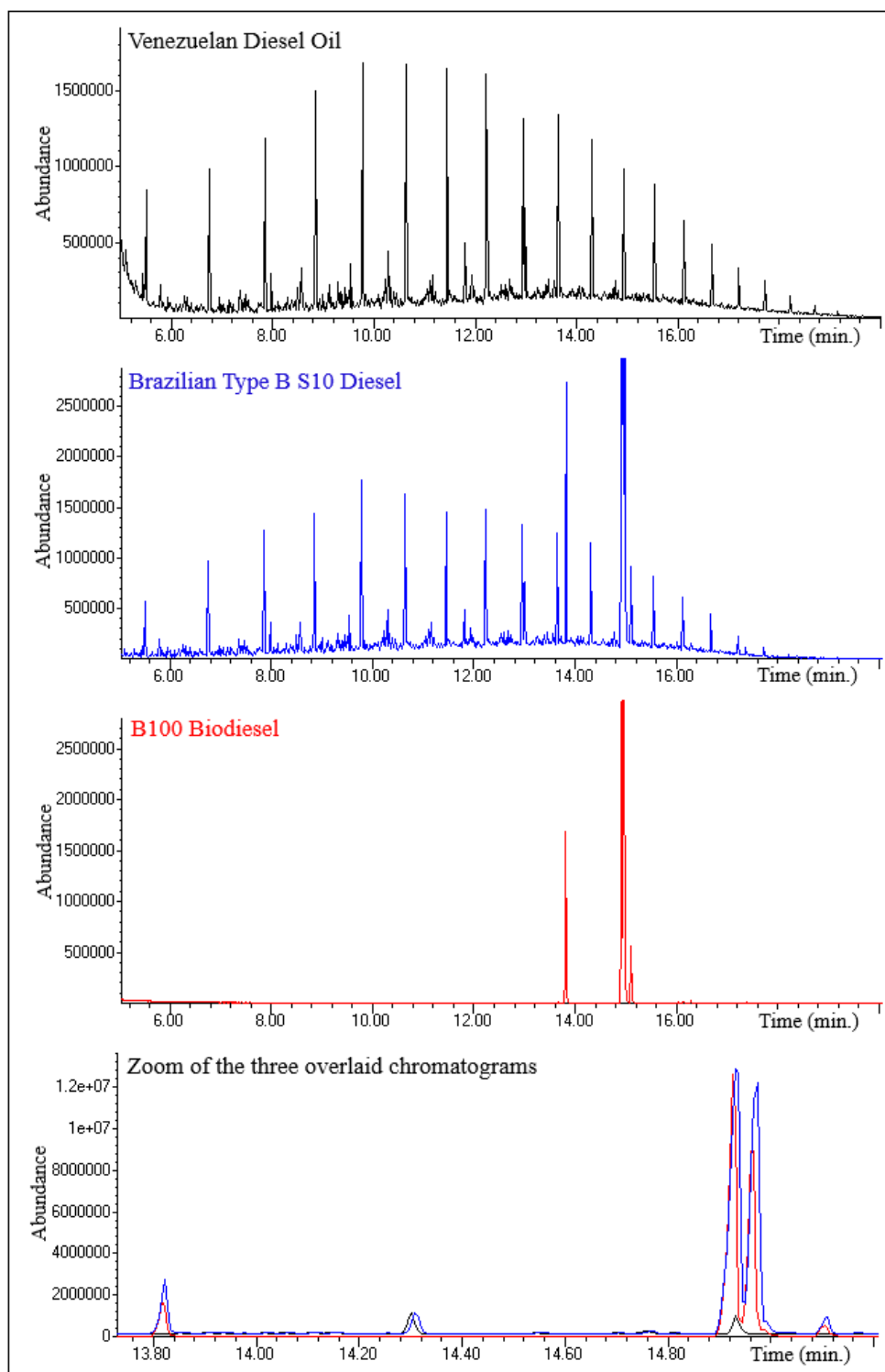
### **3.1 Diesel oil**

Concerning diesel oil, the primary objective of this study is to distinguish between Brazilian and Venezuelan fuels primarily based on the presence or absence of biodiesel, particularly within the context of smuggling from Venezuela. Figures 1 and 2 showcase the results derived from GC/MS analysis conducted on both Brazilian and Venezuelan diesel oils, along with B100 biodiesel.

