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Validation of Chromatographic Methods in the Detection of Cocaine and Diluents in Drugs Seized by the Civil Police in the State of Espírito Santo

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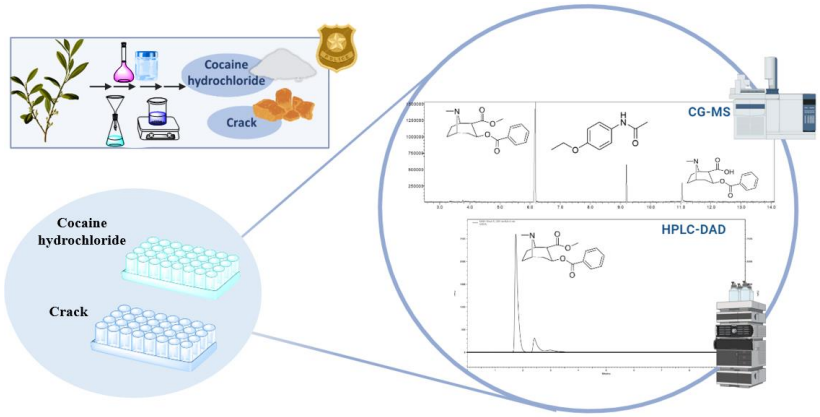
Over the years, police have seized tons of cocaine in the form of cocaine hydrochloride and crack, which are often adulterated by traffickers. This work aims to use GC/MS and HPLC to determine the limit of detection (LoD) and limit of quantification (LoQ) of 10 seized samples of crack and 10 of cocaine hydrochloride. All samples were provided by the Civil Police of Espírito Santo State (CP/ES) and each sample was analyzed in triplicate using GC/MS and HPLC-DAD. For the first technique, a correlation coefficient (R) equals 0.9939 was achieved, while for HPLC a R = 0.9984 was obtained for crack and R = 0.9907 for cocaine. Due to the validated methods, it was possible to determine the LoD and LoQ for the cocaine present in the samples, in addition to identifying some diluents, such as caffeine and phenacetin.

Graphical abstract



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1. Introduction

The amazon region is highly related to coca plant cultivation, which happens at high altitudes (450 to 1800 m above the sea), for cocaine production. Also, it is associated with cocaine trafficking between Brazil and Bolivia, Colombia and Peru. This region alone produces around 1,093,750 kt of cocaine per year [1]. In Brazil, 2021, 93.4t of cocaine (cocaine hydrochloride, base paste and crack) were seized. Also, in the first 6 months of 2022, 44.4t of cocaine (cocaine hydrochloride, base paste and crack) were seized [2].

Cocaine **1** is one of the main tropane alkaloids of the coca plant, which can also contain other alkaloids, such as Cinnamoylcocaine (cinnamoylcocaine) **2**. Beyond that, the coca plant also has a set of typical tropine derivatives lacking the methyl ester group, such as tropacocaine **3**. In addition, there are α-truxilin **4** and β-truxilin **5**, formed from truxilins, which have two acidic portions. α-truxylic and β-truxinic acids

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are obtained by the cycloaddition of two cinnamic acid units (Figure 1) [3].

