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Comparative determination of total isomeric truxillines in illicit, refined, South American cocaine hydrochloride using capillary gas chromatography–electron capture detection

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Abstract

The isomeric truxillines and their hydrolysis products are present as manufacturing impurities in most illicit, refined cocaine samples. Methodology is described for the comparative gas chromatographic determination of total isomeric truxilline manufacturing impurities/by-products, i.e., intact truxillines and their hydrolysis products, in illicit, refined cocaine samples seized in South America. These isomers, namely, alpha-, beta-, delta-, epsilon-, gamma-, omega-, zeta-, peri-+neo- and epi-truxilline, were all quantified relative to mu-truxillic acid, a structurally-related internal standard. In this method, the cocaine samples were first subjected to treatment with boron trifluoride-methanol, followed by lithium aluminum hydride reduction and then acylation with heptafluorobutyric anhydride. The resultant di-heptafluorobutyryl derivatives of the truxillyl and truxinyl diols exhibited excellent chromatography and low-picogram on-column detection when using a moderately polar fused-silica capillary column interfaced with an electron-capture detector. This methodology demonstrated good reproducibility, as determined by the repetitive analysis of a selected illicit cocaine exhibit. The analysis of 117 unadulterated, illicit, refined cocaine samples revealed that total truxilline levels ranged from 0.2–12.3% (w/w relative to cocaine), with the two most abundant truxillines being the alpha- and beta- isomers. Truxilline data for the total individual truxilline isomers in cocaine hydrochloride exhibits from five South American countries are presented.

Keywords: Truxilline; Cocaine

1. Introduction

The characterization of manufacturing impurities and by-products in illicit drugs is important for the determination of the geographic origin of certain drugs and for the comparative analyses of multiple drug seizures to assess commonality for criminal conspiracy cases [1–3]. One illicit drug of extensive forensic interest is cocaine, a powerful stimulant derived from South American coca, which is cultivated mostly in Bolivia and Peru. During the illicit cocaine manufacturing process [4], minor alkaloids present in coca leaf are co-extracted with cocaine and usually appear at trace levels in the refined product.

One group of minor tropane alkaloids present in coca leaf are the eleven isomeric truxillines, which are cyclobutane compounds that arise possibly from the 2+2 photodimerization of *cis*- and/or *trans*-cinnamoylcocaines. The two most abundant of these

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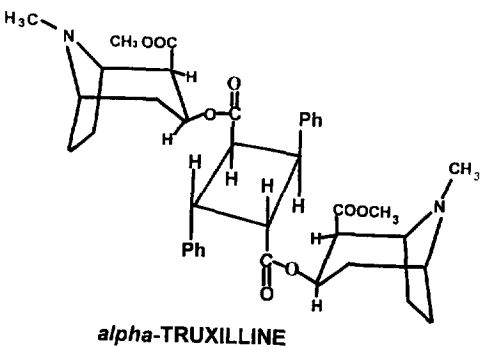

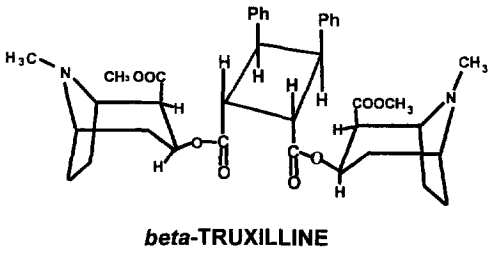

Fig. 1. alpha- and beta-truxilline.

alkaloids, alpha- and beta-truxilline, seen in Fig. 1, were structurally elucidated around the turn of the century [5,6]. More recently, all eleven truxilline isomers were detected in refined, illicit cocaine, and their presence subsequently confirmed in coca leaf [7]. In addition, quantitative data for truxillines in South American coca leaf has been reported [8], a preliminary report on their determination in illicit, refined cocaine presented [9] and the relationship between their presence in coca leaf and in illicit, refined cocaine samples reviewed [1,2]. High-performance liquid chromatographic [10,11] and thin-layer chromatographic–mass spectrometric [12] methods for the truxillines have also been reported.