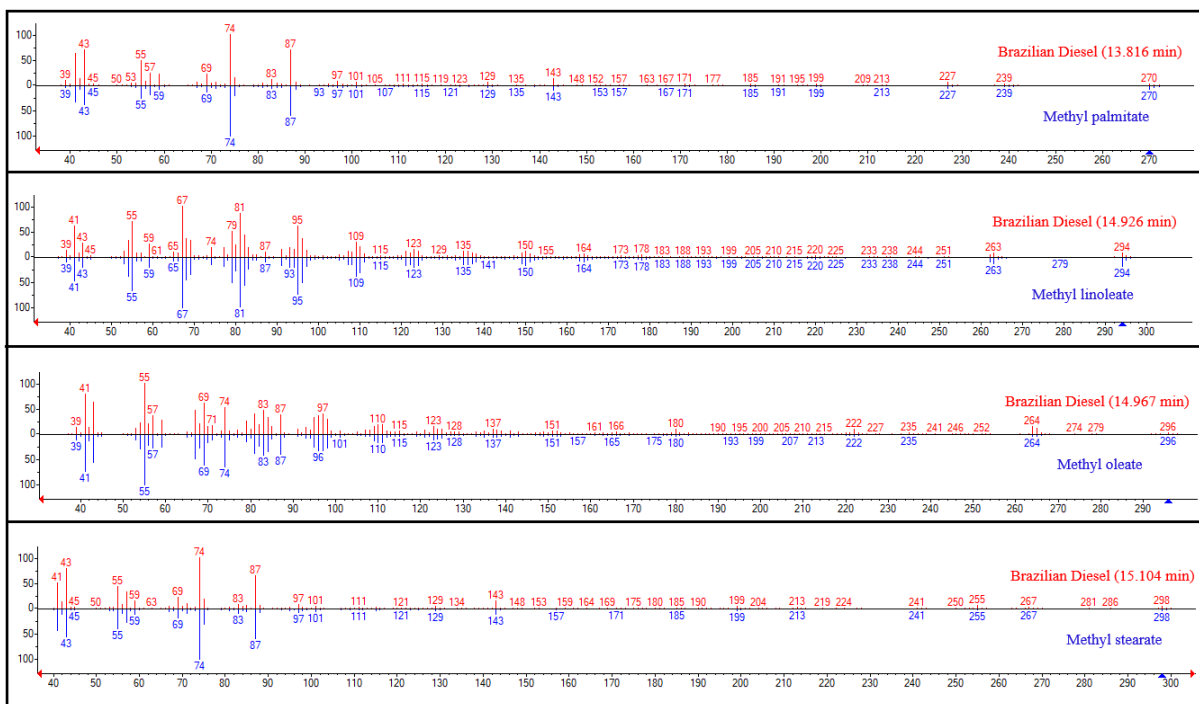
These findings unveil several characteristic peaks of diesel oil, corresponding to its distinctive hydrocarbons, in both diesel samples. Moreover, the presence of biodiesel peaks at 13.816, 14.926, 14.967, and 15.104 minutes (respectively methyl palmitate, methyl linoleate, methyl oleate, and methyl stearate, as evidenced by mass spectra comparisons in Figure 2) can be observed in Brazilian diesel, while absent in Venezuelan diesel, as better perceived in the magnified section of Figure 1. These results were derived from both retention time and mass spectra comparisons of the respective peaks. The aforementioned fatty acid methyl esters (FAME) are consistent with the composition of the analyzed B100 biodiesel <sup>30</sup>.

