



DEA LABORATORY NOTES

Date January 30, 1984

Number 83-4

DETERMINATION OF COCAINE IN COCA LEAVES

by

Emanuel Solon and Albert Sperling
Special Testing and Research Laboratory

Introduction

This laboratory has used a GC method to quantify cocaine in coca leaves that involved a dimethylsulfoxide (DMSO) extraction, followed by a number of physical and chemical steps to separate the cocaine from the DMSO and other possible interfering substances. The method is tedious, time consuming, and subject to error due to the loss of cocaine in emulsions formed during the several extractions required. A simplified procedure based on methanol extraction of cocaine and direct gas chromatographic analysis of the resultant solution was developed. A recent case provided an opportunity to compare results of both methods on aliquots of the same material.

Procedure

1. Dry coca leaves overnight in vacuum oven at 35-40°C. Grind the dried leaves to a powder. Accurately weigh approximately 500 mg of powdered coca leaves and transfer to a 13 or 15 ml, glass-stoppered centrifuge tube.
2. Add 8 ml methanol and 2.0 ml of internal standard solution containing 2 mg/ml tetracosane in chloroform. Thoroughly mix, then place tubes in a heating block at 75°C for two hours, mixing occasionally.
3. Remove the tubes and centrifuge to settle suspended material. Analyze the solution by gas chromatography using a 6' x 4mm i.d. glass column packed with 390 OV-1, on Gas Chrom Q, 100-120 mesh. Use a column temperature of about 210°C, and a nitrogen flow rate of approximately 70 ml/min. Under the above conditions the retention time of the cocaine and tetracosane should be approximately 330 and 630 seconds, respectively. Inject a standard solution prepared in chloroform:methanol containing approximately 0.4 mg/ml each of cocaine base and tetracosane.

Experimental

Two samples of coca leaves were analyzed using the DMSO extraction and the methanol:chloroform extraction. Extraction conditions were varied as described in Tables 1 and 2. The flask was used to determine if the container shape had an effect on the extraction of cocaine.

DRUG ENFORCEMENT ADMINISTRATION / U. S. DEPARTMENT OF JUSTICE

Results and Conclusions

The data obtained are shown in Tables 1 and 2; a representative sample chromatogram is shown in Figure 1. The following results were obtained and conclusions reached:

1. Each of the aliquots run that were extracted with methanol showed approximately 50% increase in the amount of cocaine found relative to that found using the DMSO procedure.
2. Heating the sample (in a centrifuge tube) in a heating block at 75°C for at least one hour and twenty minutes apparently extracts all the cocaine from the dried, ground-up coca leaves. Sonic mixing of the sample did not extract all the cocaine as additional cocaine was extracted on further sonic mixing and heating.
3. More cocaine was extracted by sonic mixing when the sample was placed in a flask than when the sample was placed in a tube, but the sample still had to be heated to extract all the cocaine. The amount of cocaine found was the same, within experimental error, as for samples extracted in tubes.
4. A lower column temperature than usual for cocaine was used. This lower temperature provided a satisfactory separation of cocaine from other extracted material. It did, however, increase the run time from four to twelve minutes.
5. To avoid the tailing methanol solvent front, an alternative procedure was tried. The methanol extracts were evaporated and reconstituted with chloroform. Analysis of the resultant chloroform solutions yielded very low results. Addition of methanol to the solutions and reanalysis yielded the much higher original amounts of cocaine. This indicated that the total cocaine extracted is not present as the free base, hydrochloride, or other chloroform-soluble form.
6. It is recommended that the methanol extraction procedure be used for determining cocaine in coca leaves because it is relatively more rapid and more accurate than the DMSO procedure

