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TECHNICAL NOTE

W. A. Trinler,[1] *Ph.D. and D. J. Reuland,*[1] *Ph.D.*

Unequivocal Determination of Cocaine in Simulated Street Drugs by a Combination of High Performance Liquid Chromatography and Infrared Spectrophotometry

Since the development of high performance liquid chromatography (HPLC) in the late 1960s papers have appeared in the literature describing the identification of drugs of abuse by this method. For example, Jane [*1*] reported a separation of a number of drugs on a Partisil column. However, while extremely useful in the forensic laboratory, HPLC has not attained its full potential because of the presumptive nature of the determination, that is, one cannot rule out the possibility that two compounds might have the same retention time. In an attempt to improve this situation a few companies are investigating the feasibility of interfacing the HPLC with a mass spectrometer. If such interfacing is successful, though, the cost probably will be prohibitive for most forensic laboratories. Also, some progress is being made in the technique of "absorbance ratioing" [*2*]. In this method the drug is monitored at more than one wavelength and the ratio of these absorbances is reported to be solely characteristic of the particular compound. Although promising, this technique requires a more elaborate and expensive detector system. Also more data need to be collected to prove statistically the premise that no two compounds will have the same absorbance ratios.

At present infrared spectroscopy, the classical method of obtaining a compound's "fingerprint," still seems to be the best method of unequivocal identification of illicit drugs, all factors being considered. Recently several techniques have been reported for obtaining infrared spectra of microgram amounts of sample. These include micro internal reflection [*3*], micro KBr disk [*4*], diamond cell [*5*], the use of computer techniques, or some combination of these. All of these methods require specialized ancillary equipment and sample handling procedures.

The purpose of this study was to ascertain the feasibility of analyzing drugs of abuse through a combination of HPLC and conventional infrared spectroscopy without having to resort to any of the aforementioned specialized techniques. Simulated street samples containing cocaine were chosen for this study as cocaine is one of the more commonly encountered drugs of abuse.

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[1] Professors, Chemistry Department, Indiana State University, Terre Haute, Ind. 47809.

Experimental Procedure

Equipment

The chromatographic separation was made on a Waters Associates 202/401 HPLC equipped with the U6K Universal Injector. The column was two sections of 3.2-mm (1/8-in.) outside diameter by 0.6-m (2-ft) stainless steel hand-packed with Waters Associates Bondapak Phenyl/Porasil B. The ultraviolet (UV) detector was operated in the 254-nm mode. The recorder was a Houston Instrument Omniscribe with one pen connected to the UV detector and one pen connected to the refractive index detector.

The infrared spectrophotometer was a Perkin-Elmer 710A. The KBr die was a Beckman Evacuable KBr Minidie.

Supplies

Cocaine was obtained from Merck. Benzocaine, procaine, and tetracaine were obtained from Sigma Chemical Co. Xylocaine® was purchased from ICN Pharmaceuticals, Inc. Acetonitrile, "distilled in glass" grade, was obtained from Burdick and Jackson Laboratories, Inc. Ammonium carbonate was reagent-grade purity, and lactose was U.S. Pharmacopeia-grade. The absolute ethyl alcohol was obtained from IMC Corp. All these chemicals were used without further purification. The water used was deionized and doubly distilled from glass.

Procedures

Simulated street samples of cocaine were prepared composed of 10% cocaine, 20% active excipient, and 70% diluent. The active excipients were either benzocaine, procaine, tetracaine, or Xylocaine (lidocaine), commonly found in cocaine street drugs, and the diluent was lactose. Each sample was extracted with sufficient solvent to give a solution of 1 mg/ml. The extracting solvent was chosen to have the same composition as the mobile phase used in the separation, 85:15 acetonitrile/water by volume, with 0.1% by weight of ammonium carbonate. Cocaine and the active excipients are readily soluble in this solvent while the lactose is only slightly soluble and must be removed by filtration. Using the same solvent for the extractant and the mobile phase precluded the possibility of lactose precipitating during the chromatographic separation. If samples were not immediately analyzed they were refrigerated to retard possible decomposition.

