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Coca Paste: Chemical Analysis and Smoking Experiments

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ABSTRACT: Several samples of Colombian and a sample of Peruvian coca paste were subjected to chemical analysis to ascertain the complexity of these products. A neutral and acid fraction and a basic fraction were analyzed by gas chromatography/flame ionization detection (GC/FID) and gas chromatography/mass spectrometry (GC/MS). The basic fraction was also analyzed as its trimethylsilyl (TMS) derivative. Several gasoline residue components were identified in the neutral fraction. In addition to cocaine (>60% in all cases), other alkaloids were identified. Lead and manganese analyses were carried out on these samples. While all the samples contained no lead (<45 ppm), most of the Colombian samples contained significant amounts of manganese (>5%). Preliminary smoking experiments with a Colombian coca paste sample indicated that it behaves more like free cocaine than like a cocaine sulfate salt.

KEYWORDS: toxicology, coca paste, cocaine

Coca paste is the most widely used product of the coca leaves today in South America [1-3]. It is the first crude extraction product obtained during the manufacture of cocaine from the leaves of the coca bush. After the fresh leaves have been mashed with alkali, kerosene and sulfuric acid (and sometimes potassium permanganate) are added. Further processing yields an off-white or light brown paste or semi-solid containing 40 to 70% cocaine (in both the salt and free-base forms), other alkaloids, benzoic acid, kerosene residue, and sulfuric acid, as well as other impurities [4].

The use of the drug started in Latin American countries in the early 1970s, reaching epidemic proportions by the end of the decade [2]. The most prominent feature which stimulated the study of coca paste smoking is the unprecedented ability of this form of drug use to induce a compulsive need in the user to repeat the experience. Jeri [1], Jeri et al. [3], and Aramayo and Sanchez [5] described the characteristics of the coca paste syndrome based on studies conducted in Peru. Euphoria, dysphoria, hallucinosis, and paranoid psychosis are the four distinct successive phases of the mental disorder associated

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with use of the drug. Similar population studies were carried out in Bolivia [5,6] and Colombia [7]. The spread of coca paste smoking went from Peru to Bolivia, Colombia, and Ecuador and, more recently, to the Netherlands Antilles [8], Panama, and the United States. The widespread use of "crack" in the United States today is probably a direct result of the coca paste experience [9].

This work is designed to generate baseline data on the chemical composition of coca paste samples to be used in subsequent toxicological evaluation of the drug. In addition, preliminary smoking experiments with coca-paste-spiked tobacco cigarettes are presented.

Experimental Materials and Methods

Coca Paste Samples

Samples 1 through 5 were obtained from different seizures made in Bogota, Colombia, while Sample 6 was obtained from Peru.

Reference Samples

The following reference samples were used:

Methyl benzoate—synthesized by methylation of benzoic acid with methyl iodide.

Ecgonine methyl ester—synthesized by reaction of ecgonine and diazomethane in methanol.

Benzoyltropine—synthesized by reaction of tropine (3- α -tropanol) and benzoic acid anhydride.

Tropacocaine—synthesized by reaction of pseudotropine (3- β -tropanol) and benzoic acid anhydride.

Ecgonine—prepared from cocaine by hydrolysis with concentrated hydrochloric acid (HCl) in water.

Benzoylcegonine—prepared from cocaine by refluxing with water.⁴

"Hydrocarbon residue"—obtained by evaporation of 1 mL of regular gasoline under a mild stream of nitrogen followed by dissolving the residue in 10 mL of hexane prior to gas chromatography (GC) and GC/mass spectrometry (GC/MS) analysis.