Regarding the FTIR analyses, the primary aim remains consistent: to ascertain the presence or absence of biodiesel in diesel oil, particularly within the context of smuggling from Venezuela. Figure 3 presents the findings garnered from FTIR

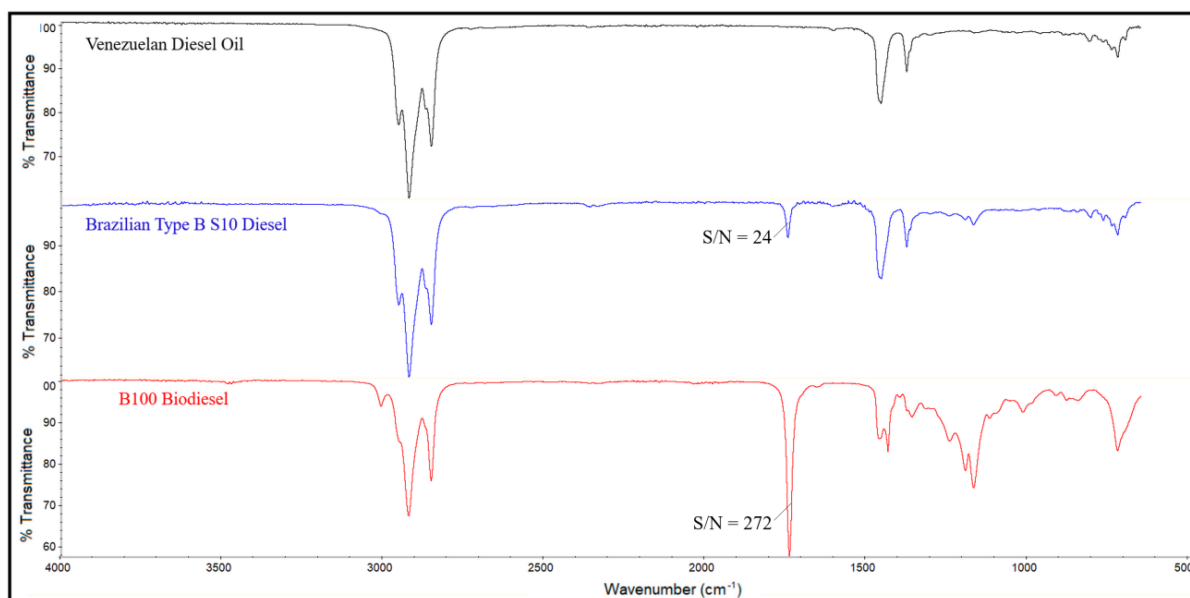
analyses conducted on both Brazilian and Venezuelan diesel oils, along with B100 biodiesel.



**Figure 1.** Comparison of chromatograms, from top to bottom, of Venezuelan diesel oil (black line), Brazilian Type B diesel oil (blue line), and B100 biodiesel (red line). Lastly, the zoomed-in view of the three overlaid chromatograms in the region of interest for B100 biodiesel.



**Figure 2.** Comparison, from top to bottom, between the mass spectra corresponding to the peaks at 13.816, 14.926, 14.967, and 15.104 minutes from Brazilian Type B diesel oil and the mass spectrum of methyl linoleate, methyl oleate, methyl palmitate, and methyl stearate, respectively, from the NIST 2.3 mass spectral library.



**Figure 3.** FTIR spectra of Venezuelan diesel oil (black line) compared to the Brazilian Type B diesel oil (blue line) and B100 biodiesel (red line).