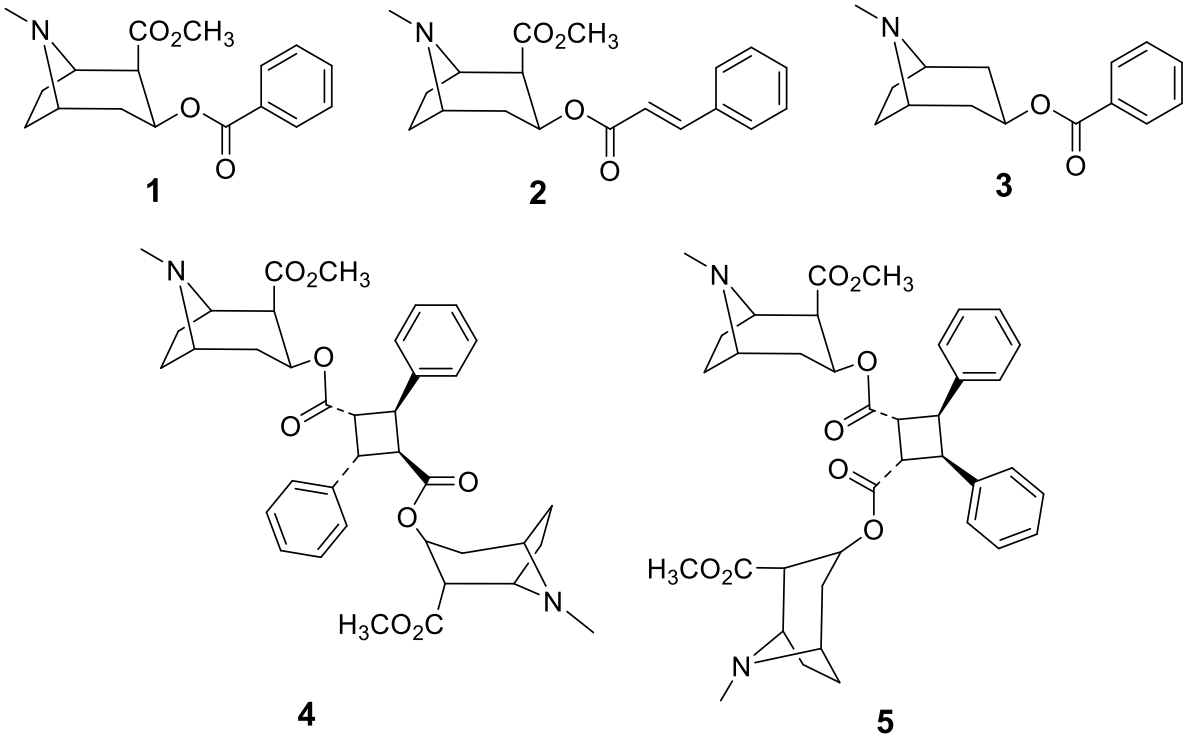

Fig. 1. Chemical structure of tropane alkaloids found in coca plants. Source: adapted from Dewick, 2002 [3].

Cocaine is found as cocaine hydrochloride or cocaine base (Figure 2). The production of cocaine hydrochloride begins with an extraction of the alkaloid from the coca plant with H_2SO_4, $CaCO_3$ and kerosene, resulting in the base paste. Its purification is conducted using $KMnO_4$, followed by filtration yielding the cocaine base. Finally, the cocaine base is crystallized with NH_4OH, acetone and HCl, resulting in cocaine hydrochloride [4].

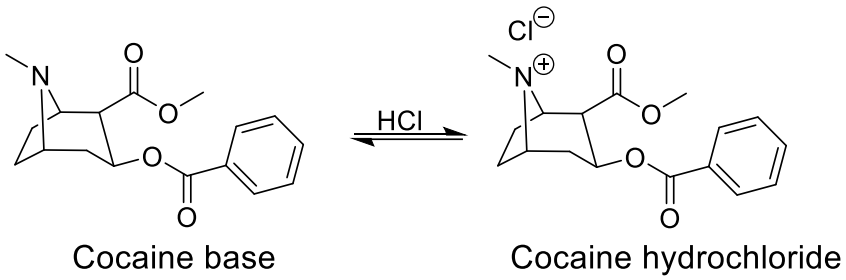

Fig. 2. Structures of cocaine. Source: adapted Vargas and Talhavini, 2000 [5].

There is also another method to produce cocaine from coca plants. The coca leaves are treated with a basic solution (usually sodium carbonate) and kerosene, then several people stomp on the mixture to help the solvent extract the cocaine alkaloids. The water and leaves are separated and the alkaloids are extracted from the kerosene into a dilute acid solution, this results in a precipitate, which is the coca paste. Then, the coca paste is treated with sulfuric acid and water, potassium permanganate solution is added to eliminate unwanted compounds, later the mixture is filtered and the liquid is treated with ammonia to form another precipitate, which is the cocaine base. The cocaine base can be transformed into hydrochloride cocaine by diluting it in ether or acetone, filtering it and treating it with HCl, the precipitate formed is dried. This precipitate is the hydrochloride cocaine.[6]

Cocaine hydrochloride and crack (cocaine base) are easily found on European streets. Cocaine hydrochloride is frequently available on the market and is often referred to as cocaine powder. Most of the crack production in Europe comes from cocaine hydrochloride, through a process called basification [4].

Meanwhile, in South America, smokable cocaine products (coca paste and cocaine base) are more available to consumers, and they are produced from intermediate products in the cocaine manufacturing process. Concerning cocaine hydrochloride, one intermediate product is the 'PBC', but it can be sold with different names, like 'merla' 'basuco', 'paco' and 'crack' [4].