Preparation of Cigarettes

For all the preparations, reference tobacco obtained from the Tobacco Research Institute, University of Kentucky, was used. Several attempts were made to mix the coca paste with bulk tobacco uniformly, but the small particles of the coca paste would adhere to the walls of the container, thus producing a product of variable coca paste content. For the purpose of the present experiments, all the cigarettes were prepared from individually weighed coca paste (50 mg) and tobacco (900 mg) aliquots. Approximately half of the tobacco was placed into a small hand roller; the coca paste was sprinkled over the tobacco, with care being taken not to place any of the paste within 1 cm of each end; and the remaining tobacco was added and rolled. With this technique, cigarettes of uniform weight and content were produced. Colombian coca paste Sample 5 was used in all smoking experiments.

⁴Bourquin, D. and Brenneisen, R., manuscript in preparation.

Smoking of Cigarettes

The smoking characteristics of the cigarettes were obtained by placing a single cigarette into a stainless steel holder attached to an ADL/II smoking system (Arthur D. Little [ADL], Inc., Cambridge, Massachusetts) with the effluent attached to a rodent exposure system (designed and constructed for the National Institute on Drug Abuse (NIDA) by Oak Ridge National Laboratories, Oak Ridge, Tennessee). The system was operated without animals present, and the animal ports were sealed with rubber stoppers. Smoke samples were obtained by placing a Cambridge filter at various locations in the smoke stream, specifically, at the cigarette butt, in a collection funnel for collecting the side stream smoke, between the ADL/II smoking system and the rodent exposure chamber, and at the exit of the rodent exposure chamber. The system was operated in such a manner that a puff (30 mL in volume) was taken on the cigarette every 60 s. This puff was then injected into the exposure chamber, where it was mixed with the chamber air and was held for 30 s, after which the residual smoke was removed by means of a small vacuum pump. The smoke sample collected at the exit of the exposure chamber was collected using this vacuum line. Furthermore, residual cocaine in the cigarette butt was determined.

Extraction

Extraction of Coca Paste Samples—All the samples were partitioned in the same manner. Approximately 10 mg of the coca paste was sonicated in 2 mL of 0.1N HCl, and the pH was adjusted to pH 2 to 3 using 1N HCl followed by partitioning with 2 mL of *n*-hexane. The hexane layer was separated and evaporated under nitrogen, and the residue was dissolved in 10 mL *n*-hexane to give “Hydrocarbon Fraction A” (Fraction A).

The aqueous acidic layer was made alkaline (pH >10) using 1N sodium hydroxide (NaOH), followed by extraction with two aliquots of 2 mL of chloroform/methanol (3:1). The combined organic extracts were then evaporated to dryness under a stream of nitrogen to give the basic “Alkaloid Fraction B” (Fraction B), which was dissolved in 10 mL of methanol prior to GC and GC/MS analysis. A 100- μ L aliquot of the methanolic solution of Fraction B was evaporated in a glass vial to dryness under a stream of nitrogen. After addition of 100 μ L bis(trimethylsilyl)trifluoroacetamide (BSTFA) (Pierce Chemical Co., Rockford, Illinois) containing 1% trimethylchlorosilane (TMCS), the mixture was sonicated, the vial tightly capped, and then heated at 50 to 60°C for 15 min. The resulting trimethylsilyl (TMS) derivative of Fraction B was designated “Alkaloid Fraction B-TMS” (Fraction B-TMS).

Extraction of Smoke Condensates and Cigarette Butts

The filter pads were removed from the Cambridge filter holders and placed into a 125-mL conical flask to which was added 10 mL ethanol, and the flask was placed into a sonic bath for 4 min. The ethanol was transferred to a 25-mL volumetric flask and the filter was again extracted, first with 10 mL and then with 5 mL of ethanol, combining the extracts. After the final volume was adjusted to 25.0 mL, a 500- μ L aliquot was removed and mixed for GC analysis with an equal volume of an ethanolic solution of codeine, which served as an internal standard.

The cigarette butts were similarly extracted with ethanol (10, 10, and 5 mL), and the extracts were combined and appropriately mixed with the codeine internal standard solution.

