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A decade of analysis of illicit street cocaine in Chile

[Una década de análisis de cocaína callejera incautada en Chile]

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Abstract

Context: Cocaine is one of the most worldwide consumed drugs of abuse. Determinate purity and adulteration profile of cocaine is very useful from the point of view of toxicology, public health, trends of misuse and for police enforcement, in order to establish the routes of drugs dealers.

Aims: To evaluate the purity profile of cocaine hydrochloride in Chile over 10 years; classify main adulterants and diluents added to cocaine, for this purpose we used collected data from all tested samples since 2006 to 2016.

Methods: In this study were used several analytical methods based in different techniques. For quantitative analysis samples were tested by Gas Chromatography with Flame Ionization Detector and High-Performance Thin Layer Chromatography. In order to confirm doubtful or extensively adulterated samples a confirmatory analysis by GC/MS was carried out.

Results: Results of this study are very alarming due to cocaine purity decreased since 2009 this fact corresponds with that cocaine has been progressively adulterated and diluted with compounds such as levamisol, caffeine, local anaesthetics, and carbonates. These events make very important this type of studies of composition of the street drug.

Conclusions: Cocaine is extensively adulterated, with substances that enhance the harm to the health of users, as in other countries in the region. According to our knowledge this is the first report in Chile about purity profile of cocaine hydrochloride in such a long period and with this large number of samples studied.

Keywords: adulterants; cocaine; diluents; purity.

Resumen

Contexto: La cocaína es una de las drogas de abuso más consumida mundialmente, la determinación de la pureza y adulteración es muy útil desde el punto de vista toxicológico, de salud pública, tendencias de abuso y para las policías, con el fin de establecer rutas de los traficantes. Por estas razones evaluamos la composición de la cocaína clorhidrato en Chile para establecer los principales adulterantes, diluyentes y compuestos tóxicos agregados intencionalmente por traficantes a la cocaína callejera.

Objetivos: Evaluar la pureza de la cocaína clorhidrato durante 10 años, categorizar los principales adulterantes y diluyentes agregados, para esto empleamos datos obtenidos de todas las muestras analizadas desde 2006 a 2016.

Métodos: Se emplearon diversos métodos analíticos; para ensayos cuantitativos se aplicaron cromatografía de gases con detector de ionización de llama y cromatografía planar de alta eficiencia. Para confirmar muestras dudosas o muy adulteradas se desarrollaron análisis confirmatorios por GC/MS.

Resultados: Los resultados son alarmantes debido a que la pureza de cocaína ha disminuido desde 2009, este hecho se corresponde con que ha sido progresivamente adulterada y diluida con componentes como levamisol, cafeína, anestésicos locales y carbonatos. Estos eventos hacen muy importante este tipo de estudios de composición de la droga callejera.

Conclusiones: La cocaína está ampliamente adulterada, con sustancias que aumentan el daño a la salud de los usuarios, según nuestro conocimiento es el primer reporte en Chile acerca de la adulteración en cocaína clorhidrato en un período de tiempo tan prolongado y con un número tan amplio de muestras.

Palabras Clave: adulterantes; cocaína; diluyentes; pureza.

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INTRODUCTION

Cocaine is a natural alkaloid extracted from *coca* plant, is one of the most frequently abused illicit drugs, and is commonly abused by inhalation, nasal insufflation, and intravenous injection. Increases in cocaine use have been reported from a number of countries in South America, Central America and the Caribbean, reflecting the development of consumer markets along the distribution chain, most clandestine laboratories for cocaine are located in South America and the main trafficking route still runs from the Andean region to North America (UNODC, 2007).

The abuse of illegal drugs is a matter that has raised both international and national alarm. In 2001 a household survey of the use cocaine in a population aged from 15 to 64 in Santiago Chile, revealed that life prevalence of use was 4.5% for cocaine, annual prevalence was 1.8% and last month prevalence was 0.7%; these numbers are growing continuously and worrying from the point of view of consumers health (González et al., 2001; Sepúlveda et al., 2011).

