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# The Anatomy of a Cocaine Comparison Case: A Prosecutorial and Chemistry Perspective

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**ABSTRACT:** Methodology used for the comparative chemical analyses of two illicit cocaine seizures, and its application in a successful criminal prosecution, is described. A description of events leading to the arrest of the defendant and an overview of the jury trial are provided. Illicit cocaine, found in the defendant's suitcase and wallet, was subjected to chemical derivatization and three distinct gas chromatographic methods for the detection and relative quantitation of cocaine manufacturing impurities/by-products. The cocaine impurities included *cis*- and *trans*-cinnamoylcocaine, the isomeric truxillines and the hydroxycocaines. Among the cocaine manufacturing byproducts detected were benzoylecgonine, ecgonine methyl ester, ecgonine, N-benzoylnorecgonine methyl ester and N-norcocaine. Chemical derivatization of the cocaine samples was accomplished using heptafluorobutyric anhydride and N,O-bis(trimethylsilyl)acetamide. The derivatized impurities/by-products were subjected to capillary gas chromatographic analysis using both flame ionization and electron-capture detectors. The comparative chemical analyses provided a positive correlation between the suitcase and wallet cocaine samples.

**KEYWORDS:** criminalistics, toxicology, cocaine, illicit drugs

The characterization of manufacturing impurities and byproducts present in illicit drugs is useful for geographic origin determinations and sample comparison analyses [*1*]. The latter has been addressed recently by a number of investigators for illicit cocaine [*1–16*]. We now describe a cocaine comparison case that is believed to be the first successful federal prosecution based largely upon data generated by indepth cocaine comparison analyses using analytical methodology described herein.

## Background and Prosecution

Each year, scores of passengers arrive at Miami International Airport (MIA) carrying not only their luggage but also illicit drugs, primarily cocaine. The cocaine is strapped on their bodies, hidden in their suitcases, secreted inside their body cavities, and even

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ingested via latex-covered pellets. Often these smugglers arrive from South American countries, are inexperienced travelers and act very nervously. They emit warning signs that result in their arrest by experienced Custom inspectors and eventual prosecution.

On the morning of December 24, 1990, John Doe, a citizen of Chile, arrived at MIA. Unlike the individuals described above, Mr. Doe was well-dressed, calm and sophisticated. Fortunately, as the plane luggage was being unloaded, an alert Customs inspector noticed that Mr. Doe's suitcase was extremely heavy. Further X-ray inspection revealed suspicious packages in the form of bricks. Finally, a narcotics-detector dog confirmed the probable presence of narcotics in the suitcase.

The Customs agents allowed the suitcase to enter the carousel, so that its owner could be identified. When Mr. Doe picked up the suitcase, a Customs inspector initiated questioning. Mr. Doe explained, and his ticket confirmed, that he was headed to Oregon for the Christmas holidays. At a secondary search area, the Customs inspectors opened the suitcase and found 30 one-half kilogram bricks, each individually wrapped in Christmas paper. Mr. Doe stated that the packages were Christmas presents. The inspectors, suspecting the presence of cocaine, field-tested the approximately 15 kg of bricks and observed a positive reaction. Mr. Doe expressed surprise, telling the agents that he thought the packages contained money. Subsequently, one of the agents found an additional 4 and one-half g of cocaine inside a folded magazine page tucked in a wallet inside Doe's briefcase.

A grand jury sitting in the Southern District of Florida charged Doe with violations of [21 U.S.C. 841(a)(1)] (possession of cocaine with intent to distribute) and [21 U.S.C. 952(a)] (importation of cocaine). The key element in the case, as in many other drug prosecutions, was to prove that John Doe intentionally and knowingly possessed and imported the cocaine.

Mr. Doe pled not guilty and proceeded to trial in early 1991. Unlike other airport smugglers, he mounted an aggressive and well-financed defense. He asserted quite effectively that he had carried the suitcase packages, which he thought contained money, as a favor for one "Roberto Rojas," supposedly a Chilean officer with close ties to a former head of the Chilean government. Mr. Doe even admitted that he had carried packages to Miami for "Rojas" on two prior occasions. As for the cocaine in his wallet, Doe explained that another Chilean acquaintance, "Rodrigo Perez," had offered the cocaine for personal consumption at a Christmas party in Chile. Adding that he never intended to actually use the cocaine, Doe claimed that he had absent-mindedly left it inside his wallet. Mr. Doe buttressed his defense by calling several witnesses, residents of Chile who corroborated major aspects of his testimony.

Mr. Doe's defense made it important for the government to prove that the suitcase and wallet samples were closely related and came from the same source, rather than the two different sources ("Rojas" and "Perez") identified by Doe. Unfortunately, no comparative chemical analyses of the suitcase and wallet cocaine were done by the government prior to trial. A Drug Enforcement Administration (DEA) field chemist testified that the wallet and cocaine samples had similarly high purities. A defense chemist, however, exacerbated the situation by claiming that his analyses, which was superficial at best, revealed that the suitcase and wallet cocaine samples were not related. This fact, combined with Mr. Doe's and other witnesses' testimony, cast sufficient doubt upon the government's case to result in a hung jury after three days of deliberation.

In December of 1991, the defendant was retried. Prior to the second trial, samples from the suitcase and wallet cocaine were sent to the DEA's Special Testing and Research Laboratory for indepth comparative chemical analysis. The results revealed that the majority of cocaine in the suitcase was markedly similar to the wallet sample in their trace chemical composition. In fact the chemical profiles for two of the suitcase samples were virtually identical with the wallet cocaine sample. The government chemist deliv-

ered his findings at the second trial. His testimony before the jury was enhanced by the presentation of enlarged gas chromatograms, which revealed the close chemical relationship the suitcase and wallet cocaine samples shared. For contrast purposes, chromatograms of an unrelated cocaine sample were also presented. As in the first trial, Mr. Doe and his supporting witnesses testified, again pointing to "Rojas" and "Perez" as the different and unrelated sources of the suitcase and wallet cocaine. Mr. Doe also presented a second defense chemist who *had not* performed his own comparative analysis of the cocaine samples. Nonetheless, the defense chemist disputed the findings of the government chemist. However, on rebuttal the DEA chemist showed how the defense chemist's testimony, in fact, supported the DEA laboratory's findings. The Assistant United States Attorney this time had ample ammunition to argue at closing that Mr. Doe's story was a fabrication, since it was highly improbable that Doe would have obtained virtually identical cocaine from two separate sources.

At the conclusion of the trial, the jury deliberated for approximately two hours, rather than three days as the first jury had done. The jury's verdict is set forth at the conclusion of this paper, as is a description of the expert opinions rendered by both the government and defense chemists concerning the relationship of the suitcase and wallet samples. Described below is the analytical methodology used by the government chemists and the results obtained in the comparative analyses of the suitcase and wallet samples.

## Chemistry

This section describes the essential elements that were considered and implemented for the chemical comparison analyses of the suitcase and wallet cocaine samples.

### *Selection of Comparison Methodology*

It is important to recognize that there are no standardized methods for conducting illicit drug comparative analyses. With respect to cocaine comparison analyses, there are a number of factors to consider when choosing the analytical methodology and evaluating the results. These include the experience and level of expertise of the chemist, knowledge of the cocaine manufacturing process, an understanding of the chemistry of cocaine manufacturing impurities, the availability of appropriate instrumentation and the size of existing cocaine impurity profile data bases. It is also recognized that there is a certain element of subjectivity that is exercised during the selection process. For example, in this paper we describe three gas chromatographic (GC) methods used in the comparison of cocaine manufacturing impurities/byproducts. It is probable, and altogether likely, that in the subjective opinion of another chemist only one or two GC methods would be necessary, and that those methods might differ from the ones described in this paper. Furthermore, a third chemist might reject the use of GC and, instead, invoke high-performance liquid chromatography (HPLC) as the method of choice for these analyses. What is of key importance, however, is that whatever methodology is chosen, the forensic chemist must be prepared to defend this selection, the results obtained and opinion rendered thereof, upon vigorous cross-examination during court testimony.