In this study, the individual and total truxilline contents were determined for 117 cocaine hydrochloride samples seized in five South American countries. Each sample was subjected to esterification with boron trifluoride–methanol, followed by extraction, lithium aluminum hydride reduction and perfluoroacylation using heptafluorobutyric anhydride; the resulting di-heptafluorobutyryl derivatives were chromatographed and quantitated using a moderately-polar fused-silica capillary column interfaced with a ^{63}Ni electron-capture detector.

2. Experimental

2.1. Cocaine hydrochloride samples

A total of 117 cocaine hydrochloride samples seized in Bolivia, Peru, Brazil, Ecuador and Colombia between 1970 and 1985 were submitted to this laboratory soon after seizure and stored in a vault under ambient conditions, mostly in the absence of light. No pre-treatment of samples was done prior to the analyses.

2.2. Solvents, chemicals and standards

All solvents were distilled-in-glass products of Burdick and Jackson Labs (Muskegon, MI, USA) and were preservative- and peroxide-free. Heptafluorobutyric anhydride (HFBA), supplied in 1-ml sealed glass ampules, and boron trifluoride-methanol (BF_3–MeOH) were products of Pierce (Rockford, IL, USA). A 1.0 *M* etherial solution of lithium aluminum hydride ($LiAlH_4$) was obtained from Aldrich (Milwaukee, WI, USA).

The methodology internal standard used for quantitative purposes was mu-truxillic acid, prepared as described in Ref. [7]. Aldrin, the gas chromatographic internal standard, was a product of Supelco (Bellefonte, PA, USA).

2.3. Capillary gas chromatography–electron capture detection (cGC–ECD)

All chromatograms were generated in the splitless mode using a Hewlett-Packard 5880A gas chromatograph (GC) fitted with a 30 m×0.25 mm I.D. fused-silica capillary column coated with DB-1701 (J&W Scientific, Rancho Cordova, CA, USA) at a film thickness of 0.25 μm. The GC was equipped with a ^{63}Ni electron capture detector (ECD) (15 mCi) and interfaced with a Hewlett-Packard Level IV data processor. The oven temperature was programmed as follows: (level 1) initial temperature, 90°C; initial hold, 1.6 min; temperature program rate, 25°C/min; final temperature, 160°C; final hold, 1.0 min; (level

2) temperature program rate, 4°C/min; final temperature, 275°C; final hold, 5 min. Injector and detector temperatures were maintained at 275°C and 300°C, respectively. Hydrogen (Zero Grade, Air Products, Tamaqua, PA, USA) was used as a carrier gas at a velocity of about 40–45 cm/s and measured for isooctane at an oven temperature of 90°C. An argon–methane (95:5) mixture (Air Products) was used as the detector make-up gas at a flow-rate of about 35 ml/min. The septae used were Thermogreen LB-2 (Supelco, Belfonte, PA, USA). During the splitless injection the solvent was vented after a 1.0 min hold.

2.4. Methyl esterification, $LiAlH_4$ reduction and HFBA derivatization of illicit cocaine samples

Approximately 50 mg of the unadulterated cocaine hydrochloride sample was accurately weighed into a 15-ml conical glass centrifuge tube containing 100.0 μg of mu-truxillic acid internal standard. To the tube was added 0.5 ml of BF_3–MeOH and the resulting solution heated at approximately 95°C for 2 h. After cooling the reaction to approximately −10°C (acetone/ice), 2 ml of ice water and 6 ml of 20% sodium hydroxide (also cooled to approximately −10°C) were added to the tube with mixing. Without delay the basified solution was extracted with 5×5-ml aliquots of ethyl ether, with each extract filtered through anhydrous sodium sulfate into a 25-ml volumetric flask, diluted to volume and mixed.

A 2.0-ml aliquot from this solution was transferred to a 15-ml glass-stoppered centrifuge tube and evaporated to dryness under a stream of nitrogen. To the residue was added 200 μl of chloroform and heated at 75°C for 3 min with occasional vortex mixing. To the tube was added 4 ml of anhydrous ethyl ether (stored over 5 Å molecular sieve) followed by 0.20 ml of 1 *M* $LiAlH_4$ in ethyl ether. After vortex mixing, the solution was reduced spontaneously in volume to less than 0.5 ml (but not to dryness) by heating at 50–55°C. A 5-ml volume of 1 *M* sulfuric acid was *carefully* added to the test tube, mixed by vortex and extracted with 3×5-ml aliquots of ethyl ether. Each extract was passed through anhydrous sodium sulfate into a 15-ml glass-stoppered centrifuge tube. The extracts were evaporated *just to dryness* under a stream of nitrogen.