Toxic effects of cocaine are very well known, in United States cocaine is the principal cause for drug–abuse-related visits to emergency departments, most of which are due to cardiovascular complaints. Through its different pathophysiological mechanisms, cocaine exerts several adverse effects on the cardiovascular system, with fatal results (Havakuk et al., 2017). Cocaine can affect all body systems and the clinical presentation may principally result from organ toxicity. Among the most severe complications are seizures, hemorrhagic and ischemic strokes, myocardial infarction, aortic dissection, rhabdomyolysis, mesenteric ischemia, acute renal injury and multiple organ failure (Zimmerman, 2012). Also, several *in vitro* models are developed in order to evaluate the toxic effects of cocaine and its adulterants in the central nervous system (Rocha et al., 2018). It is well known, from abundant studies, that cocaine causes irreversible structural changes on the brain, heart, lungs and other organs such as liver and kidney (Riezzo et al., 2012).

Most of these effects are produced by cocaine itself, but some other are made by compounds added in order to increase or mimic the stimulant effects or simulate other properties of cocaine, such as local anesthetic or typical white color and or it crystalline appearance, all these substance are known as adulterants and diluents, so street cocaine is rarely 100% pure. There are a wide number of published studies about the compounds added to cocaine (Janzen et al., 1992; Sharma et al., 2005), in that terms the most founded substances as adulterants are caffeine, lidocaine, levamisol, but by the other hand most frequent compounds known as diluents are added in order to increase the weight of cocaine (i.e. starch, carbonates and sugars). Both adulterants and diluents can be hazardous and can cause significant harm to human health (Coomber, 1997). Due to all these reasons is very significant to determinate composition of cocaine seized and consumed in a country or region. For this purpose, forensic laboratories use several analytical methods based in different techniques such as color test, chromatographic determinations (i.e. TLC, GC and LC), and spectrometric techniques based on mass spectrum or FTIR (Brosé et al., 2015). These mentioned techniques allow obtaining data about impurity profile on seized cocaine. There are a wide number of studies and surveys about chemical profile of cocaine hydrochloride mainly in Europe, Australia and North America (Darke et al., 2005; Evrard et al., 2010). On the contrary, in South America there are not many studies about the composition of street cocaine, in that aspect, the mayor number of publications about this issue are made in Brazil and Colombia with very alarming results in terms of the wide range purity of cocaine and the extensive adulteration founded in tested samples (Garzon et al., 2009; Magalhães et al., 2013; Fukushima et al., 2014; Maldaner et al., 2016).

For all these antecedents, it is very important to carry out a study of the composition of cocaine hydrochloride over time, analyzing the main adulterants and diluents and purity of this alkaloid over a period of 10 years (2006-2016). According to our data and records there are no publications

about this topic in our country with such an important number of seized samples.

MATERIAL AND METHODS

Reagents, reference materials and samples

Ammonia, methanol, toluene, diethylamine, and cyclohexane were high-performance liquid chromatography (HPLC) grade and were purchased from Merck (Darmstadt, Germany). Reference material of cocaine hydrochloride was purchased from Cerilliant®, Texas. Reference standards from adulterants were kindly provided by United Nations (UNODC), Vienna.

This study was performed over 61 852 samples of cocaine hydrochloride seized by Chilean police since 2006 to 2016.

Sample preparation

Sample preparation for cocaine purity profiling consisted in dissolving 10 mg of sample in 10 mL of methanol. This solution was sonicated for 5 min. Then almost 2 mL of this solution was added to a glass vial for chromatographic examination. Samples were tested by FTIR-ATR, and Scott´s reagent following procedures recommended by UNODC, in order to establish the presence of cocaine base or hydrochloride and its main diluents (Tsumura et al., 2005; Oguri et al., 2011).

Quantitative analysis

For quantitative analysis samples were tested by Gas Chromatography with Flame Ionization Detector (GCFID, Agilent Technologies®) and High-Performance Thin Layer Chromatography (HPTLC, CAMAG®), under following conditions:

GCFID

Inlet: T°: 250°C, Split 1:25.

Injection volume of Samples and Standards: 1 µL

Column: HP-5 30 m x 0.32 µm x 0.25 mm

Mobile Phase: Helium, 1.0 mL/min.