Gas Chromatography

The following conditions were used to generate the GC profiles: A Hewlett-Packard (HP) 5890 gas chromatograph with a flame ionization detector (FID) was used. The instrument was connected with an HP 5895A GC ChemStation, an HP 2225A Think-Jet printer, and an HP 7470A X/Y plotter. The column was a 30-m by 0.25-mm inside-diameter DB-1 Durabond fused silica capillary column with 0.25- μ m film. The oven temperature was programmed from 70 to 250°C at 5°C/min; the detector temperature was kept at 300°C; and the injector temperature was 250°C. Injections of 2 μ L were made in the splitless mode (30-s delay). The flow of the carrier gas (helium) was maintained at 25 cm/s.

Quantitative Determination of Cocaine in Coca Paste Extracts

Quantitation of cocaine was carried out following a modification of the method used by Turner et al. [10] for the analysis of coca leaves. An accurately weighed amount (about 10 mg) of the coca paste was dissolved in 1 mL of an ethanolic solution of the internal standard (10 mg/mL 3-androstene-3,17-dione) prior to GC injection. The concentration was determined from a calibration curve prepared from cocaine and the internal standard. The GC system was a Varian 3300 gas chromatograph fitted with a 15-m by 0.25-mm DB-1 capillary column (J&W Scientific) operated at 170°C (1 min) to 250°C at 5°C/min. Injections were made in the split mode (60:1).

Analysis of Smoke Condensates and Cigarette Butts

These extracts of the filter pads (smoke condensate) and cigarette butts were analyzed using a Varian 1400 gas chromatograph fitted with a flame ionization detector. The chromatograph was fitted with a 1.8-m glass column (2-mm inside diameter) packed with 3% OV-17 on Gas Chrom Q (100/120 mesh) and operated isothermally at 190°C. Under these conditions, the retention times for the cocaine and codeine were 10.8 and 21.7 min, respectively. The oven was operated isothermally at 190°C.

Gas Chromatography/Mass Spectrometry

An HP 5890 GC was connected to an HP 5970B mass selective detector (MSD) with a ChemStation data system (Pascal rev. 3.1) and an HP 2225A Think-Jet printer. The injector temperature was maintained at 250°C and the transfer line at 260°C. The mass spectrometer was operated in the full scan mode between m/z 30 and 400 at 1.16 scans/s. A solvent delay of 5 min was used for Fractions A and B and a delay of 9 min for Fraction B-TMS. Injections of 1.0 μ L were made in all cases. For GC/MS analysis of Fraction A, a Durabond DB-5 capillary column (20 m by 0.18 mm inside diameter and 0.40- μ m film) was used. The column temperature was programmed from 50°C (2 min hold) to 250°C at 5°C/min. For Fractions B and B-TMS, the column used was an HP-1 capillary column (12 m by 0.33-mm inside diameter and 0.25- μ m film) with the oven temperature programmed from 70 to 250°C at 5°C/min.

Determination of Manganese and Lead in Coca Paste Samples

Quantitation of manganese and lead was carried out by Galbraith Laboratories, Knoxville, Tennessee.

Results and Discussion

Five coca paste samples were obtained from Colombia and one sample was obtained from Peru. These samples were analyzed for their cocaine content as well as for manganese (Mn) and lead (Pb) residues as a result of the possible use of potassium permanganate and leaded gasoline during the preparation of the paste in the clandestine coca paste laboratories. Table 1 shows the results of these analyses.

All coca paste samples were then analyzed by capillary GC/FID and GC/MS. Each sample was extracted and fractionated into two major fractions (Fig. 1). Hydrocarbon Fraction A (Fraction A) is essentially the acidic and neutral fraction made by partitioning the paste between acid and *n*-hexane. The main purpose of this fraction was to determine the presence and extent of any gasoline residues in the paste. On the other hand, Alkaloid Fraction B (Fraction B) was obtained by basifying the acidic layer above and extracting with a polar solvent (chloroform/methanol, 3:1). Therefore, Fraction B contains the basic substances and other polar components. This fraction was analyzed underivatized and as its TMS derivative. The latter was designated Alkaloid Fraction B-TMS (Fraction B-TMS).