### *Comparative Chemical Analyses of Illicit Cocaine Samples—General Approach*

As mentioned previously, the illicit contraband seized in this case consisted of a suitcase holding 30 one-half kg packages and a wallet containing about 4 and one-half g of cocaine hydrochloride. Approximately one gram of cocaine from each of 18 suitcase packages and 1 gm from the wallet sample were placed in glass vials and submitted to

this laboratory for comparison analyses. Twelve of the 30 suitcase packages had been previously composited and were, therefore, not suitable for comparative analyses.

The analytical approach used in these comparison analyses was three-fold. First, each of the 18 suitcase samples and the wallet sample were subjected to a cocaine quantitative analysis. Second, each sample was "screened" for the presence of adulterants and diluents. Third, the samples were subjected to a manufacturing impurity profile analysis using three independent gas chromatographic methods. Upon completion of the foregoing analyses, all data was reviewed and an opinion offered regarding the relationship between the cocaine found in the suitcase and the cocaine found in the wallet.

### *Cocaine Quantitative Analysis*

Each of the 18 suitcase samples and the wallet sample were subjected to quantitative analyses using capillary gas chromatography-flame ionization detection (cGC-FID). Each sample was dissolved directly in chloroform containing a small volume of methanol and an appropriate internal standard. The sample solutions were injected into a 30 M × 0.25 mm i.d. fused-silica capillary column coated with DB-1 (0.25 μm) and quantified using a primary cocaine hydrochloride standard. These results are given in Table 1.

There were a number of reasons for determining the % purity of the cocaine samples. First, this quantitative data was necessary to determine the quantity of cocaine that was to be subjected to subsequent manufacturing impurity/byproduct profile analyses (Methods I, II and III). Secondly, knowing the cocaine content of the samples was helpful in determining whether significant levels of cocaine adulterants/diluents were present. Finally, the cocaine quantitative results could, in a very small measure, be included with other data when a final opinion was rendered regarding common origin of the suitcase and wallet samples. The cocaine quantitative data is given in Table 1.

Given the different storage conditions for the wallet sample versus the suitcase cocaine samples, the quantitative results from Table 1 did not dispute their commonality.

TABLE 1—*Quantitative cocaine results for the suitcase and wallet samples using capillary gas chromatography-flame ionization detection.*

| Sample | % Cocaine HCL |
|---|---|
| Suitcase Ex. 1 | 93.6 |
| Suitcase Ex. 2 | 94.0 |
| Suitcase Ex. 3 | 93.8 |
| Suitcase Ex. 4 | 94.3 |
| Suitcase Ex. 5 | 92.0 |
| Suitcase Ex. 6 | 93.1 |
| Suitcase Ex. 7 | 93.2 |
| Suitcase Ex. 8 | 93.3 |
| Suitcase Ex. 9 | 92.8 |
| Suitcase Ex. 10 | 92.6 |
| Suitcase Ex. 11 | 92.2 |
| Suitcase Ex. 12 | 92.5 |
| Suitcase Ex. 13 | 92.8 |
| Suitcase Ex. 14 | 92.1 |
| Suitcase Ex. 15 | 93.2 |
| Suitcase Ex. 16 | 92.4 |
| Suitcase Ex. 17 | 93.0 |
| Suitcase Ex. 18 | 92.6 |
| Wallet Ex. 1 | 91.4 |

*Detection of Adulterants and Diluents*

The cocaine quantitative results in Table 1 indicated that there was no significant adulteration or dilution of the cocaine samples. Indeed, routine "screening" of the samples did not reveal the presence of added substances. However, when Comparison Method II, described in the following, was applied to the wallet and suitcase cocaine samples, a peak appeared in the chromatograms that could not be ascribed to a cocaine manufacturing impurity or byproduct. Subsequent mass spectral analysis of the peak revealed it to be the tetra-trimethylsilyl derivative of citric acid. The citric acid was present in the wallet sample and most of the suitcase samples at levels under 1%.

The presence of citric acid in the wallet and suitcase samples was considered quite unique. The government chemist asserted that in his many years of experience in conducting indepth cocaine analyses, he was aware of only one other reporting of citric acid in a cocaine sample. Therefore, citric acid proved to be a useful parameter in supporting the relationship of the wallet and suitcase cocaine samples.

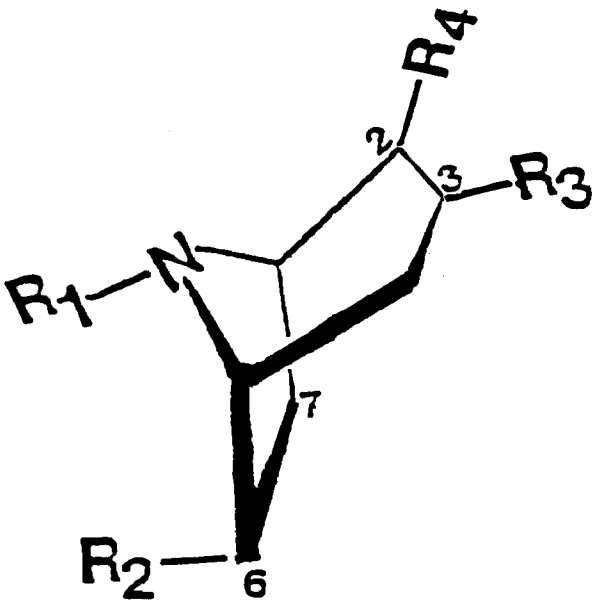
*Comparative Chemical Analyses of Cocaine Manufacturing Impurities/Byproducts*

The suitcase and wallet samples were subjected to three different capillary gas chromatographic procedures for the detection and relative quantitative analyses of their cocaine manufacturing impurities/byproducts. These comparison methods were, by far, the most important criteria for establishing the commonality of the wallet and suitcase cocaine samples. Since each of these methods used cocaine equivalents for the analyses, a direct quantitative comparison of the levels of manufacturing impurities/byproducts between the wallet and suitcase cocaine samples was possible. In Figs. 1 and 2 are given the structures for some of the compounds detected by the three comparison methods. Although 18 suitcase cocaine exhibits were analyzed, for the purposes of this paper, only the chromatograms for Exhibit #'s 8 and 14 were chosen for comparison with the wallet sample.

*Comparison Method I (Hydroxycocaine Method)*

Of the three comparison methods, this one was believed to be the most discriminatory. The Hydroxycocaine Method involved the direct chemical derivatization of the cocaine sample with heptafluorobutyric anhydride (HFBA)/acetonitrile followed by analysis using capillary gas chromatography-electron capture detection [*14*]. This method was a modification of a procedure for the detection of manufacturing impurities/byproducts in illicit heroin [*17*]. The Hydroxycocaine Method has been shown previously to exhibit excellent reproducibility [*14*]. This method was run repetitively on a suitcase cocaine exhibit to establish its homogeneity with respect to manufacturing impurities/byproducts. The sample proved to be homogeneous.