TABLE 1

Comparison of DMSO and Various Methanol Extraction Procedures

| Treatment ⁽¹⁾ | Sample | Percent Cocaine Found (as Base) |
|--|----------------------|---------------------------------|
| 1. DMSO Extraction | 25772 | 0.39 |
| | 25773 | 0.40 |
| 2. CH ₃ OH Extraction, Flask, Steam bath | 25772 | 0.61 |
| | 25773 | 0.58 |
| 3. CH ₃ OH Extraction, Tube 30 min. heat 40 min. heat Standing overnight then 1 hr heat | 25772 ⁽²⁾ | 0.53 |
| | | 0.60 |
| | | 0.65±0.02 |
| | | |
| 4. CH ₃ OH Extraction, Tube 10 min. heat 20 min. heat Standing overnight Additional 1 hr heat | 25772 ⁽³⁾ | 0.54 |
| | | 0.54 |
| | | 0.56±0.01 |
| | | 0.66±0.02 |
| 5. CH ₃ OH Extraction, Tube Sonic mixed 15 min. 1 hr heat | 25772 ⁽³⁾ | 0.26 |
| | | 0.51 |

- Notes: 1. Each numbered treatment done on a separate 500 mg aliquot of the sample.
2. Internal standard added after initial heating.
3. Internal standard added prior to initial heating.

TABLE 2

Comparison of Sonic Mixing and Heating of Aliquots of 25773 in
Tube and Flask

| Treatment | <u>Percent Cocaine Base Found</u> | | |
|--|-----------------------------------|--------|-------|
| | Tube 1 | Tube 2 | Flask |
| 1. 1 hr sonic mixing | 0.43 | 0.45 | 0.49 |
| 2. 2 hrs sonic mixing | 0.51 | | 0.55 |
| 3. 1 hr sonic mixing plus 1 hr heating in block | | 0.63 | |
| 4. 2 hrs sonic mixing plus 1.5 hrs heating in block | 0.61 | | 0.58 |
| 5. Additional 1.5 hrs heating | 0.64 | 0.63 | 0.61 |

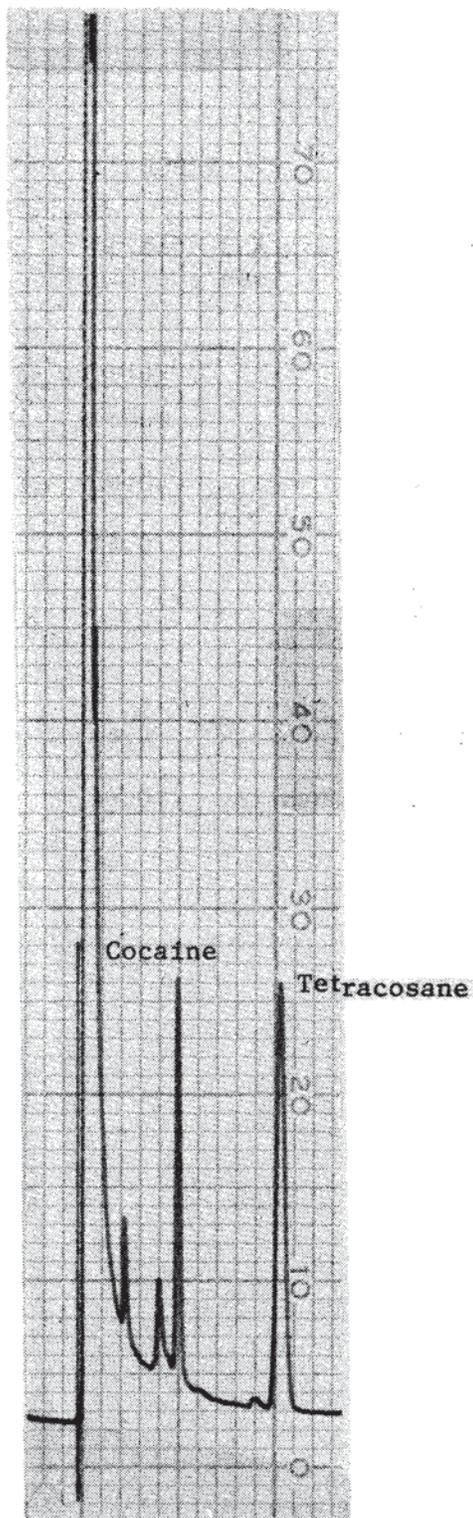


Figure 1. Chromatogram of Sample 25773 after 1 hour sonic mixing plus 1 hour heating in block. (See Table 2, Treatment 3). Column 210°C. (Chart speed 10 min./in.)