All the fractions were analyzed by GC and GC/MS, and attempts were made to characterize as many components as possible by GC/MS. This was achieved either by using reference samples or by comparing the mass spectra of each component against the data available in the National Bureau of Standards (NBS) mass spectral (MS) library and in the literature [11,12].

Table 2 shows a comparison between the composition of the hydrocarbon residue from Fraction A of the six coca paste samples and that of a gasoline residue (Sample 7). It is evident that all the samples contain aromatic hydrocarbons (alkyl derivatives of benzene and naphthalene) similar to those present in gasoline. Figure 2 shows a typical GC profile for Fraction A of Sample 5, and Fig. 3 shows the chromatogram from a gasoline residue. Peaks appearing before 8 min are mainly due to hexane used to dilute the gasoline residue. It was noticed, however, that although Fraction A of Sample 6 (Peruvian) was similar in composition to that of the other coca paste samples, it contained a significant amount of benzoic acid (broad peak at 18.6 min with *m/z* 122 (M⁺) and 105 (base peak)).

Table 3 shows a comparison of the alkaloidal composition of the six coca paste samples when the basic Fraction B is analyzed underivatized; Table 4 shows the identification of benzoic acid, ecgonine, and benzoylecgonine as their TMS derivatives in Fraction B-TMS. The appearance of benzoic acid in Fraction B was not expected on the basis of the partitioning scheme and, therefore, was initially thought of as an artifact, possibly through hydrolysis of cocaine as benzoylecgonine during derivatization. However, partitioning experiments under conditions similar to those in Scheme 1, using 1 mg pure benzoic acid, showed approximately one third of the benzoic acid remaining in the aqueous acid fraction (Fraction B). This supports the conclusion that benzoic acid is an

TABLE 1—Content of cocaine, manganese, and lead in coca paste samples.

| Sample No. | Origin | Cocaine, % w/w | Manganese, % w/w | Lead, ppm |
|------------|----------|----------------|------------------|-----------|
| 1 | Colombia | 75.1 | 6.08 | <45 |
| 2 | Colombia | 68.2 | 5.86 | <45 |
| 3 | Colombia | 75.0 | 6.20 | <45 |
| 4 | Colombia | 104.6 | 0.07 | <45 |
| 5 | Colombia | 67.6 | 8.15 | <45 |
| 6 | Peru | 77.8 | 0.004 | <45 |

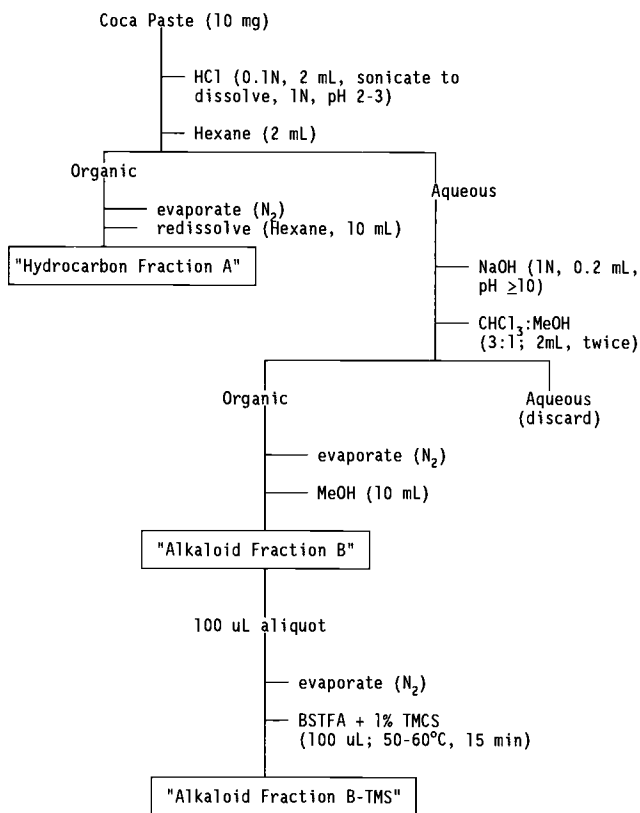


FIG. 1—Partition scheme.