These results indicate a high correlation between Brazilian and Venezuelan diesel oils, with the main difference observed at  $1741\text{ cm}^{-1}$ , precisely in the region of interest in this case, corresponding to the Carbonyl C=O stretching band of methyl



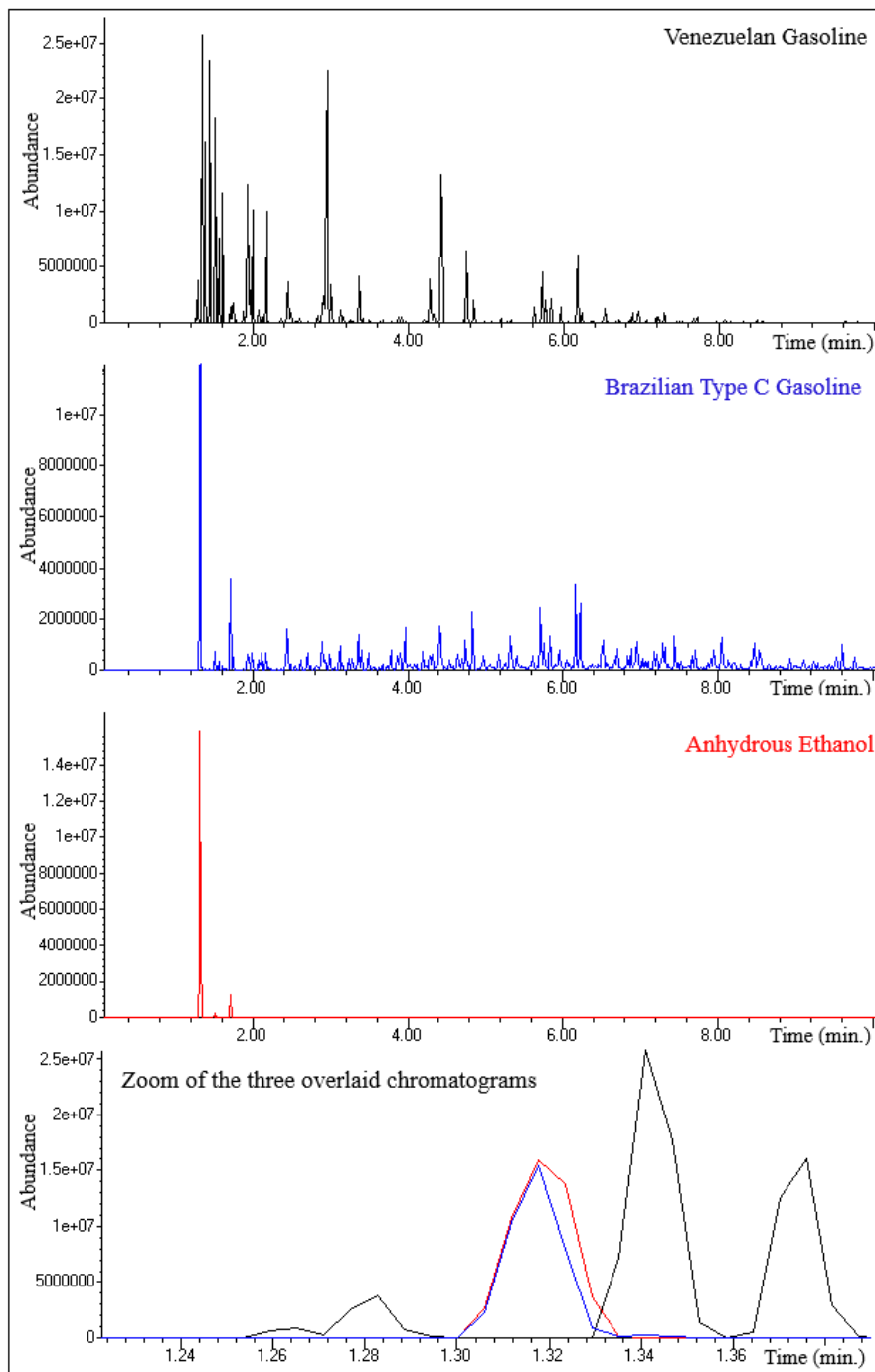
esters composing B 100 biodiesel. The signal-to-noise ratios (S/N) for the signals at  $1741\text{ cm}^{-1}$  are indicated in Figure 3. Therefore, the absence of the band at  $1741\text{ cm}^{-1}$ , in the context of diesel oil smuggling from Venezuela, is inconsistent with diesel oil commercially available in Brazil, indicating compatibility with Venezuelan diesel oil. The remaining bands are primarily characteristic of diesel hydrocarbons, such as the Aliphatic C-H stretching ( $2953\text{ cm}^{-1}$ ,  $2921\text{ cm}^{-1}$ , and  $2852\text{ cm}^{-1}$ ) and  $\text{CH}_2$  bending band ( $1456\text{ cm}^{-1}$  and  $1377\text{ cm}^{-1}$ ).