Temperature program: Initial 150°C per 0 minute then 25°C per minute to 300°C and hold for 5 minutes.

HPTLC

HPTLC was performed on 20 × 10 cm precoated silica gel F254 plates (Merck, Darmstadt, Germany) previously activated at 80°C for 30 min. Standards (1 µL) and samples (1 µL) were applied in 3 mm bands with an ATS 4 automatic TLC sampler (CAMAG, Muttenz, Switzerland), using a spray band technique. The plates were developed in an automatic developing chamber (ADC-2, CAMAG) to a distance of 70 mm with cyclohexane diethylamine (90:10) as the mobile phase (10 mL, without saturation of the chamber). After a drying time of 3.0 min, the bands were scanned with a CAMAG TLC Scanner 4 densitometer by absorbance at 230 nm, all procedure was controlled with WinCATS® Planar Chromatography Manager version 1.4.7 software (CAMAG).

Confirmatory analysis

In order to confirm doubtful or extensively adulterated samples, an analysis by GCMS (Agilent® 6890N coupled with mass detector 5973B) was carried out under next conditions:

Injection volume of 1.0 µL split 1:50. Chromatographic separations were achieved on an HP-5 MS capillary column (30 m length, 0.25 mm, and 0.25 µm). The carrier gas was helium. Oven temperature program was the same as used in the GC FID program. The separated molecules were ionized by EI to 70 eV; mass spectra were scanned from 20 to 500 m/z in the full scan mode. All spectrums were matched with NIST and SWGDRUG spectrum libraries (Marchei et al., 2008).

Statistical analysis

The data were examined using the Statgraphics Centurion XVII®, Software R 3.5 (Core Team, 2018) and Microsoft Excel 2010 ® in order to establish average, standard deviation and relative standard deviation of cocaine hydrochloride purity over the evaluated period.

RESULTS AND DISCUSSION

Between 2006 and 2016 our laboratory analyzed a total of 61 852 samples with presence of cocaine hydrochloride, with an average of 5622 samples per year. The 55.4% of the samples were sent by police enforcement of Metropolitan Region (Santiago), followed by regions of Valparaíso (9.6%) and Tarapacá (8.8%). The samples that came from the northern regions of Chile (Arica, Tarapacá, Antofagasta and Atacama) revealed a concentration (w/w %) much higher than the other regions of the country, with a purity average greater than 70% w/w. These regions are very close to countries where the *coca* bushes are grown, and cocaine alkaloid is extracted from coca leaves for illicit drug market. As we detailed before, from 2006 to 2016, cocaine hydrochloride was detected in 61 852 seized samples, from this total 39 804 samples were studied in order to determinate the purity of cocaine in each seized sample. Fig. 1 shows purity profile over 10 years comparing evolution of cocaine concentration (%w/w) from 2006 to 2016. Purity has gradually steady about 55% w/w to 2009; this trend has also been reported in several countries in Europe and Brazil in South America (Evrard et al., 2010).

The highest purity was detected in 2009 and then this value has progressively declined near to 40%. Meanwhile the lowest concentration of cocaine was observed in 2015 with 37.17%. This fact is correlated with arise of adulterants such as levamisole, that appears in illicit seized cocaine since 2009, from this point purity of cocaine was rapidly decreasing. Table 1 displays the purity of cocaine per year and dispersion of the data over 10 years.

Levamisole is highly toxic and is no longer utilized in humans, it is a common veterinary pharmaceutical used to treat worm infestations in sheep, and pigs. An increase in the prevalence of levamisole in cocaine was noted in early 2008 in United States (Buchanan et al., 2010; Morris et al., 2012). Exposure to levamisole can cause agranulocytosis patience with this syndrome are more susceptible to fulminant and opportunistic infections (Crocker and Tremaglio, 2012). Fig. 2 shows the frequency of levamisole *versus* purity of cocaine from its first appearance in 2009 to 2016. On the other side in countries like Luxembourg from 2005-2010 this scenario is different due to the purity of cocaine; the mean concentration was lowest in 2009 and highest in 2005. In 2010, levamisole has become the most abundant adulterant detected in cocaine samples in Luxembourg (Schneider and Meys, 2011).

