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Technical note

Evaluation of the composition of street cocaine seized in two regions of Brazil

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ABSTRACT

This work evaluates cocaine purity and the concentration ranges of adulterants and inorganic constituents for 31 street cocaine samples seized in two different regions of Brazil from July 2008 to May 2010. Cocaine and adulterants, such as caffeine, lidocaine and benzocaine, were quantified by Gas chromatography–mass spectrometry (GC–MS), and the inorganic constituents were determined by Inductively Coupled Plasma–Optical Emission Spectrometry (ICP–OES) and ion chromatography (IC). The cocaine concentrations in the samples seized in the Amazonas state (AM samples) ranged from 154 to 978 mg g⁻¹, and these samples did not contain any of the adulterants studied. The cocaine concentrations in the samples seized in the Minas Gerais state (MG samples) ranged from 63.9 to 753 mg g⁻¹. Caffeine was the main adulterant found in 76% of the MG samples, ranging in concentration from 5.5 to 645.3 mg g⁻¹. Lidocaine was found in 66.7% of the MG samples, with concentrations ranging from 16.3 to 576.7 mg g⁻¹. Benzocaine was found in only one MG sample, at a concentration of 84.8 mg g⁻¹. Fourteen elements were identified by ICP–OES, and a wide variation was observed in the concentrations of Ca, Mg, Na, P, Al, Fe, Mn and Zn. Pearson Product–moment Correlations between the analytes allowed the constituents to be associated with the chemicals used in the manufacturing of cocaine and with some common diluents. The study of the purity of cocaine and the presence and concentration of adulterants and inorganic constituents is important because the latter can have deleterious effects on health.

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1. Introduction

Cocaine is an illicit drug consumed by 0.7% of the Brazilian population between 12 and 65 years of age. In general, cocaine is one of the most usually used drugs worldwide [1]. Street cocaine is commonly illicitly sold as a white powder, and it typically contains cocaine hydrochloride, freebase and several other substances, such as contaminants, adulterants and diluents. Some contaminants derived from the refining process are often found in samples. As a result, street cocaine is rarely 100% pure [2]. These impurities can be generated from compounds that are present in the leaves of the coca plant (any of the four cultivated plants that belong to the family Erythroxylaceae, native to western South America), i.e. cis and trans-cinnamoylcocaine, hydroxycocaine, tropocaine, trimethoxycocaine and truxiline [3], or added to cocaine to increase the available volume of the drug, dilute the active ingredient and/or modify the pharmacological effects of the final product [4,5]. Many adulterants are other psychoactive

drugs that are much cheaper than the main substance and have similar or complimentary effects when taken with cocaine, which help to hide the fact that the substance has been diluted [6]. Diluents, such as glucose and sodium carbonate, are added simply to increase the amount of drug available to be sold [6,7]. Some other substances that are found in street drugs are the result of the particular manufacturing process used to make the drug [6]. The extraction of cocaine from coca leaves usually employs not only available organic solvents (such as fuels: kerosene or gasoline) but also inorganic bases (e.g. calcium oxide- quicklime- and sodium carbonate) or acids (e.g. sulfuric or hydrochloric). The product of this first extraction step is internationally known as coca paste. In a second step, which may occur immediately after the first one, coca paste is dissolved in dilute sulfuric acid and treated with an oxidizing solution, usually containing potassium permanganate. This is done in order to oxidize undesirable alkalis or impurities and a refined and light colored solid substance, named coke base and more valuable than coca paste in the illicit market, is produced [8]. All these extraction steps may therefore append organic and inorganic impurities into the final product.

Knowledge of the quality (purity and adulterants) of seized cocaine and its variations in quality could be useful both in controlling supply and in preventing and controlling some of the health effects associated with its use. Specifically, this knowledge could help in

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the following ways: (a) to formulate hypotheses about the structure of the illegal cocaine market (e.g., dilution at different stages in the chain of distribution, supply points, etc.) and to estimate the quantities of cocaine imported, when used in conjunction with other data [9]; (b) to estimate trends in cocaine availability on the illicit market when used together with price (i.e., the price/purity ratio); (c) to explain the establishment or spread of certain patterns of cocaine use and (d) to explain the occurrence of acute reactions or 'overdoses' from this drug, especially with respect to fatal reactions [10].

Previous research has identified caffeine, lidocaine, benzocaine, diltiazem, levamisole, procaine and phenacetin as adulterants in cocaine samples [11–14]. Adulterants may influence the perception of cocaine quality by users because they have effects that can mimic cocaine (for example, a local anesthetic effect) or alter its pharmacological effects [15]. Moreover, several diluents have been described in the literature, such as starch, sugar, sodium carbonate, silicates, sulfates and limestone [6,7]. Both adulterants and diluents can be dangerous and can cause considerable harm.

To determine the composition of street cocaine, several analytical techniques are necessary. The analysis of drugs of abuse, adulterants and impurities in street samples has been deeply studied from several points of view using different techniques, including simple color tests [16], thin-layer chromatography [2,7], microcrystal analysis [17], gas chromatography [15,18], liquid chromatography [19] and direct analysis using ambient mass spectrometry [20,21]. Few reports have described the use of inductively coupled plasma optical emission spectrometry (ICP-OES) and ionic chromatography (IC) for the analysis of street drugs. However, these techniques can provide important information about the inorganic constituents in these samples.