Without delay, 1.0 ml of acetonitrile and 50 μl of HFBA were added to the tube which was mixed by vortexing and heated at 75°C for 10 min. To the tube was added 10 μl of pyridine and heated for an additional 2 min. After cooling, 8.0 ml of isooctane (containing aldrin internal standard at 200 pg/μl) was added to the tube, followed by 5 ml of an aqueous solution saturated with sodium bicarbonate; *without delay* the contents of the tube were vigorously mixed. The tube was centrifuged and the isooctane layer transferred to another tube and dried over anhydrous sodium sulfate. About 2 μl of the isooctane extract was injected into the cGC–ECD under conditions described previously (Section 2.3).

3. Results and discussion

3.1. Chromatography of the truxillines

The truxillines are difficult to chromatograph directly by GC, due to their large mass and thermal lability [7]. Our attempts to chromatograph the truxillines resulted in two or more peaks, representing the degradation products ecgonidine methyl ester (major GC peak) and various truxillic/truxinic acids. Although some of the truxillines have been chromatographed successfully using HPLC [10,11] and TLC [12] those methods were unable to detect/resolve all eleven isomers. Furthermore, those studies did not provide quantitative data.

3.2. Truxilline hydrolysis and sample shelf-life

alpha-Truxilline and its possible hydrolysis products (a–e) are illustrated in Fig. 2. It has been previously established that the truxillic/truxinic acid hydrolytes (e.g., structure (e) in Fig. 2) are present in illicit cocaine hydrochloride [7]. In this study, when an illicit cocaine sample was subjected to alumina column chromatography (in order to isolate the truxillines), HPLC–MS–MS analysis of the most polar column eluate (methanol) revealed significant truxilline hydrolysis, with the primary hydrolytes being of the truxilloyl/truxinoyl type, e.g., structure (a) in Fig. 2; there was also evidence for the presence of compounds (b), (d) and (e). In all likelihood, these