The cocaine impurities/byproducts detected using the Hydroxycocaine Method included ecgonine methyl ester, N-norcocaine, N-norecgonine methyl ester, N-nortropacocaine, N-benzoylnorecgonine methyl ester and at least seven suspected hydroxycocaine impurities, e.g., 6-hydroxycocaine (See Fig. 1). The comparative chromatographic profiles for the wallet sample and Exhibit #'s 8 and 14 of the suitcase samples are illustrated in Fig. 3*a*. In Figure 3*b* are shown chromatographic profiles of the wallet cocaine sample, Exhibit #8 of the suitcase cocaine sample and an unrelated cocaine sample. Figure 3*c* illustrates the chromatographic profiles of the wallet sample, Exhibit #8 of the suitcase sample and an unrelated sample known to have a purity level >99%. The Fig. 3 chromatograms illustrate only the retention time window of 26 to 36 minutes, as this section




1. $R_1$=$CH_3$; $R_2$=H; $R_3$=Ph-C(=O)-O; $R_4$=-C(=O)-$OCH_3$

2. $R_1$=$CH_3$; $R_2$=H; $R_3$=OH; $R_4$=-C(=O)-$OCH_3$

3. $R_1$=Ph-C(=O); $R_2$=H; $R_3$=OH; $R_4$=-C(=O)-$OCH_3$

4. $R_1$=$R_2$=H; $R_3$=Ph-C(=O)-O; $R_4$=-C(=O)-$OCH_3$

5. $R_1$=$R_2$=H; $R_3$=OH; $R_4$=-C(=O)-$OCH_3$

6. $R_1$=$CH_3$; $R_2$=OH; $R_3$=Ph-C(=O)-O; $R_4$=-C(=O)-$OCH_3$

7. $R_1$=$R_2$=H; $R_3$=Ph-C(=O)-O; $R_4$=H

8. $R_1$=$R_2$=H; $R_3$=OH; $R_4$=H

9. $R_1$=C(=O)-H; $R_2$=H; $R_3$=OH; $R_4$=-C(=O)-$OCH_3$

10. $R_1$=$CH_3$; $R_2$=H; $R_3$=Ph-C(=O)-O; $R_4$=-C(=O)-OH

11. $R_1$=$CH_3$; $R_2$=H; $R_3$=OH; $R_4$=C(=O)-OH

FIG. 1—*Structures of cocaine and some of its manufacturing impurities and byproducts*[a]

[a] *1* = Cocaine, *2* = Ecgonine Methyl Ester, *3* = N-Benzoylnorecgonine Methyl Ester, *4* = N-Norcocaine, *5* = N-Norecgonine Methyl Ester, *6* = 6-Hydroxycocaine, *7* = N-Nortropacocaine, *8* = N-Nor-ψ-Tropine, *9* = N-Formylnorecgonine Methyl Ester, *10* = Benzoylecgonine and *11* = Ecgonine.

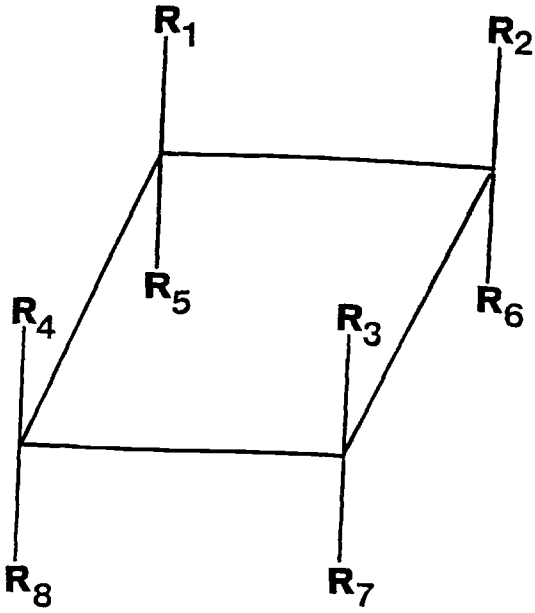



FIG. 2—*Structures for the eleven isomeric turxillines and their turxillic and turxinic acid hydrolysis products.*

of the chromatogram had been shown previously to be the most diagnostic and reproducible. In Table 2 are found peak identity and retention times for these chromatograms.

A review of the chromatograms in Fig. 3*a* revealed the marked similarity between the wallet cocaine sample and Exhibit #'s 8 and 14 of the suitcase samples provided by the Hydroxycocaine Method. Conversely, the dissimilarity between the wallet/suitcase samples and an unrelated cocaine sample is evident in Fig. 3*b*. The inclusion of Fig. 3*c* demonstrates the relatively peak-free chromatogram of a cocaine sample that had been refined using alumina column chromatography. This supported the proposition that the peaks present in the wallet and suitcase sample chromatograms represented bona-fide cocaine manufacturing impurities/byproducts and were not generated as method artifacts.

*Comparison Method II (Cinnamoylcocaine Method)*

In the Cinnamoylcocaine Method the cocaine samples were derivatized directly with N,O-bis(trimethylsilyl)acetamide (BSA) followed by analysis using capillary gas chromatography-flame ionization detection. This procedure was a modification of existing methodology [*10,18–20*]. The reproducibility of this method had been established previously.

Some of the cocaine impurities/byproducts that could be detected using the Cinnamoylcocaine Method included in the coca alkaloids *cis*- and *trans*-cinnamoylcocaine [*18*] and tropacocaine [*10*], and byproducts such as N-norcocaine and the cocaine hydrolysis products ecgonine, ecgonine methyl ester and benzoylecgonine (see Fig. 1). As mentioned previously, the presence of the contaminant citric acid was detected in both the wallet and suitcase cocaine samples using this methodology. The comparative chromatographic profiles for the wallet cocaine sample and Exhibit #'s 8 and 14 of the suitcase samples are illustrated in Fig. 4*a*. In Fig. 4*b* are shown chromatographic profiles of the wallet sample, Exhibit #8 of the suitcase sample and an unrelated cocaine sample.

| | Truxillic/Truxinic Acids | Truxillines[a] |
|---|---|---|
| 1) | *alpha*-[b], $R_1 = R_7 = COOH$, $R_4 = R_6 =$ phenyl, $R_2 = R_3 = R_5 = R_8 = H$ | *alpha*-, $R_1 = R_7 =$ Me ecgonine ester, $R_4 = R_6 =$ phenyl, $R_2 = R_3 = R_5 = R_8 = H$ |
| 2) | *beta*-[c], $R_5 = R_6 = COOH$, $R_3 = R_4 =$ phenyl, $R_1 = R_2 = R_7 = R_8 = H$ | *beta*-, $R_5 = R_6 =$ Me ecgonine ester, $R_3 = R_4 =$ phenyl, $R_1 = R_2 = R_7 = R_8 = H$ |
| 3) | *delta*-[c], $R_2 = R_5 = COOH$, $R_4 = R_7 =$ phenyl, $R_1 = R_3 = R_6 = R_8 = H$ | *delta*-, $R_2 = R_5 =$ Me ecgonine ester, $R_4 = R_7 =$ phenyl, $R_1 = R_3 = R_6 = R_8 = H$ |
| 4) | *epsilon*-[b], $R_5 = R_7 = COOH$, $R_2 = R_4 =$ phenyl, $R_1 = R_3 = R_6 = R_8 = H$ | *epsilon*-, $R_5 = R_7 =$ Me ecgonine ester, $R_2 = R_4 =$ phenyl, $R_1 = R_3 = R_6 = R_8 = H$ |
| 5) | *mu*-[c], $R_1 = R_6 = COOH$, $R_4 = R_7 =$ phenyl, $R_2 = R_3 = R_5 = R_8 = H$ | *mu*-, $R_1 = R_6 =$ Me Ecgonine ester, $R_4 = R_7 =$ phenyl, $R_2 = R_3 = R_5 = R_8 = H$ |
| 6) | *gamma*-[b], $R_1 = R_4 = COOH$, $R_4 = R_6 =$ phenyl, $R_2 = R_5 = R_7 = R_8 = H$ | *gamma*-, $R_1 = R_3 =$ Me ecgonine ester, $R_4 = R_8 =$ phenyl, $R_2 = R_5 = R_7 = R_8 = H$ |
| 7) | *neo*-[c], $R_2 = R_5 = COOH$, $R_3 = R_4 =$ phenyl, $R_1 = R_6 = R_7 = R_8 = H$ | *neo*-, $R_2 = R_5 =$ Me ecgonine ester, $R_3 = R_4 =$ phenyl, $R_1 = R_6 = R_7 = R_8 = H$ |
| 8) | zeta-[c], $R_5 = R_6 = COOH$, $R_4 = R_7 =$ phenyl, $R_1 = R_2 = R_3 = R_8 = H$ | *zeta*-, $R_5 = R_6 =$ Me ecgonine ester, $R_4 = R_7 =$ phenyl, $R_1 = R_2 = R_3 = R_8 = H$ |
| 9) | *epi*-[b], $R_1 = R_7 = COOH$, $R_2 = R_4 =$ phenyl, $R_3 = R_5 = R_6 = R_8 = H$ | *epi*-, $R_1 = R_7 =$ Me ecgonine ester, $R_2 = R_4 =$ phenyl, $R_3 = R_5 = R_6 = R_8 = H$ |
| 10) | *peri*-[b], $R_1 = R_3$-COOH, $R_2 = R_4 =$ phenyl, $R_5 = R_6 = R_7 = R_8 = H$ | *peri*-, $R_1 = R_3 =$ Me ecgonine ester, $R_2 = R_4 =$ phenyl, $R_5 = R_6 = R_7 = R_8 = H$ |
| 11) | *omega*-[c], $R_1 = R_2 = COOH$, $R_3 = R_4 =$ phenyl, $R_5 = R_6 = R_7 = R_8 = H$ | $R_1 = R_2 =$ Me ecgonine ester, $R_3 = R_4 =$ phenyl, $R_5 = R_6 = R_7 = R_8 = H$ |

[a]See Reference 2.
[b]Truxillic acid.
[c]Truxinic acid.

