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A survey of adulterants used to cut cocaine in samples seized in the Espírito Santo State by GC–MS allied to chemometric tools

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ABSTRACT

Cocaine is a stimulant drug of the central nervous system (CNS) extracted from the leaves of *Erytroxylum coca*. It is defined as a tropane alkaloid containing 1R-(exo,exo)-3-(benzoyloxy)-8-methyl-8-azabicyclo[3.2.1]octane-2-carboxylic acid methyl esther. However, despite its defined composition, a wide variety of chemical additives are present in cocaine found in the illicit market, such as benzocaine, lidocaine, caffeine, procaine and phenacetin. In this work, 512 cocaine samples seized by the Civil Police of Espirito Santo state (PC-ES, Brazil) were analyzed by gas chromatography mass spectrometry (GC–MS) allied to principal component analysis (PCA) in order to classify the samples as a function of seizure year (2008, 2009, 2010, 2011 and 2012) and location (metropolitan, north, south and central). The cocaine content (wt.%) and its adulterants were also estimated. Analyzing the samples seized between 2008 and 2011, three sample sets are clearly grouped according to the degree of adulteration with caffeine and lidocaine: 100–50 wt.% of cocaine; 50–20 wt.% of cocaine; and 20–80 wt.% of lidocaine and 60–80 wt.% of caffeine, simultaneously. The last group is formed by samples seized between 2008 and 2009, which proves the higher degree of adulteration during this period. In 2012, higher cocaine content was observed for the 191 analyzed samples than in samples from previous years. The PCA data also suggests that the metropolitan region samples had a higher degree of adulteration than the state countryside samples.

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1. Introduction

According to the 2011 report from the United Nations Office on Drugs and Crime (UNODC), cocaine is the second most consumed drug in the world [1,2]. Moreover, cocaine consumption is increasing significantly in South America, and estimates show that in 2011, South America, the Caribbean and Africa were home to 36% of total cocaine users. The UNODC estimates that 17 million people used cocaine in 2011. This number corresponds to 0.37% of the world population aged between 15 and 64 years [2].

In 2011, 54% of the cocaine seized in Brazil came from Bolivia, 38% from Peru and 7.5% from Colombia. These numbers are unsurprising as Brazil borders the three main cocaine-producing countries: Bolivia, Colombia and Peru. In addition, Brazil has a long seaside coast that facilitates access to the Atlantic Ocean, enabling traffic to Europe and Africa [2,3].

1.1. Cocaine

Cocaine is a stimulant drug of the central nervous system (CNS) extracted from the leaves of *Erytroxylum coca*. It is a powerful local anesthetic, though its stimulant effects are short and depend on the method of consumption—and afterwards it provokes depression [4]. The structure of cocaine includes a tropane nucleus, which inhibits the action of acetylcholine [5].

Cocaine is mainly consumed as a salt (cocaine hydrochloride) or in a freebase form (crack). Cocaine hydrochloride is a water-soluble salt obtained as a powder, and it can be administered via aspiration or intravenously. Crack, however, appears as a rock and is slightly soluble, but it is easily volatilized when heated because of its low melting point (approximately 95 °C) [3,6]. Fig. 1 shows the structural formula of cocaine as hydrochloride and freebase cocaine (crack).

According to the International Union of Pure and Applied Chemistry (IUPAC), cocaine is a 1R-(exo,exo)-3-(benzoyloxy)-8-methyl-8-azabicyclo[3.2.1]octane-2-carboxylic acid methyl esther. Its molecular formula is $C_{17}H_{21}NO_4$, and its molar mass is 303.4 g mol^{-1}. Cocaine's melting point range is 96–98 °C, and its $pK_b = 5.4$ [7].

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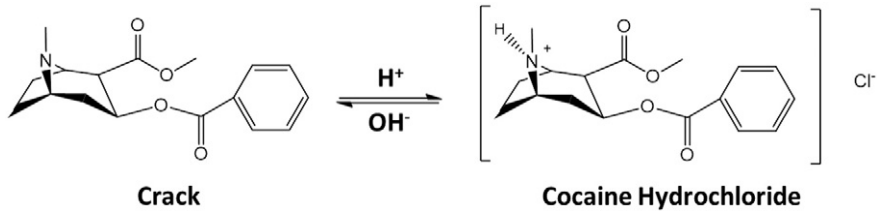

Fig. 1. Acid-base equilibrium of Cocaine structure.