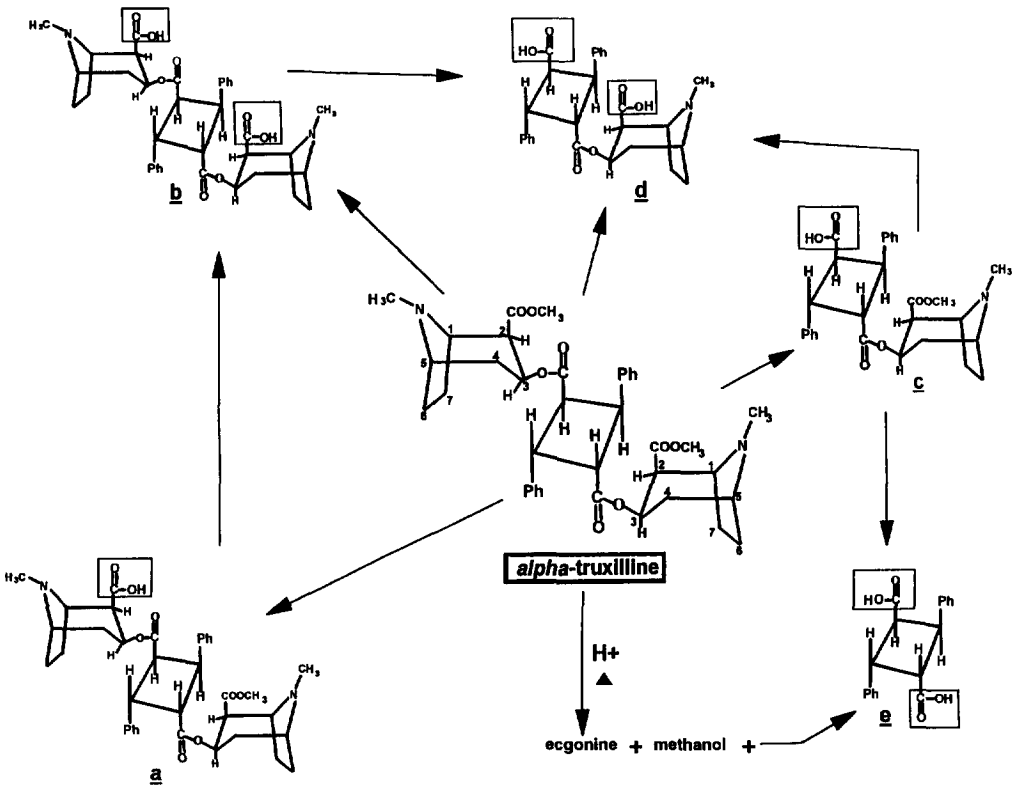

Fig. 2. alpha-Truxilline hydrolysis products believed present in illict, refined cocaine samples.

hydrolytes are also present at widely varying levels in most illicit cocaine.

As a prelude to the development of the methodology described herein, a survey of illicit cocaine exhibits of varying shelf-life (1–15 yr) stored in our laboratory was conducted, and revealed a direct relationship between sample "age" and the level of truxillic/truxinic acids (e.g., structure (e)) in those samples. Not surprisingly, the older cocaine samples generally had lower intact truxilline levels and higher amounts of the truxillic/truxinic acids. This relationship is expected to hold for the other truxilline hydrolytes (a–d).

3.3. BF_3–methanol esterification, $LiAlH_4$ reduction and HFBA derivatization

To render sample age irrelevant in the quantitation of truxillines in illicit cocaine samples, the methodology herein was developed to allow for the determination of the "total" truxillines for each isomer. This was accomplished by first subjecting the samples to methyl esterification using BF_3–methanol. These reactions are illustrated in Fig. 3 for two of the more abundant types of truxilline hydrolytes, (a) and (e). The methyl esterification rendered the hydrolytes suitable for high-yield extraction from aqueous base into chloroform. As shown, the intact truxillines do not participate in the reaction. Without prior esterification, high-yield extraction of some of the truxilline hydrolytes from basic solutions is problematic. Conversely, the "intact" truxillines are extracted in high yield from aqueous base into chloroform.

Once the truxillines and methylated hydrolytes were isolated in their free base form (or, in the case of truxillic/truxinic acids, as neutral compounds), they were subjected to $LiAlH_4$ reduction. This step proceeded smoothly and in high yield, giving truxillyl and truxinyl alcohols (i.e., diols). HFBA de-