Acetonitrile and water mixtures have been demonstrated to be effective in separating many mixtures containing drugs of abuse [*6,7*]. The composition of 85:15 was found to separate cocaine from any one of the active excipients used in the simulated street samples. Ammonium carbonate was added to insure the presence of the drugs as the free bases. A flow rate of 2 ml/min was chosen and yielded a pressure of approximately 20 MPa (3000 psi). The preparative-type packing was used in an analytical column to insure sufficient capacity for the macro size sample used. Injections of 0.25 ml were collected. In order to have sufficient compound for a satisfactory infrared spectrum two collections were made for each sample. The fraction of the eluate collected will depend on the dead volume from the detector to the outlet of the collection tube. For our system this dead volume was about 1 ml. Therefore each peak was collected in the interval one-half minute from the time it appeared to one-half minute after either it returned to baseline or another peak emerged.

Following collection the samples were placed in a warm bath at approximately 50°C and evaporated to dryness under the reduced pressure of a water aspirator. The residue was taken up in 0.5 ml of absolute ethyl alcohol and this solution was added to 170 mg of potassium bromide. This slurry was placed in a vacuum desiccator and dried by means of

a mechanical pump. The dry powder was ground in an agate mortar and mixed thoroughly in a Wig-L-Bug® amalgamator. The sample was then pressed into a pellet and analyzed.

Results and Discussion

The results of this study are depicted in Figs. 1 to 6. In Figs. 1 to 4 the chromatographic separations of each street sample under the conditions described above are shown.

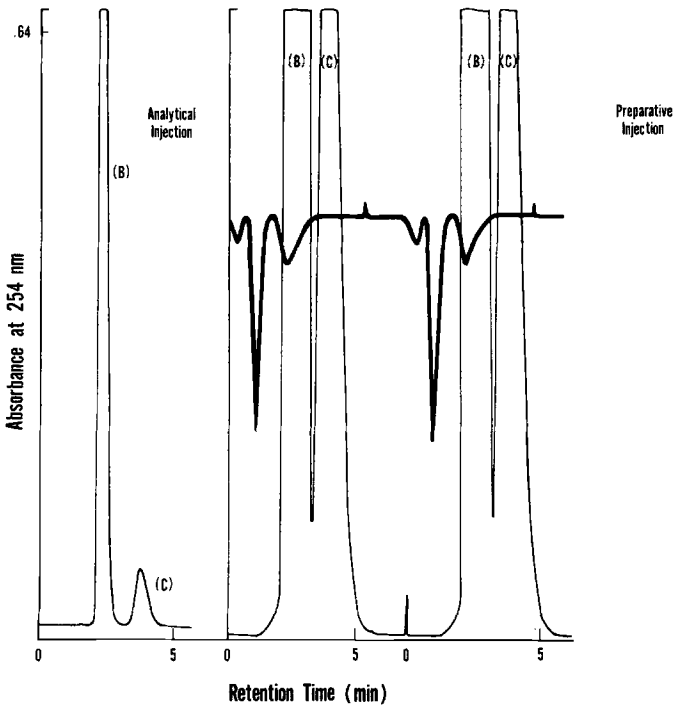

FIG. 1—*Chromatographs of benzocaine* (B) *and cocaine* (C) *sample.*

In each figure the first trace represents that of a 10 to 15 μg analytical-size sample, displayed to better illustrate the chromatographic separation. Adjacent to it are the traces of the two preparative-size samples chromatographed and collected for analysis. The upper trace is from the output of the refractive index detector. Because of the low solubility of the lactose in the extracting solvent and the low sensitivity at which the refractive index detector was set no lactose peak was apparent in any of the chromatographs. In some cases a peak is present which is thought to be due to an impurity or decomposition product. Figures 5 and 6 show the infrared spectra of each of the collected fractions. As evidenced from these spectra no measurable amounts of interfering substances are observed and the spectra provide an unequivocal identification of each drug.