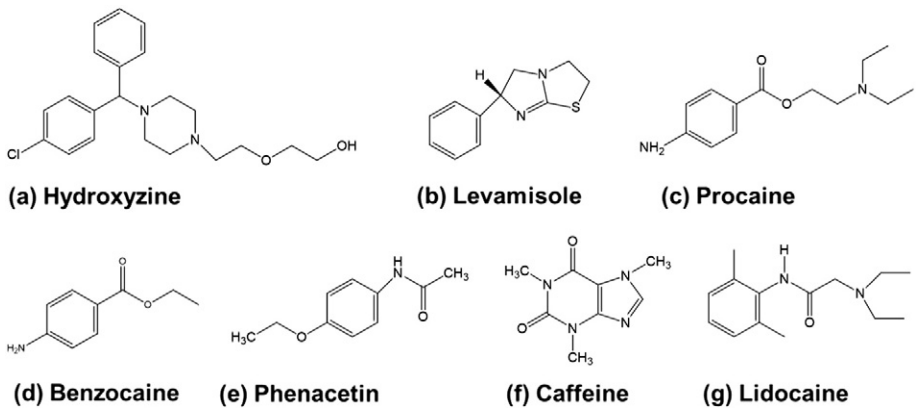

Fig. 2. Structure of some adulterants present in the cocaine seized in the Brazilian illicit trade: (a) hydroxyzine, (b) levamisole, (c) procaine, (d) benzocaine, (e) phenacetin, (f) caffeine and (g) lidocaine.

1.2. Cocaine adulterants

In the last few years, the purity of "commercial" cocaine has been decreasing. To increase drug volume and therefore drug trafficking profits, chemical additives are blended to the alkaloid as adulterants and/or diluents. Among the chemical additives, psychoactive substances, commonly anesthetics, are used to mimic or increase the drug's effect for users [8,9]. Fig. 2a–g shows the chemical structures of some adulterants found in cocaine samples seized in the Brazilian illicit market. The effects of these molecules on the central nervous system are demonstrated in Table 1.

1.3. Analytical methods for cocaine identification

The most analytical methodologies for cocaine analysis are mostly qualitative. At least two examinations are performed: a preliminary test using wet chemistry and an analytical test with higher selectivity. Colorimetric tests or pre-tests have the advantages of rapidity, low cost and ease of execution and interpretation. However, they feature low specificity. Among the colorimetric tests used to determine the presence of alkaloids, the test employing a solution of cobalt thiocyanate and the Scott test stand out [9,10]. Nevertheless, positive results can be found when some adulterants or diluents are present, such as lidocaine, powdered milk and promethazine [9].

Table 1
Effects on the CNS of the adulterants present in the cocaine seized in the Brazilian illicit trade.

Hydroxyzine	Antihistamine
Levamisole	Stimulant synergy with cocaine
Procaine	Local anesthetic
Benzocaine	Local anesthetic
Phenacetin	Analgesic similar to paracetamol
Caffeine	Stimulant
Lidocaine	Local anesthetic (similar do cocaine)

Other analytical methodologies with high selectivity and sensibility are routinely applied for cocaine analysis, including thin layer chromatography [11–14], gas chromatography coupled with mass spectrometry (GC–MS) [15] and a gas chromatography-flame ionization detector (GC-FID) [16,17].

Cocaine analysis by GC–MS has been widely explored for cocaine quantification and its adulterants due to the analytical methodology recommended by UNODC. GC–MS enables simultaneous qualitative and quantitative studies of cocaine and adulterants with molar masses up to 500 Da.

Mapping the distribution of cocaine seized in Espirito Santo state, Fig. 1S (see in supplementary material), is a challenge since the frequency of seizures has been growing. Fig. 3 highlights the growth in the number of reports registered by the Civil Police of Espírito Santo state (PC-ES) between 2008 and 2013. The growth in the number of seizures is evident, reaching an increase of more than 100% in 2013 when compared to year 2008.

In this work, the chemical profiles of 192 cocaine samples seized in the ES state in 2008, 2009, 2010 and 2011 as well as 321 samples distributed according to the four macroregions (metropolitan, north, south and central) were analyzed, Fig. 1S. The chromatographic profile obtained was classified as a function of the percentage of cocaine and its adulterants via multivariate analysis using principal component analysis (PCA).

2. Experimental section

2.1. Sample selection

Cocaine samples seized in 15 cities of the ES state in 2012 (maximum of 20 samples per city) were selected and analyzed by GC–MS and PCA. The cities were chosen based on the volume of seizures and the local population and included the four administrative macroregions of the state (metropolitan, north, central and south), totaling 321 samples. The cities selected were: i) *metropolitan region*: Cariacica, Guarapari, Vitória, Serra and Vila Velha; ii) *south region*: Alegre,

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