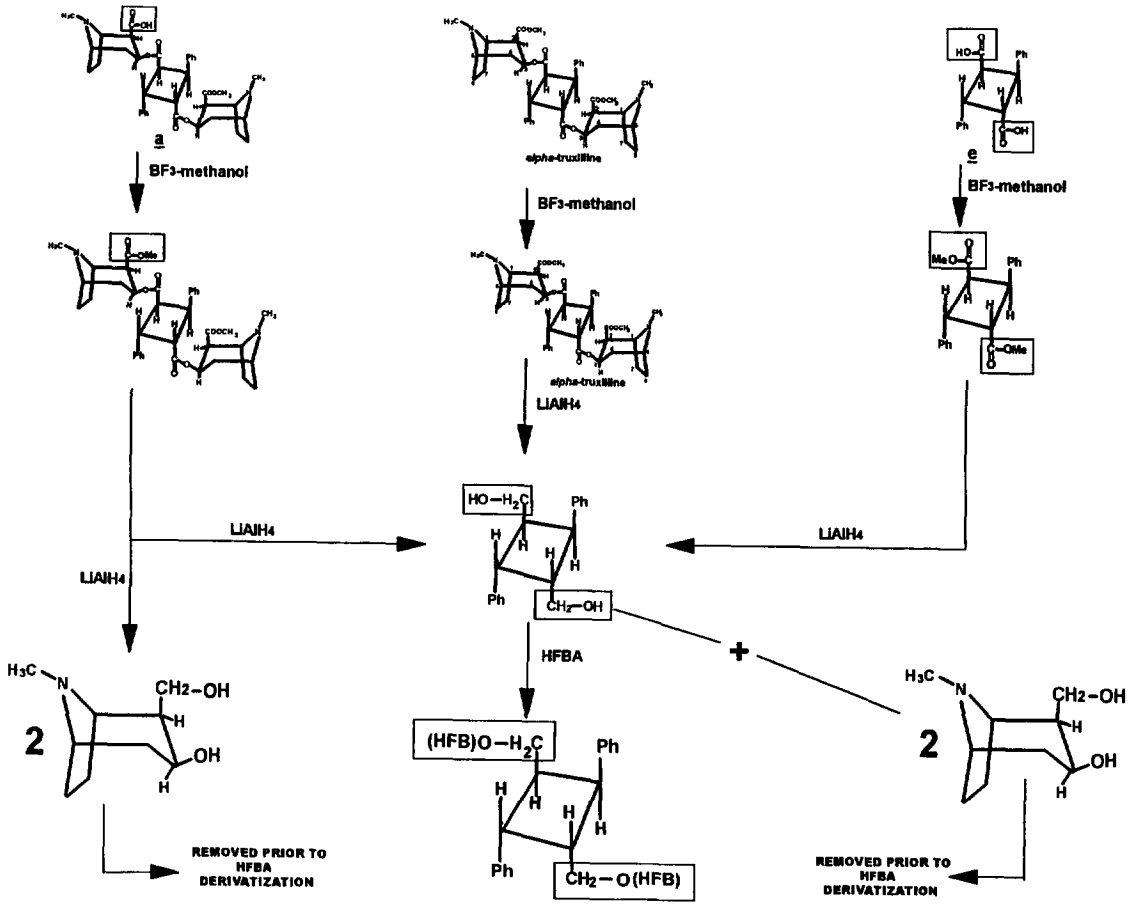

Fig. 3. Reaction pathways illustrating the BF_3–MeOH treatment, $LiAlH_4$ reduction and HFBA derivatization of alpha-truxilline and its two most abundant hydrolysis products.

rivatization of these diols (Fig. 3) proceeded quantitatively and virtually instantaneously. The di-HFB derivatives of the eleven isomeric diols were then extracted in high yield into isooctane while the excess HFBA was concomitantly hydrolyzed and retained in the sodium bicarbonate/water phase as sodium heptafluorobutyrate.

It should be noted that diols could alternatively have been trimethylsilylated (in lieu of perfluoroacylation) and their trimethylsilyl (TMS) derivatives chromatographed; however, since di-TMS derivatives are not sufficiently electrophilic to provide a significant ECD response, a flame ionization detector (FID) would be required. This would result in a 2–3 order-of-magnitude reduction in method sensitivity. Furthermore, unlike their HFB counterparts, the eleven di-TMS diol isomers are not all resolved on capillary GC columns [13].

3.4. Chromatography and detection levels for di-HFB derivatives of the truxillyl/truxinyl alcohols

After evaluating several capillary columns of differing polarity (DB-1, DB-5 and DB-1701) only DB-1701 allowed for detection of all eleven $LiAlH_4$-reduced/HFBA-derivatized isomeric truxillines [7]. This chromatogram is illustrated in Fig. 4. Table 1 gives retention time data for the isomeric di-HFB derivatives of the truxillyl and truxinyl alcohols.

Due in part to the inherent sensitivity of the ECD towards di-O-HFB electrophiles, the reduced/derivatized truxillines were readily detected in all 117

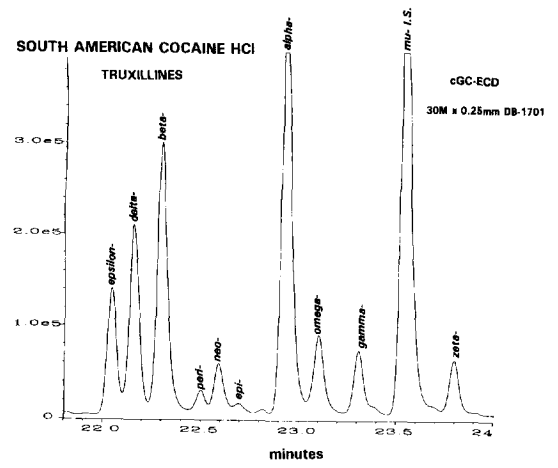

Fig. 4. Partial capillary gas chromatographic–electron capture detection chromatogram illustrating the isomeric truxillines after their $LiAlH_4$ reduction and HFBA derivatization.