If the chromatograph is set up with the proper solvent and column and a satisfactory baseline has been established the analysis time from initial extraction to final infrared spectrum requires approximately 1 h for cocaine only and an additional 15 min per excipient.

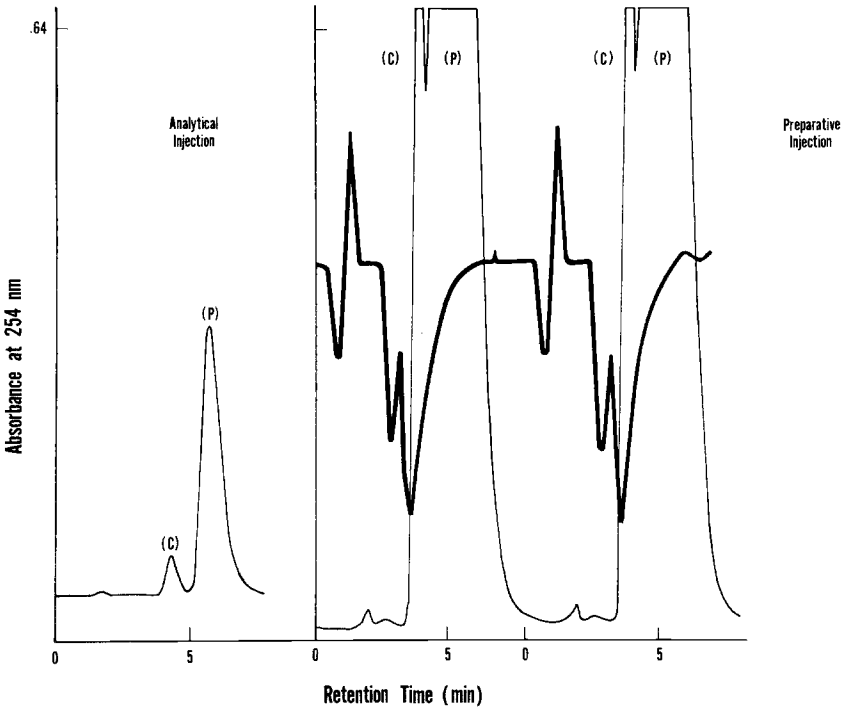

FIG. 2—*Chromatographs of procaine* (P) *and cocaine* (C) *sample.*

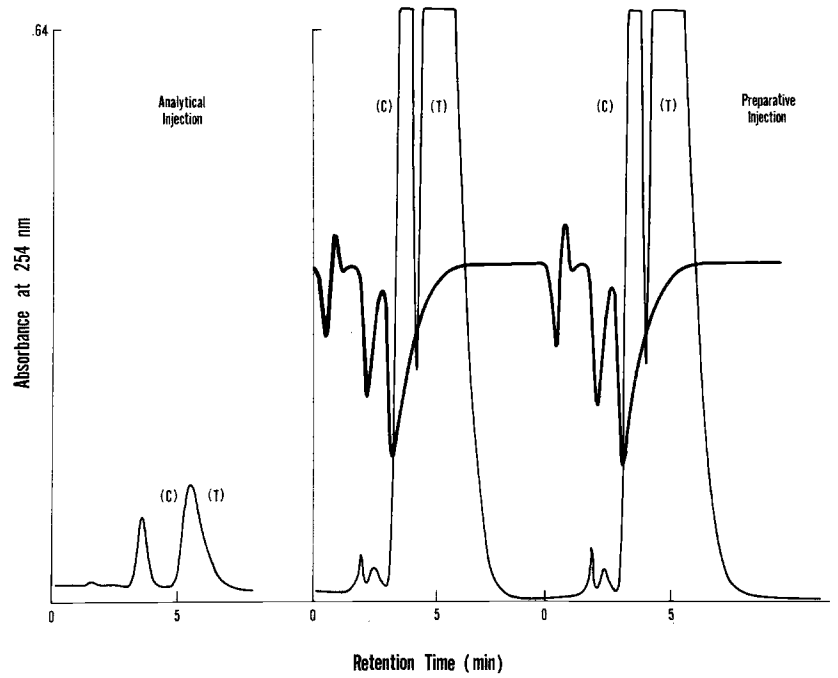

FIG. 3—*Chromatographs of tetracaine* (T) *and cocaine* (C) *sample.*