The present study deals with the quantification of organic and inorganic constituents in cocaine samples seized in two different regions of Brazil, the Minas Gerais and Amazonas states.

2. Materials and method

2.1. Reagents and samples

HPLC grade methanol was supplied by Burdick & Jackson® (Muskegon, MI, USA). Caffeine, lidocaine and benzocaine were obtained from Sigma-Aldrich (St. Louis, MO, USA). Cocaine was supplied by Cerilliant Corp. (Round Rock, TX, USA). Nitric acid 65% w v⁻¹ and reference solutions of aluminum, calcium, cobalt, copper, chromium, iron, potassium, magnesium, manganese, molybdenum, sodium, lead phosphorus, zinc, nitrate, chloride, nitrite, bromide, fluoride and sulfate at 1000 mg L⁻¹ were obtained from Merck (Darmstadt, Germany). Thirty-one illicit street cocaine samples, which were seized by the police of the Minas Gerais state (21 samples) and the Amazonas state (10 samples) between July 2008 and May 2010, were studied. The samples were analyzed by GC–MS, ICP-OES and IC.

2.2. Sample preparation

The sample preparation for the determination of organic constituents consisted in dissolving 10.00 mg of street sample in 10.00 mL of methanol. The mixture was sonicated for 5 min and the insoluble compounds were removed by centrifugation. The solution was further diluted to 1 µg mL⁻¹ for analysis. Fenproporex was used as internal standard at the concentration of 0.75 µg mL⁻¹. Volumes of 1.0 µL were injected in the chromatographic system.

For the determination of inorganic constituents 100.0 mg of each sample was solubilized in 10.00 mL of water. The mixture was sonicated for 5 min and the insoluble compounds were removed by centrifugation. An aliquot of 5.00 mL was removed for analysis by ion chromatography. The aliquots were filtered through a 0.45 µm membrane filter before injection. The remaining 5.00 mL was acidified

with 1.000 mL of concentrated nitric acid and diluted to 10.00 mL with deionized water obtained from a Milli-Q system (Millipore Direct-Q 3, France) and used for analysis by ICP-OES.

2.3. Analyses by GC–MS

The GC–MS analyses were performed on a Focus Model gas chromatograph (Thermo Electron Corporation, San Jose, CA) equipped with a mass selective detector (DSQ II) and an autosampler (Triplus). Xcalibur 2.07 software was used to control the chromatographic system and the data acquisition. An injection volume of 1.0 µL and the splitless injection mode were chosen. The chromatographic separations were achieved on an OV-5 MS capillary column (30 m length, 0.25 mm i.d. and 0.25 µm film thickness). The carrier gas was helium and was used at a flow rate of 1.2 mL min⁻¹. The oven temperature program was as follows: 100 °C for 2 min, followed by heating to 230 °C at a rate of 30 °C min⁻¹, held at 230 °C for 2 min, then heated at a rate of 70 °C min⁻¹ to 300 °C and finally, held for 2 min at isothermal conditions. The injector and GC–MS interface temperatures were set at 240 °C. The column effluent was directed into the ion source where the molecules were ionized by electrons (with an energy of 70 eV). For the analyses, mass spectra were scanned from 50 to 500 *m/z* in the full scan mode. For the determination of the cocaine, benzocaine, lidocaine, caffeine and fenproporex (the internal standard) concentrations, the following ions were selected, respectively: 182, 120, 86, 194 and 97. For quantification purposes the standard addition method was employed because remarkable differences were observed between the slopes of the calibration curves prepared in methanol and in the presence of matrix (matrix-matched standard).

2.4. Analyses by ICP-OES

An ICP-OES with an axial viewed configuration (ICP-OES, VISTA RL, Varian, Mulgrave, Australia) was used for the Al, Ca, Co, Cr, Cu, Fe, K, Mn, Mg, Mo, Na, P, Pb and Zn determination. The sample introduction system was composed of a Sturman–Masters spray chamber and a V-groove nebulizer. The operational parameters are described in Table 1. A multielement calibration curve was prepared by diluting standard solutions at 100 mg L⁻¹ of each metal (CertiPur, Merck).

2.5. Analyses by IC

These separations were performed with a Shimadzu system (Kyoto, Japan) equipped with an LC10A Shimadzu pump, a 7125 Rheodyne injection valve with a sample loop of 50 µL and an LC10A Shimadzu conductivity detector. A solution of 250 mmol L⁻¹ phthalate at pH 3.80 was passed through at 0.5 mL min⁻¹ for 2 h before starting

Table 1

Instrumental parameters for elemental determination using ICP-OES with an axial viewed configuration.

| Instrumental parameters | |
|---|---|
| Generator frequency (MHz) | 40 |
| RF power (kW) | 1.2 |
| Plasma gas flow (L min ⁻¹) | 1.5 |
| Auxiliary gas flow (L min ⁻¹) | 1.5 |
| Nebulizer gas flow rate (mL min ⁻¹) | 0.7 |
| Integration time (s) | 1.0 |
| Stabilization time (s) | 15 |
| Replicates | 3 |
| Analytical wavelengths (nm) | Al(I) 396.153; Ca(II) 422.673; Co(I) 228.616; Cu(I) 327.393; Cr(I) 283.563; Fe(II) 259.939; K(I) 766.940; Mn(II) 257.610; Mg(II) 279.077; Mo(II) 202.031; Na(I) 589.592; P(I) 213.617; Pb(II) 220.353; Zn(II) 213.857 |

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