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Note

Gas–liquid chromatographic–mass spectrometric determination of lidocaine in an illicit sample of cocaine

SATYA P. JINDAL, THERESA LUTZ and PER VESTERGAARD
Rockland Research Institute, Orangeburg, N.Y. 10962 (U.S.A.)
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Cocaine, a pharmacologically active alkaloid, is one of the most powerful naturally occurring stimulants known. Its euphoric and stimulant effects induce a high level of physiological dependence[1,2]. Detailed information, concerning the historical, chemical, physiological and treatment aspects of cocaine use and abuse, has recently been published[3,4]. The changing nature of cocaine use in our society has recently been reviewed[5,6]. Cocaine use appears to have greatly increased over the last 10 years. Because of its price and its distribution through black market channels, less expensive, more available materials are likely to be misrepresented as cocaine. This "drug deception" is, in fact, fairly common[7]. Much of what is sold as cocaine is actually amphetamines and/or other white substances. Common substitutions for cocaine, as analyzed by street drug analyses programs, are amphetamines, tetracaine and other local anesthetics. This report describes a gas–liquid chromatographic (GLC)–mass spectral identification and quantitation of lidocaine as a major component in an illicit sample of cocaine.

MATERIALS AND METHODS

Analytical grade cocaine hydrochloride, lidocaine (Mallinckrodt, New York, N.Y., U.S.A.), trifluoroacetic anhydride and all solvents used in this work were of analytical grade (Pfaltz and Bauer, Flushing, N.Y., U.S.A.). The illicit sample of cocaine was supplied by the New York State Drug Addiction Control Commission. The stock solution of cocaine, the illicit sample of cocaine and of lidocaine were made in chloroform.

INSTRUMENTATION

A magnetic sector, single-focusing mass spectrometer, LKB 9000, (LKB, Stockholm, Sweden) interfaced with a gas chromatograph and equipped with a multiple ion detector–peak matcher accessory (MID–PM) was used in this investigation[8,9]. GLC was performed on a glass column (1.8 m × 2 mm I.D.) silanized with 5% dimethyldichlorosilane in toluene and packed with 3% OV-17 on 100–200 mesh Gas-Chrom Q. The column was conditioned for 24 h at 280° with a flow-rate of 20 ml of helium/min. The column temperature was 200°, the flash heater was at 230°,

the separator was at 235° and the ion source was at 250°. The accelerating voltage was 3.5 kV, the ionization potential was 70 eV and the trap current was set at 60 μA.

RESULTS AND DISCUSSION

Gas chromatographic analysis of illicit cocaine with flame ionization detector (Fig. 1) shows two peaks with retention times of 2 min and 6 min, respectively. The peak at 6 min was identified as cocaine, its mass spectrum[10,11] and GLC retention time were identical to that of authentic cocaine. GLC analysis of illicit cocaine with a nitrogen detector (Fig. 1) also shows two peaks as above, however, the relative intensity of the first peak is almost doubled, indicating the material comprising the first peak with at least two nitrogen atoms. The illicit cocaine was treated with trifluoroacetic anhydride, GLC analysis of the resulting product again gave two peaks with identical retention times indicating the material, corresponding to the first peak, has nitrogen atoms as tertiary amine, amide and/or nitrile and that all of the nitrogen atoms in the molecule are devoid of ionizable hydrogen atoms.

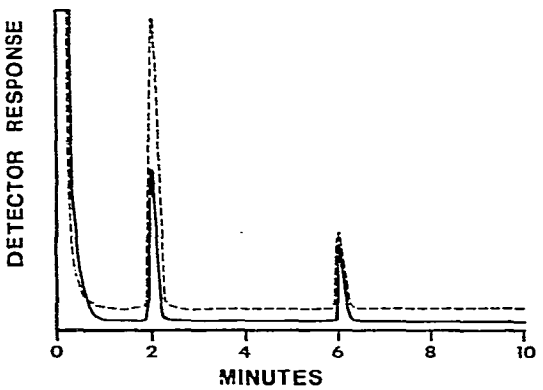

Fig. 1. Gas chromatograph of illicit cocaine; 1 μl (100 ng/μl) of the stock solution in chloroform as injected; flame ionization detection (——), specific nitrogen detection (- - - -).

The mass spectrum of the material representing the first peak (Fig. 2), shows a molecular ion at *m/e* 234, a base peak at *m/e* 86 and peaks of minor intensities at *m/e* 132, 147 and 217, respectively. The mass spectrum and the GLC analysis presented above leads to lidocaine as the likely structure of this material. Assuming charge localization at the nitrogen atom, as a result of electron impact ionization and following the established ion fragmentation mechanisms[12], the spectrum can be rationalized (see Fig. 3). Exact mass measurements of the material (234.1703 $\pm$ 12 ppm) and the identity of the mass spectrum with that of the authentic lidocaine further confirmed the structure of the material.

GLC standard curve of lidocaine using cocaine as an internal standard was made; the plot of the peak height ratio of lidocaine–cocaine *versus* the amount of lidocaine per fixed amount of cocaine was linear. The amount of lidocaine calculated in the illicit cocaine sample was found to be 37% by weight.

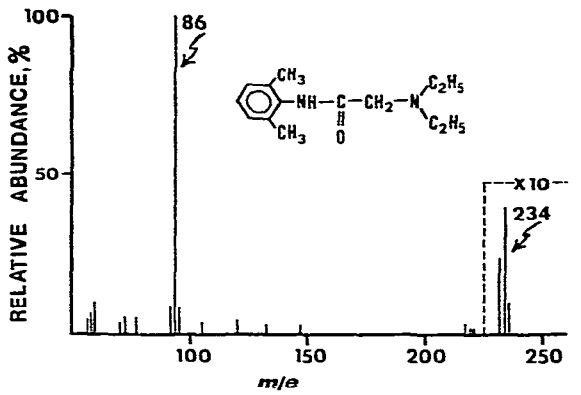

Fig. 2. Electron ionization (70 eV) mass spectrum of lidocaine (peak 1, retention time, 2 min) present in illicit cocaine.

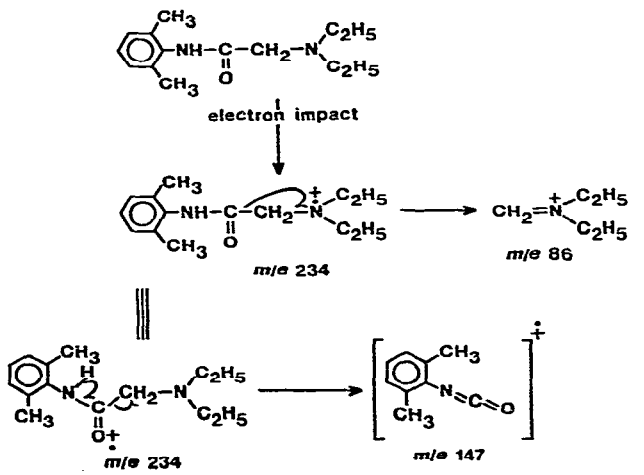

Fig. 3. Electron impact fragmentation of lidocaine.

There is some evidence that lidocaine potentiates the central nervous system effects of cocaine and the patients using this illicit sample experienced longer lasting and much greater stimulation. Further work in this area, particularly the cocaine interactions with lidocaine and other synthetic and naturally occurring local anesthetics needs to be undertaken.

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