FIG. 2—*Continued.*

Figure 4*c* illustrates the chromatography for a standard of Merck cocaine hydrochloride. Peak identity and retention times for these chromatograms are found in Table 3.

A review of the chromatograms in Fig. 4*a* revealed the striking resemblance between the wallet cocaine sample and Exhibit #'s 8 and 14 of the suitcase sample using the Cinnamoylcocaine Method. Citric acid is represented by peak #3 in these chromatograms. In Figure 4(b) are shown the dissimilar chromatographic profiles of the wallet and suitcase Exhibit #8 versus an unrelated cocaine seizure. The absence of citric acid in the unrelated sample is noted. Figure 4*c* illustrates the comparatively low levels of manufacturing impurities/byproducts normally associated with pharmaceutical-grade cocaine.

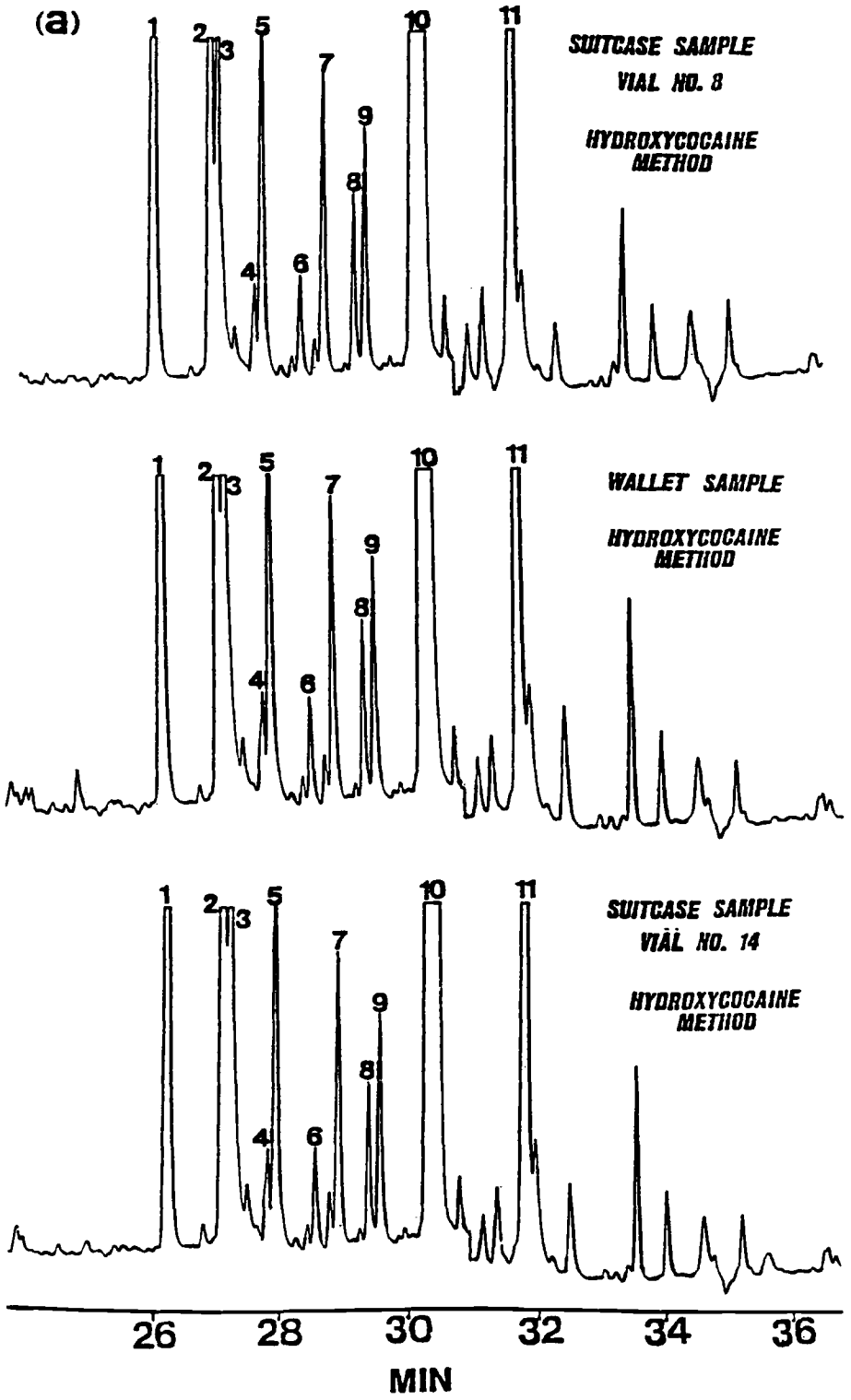



FIG. 3—*The HFBA derivatization and CGC-ECD analyses of* (a) *the wallet cocaine sample and exhibit numbers 8 and 14 of the suitcase cocaine sample,* (b) *the wallet cocaine sample, exhibit number 8 of the suitcase cocaine sample and an unrelated cocaine sample, and* (c) *exhibit number 8 of the suitcase cocaine sample, the wallet cocaine sample and an unrelated highly refined cocaine sample. See table 2 for identification and retention times of enumerated peaks.*