illicit cocaine samples examined. An added factor in their facile determination was their relatively high "carry-through" (along with cocaine) in the illicit manufacturing process. On-column minimum detectable quantity (GC–MDQ) levels for the di-O-HFB derivatives (Fig. 3) were estimated to be 1–5 pg. Methodology minimum detectable quantity (MTHD–MDQ) levels were well below 0.001% (relative to 50 mg cocaine).

Table 1
Gas chromatographic retention times of the di-heptafluorobutyryl derivatives of the truxillyl and truxinyl diols[a]

Truxilline isomer	GC retention times[b]
Aldrin I.S.[c]	18.84
Epsilon-	22.03
Delta-	22.15
Beta-	22.29
Peri-	22.49
Neo-	22.58
Epi-	22.68
Alpha-	22.92
Omega-	23.09
Gamma-	23.30
Mu-I.S.[d]	23.54
Zeta-	23.79

[a] Truxillines subjected to $LiAlH_4$ reduction and HFBA derivatization, yielding di-O-HFB derivatives.
[b] See Section 2.3 for GC conditions; retention times in min.
[c] Aldrin used as an instrumental internal standard.
[d] mu-Truxillic acid used as methodology internal standard.

3.5. Determination of the truxillines using mu-truxillic acid

In order to provide acceptable quantitative data for the truxillines, the structurally-related mu-truxillic acid was incorporated in the method. In a previous survey of illicit cocaine samples, it was determined that the level of mu-isomer was negligible, allowing for its use as a methodology internal standard [13]. It was also established that the molar ECD response of the di-O-HFB derivatives of the reduced mu-truxillic acid was comparable to those for both alpha-truxillic and beta-truxinic acids. The mu-truxillic acid internal standard was introduced just prior to the BF_3–methanol treatment of the sample.

3.6. Incorporation of a standard sample

The analysis of the 117 samples proceeded over a four-month period. Over such a prolonged time frame, minor variations in chromatographic conditions and small anomalies in reduction, derivatization and extraction steps are expected. Therefore, in order to further enhance quantitative accuracy for the truxillines, and to normalize results over this time period, an illicit cocaine standard sample containing nominal levels of the truxillines was interspersed throughout the sample population and analyzed repetitively. An average of 4 or 5 illicit samples along with the standard sample were analyzed daily. After completion of the analyses of all 117 cocaine samples, the average truxilline content for the 10 isomers in the standard sample (representing about 23 repetetive analyses) was calculated. The average individual truxilline results obtained for the standard sample over the four-month period were then ratioed to the results obtained for the daily standard sample run. In this manner, a numerical factor was generated that could be applied to the individual truxilline results for those illicit samples run on any given day. This, in effect, normalized all results for the four-month period.

3.7. Method reproducibility

To ascertain reproducibility of the total truxillines methodology, the standard sample was subjected to 12 repetitive analyses over 2–3 days. Each analysis

Table 2
Reproducibility of $LiAlH_4$-reduced and HFBA-derivatized truxilline methodology[a,b]

Truxilline/mu-internal standard (Peak Area/Peak Area)	Relative standard deviation (%)
Epsilon-/mu-	±1.89
Delta-/mu-	±2.06
Beta-/mu-	±2.28
Peri-+neo-/mu-	±1.80
Epi-/mu-	±4.67
Alpha-/mu-	±1.71
Omega-/mu-	±1.64
Gamma-/mu-	±1.95
Zeta-/mu-	±3.96

[a] Based upon the repetitive analysis ($N=12$) of a selected illicit, refined cocaine HCl sample and duplicate injections for each sample run.
[b] Data generated by ratioing the peak areas of the individual truxillines to the peak area of the mu-truxillic acid internal standard.

incorporated duplicate GC injections. All data was generated by ratioing the peak areas for the truxilline isomers to that for the mu-isomer. These results are given in Table 2. As seen, all isomers fell within a relative standard deviation range of ±1.64–4.67%. When the standard sample was repetitively analyzed ($n=31$) over the four-month period, the relative standard deviation for the total truxilline content was determined to be ±5.53%.