Table 1. Purity of cocaine and dispersion of the data over 10 years.

Year	Mean(%w/w)	Standard Deviation	Relative Standard Deviation% (RSD)	Minimum (%w/w)	Maximum (%w/w)
2006	39.5	30.9	78.1	Lower than 1.0	100.0
2007	45.9	32.1	69.8	1.0	99.0
2008	39.3	30.2	76.8	1.0	100.0
2009	53.4	28.0	52.5	2.0	99.0
2010	44.2	30.4	68.8	1.0	99.0
2011	41.7	30.2	72.5	1.0	100.0
2012	41.6	29.3	70.4	2.0	100.0
2013	39.4	25.5	64.7	2.0	100.0
2014	39.2	26.2	67.0	1.0	99.0
2015	37.2	26.7	71.9	1.0	99.0
2016	39.2	29.4	75.0	2.0	99.0

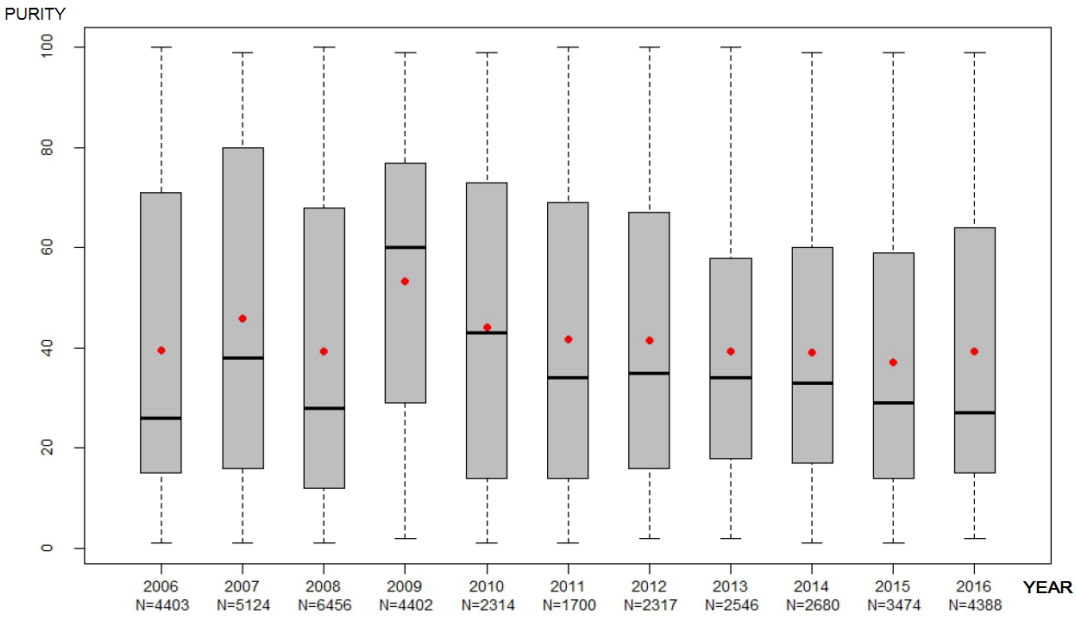

Figure 1. Purity profile over 10 years comparing the evolution of cocaine concentration (%w/w) from 2006 to 2016.