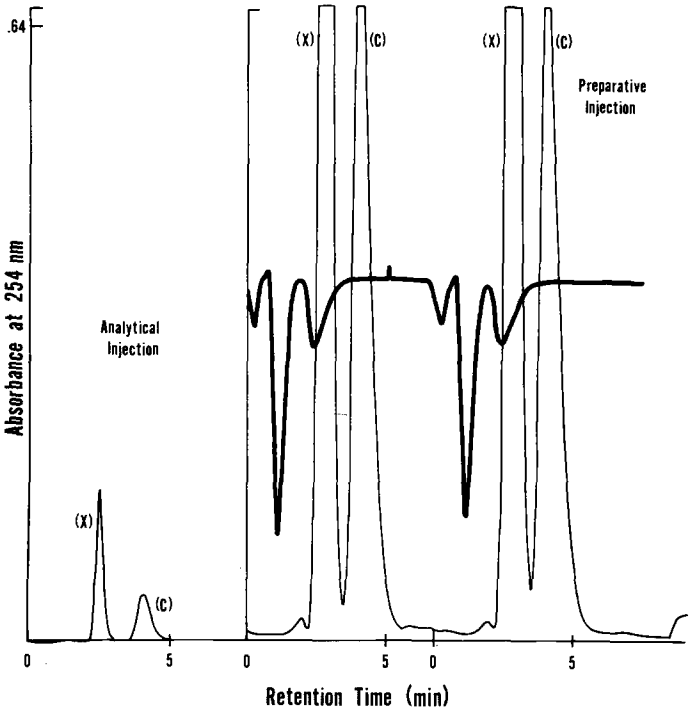

FIG. 4—*Chromatographs of Xylocaine* (X) *and cocaine* (C) *sample.*

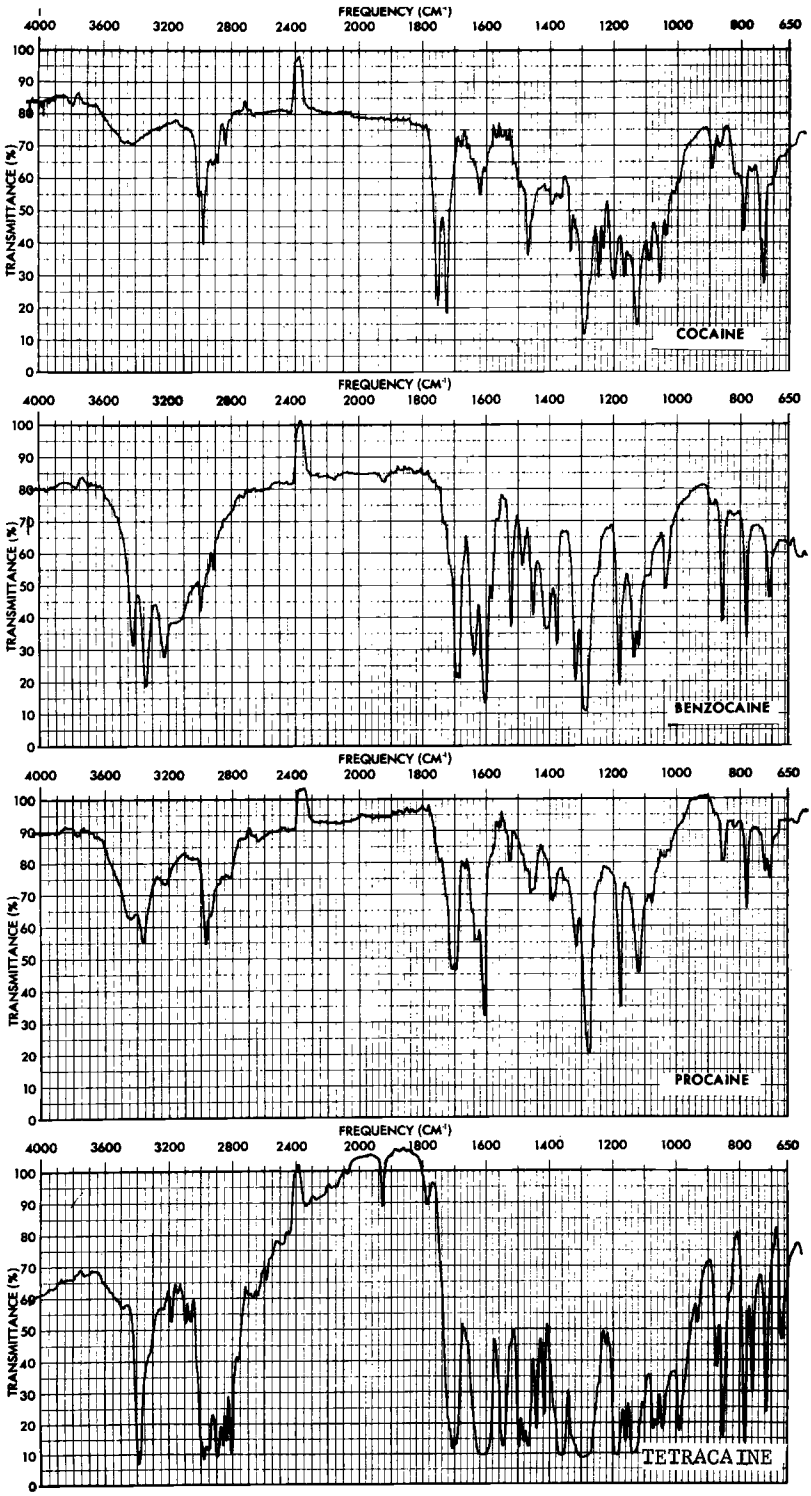

FIG. 5—*Infrared spectra of chromatographed drugs: cocaine, benzocaine, procaine, and tetracaine.*

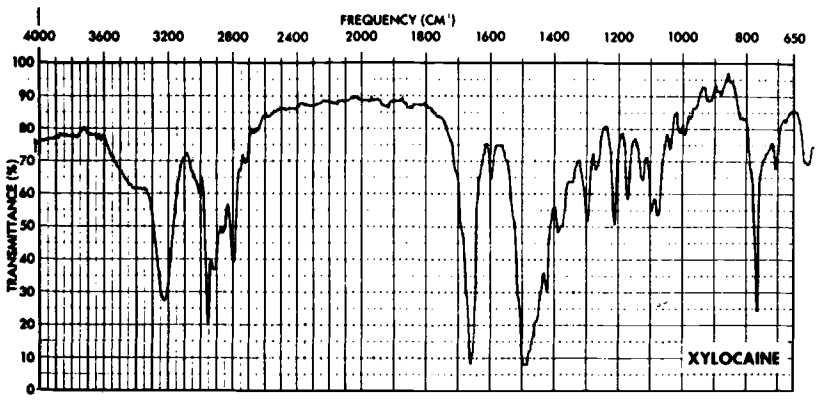

FIG. 6—*Infrared spectra of chromatographed Xylocaine.*

Summary

A technique for the unequivocal identification of cocaine in street samples adulterated with the commonly encountered excipients benzocaine, procaine, tetracaine, lidocaine, and lactose has been described. Milligram quantities of samples were analyzed by a combination of HPLC and infrared spectrophotometry. Conventional sampling techniques and equipment were used. Analysis time was approximately 1 h per sample.

Acknowledgments

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Address requests for reprints or additional information to
W. A. Trinler, Ph.D.
Chemistry Department
Indiana State University
Terre Haute, Ind. 47809