actual component of the coca paste and not an artifact. Ecgonidine methyl ester, on the other hand, is probably a GC artifact formed in the injection port from decomposition of cocaine [13]. The differences between the samples are more of quantitative nature. Figure 4 shows a typical GC profile for Fraction B of Sample 5, and Fig. 5 shows the chromatogram for Fraction B-TMS of the same sample.

The data presented in Tables 1 through 4 show that coca paste is a chemically complex product that should not be considered as having the same biological and toxicological properties as cocaine, even though cocaine is the major component.

Heavy metal analysis of the samples used in this study for Mn and Pb showed that all of Colombian Samples 1 through 5 contained significant amounts of Mn ($\geq 5.8\%$) (Table 1), with the exception of Sample 4, which was highly purified (almost pure cocaine). On the other hand, the Peruvian Sample 6 contained only traces of Mn. This reflects the use of potassium permanganate during the preparation of the paste in the clandestine laboratories in Colombia. That does not mean, however, that potassium permanganate is not used in Peru; rather, it emphasizes the existence of two types of coca pastes, with and without Mn. No significant amounts of Pb were observed in any of the samples (< 45 ppm) (Table 1).

Since coca paste is mainly abused by smoking it with tobacco, it was necessary to study the behavior of coca paste under these smoking conditions and to determine whether it behaves as free cocaine or as a cocaine salt. The coca paste utilized in the smoking experiments was coca paste Sample 5, which contained 68% cocaine (see Table 1). Thus,

TABLE 2—GC profiles and mass spectral data of coca paste samples—Hydrocarbon Fraction A.

| Sample No. ^a | | | | | | | Peak No. | Retention Time, min | MS | | Compound ^b |
|-------------------------|---|---|---|---|---|---|----------|---------------------|-----|------|-----------------------|
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | | | M+ | 100% | |
| X | X | X | X | X | X | X | 1 | 8.15 | 106 | 91 | ethylbenzene |
| X | X | X | X | X | X | X | 2 | 8.42 | 106 | 91 | xylene |
| X | X | X | X | X | X | X | 3 | 9.02 | 106 | 91 | xylene |
| X | X | | X | X | X | X | 4 | 10.02 | 120 | 105 | methylethylbenzene |
| X | X | | | X | X | X | 5 | 10.93 | 120 | 105 | propylbenzene |
| X | X | | X | X | X | X | 6 | 11.19 | 120 | 105 | methylethylbenzene |
| | X | | X | X | | X | 7 | 11.43 | 120 | 105 | |
| X | X | | X | X | X | X | 8 | 11.73 | 120 | 105 | |
| X | X | X | X | X | X | X | 9 | 12.24 | 120 | 105 | trimethylbenzene |
| X | X | X | X | X | X | X | 10 | 13.09 | 120 | 105 | trimethylbenzene |
| | | | | X | | X | 11 | 13.45 | 117 | 117 | |
| | | | | | | X | 12 | 14.11 | 134 | 119 | |
| | | | | | | X | 13 | 14.23 | 134 | 119 | methylpropylbenzene |
| | | | | | | X | 14 | 14.32 | 134 | 119 | ethylidimethylbenzene |
| | | | | | | X | 15 | 14.60 | 154 | 57 | |
| | | | | | | X | 16 | 14.75 | 134 | 105 | methylpropylbenzene |
| | | | | | | X | 17 | 14.99 | 134 | 119 | |
| | | | | | | X | 18 | 15.18 | 134 | 119 | |
| | | | | | | X | 19 | 15.83 | 134 | 119 | |
| | | | | | | X | 20 | 16.23 | 134 | 119 | |
| | | | | | | X | 21 | 16.34 | 134 | 119 | tetramethylbenzene |
| | | | | | | X | 22 | 16.88 | 132 | 117 | |
| | | | | | | X | 23 | 17.18 | 132 | 117 | |
| | X | X | X | X | | X | 24 | 18.10 | 128 | 128 | naphthalene |
| | X | | | | | X | 25 | 21.57 | 142 | 142 | methylnaphthalene |
| | X | | | | | X | 26 | 22.02 | 142 | 142 | methylnaphthalene |