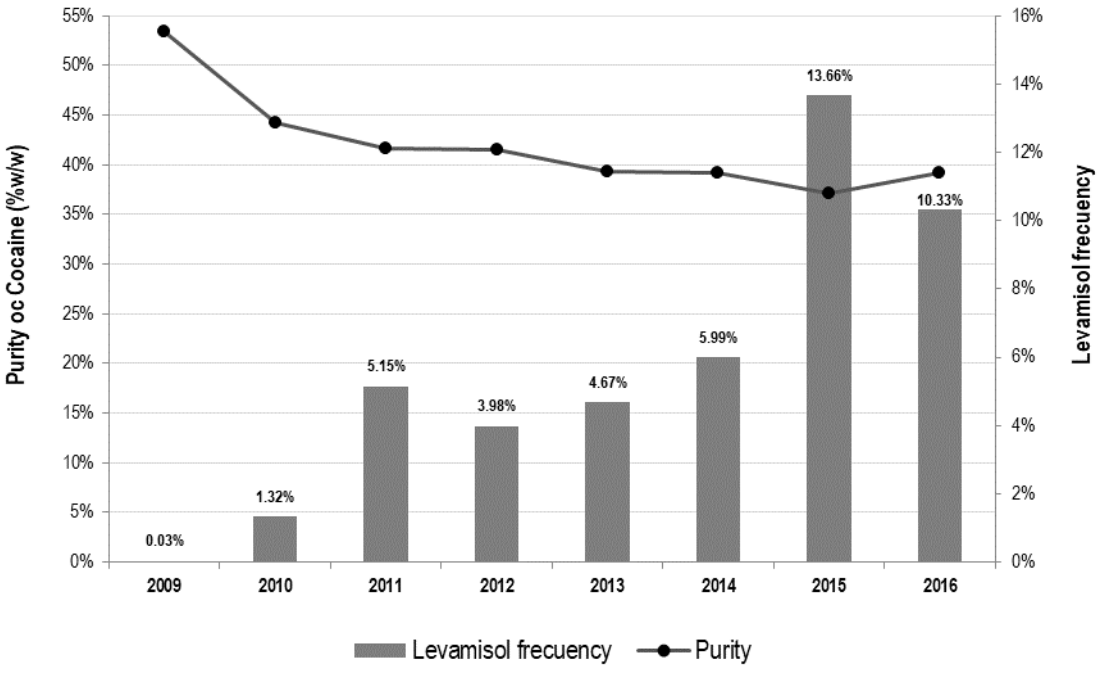

Figure 2. Frequency of levamisole *versus* purity of cocaine (%w/w).

Since 2003, levamisole was regularly introduced as a cocaine adulterant in the United States, with a rapid spread in other countries in South America and Europe. Today, it seems that almost 70% of all the cocaine hydrochloride seized worldwide is containing levamisole (Hantson, 2015). Chile is not immune to this problem, so levamisole is one of the main adulterants found in cocaine samples since 2009. A different situation occurs with lidocaine (a local anesthetic), this substance was the most used as adulterant in seized cocaine in 2006, where 40.4% of the samples with cocaine hydrochloride were also confirmed for the presence of lidocaine, and however its presence has been decreased in order to 11.8% in 2016. This fact is mainly explained by the important raise of caffeine as adulterant (Fig. 3). Thus from 2009 to 2011 more than 30% of cocaine samples were adulterated with caffeine. The combination of cocaine and caffeine enhance the stimulant effects and produce additive effects between the two substances, caffeine is also addictive, and chronic abuse can lead to cardiac injury (Mehta et al., 2004). Benzocaine and procaine were detected in less than 3% from the total.

In terms of the less common adulterants is very interesting the presence of diphenhydramine, detected in only 0.04% from the total, however this combination of cocaine and histamine-H1 receptor antagonists would have enhanced potential for abuse relative to each drug alone, cocaine and H1-antihistamines can be synergistic in terms of reinforcing effects and that the mixture may have important potential for abuse (Wang and Woolverton, 2007). Another important issue to notice was the fact that ephedrine was present in 0.05% from the total. It is a well-known fact that the action of cocaine and this substance on the blood pressure is synergistic. It has been shown that this synergy between cocaine and ephedrine includes toxicity and significantly increase the dangerous effects of cocaine (Munarriz et al., 2003).