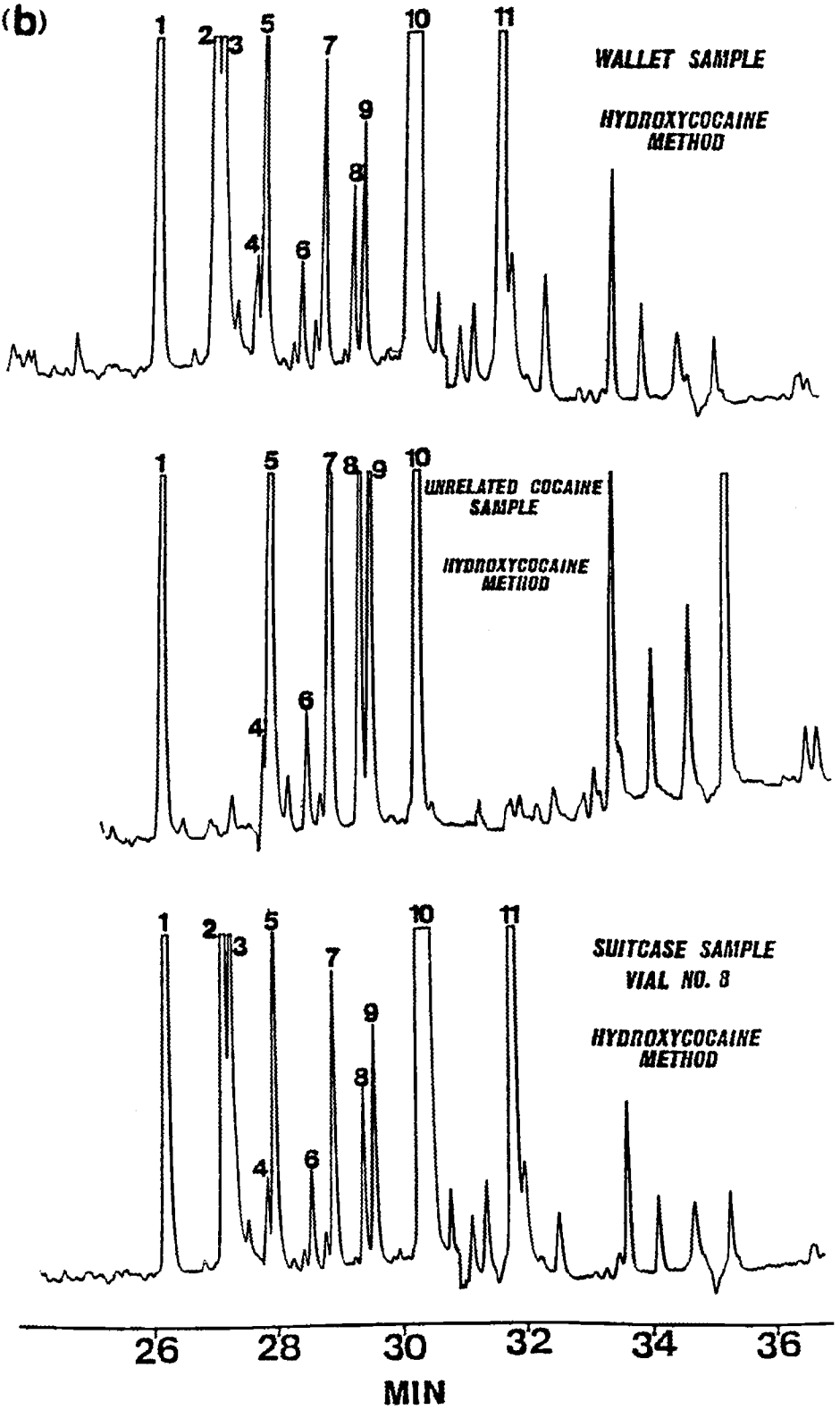



FIG. 3—*Continued.*

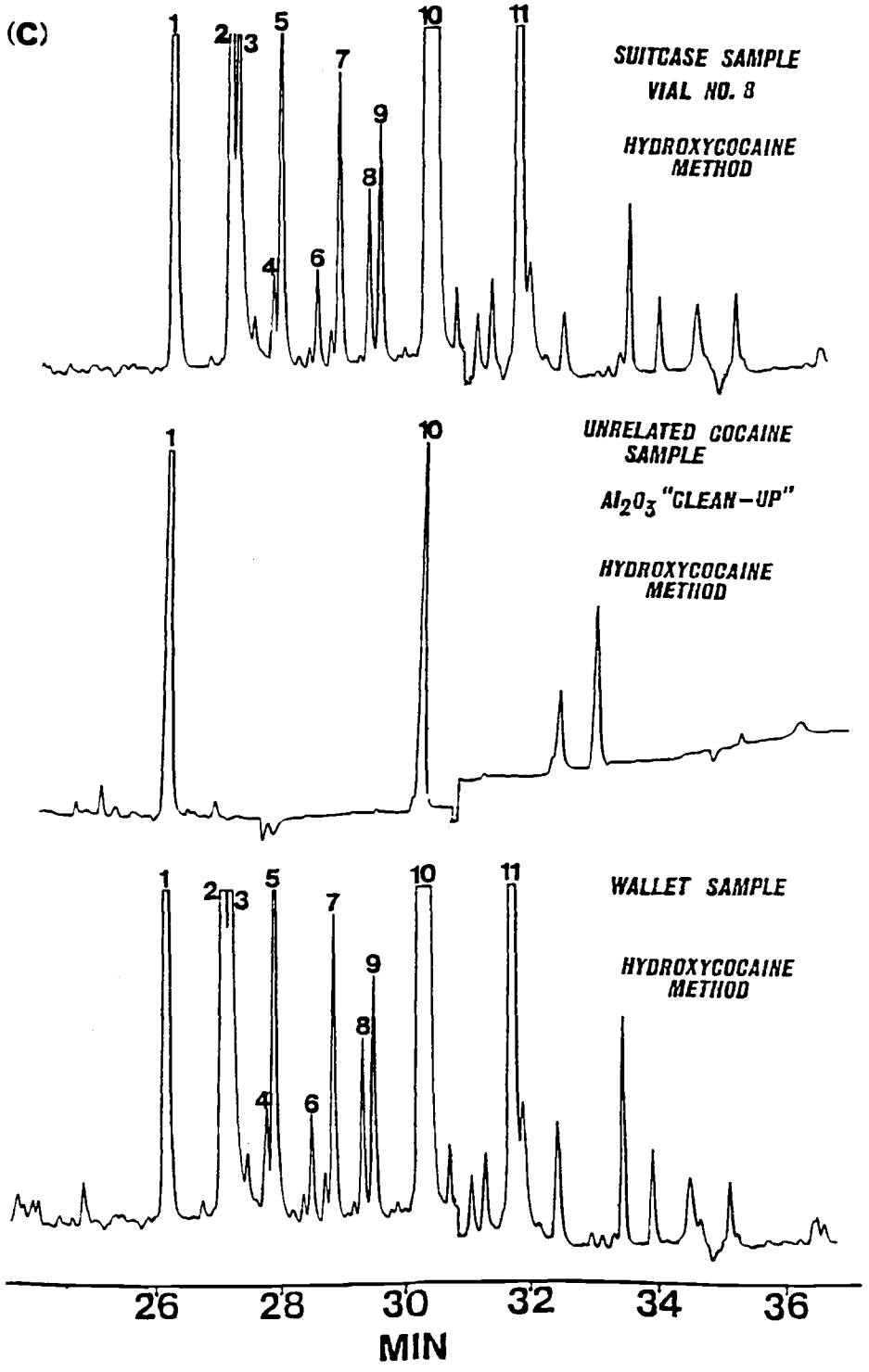



FIG. 3—*Continued.*

TABLE 2—*Retention times for the heptafluorobutyryl derivatives of some illicit cocaine manufacturing impurities/byproducts using capillary gas chromatography-electron capture detection: wallet and suitcase cocaine samples (see Fig. 3a–c for comparison Method I chromatographic profiles).*

| Peak #[a] | Compound Name[b] | Retention Time (Min) |
|---|---|---|
| 1 | Heneicosanol Internal Standard[c] | 26.32 |
| 2 | *a*[d] | 27.22 |
| 3 | *a*[d] | 27.35 |
| 4 | *a*[d] | 27.93 |
| 5 | *a*[d] | 28.04 |
| 6 | *a*[d] | 28.64 |
| 7 | *a*[d] | 28.99 |
| 8 | N-Benzoylnorecgonine Methyl Ester[c] | 29.47 |
| 9 | *a*[d] | 29.64 |
| 10 | N-Norcocaine[e] | 30.41 |
| 11 | *f*[f] | 31.86 |

[a]Refer to Fig. 3(*a–c*) for cGC-ECD chromatograms of numbered peaks.

[b]Retention times of other cocaine manufacturing byproducts (see Fig. 1): *8* = 10.90 min, *ψ-ecgonine methyl ester* = 11.24 min, *2* = 11.55 min, *9* = 13.62 min, *5* = 13.17 min, and *7* = 25.47 min.

[c]Chromatographed as O-HFB derivative (see Fig. 3).

[d]Compound *a* = A suspected hydroxycocaine, e.g. *6* in Fig. 1, chromatographed as O-HFB or di-O-HFB derivative.

[e]Chromatographed as N-HFB derivative.

[f]Hydroxy-containing tertiary amine impurity or byproduct chromatographed as O-HFB or di-O-HFB derivative (hydroxycinnamoylcocaine?).

*Comparison Method III (Truxilline Method)*

The Truxilline Method provided for the relative determination for 10 of the 11 isomeric truxillines, all of which are alkaloidal constituents of the coca leaf. These truxilline analyses were similar to those described by Moore et al. [*2,5*] and involved chemical reduction of the isolated truxillines using lithium aluminum hydride, followed by heptafluorobutyrylation and analysis using capillary gas chromatography-electron capture detection. The reproducibility of total truxillines analyses was found to be good [*5*]. Illustrated in Fig. 2 are the structures for the eleven truxillines and their truxillic and truxinic acid hydrolysis products.