Crack is considered one of the most dangerous drugs due to its powerful central nervous system stimulant properties, making it highly addictive. The effect of crack and its dependence is faster than other forms of cocaine [7]. When cocaine, in the form of hydrochloride, is administered

intranasally, its effects last between 30 and 60 minutes, but when administered intravenously, this time reduces to 10 to 20.[8] Crack, on the other hand, is smoked in a pipe and its effects lasts for 5 to 10 minutes. Due to the shorter effect time, crack is more addictive than cocaine [8, 9]. Furthermore, crack has some diluents such as caffeine, phenacetin, lidocaine, levamisole, hydroxyzine, benzocaine, and procaine [10].

In the literature, several ways of determining and quantifying a series of compounds are reported, using different methods and equipment. Maldaner et al used GC-FDI to validate a method for the simultaneous analysis of tropane alkaloids (anhydroecgonine methyl ester, anhydroecgonine, methylecgonine, tropacocaine, norcocaine, N-formylcocaine, trimethoxycocaine, ecgonine, benzoylecgonine, trans- and cis-cinnamoylcocaine). The method was used to detect alkaloids in eleven samples of cocaine seized by the Brazilian police. As a result, it was possible to notice that the cocaine samples were probably prepared from different coca leaves and origins (Peru, Bolivia or Colombia).[11]

To investigate the composition of heroin and cocaine seized by the Luxembourg police during 2019 and 2020, Burmaud et al evaluated these samples. All the analyzes were carried out using GC/MS, HPLC-UV and LC-Q-ToF. The diluents found in the cocaine samples were levadamisole, phenacetin and caffeine. Meanwhile, for the heroin samples paracetamol and caffeine were detected.[12]

One of the most common practices in quantification is method validation, which is necessary to guarantee the quality of results. According to ANVISA, method validation must follow some parameters, such as linearity, detection limit and quantification limit. Linearity is the ability to demonstrate the response of the analyte at different concentrations, within the presented method. It is recommended that there be at least 5 different concentrations of independently prepared solutions, which can be obtained from dilution of a stock solution. The results must be expressed in a graphical representation, obtaining the equation of the regression line of y in x, using the least squares method. Therefore, the linear association must be evaluated using the correlation coefficient (R) and coefficient of determination (R^2). [13]

The limit of detection (LoD) is the lowest quantity of analyte that can be detected on a sample. In instrumental methods, like chromatographic ones, it is possible to obtain the LoQ by calculating three times the residual standard deviation of the regression line by the inclination from the analytic curve. Meanwhile, the limit of quantification (LoQ) is the lowest quantity of analyte on a sample that can be precisely and accurately determined under experimental conditions. The LoQ can be calculated by ten times the residual standard deviation of the regression line by the inclination from the analytic curve. [13]

Therefore, this work aims to compare two different techniques: Gas Chromatography/Mass Spectrometry (GC/MS) and High Performance Liquid Chromatography with diode array detection (HPLC-DAD), for the determination, limit of detection and limit de quantification of cocaine present in 10 samples of crack and 10 samples of street cocaine (cocaine hydrochloride) seized by the Civil Police of Espírito Santo State.

2. Material and Methods

2.1. Reagents and chemicals

Methanol (liquid chromatography gradient grade) LiChrosolv® (Darmstadt, Germany), glacial acetic acid P.A. of Alphatec (Brazil). Cocaine, phenacetin, and caffeine were purified in the laboratory.

2.2. Sample

The samples of cocaine and crack, 10 g each, used in this work were obtained from the Civil Police from the Espírito Santo state and were seized during 2020. According to the Technical Cooperation Agreement nº 23068.022157/2020-69.

2.3. Sample preparation

All samples (10 cocaine-seized samples and 10 crack samples) were prepared following the same method. For each sample it was weighted 1 mg on a vial, using an analytical balance. Then, 1000 µL of methanol-HPLC was added to each vial and with the assistance of an ultrasonic bath the samples were dissolved. The concentration for each sample was 1 $mg.mL^{-1}$. Once the samples were ready, they were analyzed by GC/MS and HPLC-DAD.