^aThe identity of Samples 1 through 6 is shown in Table 1. Sample 7 is the "hydrocarbon residue" (see Experimental Materials and Methods section).

^bTentatively identified on the basis of MS library (NBS) search or literature data [12,13] or both.

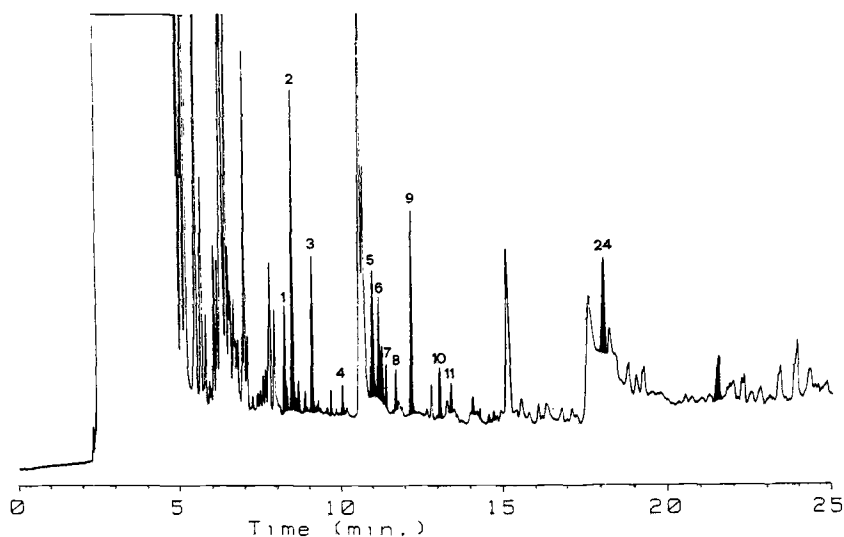


FIG. 2—GC profile of coca paste Sample 5 (Colombian)—Hydrocarbon Fraction A. For the peaks, see Table 2. Peaks corresponding to gasoline components are colored black.

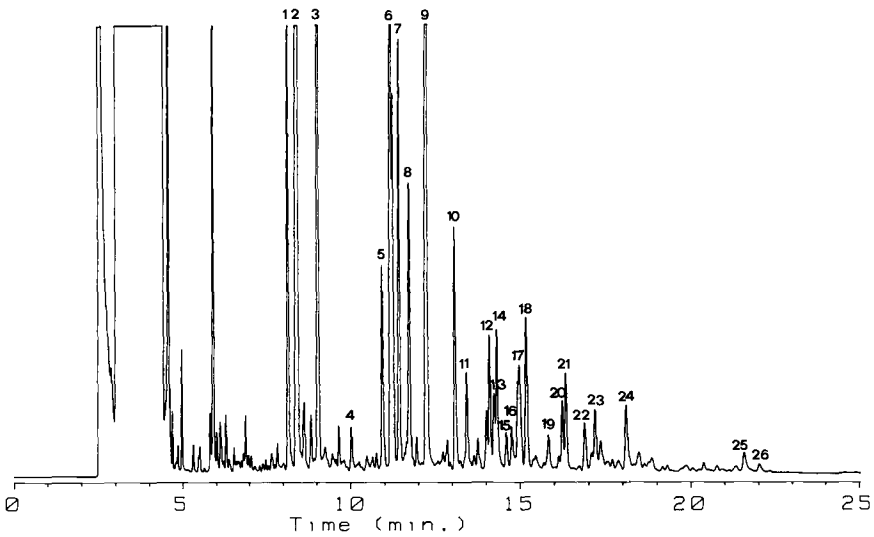


FIG. 3—GC profile of a gasoline sample—hydrocarbon residue. For the peaks, see Table 2.