The comparative chromatographic profiles generated by the Truxilline Method for the wallet sample and Exhibit #'s 8 and 14 of the suitcase cocaine sample are seen in Fig. 5*a*. The comparison of the chromatography for the wallet sample, Exhibit #8 of the suitcase sample and an unrelated cocaine sample is illustrated in Fig. 5*b*. Table 4 gives peak identity and retention times for the isomeric truxillines seen in the Fig. 5 chromatograms.

The three chromatographic profiles, shown in Fig. 5*a*, using the Truxilline Method are virtually identical. This confirmed the positive correlation between the wallet cocaine sample and Exhibit #'s 8 and 14 of the suitcase cocaine sample. Figure 5*b* compares the chromatographic profiles of the wallet and Exhibit #8 of the suitcase sample with an unrelated cocaine sample.

## Testimony and Conclusions

In arriving at a conclusion/expert opinion regarding the relationship between the suitcase and wallet cocaine samples, three elements were considered. Undoubtedly, the most

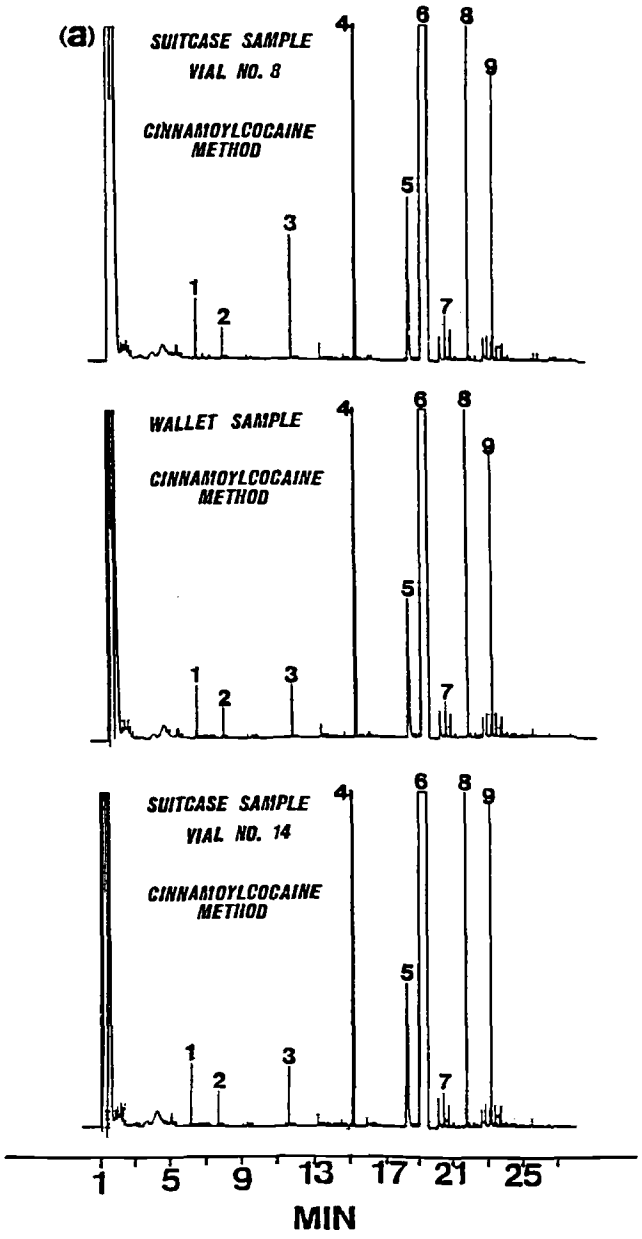



FIG. 4—*The BSA derivatization and CGC-FID analyses of:* (a) *the wallet cocaine sample and exhibit numbers 8 and 14 of the suitcase cocaine sample,* (b) *the wallet cocaine sample, exhibit number 8 of the suitcase cocaine sample and an unrelated cocaine sample and* (c) *the wallet cocaine sample, exhibit number 8 of the suitcase cocaine sample and standard Merck cocaine hydrochloride. See Table 3 for identification and retention times of enumerated peaks.*