2.4. Gas Chromatography/Mass Spectrometry

For the analyses, an GCMS-QP2010 ultra (Shimadzu Corporation) system equipped with a capillary column Agilent (USA) DB5-ms (30 m x 250 µm x 0,25 µm) was used. 1 µL of each sample was injected in split mode (10:1), injection temperature of 280 ºC and automatic injection. The initial oven temperature was held at 150 ºC for 3 minutes, then programmed to rise to 290 ºC, at a 20 $ºC.min^{-1}$ ratio. It was held for 10 min at 290 ºC, the whole analysis took 20 minutes. The carrier gas was helium (99.999%, White Martins), at a constant ratio of 1.5 $mL.min^{-1}$. The mass spectrometer was operated in the electron impact ion source mode at 70 eV, the ion source temperature was 230 ºC, with an acquisition range (m/z) from 50 to 500, with solvent cut at 2.5 min. For the data, the software LabSolutions - Data Analysis (GCMS Solutions, version 2.72, Shimadzu Corporation).

2.5. High Performance Liquid Chromatography

The analysis was performed on a 1260 Infinity Agilent (Santa Clara, California - USA) with a DAD detector and analytical column Agilent C18 (5 mm x 4,6 mm x 150 mm). The solvents used were A: water/ acetic acid 0,1% and B: methanol. For the crack samples the isocratic elution was used: 0 - 10 min, solvent A 5% and solvent B 95%, ratio of 1.0 $mL.min^{-1}$, wavelength of 235 nm, injection volume of 5 µL and manual injection (Method 1). For cocaine, the samples were performed in an isocratic elution: 0 - 10 min, solvent B, ratio of 1.0 $mL.min^{-1}$, wavelength of 202 nm, injection volume of 5 µL and manual injection (Method 2). For both methods the absorption range was from 200 to 290 nm. For the data, the software Agilent OpenLAB.

2.6. Validation

Validation was performed according to National Health Surveillance Agency (ANVISA - Agência Nacional de Vigilância Sanitária) regulations [13]. In the validation process, detection limit (LoD), quantitation limit (LoQ) and linearity were evaluated. To validate the method, an analysis of the working range at concentrations of 0.1, 10 and 100 µg.mL-1 was carried out on samples of cocaine, caffeine and standard phenacetin.

Linearity was determined from the correlation coefficient obtained from the calibration curve parameters, generated from 10 different concentrations of a cocaine standard (0.1 to 949.6 mg.mL^{-1}). The solutions were analyzed on GC/MS and HPLC, two chromatographic methods. The same procedure was performed for caffeine (52 to 82.6 mg.mL^{-1}) and phenacetin (1.5 to 181.5 mg.mL^{-1}), which were analyzed only on GC/MS.

The Limit of Quantitation (LoQ) and Limit of Detection (LoD) were determined from equation 1 and 2, respectively.

$$LoD = \frac{3,3\ x\ \sigma}{IC} \qquad Eq.1$$

$$LoD = \frac{10\ x\ \sigma}{IC} \qquad Eq.2$$

3. Results and Discussion

3.1. Gas Chromatography – Mass Spectrometry

In order to construct the calibration curve, 10 different concentrations of cocaine, phenacetin and caffeine were used. From this it was possible to obtain the determination coefficient (R^2) and consequently the correlation coefficient (R). However, for the linearity evaluation is necessary to estimate according to the results acquired by the Analysis of Variance (ANOVA). In this case, the tabulated value of F (F_{tab}) is 5.318 and the calculated value of F (F_{calc}) is higher in all cases. Therefore, the null hypothesis (in which there is no relationship between the measurements) is rejected, and the alternative hypothesis is accepted, expressing the veracity of a significant correlation between the measurements (concentration and the response obtained by the equipment).

Furthermore, the homoscedasticity and heteroscedasticity of the results can be determined by the Cochran Test. This parameter indicates if there is a constant variation between the errors or if it is aleatory. The Cochran Test may be executed by comparing the calculated value (Q_{cal}) with the theoretical (Q_{tab}), the calculation is done by the residue's dispersion graphic. If Q_{cal} is lower than Q_{tab} a homoscedasticity is present in the error's variance, while if Q_{cal} is higher than Q_{tab} a heteroscedasticity is present. Since cocaine, caffeine and phenacetin all have $Q_{cal}<Q_{tab}$, it is correct to say their methods are homoscedastic.