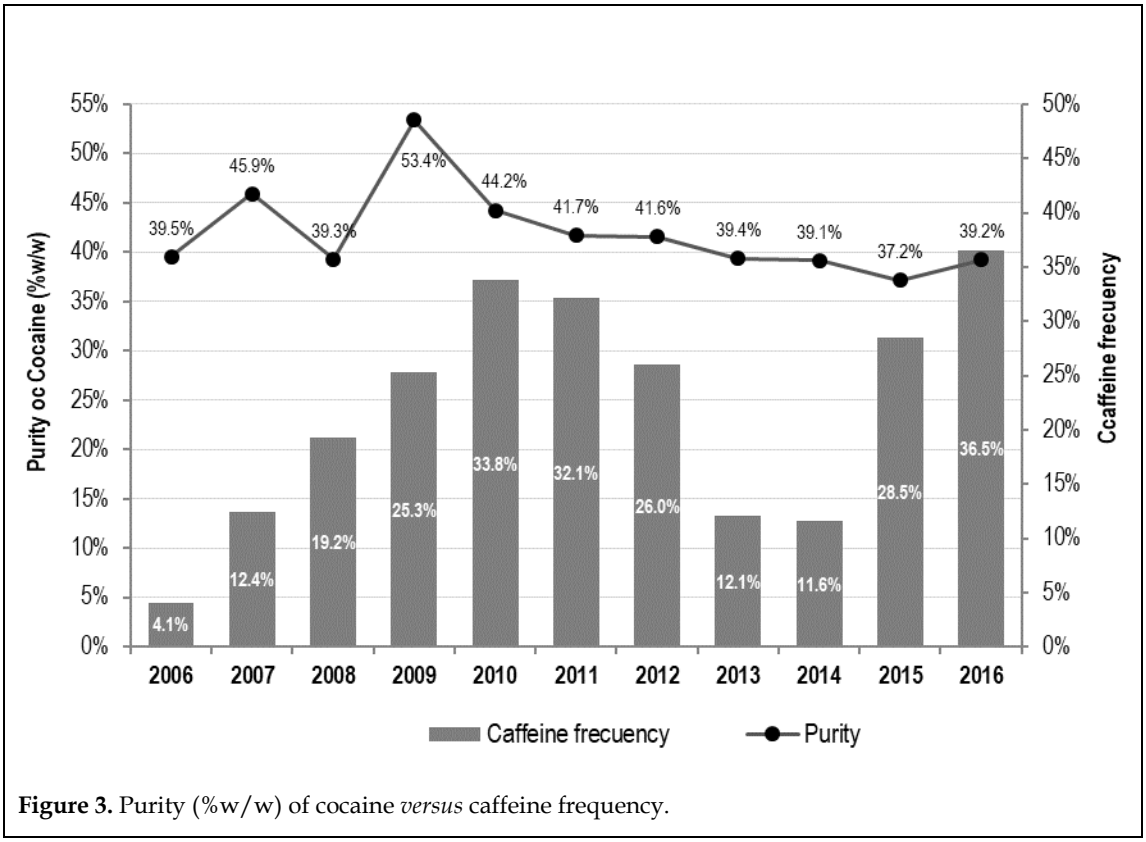

Figure 3. Purity (%w/w) of cocaine *versus* caffeine frequency.

Table 2. Most important adulterants in seized cocaine hydrochloride, 2006-2016.

Adulterants	Year											
	2006	**2007**	**2008**	**2009**	**2010**	**2011**	**2012**	**2013**	**2014**	**2015**	**2016**	**Total samples**
Caffeine	204	721	1559	1775	1890	1435	1430	583	540	1535	2006	13678
Lidocaine	2022	1406	1870	1689	1740	1238	1321	587	408	702	648	13631
Levamisole	0	0	0	2	74	230	219	225	279	735	568	2332
Phenacetin	210	330	368	237	123	85	82	61	75	38	36	1645
Benzocaine	147	221	334	257	219	123	56	29	26	28	7	1447
Procaine	314	73	38	6	19	14	13	14	9	12	1	513
Aminopyrine	13	4	12	14	45	10	3	4	10	1	3	119
Acetaminophen	10	27	14	7	0	6	8	2	3	1	7	85
Dipyrone	8	10	14	2	0	3	1	0	1	0	0	39
Ephedrine	1	0	1	29	0	0	0	0	0	0	0	31
Diphenhydramine	0	0	17	0	2	0	0	2	0	0	1	22