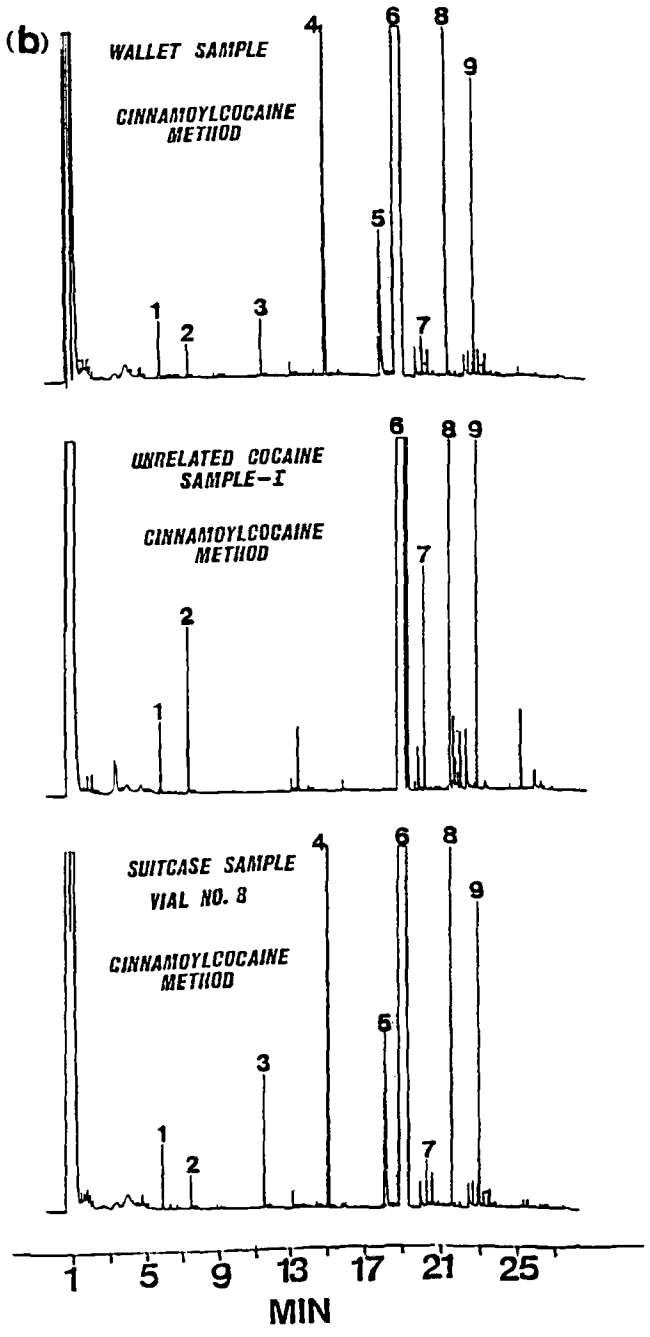



FIG. 4—*Continued.*

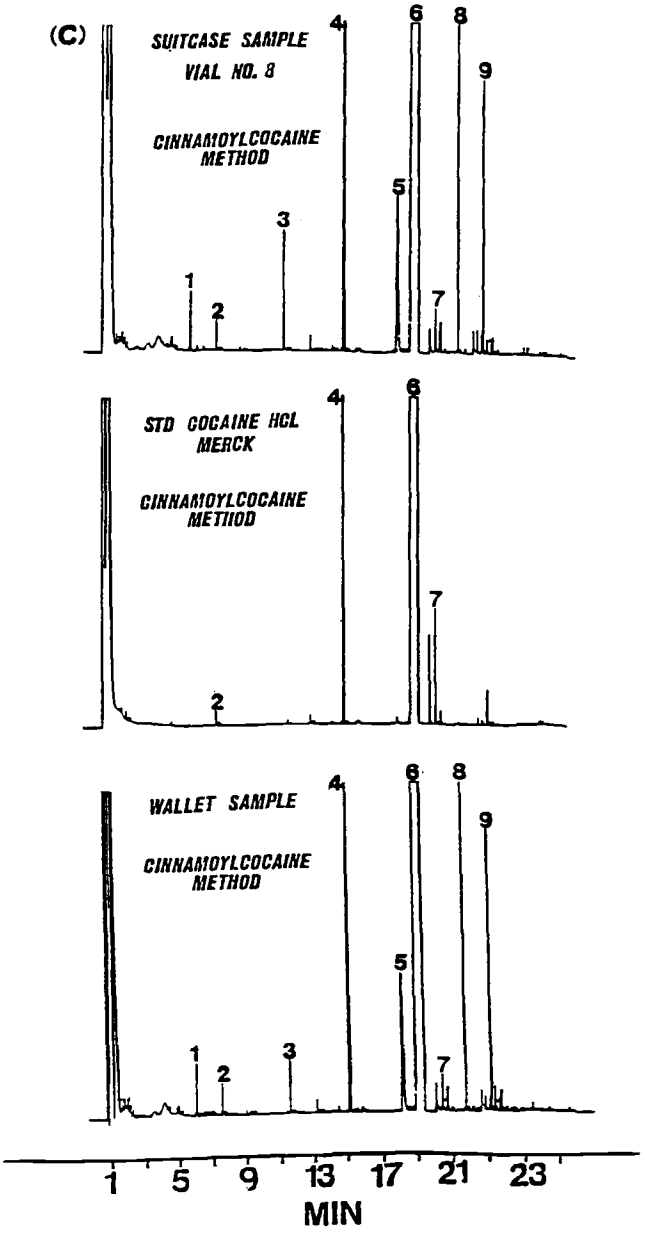



FIG. 4—*Continued.*

important element in these comparison analyses was the detection and relative determination of cocaine manufacturing impurities and byproducts using Methods I, II and III. In fact, these methods could stand alone in rendering a scientific opinion regarding the intimate chemical relationship between the wallet cocaine sample and the suitcase cocaine samples, especially exhibit #'s 8 and 14.

The element involving the detection and quantitation of adulterants and diluents usually carries less weight when formulating an opinion regarding the relationship between

TABLE 3—*Retention times for some illicit cocaine manufacturing impurities/byproducts subjected to trimethylsilylation and capillary gas chromatographic-flame ionization detection analysis: wallet and suitcase cocaine samples (see Fig. 4a–c for comparison Method II chromatographic profiles).*

| Peak/Compound # | Compound name | Retention time (Min) |
|---|---|---|
| 1 | Ecgonine Methyl Ester[a] | 6.18 |
| 2 | Ecgonine[b] | 7.71 |
| 3 | Citric acid[c] | 11.64 |
| 4 | Eicosane Internal Standard | 15.19 |
| 5 | N-Norcocaine | 18.16 |
| 6 | Cocaine | 19.25 |
| 7 | Benzoylecgonine[d] | 20.32 |
| 8 | *cis*-Cinnamoylcocaine | 21.66 |
| 9 | *trans*-cinnamoylcocaine | 23.10 |

[a]Chromatographed as an O-TMS derivative of a hydroxyl moiety (see Fig. 1 for structures and Fig. 4(*a–c*) for cGC-FID chromatograms).
[b]Chromatographed as a di-O-TMS derivative of a hydroxyl and carboxyl moieties.
[c]Chromatographed as a tetra-O-TMS derivative.
[d]Chromatographed as an O-TMS derivative of a carboxyl moiety.

samples. However, in this case, the presence of trace levels of citric acid in both the suitcase and wallet samples was so unusual that this element was given additional weight.

Although not trivial, the cocaine quantitative results was the element of least importance, especially given the presence of citric acid in these samples. At worst, it can be stated that the cocaine quantitative results did not dispute the commonality of the suitcase and wallet samples.

Prior to the testimony of the government chemist at the second trial, selected chromatograms from comparison Methods I, II and III were enlarged to poster size for presentation to the jury. This proved an effective tool in that it allowed the government chemist to more readily explain the relationship between the suitcase and wallet cocaine samples. This presentation to the jury was bolstered by demonstrating the dissimilarity of the wallet/suitcase samples with an unrelated cocaine sample. After several hours of testimony by the government chemist, the following scientific opinion paraphrased below was rendered regarding the relationship between the suitcase and wallet cocaine samples.

> After a review of all analytical data it can be stated with a high level of scientific certainty and beyond a reasonable scientific doubt that a close chemical relationship exists between the cocaine in the wallet sample and the cocaine in 17 of 18 suitcase exhibits, strongly suggesting that they were derived from the same manufacturing process. This relationship is especially so for the cocaine in the wallet sample and suitcase Exhibit #'s 8 and 14, in that they were probably derived from the same production batch.

After the testimony of the government chemist, a second defense chemist testified that in his opinion the wallet and suitcase samples *were not* related. This chemist had not performed any chemical comparison analyses on the suitcase and wallet samples, so his opinion was rendered solely upon his own review of the government chemist's analytical data. Under direct examination the defense chemist testified that, although the suitcase and wallet cocaine samples bore a close chemical relationship to one another, they were not derived from the same process or the same production batch. His opinion was rendered after close examination of only the relative quantitative truxilline data presented by the government chemist. He also testified that although the government chemist had presented the average of duplicate quantitative analyses for cocaine, he (government chemist) had done only single truxilline analyses on the suitcase and wallet exhibits.

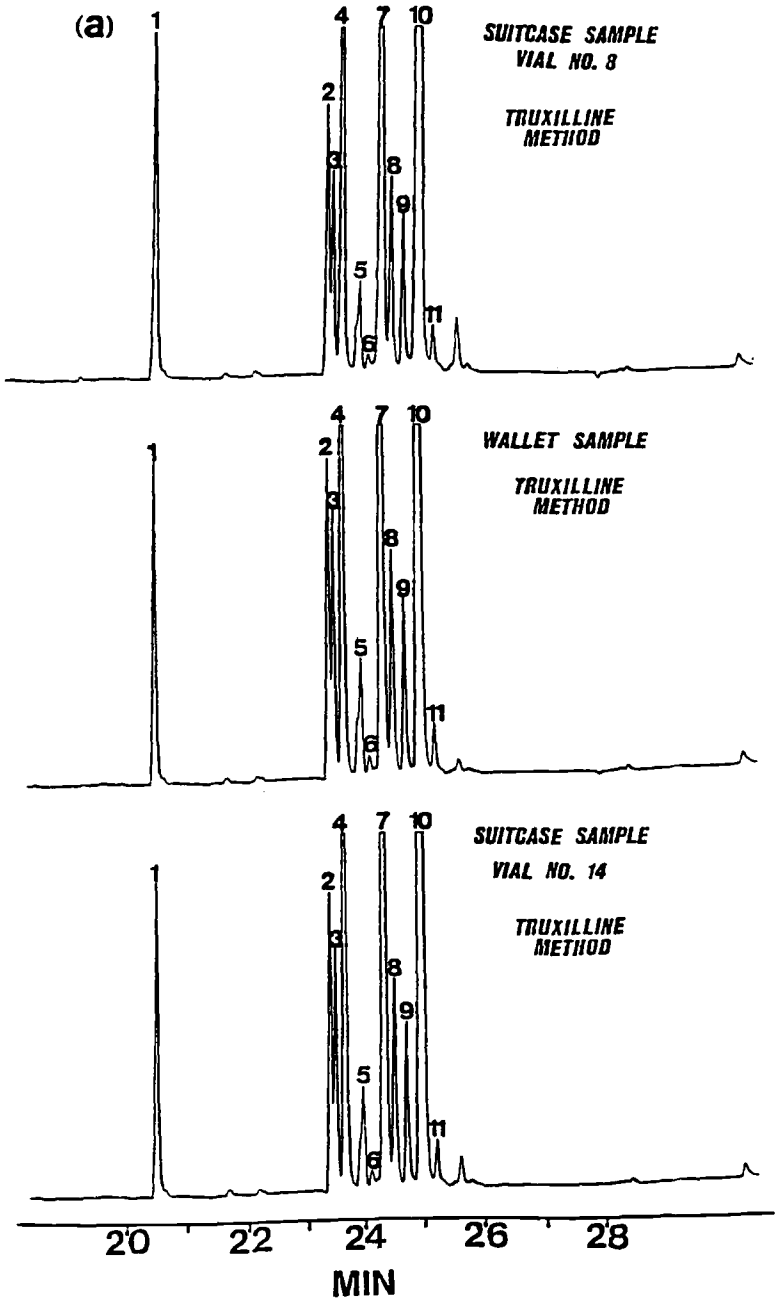



FIG. 5—*The $LIAlH_4$ reduction, heptafluorobutyrylation and CGC-ECD analyses of:* (a) *the wallet cocaine sample and exhibit numbers 8 and 14 of the suitcase cocaine sample and* (b) *the wallet cocaine sample, exhibit 8 of the suitcase cocaine sample and an unrelated cocaine sample. See Table 4 for identification and retention times of enumerated peaks.*