The Limits of Detection (LoD) and Limit of Quantification (LoQ) were obtained from the calibration curve parameters. For cocaine, LoD was 123.42 mg.mL^{-1}, and LoQ was 374.02 mg.mL^{-1}. For caffeine, the values of LoD and LoQ were 8.92 mg.mL^{-1} and 27.03 mg.mL^{-1}, respectively. While for phenacetin, LoD and LoQ was 28.57 mg.mL^{-1} and 86.59 mg.mL^{-1}, respectively. Table 1 shows the line equation, correlation coefficient (R), homoscedasticity and the value found for F_{cal}, LoD and LoQ for cocaine, phenacetin and caffeine.

Table 1. Results obtained for cocaine, caffeine and phenacetin from GC/MS.

GC/MS	F	R	Line Equation	LoD (mg.mL-1)	LoQ (mg.mL-1)	Homoscedasticity**
Cocaine	652.598	0.9939	y = 5219.6x + 1169	123.42	374.02	0.298
Caffeine	130.486	0.9692	y = 1582.7x + 212829	8.92	27.03	0.381
Phenacetin	440.281	0.9910	y = 5634.9x - 3473.1	28.57	86.59	0.239

*F_{tab} = 5.318; ** Q_{tab} = 0.445

3.2. HPLC

For the linearity analysis performed on HPLC, the correlation coefficient (R) and the calculated value of F (F_{cal}) were observed. The F_{cal} value found for both chromatographic methods (method 1: crack samples; method 2: seized cocaine samples) was higher than the tabulated value of F (F_{tab} = 5.318). Furthermore, the Cochran Test was applied and as a result the value of Q_{cal} showed up to be higher than Q_{tab}, indicating that a homoscedasticity was present.

For HPLC, the LoD and LoQ were also obtained from the calibration curve for cocaine. The observed LoD was 61.56 mg.mL^{-1} and LoQ was 186.55 mg.mL^{-1} for crack samples, denoted as method 1. For the seized cocaine (method 2), the values of LoD and LoQ were 152.82 mg.mL^{-1} and 463.11 mg.mL^{-1}, respectively. Table 2 shows the line equation, correlation coefficient (R), homoscedasticity and the value found for F_{cal}, LoD and LoQ for cocaine, phenacetin and caffeine.

Table 2. Results obtained for crack and cocaine seized samples from HPLC.

HPLC	F***	R	Line Equation	LoD (mg.mL-1)	LoQ (mg.mL-1)	Homoscedasticity****
Method 1*	2623.342	0.9984	y = 49064x + 72383	61.56	186.55	0.335
Method 2**	425.669	0.9907	y = 583.65x + 28532	152.82	463.11	0.125

*Method 1: for crack samples; **Method 2: cocaine seized samples; ***F_{tab} = 5.318, and **** Q_{tab} = 0.445

The values presented in tables 1 and 2 show a lower limit of detection (LoD) and quantification (LoQ) for cocaine when using method 1 in HPLC instead of method 2 in gas chromatography analyses. However, with mass analysis coupled with gas chromatography, it was able to verify the composition of the 20 seizures. Table 3 shows the compounds present in the cocaine and crack seizures, as well as the diluents.

Table 3. Compounds found in the seized cocaine and crack by GC/MS analysis.

Sample	Compounds type	
	Tropane Alkaloides	**Diluents**
Cocaine hydrochloride	Ecgonine methyl ester	Phenacetin
	Ecgonine anhydrous methyl ester	Lidocaine
	Norcocaine	Caffeine
	Cocaine	Amphetamine derivative
	Cinnamoylcocaine	Aminopyrine
	Benzoylecgonine	
	Tropacocaine	
Crack	Ecgonine anhydrous methyl ester	Cinnamic acid
	Anhydroecgonine	3-methylpyridine-4-one
	Tropacocaine	Resorcinol
	Cocaine	Phenacetin
	Cinnamoylcocaine	Methyl cinnamate
	Benzoylecgonine	3-methylpyridine-4-one
		2-Hexadecyl-5-methylpyrrolidine

The compounds listed in the first row in table 4 are tropane alkaloids found in *Erythroxylum* (Erythroxylaceae) species or from reactions that happened during the drug production process.[3, 11] The second row in table 4 shows the most common diluents in crack and seized cocaine, like phenacetin, caffeine and lidocaine. Substances of lower value, generally with some effect similar to cocaine are mixed with the drug to increase the volume and then be profitable for illegal sale. Amphetamine derivatives are not common diluents. In this case, there may also be products mixed with the drug unintentionally, which can be considered as diluents. Cinnamic acid and methyl cinnamate are substances that can come from both the extraction process and parallel reactions during the drug production process.[3]

From the calibration curve, the amount of cocaine present in each seized sample can be observed. Table 4 shows the mass of cocaine for crack and seized cocaine when analyzed by gas chromatography and liquid chromatography.