TABLE 3—GC profiles and mass spectral data of coca paste samples—Alkaloid Fraction B.

| Sample No. | | | | | | Peak No. | Retention Time, min | MS | | Compound |
|------------|---|---|---|---|---|----------|---------------------|-----|------|--------------------------------------|
| 1 | 2 | 3 | 4 | 5 | 6 | | | M+ | 100% | |
| X | X | X | X | X | X | 1 | 10.3 | 136 | 105 | methyl benzoate ^a |
| X | X | X | X | X | X | 2 | 18.7 | 181 | 152 | ecgonidine methyl ester ^b |
| X | X | X | X | X | X | 3 | 20.9 | 199 | 82 | ecgonine methyl ester ^a |
| | | | | X | | 4 | 32.1 | 245 | 124 | benzoyltropine |
| X | X | X | X | X | X | 5 | 32.5 | 245 | 124 | tropacocaine |
| | | X | X | X | | 6 | 36.6 | 303 | 182 | pseudococaine ^b |
| X | X | X | X | X | X | 7 | 37.8 | 303 | 182 | cocaine ^a |
| X | X | X | | | X | 8 | 41.7 | 329 | 82 | cis-cinnamoylcocaine ^b |
| X | X | X | X | | X | 9 | 45.8 | 329 | 82 | trans-cinnamoylcocaine ^b |

^aIdentified with reference samples (see Experimental Materials and Methods section).

^bTentatively identified on the basis of MS library (NBS) search or literature data [12,13] or both.

TABLE 4—GC profiles and mass spectral data of coca paste samples—Alkaloid Fraction B-TMS.

| Sample No. | | | | | | Peak No. | Retention Time, min | MS | | Compound ^a |
|------------|---|---|--------|---|---|----------|---------------------|-----|------|-----------------------|
| 1 | 2 | 3 | 4 | 5 | 6 | | | M+ | 100% | |
| X | X | X | X | X | X | 1 | 14.9 | 194 | 179 | benzoic acid-TMS |
| X | X | X | X | X | X | 2 | 26.1 | 329 | 83 | ecgonine-TMS |
| | | | (X, X) | | | 3 | 40.2 | 361 | 82 | benzoylcegonine-TMS |

^aAll identified with reference samples (see Experimental Materials and Methods section).

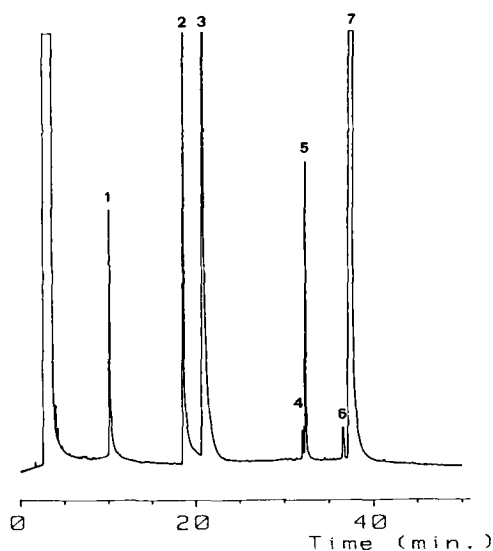


FIG. 4—GC profile of coca paste Sample 5 (Colombian)—Alkaloid Fraction B. For the peaks, see Table 3.