### 3.2 Gasoline

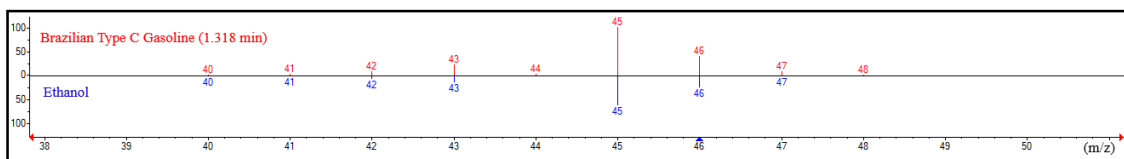
Regarding gasoline, this study aims to primarily differentiate between Brazilian and Venezuelan fuels based on the presence or absence of ethanol within the context of smuggling from Venezuela. Thus, Figures 4 and 5 display the results obtained through GC/MS analysis of both Brazilian and Venezuelan gasolines, as well as anhydrous ethanol.

These results reveal various characteristic peaks of gasoline, corresponding to its characteristic hydrocarbons, in both gasoline samples. Additionally, it is possible to observe the presence of the ethanol peak at 1.318 minutes in Brazilian gasoline and its absence in Venezuelan gasoline, as further detailed in the zoomed-in section of Figure 4. These results were obtained through both retention time and mass spectra comparisons of the respective peaks.

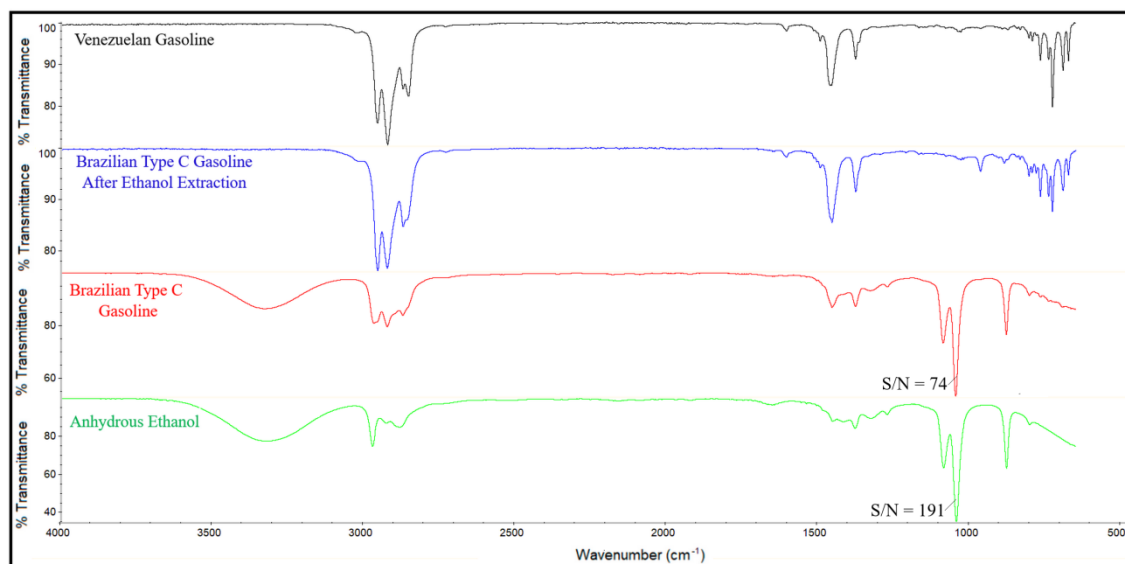
Regarding the FTIR analyses, the objective is essentially the same, namely, to verify the presence or absence of ethanol in gasoline within the context of smuggling from Venezuela. Thus, Figure 6 displays the results obtained through FTIR analyses of both Brazilian and Venezuelan gasolines, as well as anhydrous ethanol. Additionally, the organic phase obtained after ethanol extraction from gasoline Type C was subjected to analyses under the same conditions.