3.8. Truxilline calculations

The following equation, with the alpha-isomer as an example, was used to calculate truxilline quantitative data:

%alpha − truxilline = [(alpha − truxillic acid area × mu − truxillic acid std conc (μg/ml) × normalization factor × 2.22/(mu − truxillic acid std area × cocaine weight (μg))] × 100

The use of the normalization factor was discussed previously. A multiplier of 2.22 was applied to convert the truxillic/truxinic acid values to their respective intact truxillines. All truxilline results were % w/w relative to the cocaine content.

3.9. Truxilline data

The samples analyzed in this study were seized in Colombia (52 samples), Brazil (14 samples), Ecuador (16 samples), Peru (16 samples) and Bolivia (19 samples). The average individual truxilline results for those samples, along with their quantitative ranges and relative standard deviations (R.S.D.) are found in Tables 3–5. At the time of this study, the resolution between the peaks representing the peri- and neo-isomers was less than satisfactory on the DB-1701 column, so their peak areas were summed for calculation purposes.

Quantifiable amounts of the truxillines were present in all 117 samples. This is in contrast to cocaine impurities such as tropacocaine and the cinnamoylcocaines, both of which can be below quantitative levels in illicit samples [1]. As seen in Table 3, the two isomers of greatest abundance in samples from the five countries were alpha- and beta-truxilline. These were followed in descending order of concentration by the delta-, epsilon-, omega- and gamma-isomers (excluding the summed peri-+neo-trux-

Table 3
Average truxilline isomer content for illicit cocaine HCl seizures from South America[a,b]

Country	Average									
	Alpha-	Beta-	Delta-	Epsilon-	Omega-	Gamma-	Zeta-	Epi-	Peri-+Neo-	Total
Peru	0.41	0.43	0.21	0.15	0.05	0.04	0.004	0.01	0.07	1.38
Bolivia	0.51	0.49	0.25	0.19	0.07	0.05	0.01	0.02	0.08	1.66
Brazil	0.63	0.54	0.27	0.22	0.09	0.07	0.01	0.02	0.09	1.92
Ecuador	1.21	1.05	0.46	0.34	0.19	0.15	0.04	0.03	0.20	3.66
Colombia	1.33	1.07	0.51	0.39	0.23	0.19	0.04	0.03	0.22	4.01

[a] All results are % w/w relative to cocaine content.
[b] Samples analyzed: Peru ($N=16$), Bolivia ($N=19$), Brazil ($N=14$), Ecuador ($N=16$) and Colombia ($N=52$).

Table 4
Ranges for truxilline isomer content of illicit cocaine HCl seizures from South America[a,b]

Country	Average									
	Alpha-	Beta-	Delta-	Epsilon-	Omega-	Gamma-	Zeta-	Epi-	Peri-+Neo-	Total
Peru	0.05–0.77	0.04–1.02	0.06–0.49	0.03–0.33	0.01–0.11	0.01–0.07	0.00–0.02	0.00–0.02	0.02–0.12	0.22–2.77
Bolivia	0.19–0.95	0.17–0.81	0.09–0.42	0.08–0.31	0.02–0.13	0.02–0.10	0.00–0.03	0.01–0.03	0.03–0.17	0.63–2.56
Brazil	0.31–1.06	0.27–1.19	0.10–0.49	0.12–0.41	0.03–0.14	0.03–0.14	0.00–0.02	0.01–0.03	0.05–0.19	1.16–3.61
Ecuador	0.12–3.95	0.10–3.38	0.08–1.66	0.04–1.02	0.02–0.65	0.01–0.54	0.00–0.21	0.00–0.08	0.00–0.79	0.39–12.3
Colombia	0.17–3.06	0.10–3.20	0.11–1.26	0.09–0.81	0.03–0.67	0.00–0.49	0.00–0.20	0.01–0.08	0.04–0.65	0.57–9.56