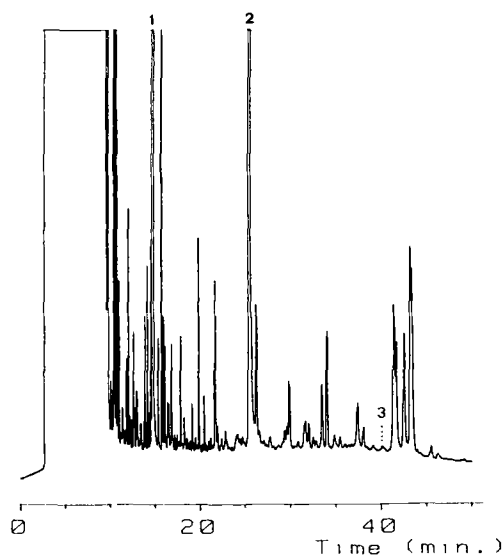


FIG. 5—GC profile of coca paste Sample 5 (Colombian)—Alkaloid Fraction B-TMS. For the peaks, see Table 4.

when 50 mg of coca paste was mixed with the 900 mg of tobacco, each cigarette then contained 34 mg of cocaine. The cocaine recoveries based on the analyses of the various samples obtained during and after smoking these cigarettes in the smoking system (described earlier in this paper) are shown in Table 5. Preliminary observations on these data indicate that 5.6% of the cocaine remained in the cigarette butt even though no coca paste was placed in the last 1 cm of the cigarette when it was prepared. This suggests that there is some distillation and condensation of the material in the butt, which acts

TABLE 5—*Smoking Study results.*

| Collection Site for Cocaine | Amounts of Cocaine Recovered ^a |
|----------------------------------|---|
| Smoke at butt | 2.08 ± 0.20 mg (<i>n</i> = 7) |
| Smoke side stream | 1.27 ± 0.56 mg (<i>n</i> = 7) |
| Butt | 1.89 ± 1.84 mg (<i>n</i> = 7) |
| Smoke between ADL/II and chamber | 1.40 ± 0.35 mg (<i>n</i> = 5) |
| Smoke at exit of chamber | 1.12 ± 0.45 mg (<i>n</i> = 5) |

^aCigarettes = 900 mg tobacco + 50 mg coca paste (34 mg cocaine).

as a filter. Approximately 3.7% of the original cocaine was collected in the side-stream smoke in the form of free cocaine. Finally, only about 6.1% of the initial cocaine was trapped in the smoke leaving the cigarette butt. The totaled values of the three samples indicate that approximately 15.4% of the initial cocaine in the cigarette has survived the pyrolysis. These observations are consistent with those reported by Cook et al. [14] in their pyrolysis experiments with free-base cocaine and cocaine hydrochloride. It was reported [14] that 16% of the pyrolysis product from cocaine free base was the parent drug at 800°C (the temperature of a glowing end of a tobacco cigarette) and that 6% of cocaine was recovered in the trap when the drug was smoked as a mixture with tobacco. In addition, cocaine hydrochloride completely decomposed by pyrolysis at that temperature.

The data in Table 5, therefore, indicate that coca paste contains free cocaine and not cocaine sulfate. Furthermore, the data illustrate that there was some loss of cocaine during the movement of the aerosol through the smoke exposure system. Only about 67% of that leaving the cigarette butt was expelled from the ADL/II, and, further, only about 80% of that entering the exposure chamber for the animals was collected at the exit 30 s later. These numbers reflect the condensation or precipitation of the aerosol on the surfaces of the apparatus, and any subsequent experiments with animals must take this factor into account.

Conclusions

The data presented in this study underscore the need for pharmacological and toxicological evaluation of coca paste samples in comparison with cocaine. Coca paste is a mixture of many components, with cocaine being the major constituent. Samples with and without manganese should be used for such studies. The data also show that when coca paste is smoked with tobacco, a very small percentage (about 6%) of the cocaine survives the smoking process. Therefore, the bioavailability of cocaine from coca paste used in that fashion is expected to be very low.

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