**Figure 4.** Comparison of chromatograms, from top to bottom, of Venezuelan gasoline (black line), Brazilian Type C gasoline (blue line) and anhydrous ethanol (red line). Lastly, the zoomed-in view of the three overlaid chromatograms in the region of interest for ethanol.



**Figure 5.** Comparison between the mass spectra of the peak at 1.318 minutes from Brazilian Type C gasoline and the mass spectrum of ethanol from the NIST 2.3 mass spectral library.



**Figure 6.** FTIR spectra of Venezuelan gasoline (black line) compared to the Brazilian Type C Gasoline after ethanol extraction (blue line), Brazilian Type C gasoline (red line) and anhydrous ethanol (green line).

These results show that Type C gasoline exhibits a higher correlation with anhydrous ethanol, with the main characteristic bands being O-H stretching vibrations ( $3324\text{ cm}^{-1}$ ), C-H bond stretching ( $2972\text{ cm}^{-1}$  and  $2883\text{ cm}^{-1}$ ) and stretching vibrations of the C-O bond ( $1379\text{ cm}^{-1}$ ,  $1087\text{ cm}^{-1}$  and  $1045\text{ cm}^{-1}$ ). The signal-to-noise ratios (S/N) for the signals at  $1045\text{ cm}^{-1}$  are indicated in Figure 6.

However, after ethanol removal, the correlation with Venezuelan gasoline becomes evident, with the main characteristic bands being C-H bond stretching ( $2955\text{ cm}^{-1}$ ,  $2924\text{ cm}^{-1}$ , and  $2871\text{ cm}^{-1}$ ) and  $\text{CH}_2$  bending bands ( $1456\text{ cm}^{-1}$  and  $1377\text{ cm}^{-1}$ ). Thus, the presence of ethanol in gasoline can also be easily observed by this technique.

It is important to emphasize that in cases of adulteration with ethanol and biodiesel, respectively added to Venezuelan gasoline and diesel oil, at the exact percentage required by regulations, the methodology presented in this study is not applicable. In these rare situations, the use of reference standards for profile comparisons and/or the application of chemometric tools, as demonstrated in previous studies<sup>1</sup>, becomes necessary.

#### 4. Conclusion

The results of the analyses clearly demonstrate that the presence or absence of biofuels, specifically ethanol and biodiesel, is a key differentiator between Brazilian and Venezuelan fuels. In the majority of cases, the absence of these mandatory

additives in Venezuelan fuels allowed for straightforward differentiation using both GC/MS and FTIR techniques. While the study focused on the comparison between fuels from Venezuela and Brazil, the approach is applicable to other countries with similar smuggling challenges. The analysis shows that the detection of biofuels can be achieved using either GC/MS or FTIR, though the quantification of biofuels may be necessary in cases where both countries' fuels contain biofuels in varying concentrations.

These instrumental techniques are widely available in forensic chemistry laboratories, as they are applicable to a range of other analyses, making them a reliable tool for rapid differentiation in fuel smuggling investigations. Therefore, this methodology offers an effective, simple, and readily applicable approach for combating fuel smuggling, contributing significantly to forensic investigations, both in Brazil and internationally.

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