[a] All ranges for truxillines are % w/w relative to cocaine content.
[b] Samples analyzed: Peru ($N=16$), Bolivia ($N=19$), Brazil ($N=14$), Ecuador ($N=16$) and Colombia ($N=52$).

illines). The zeta- and epi-isomers were at lower levels in most samples. As stated previously, the presence of mu-truxilline is negligible in illicit cocaine, allowing it to be used as a methodology internal standard. The lowest total truxilline content, 0.22%, was found in a Peruvian sample, whereas the highest level, 12.3%, belonged to a sample from Ecuador.

The greatest ranges and R.S.D. values for the truxilline isomers, shown in Tables 4 and 5, were found in samples from Ecuador and Colombia. It is interesting to note that for the most abundant truxilline isomers, samples from Peru had markedly higher R.S.D.s than cocaine seized in Bolivia (see Table 5). This might reflect diversity in the manufacturing process or coca leaf variety. However, many more samples need to be analyzed to allow for more meaningful statistical analyses.

Though not seen in Table 3, only one sample (Brazil) from the 49 exhibits representing Peru/Bolivia/Brazil had a total truxilline content over 3%. This is in marked contrast to 68 cocaine exhibits from Colombia/Ecuador, in which 36 samples had total truxilline levels of over 3%. Furthermore, Colombia and Ecuador had much wider ranges in truxilline content compared to the other countries. This allowed for the division of samples into two groups based upon truxilline levels; one representing Colombia/Ecuador and the other Peru/Bolivia/Brazil. As seen in Table 3, the samples from Colombia/Ecuador have markedly higher amounts of all truxilline isomers. One reason could be that many of the samples from Colombian/Ecuador were derived from coca leaf that was chemotaxinomically different than leaf used to produce the cocaine from the other three countries. Another reason could be that the samples from Peru/Bolivia/Brazil were subjected to a manufacturing methodology that was superior in the removal of truxillines from the final cocaine product. The latter explanation appears less probable, however, because cocaine samples have been encountered that possessed high truxilline levels and, concomitantly, much lower amounts of other alkaloidal impurities (e.g., the cinnamoylco-

Table 5
Relative standard deviations for truxilline isomer content in illicit cocaine HCl seizures[a]

Country	Relative Standard Deviation[b]								
	Alpha-	Beta-	Delta-	Epsilon-	Omega-	Gamma-	Zeta-	Epi-	Peri-+Neo-
Peru	51.3	64.5	53.5	56.3	53.0	44.7	≥100	45.5	42.9
Bolivia	41.1	35.5	38.5	40.2	46.2	45.7	≥100	44.2	22.3
Brazil	35.9	45.2	39.7	37.7	34.4	48.0	70.0	35.4	41.8
Ecuador	78.1	79.1	85.4	74.1	90.4	89.0	≥100	73.0	95.9
Colombia	60.1	63.1	58.7	52.9	69.8	73.0	≥100	56.8	66.5

[a] Samples analyzed: Peru ($N=16$), Bolivia ($N=19$), Brazil ($N=14$), Ecuador ($N=16$) and Colombia ($N=52$).
[b] Relative standard deviation data given as ±%, e.g., ±50%.

caines), and visa versa. We are continuing to investigate these issues.

3.10. Sample individuality

An important factor in the application of sample comparison analyses to criminal conspiracy cases, and eventual expert court testimony, is the uniqueness of chromatographic profiles generated by a specific analytical method. In this study, the 117 samples analyzed generated an equal number of truxilline chromatographic profiles. For each profile (and, therefore, for each sample) the quantitative levels of indiviual isomeric truxillines and the total truxilline content were determined. The samples were then listed in order of decreasing total truxilline content; for those samples with similar similar total truxilline contents, i.e., within 5%, the individual isomeric truxilline levels were compared to determine whether those samples had profiles that "matched." An analysis of all the truxilline data revealed that, within experimental error (see Table 2), each of the 117 samples was uniquely individual. Given the good reproducibility of this method (Table 2), it appears to be ideally suited for comparative analyses.

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