Another very well identified toxic agent present in seized samples was Acetaminophen, from the total only 85 samples were confirmed for this compound, which could cause liver failure, particularly when is mixed with alcohol. Dipyrone was found in 0.06% from the total, previous reports indicates that this substance is in a very small amount comparing with the most frequent adulterants such as caffeine or lidocaine (Mariotti et al., 2016), the same scenario was established for dimethylaminoantipyrine or also known as aminopyrine with a 0.19% (119 samples) form total, by the other hand phenacetin was found in almost 2.7% from all tested samples with more than 1640 samples adulterated this toxic compound, these drugs (aminopyrine and phenacetin) are not available in Chile for almost 40 years because their toxicity is very well known (Fucci, 2004).

Table 2 shows the main adulterants tested over the period. "*Speedball*", name given to the mix of cocaine and morphine was found in only one sample in 2006, since this year there are no more samples containing this deleterious combination (Cunha-Oliveira et al., 2013). In terms of diluents, nearly 34% from all samples were mixed with carbonates (20 926 samples), followed by sugars (0.66%) and starch with 0.58% from the total. Finally, the use of some substances as cutting agents could be related to their higher availability at a given time in our country for example carbonates and caffeine (both are non-controlled substances).

Similar to the rest of the world cocaine sold in Chile has an extensive grade of adulteration and dilution. Cocaine seizures are increasing steadily over time, according to national studies the consumption of this drug has also increased, and unfortunately consumers are not informed about the toxic components added to street cocaine by drug dealers. As we mentioned before, there are many reasons behind the addition of adulterants and diluents to street cocaine. Most of the diluents found in street samples can be acquired in national territory, without need for authorization from government agencies. In terms of adulterants, the scenario is a little different; the most common adulterants confirmed in seized cocaine were added at the place of fabrication or extraction of cocaine hydrochloride, due to phenacetin, aminopyrine are forbidden in our country since decades, while levamisole has no longer use in humans and is used only in animals. However, caffeine, lidocaine and acetaminophen can be purchased in our country without mayor difficulties. Comparing results with other countries, there are many similarities in terms of adulteration, the main substances are almost the same reported in Europe and United States of America, this fact confirms that some adulterants, specially forbidden com-

pounds, are added at the places of production. A different situation may occur with some diluents such as sugars, starch or carbonates that could be added almost in any country all over the world. Referring to of analytical methods, all implemented and validated techniques were suitable for chemical profiling of cocaine samples and allowed to us to perform this research.

CONCLUSIONS

The purity of cocaine varied significantly from 2006 to 2016. This could be a very dangerous problem for users who cannot estimate the appropriate amount for consumption; and are unaware of the toxic effects of adulterants added to the illicit cocaine sold in Chile. Temporal evolution about adulterants frequencies explains the dynamism of illicit drug market. Details behind addition of some adulterants to cocaine could help to elucidate the use of all these compounds; knowing that these substances are added with aim to enhance or mimic effects, increase addiction of cocaine and weight of the bulk material. This is a very important issue, especially when users are not able to distinguish the presence of any adulterants or diluents in street cocaine; and also have a partial or no knowledge about cocaine chemical profile and toxic effects of these substances present in a cocaine dose. The identification of considerable adulteration with substances responsible to produce acute or chronic toxicity is an important subject for consumers and health professionals. This type of studies also can allows to police enforcement to identify important data about drug traffic and which adulterants and/or diluents are added to cocaine, this research yields applicable information to understand and to compare the organization of illicit drug markets and border controls or internal diversion of non-controlled substances used as diluents or adulterants. To the best of our knowledge this is the first report in our country about purity profile of cocaine hydrochloride and substances added in such a long period and with a large number of samples analyzed.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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AUTHOR CONTRIBUTION:

Contribution	Duffau BE	Rojas SA	Ayala SA
Concepts or ideas	x	x	x
Design	x	x	x
Definition of intellectual content	x	x	
Literature search	x		
Experimental studies	x	x	
Data acquisition	x	x	x
Data analysis	x		x
Statistical analysis			x
Manuscript preparation	x	x	
Manuscript editing		x	
Manuscript review	x	x	x

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