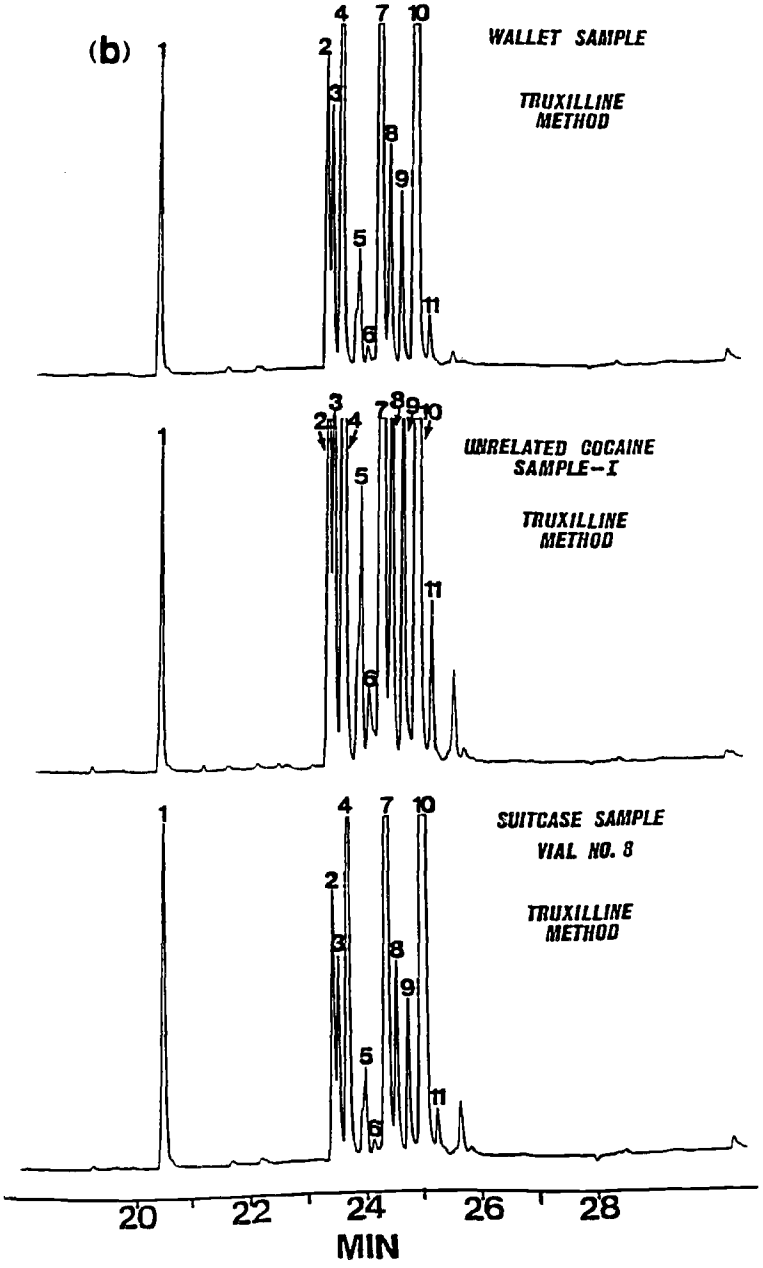



FIG. 5—*Continued.*

TABLE 4—*Retention times for the isomeric truxilline impurities subjected to lithium aluminum hydride reduction, heptafluorobutyrylation and capillary gas chromatographic-electron capture detection analysis: wallet and suitcase cocaine samples (see Fig. 5a,b for comparison Method III profiles).*

| Peak/Compound # | Compound name[a] | Retention time (Min) |
|---|---|---|
| 1 | Aldrin Internal Standard | 20.56 |
| 2 | *epsilon*-Truxilline | 23.45 |
| 3 | *delta*-Truxilline | 23.54 |
| 4 | *beta*-Truxilline | 23.70 |
| 5 | *peri-* + *neo*-Truxilline[b] | 24.01 |
| 6 | *epi*-Truxilline[b] | 24.16 |
| 7 | *alpha*-Truxilline | 24.37 |
| 8 | *omega*-Truxilline[b] | 24.55 |
| 9 | *gamma*-Truxilline | 24.75 |
| 10 | *mu*-Truxilline[b,c] | 24.98 |
| 11 | *zeta*-Truxilline[b] | 25.25 |

[a]All truxilline isomers were reduced with lithium aluminum hydride, derivatized with heptafluorobutyric anhydride and chromatographed as di-O-HFB derivatives (see Fig. 2 for structures and Fig. 5*a,b* for cGC-ECD chromatograms).

[b]The identification of this isomer was presumptive (see Ref *2*).

[c]Used in this analysis as a structurally related internal standard (see Ref *5*).

Finally, the defense chemist testified that there was a significant difference in the cocaine quantitative analyses between the wallet and suitcase samples, thus supporting his opinion that the wallet and suitcase samples were not related.

After the defense chemist concluded his direct testimony, it was brought out by the prosecution under cross-examination of the defense chemist that he had no practical experience in conducting cocaine comparative analyses and was mostly unfamiliar with recent scientific literature pertaining to the subject. Subsequently, the government chemist took the stand for rebuttal testimony, which focused upon the fact that in his analysis of the chemical comparative data, the defense chemist was using insignificant figures in forming his expert opinion. In fact, the prosecution entered as a government exhibit the defense chemist's own calculations, which were effectively discredited during rebuttal testimony.

After closing arguments, the jury returned guilty verdicts on both counts—possession with intent to distribute and importation of cocaine. The comparative cocaine analyses not only contributed to the verdict but also supported the Assistant United States Attorney's argument at sentencing that John Doe had obstructed justice by committing perjury. The sentencing judge agreed, noting that he could count on one hand the number of times he had found that a testifying defendant had committed perjury. The enhancement of John Doe's sentence for obstruction of justice resulted in a term of incarceration of 151 months. John Doe initially appealed his conviction, but, after studying the record and gauging his chances for success, decided to abandon his appeal. He is currently serving his sentence.

This case is believed to be the first successful federal narcotics prosecution in which this type of comparative cocaine analyses has been accepted by the jury and the court and which weighed heavily in determining the outcome of the trial.

*Acknowledgments*

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this case. We also want to thank Edward S. Franzosa and Charles Harper for their assistance in preparing this manuscript.

*Addendum*

The cocaine comparison case described above has been recently cited as a precedent by the United States District Court for the Eastern District of North Carolina (July 1992). The North Carolina case, which also included cocaine comparison analyses, involved the successful prosecution of individuals who participated in a "crack cocaine" conspiracy. Two chemists, John Casale, now of this laboratory, and Richard Waggoner, Jr., of the North Carolina State Bureau of Investigation, compared three "crack" samples, from different sources, using cGC-FID methodology they developed and cited herein as Ref [*10*]. They concluded that the three samples were from the same manufacturing batch. Their comparative analyses and expert opinion testimony were fully accepted by the court. The chemists' findings in the North Carolina case corroborated the police officers' testimony and was a major factor in the resulting conviction of the defendant.

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