Table 4. Mass of cocaine present in crack and cocaine seized at GC/MS and HPLC.

Cocaine			Crack		
Sample	**GC/MS (mg.g^{-1})**	**HPLC (mg.g^{-1})**	**Sample**	**GC/MS (mg.g^{-1})**	**HPLC (mg.g^{-1})**
C1	543.6	297.4	CR1	121.3	174.6
C2	514.9	518.9	CR2	360.6	554.3
C3	633.2	233.0	CR3	414.6	638.1
C4	184.5	678.1	CR4	432.4	408.3
C5	656.0	730.0	CR5	282.6	424.6
C6	606.8	693.4	CR6	344.9	673.3
C7	*	880.1	CR7	482.9	617.7
C8	624.6	128.4	CR8	396.6	505.2
C9	176.8	328.0	CR9	400.7	554.0
C10	585.0	756.2	CR10	756.7	946.0

* not detected.

The quantities of cocaine in the samples vary greatly, this result shows the difference between seizures. The highest amount of cocaine was around 800 - 900 mg.g-1 and the lowest amount was around 100 - 200 mg.g-1. The calibration curve was also constructed for the diluent's phenacetin and caffeine.

For phenacetin, it was observed that the quantity varied from 1.1 to 74.8 mg.g^{-1} in the seized cocaine. For crack samples, phenacetin concentrations were observed between 3.7 and 687.1 mg.g^{-1}. Demonstrating that crack samples contain more phenacetin than seized cocaine ones. Caffeine was detected only in cocaine samples, with amounts varying between 13.7 and 22.5 mg.g^{-1}.

When comparing the cocaine concentration obtained by GC/MS and HPLC in each sample, there is a difference in some samples. This difference may be related to the effect of the sample matrix in each technique, since cocaine was not isolated.

Vinkovic et al. validated a method for micro-HPLC to detect diluents, such as caffeine, procaine, levamisole,

phenacetin, lidocaine and benzocaine, in 110 samples of cocaine seized by Australian police from 2012 to 2017. From 110 samples, only 5 samples had 100 w% of cocaine. Furthermore, the lowest concentration detected was 3 w%, in addition to having samples in which cocaine was not detected, only diluents. [14] As well as the work developed, the amount of cocaine varies greatly between different samples.

4. Conclusions

As it is well known, it is possible to observe that the amount of cocaine present in seized crack and cocaine can be quantified by chromatography techniques. Both GC/MS and HPLC techniques have been proven to be excellent techniques for detecting and quantifying cocaine and its diluents, specifically phenacetin and caffeine. The amounts found do not follow a pattern, varying considerably for both types of illicit drugs. Some samples have shown a large amount of cocaine in crack samples, despite the crack being produced with leftovers from the production of cocaine hydrochloride.

The validated methods follow ANVISA guidelines, which have a correlation coefficient above 0.990, and linearity and homoscedasticity are evaluated. HPLC has presented a higher correlation coefficient (0.9984) for method 1 compared to GC/MS. The GC/MS and HPLC analyses proved to be promising for identifying and quantifying cocaine, metabolites and diluents, and also being economically viable.

Common diluents in Brazilian seizures were found being caffeine, phenacetin and lidocaine. Levamisole was not observed, despite being a commonly used diluent.

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Author Contributions

Karoline P. Miossi (Investigation, Methodology, Data curation, Formal Analysis, Validation, Visualization, Writing – original draft, Writing – review & editing); Thays C. Valim (Visualization, Writing – original draft, Writing – review & editing); Ramon R. T. Bottocim (Visualization, Writing – original draft, Writing – review & editing); Bruno Q. Araújo (Formal Analysis, Methodology, Visualization); Wanderson Romão (Conceptualization, Supervision, Project administration, Visualization); Eustaquio V. R. de Castro (Supervision, Visualization); Valdemar Lacerda Jr. (Conceptualization, Methodology, Project administration, Supervision, Visualization, Writing – original draft) and Álvaro C. Neto (Conceptualization, Methodology, Project administration, Supervision, Visualization, Writing – original draft, Writing – review & editing).

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