TECHNICAL NOTE

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Efficiency of Capillary Column Gas Chromatography in Separating Lysergic Acid Diethylamide (LSD) and Lysergic Acid Methylpropylamide (LAMPA)

REFERENCE: Stafford, D. T., Nichols, H. S., and Anderson, W. H., **"Efficiency of Capillary Column Gas Chromatography in Separating Lysergic Acid Diethylamide (LSD) and Lysergic Acid Methylpropylamide (LAMPA),"** *Journal of Forensic Sciences*, JFSCA, Vol. 29, No. 1, Jan. 1984, pp. 291–298.

ABSTRACT: A question frequently asked of forensic drug chemists when they go to court on lysergic acid diethylamide (LSD) cases is, "How do you know this sample was not lysergic acid methylpropylamide (LAMPA) instead of LSD?" There are chromatographic means of separating the two compounds, but some of these are not very chromatographically efficient and others require time-consuming preparation of the chromatographic system. The separation on methyl silicone fused silica capillary columns described here can be performed routinely in a very time effective manner. One of the more interesting aspects of this work is the efficiency of the capillary system in this application. This combined with the inertness of the fused silica column and injection port liner, makes a very powerful and flexible means for addressing the separation problem and providing an answer to the original question.

KEYWORDS: toxicology, lysergic acid, chromatographic analysis

One of the problems that has plagued drug chemists for some time is the question of whether or not a sample identified as lysergic acid diethylamide (LSD) could actually be lysergic acid methylpropylamide (LAMPA). Each has a molecular weight of 323, and they exhibit nearly identical infrared (IR) and mass (MS) spectra.

Thin-layer chromatographic (TLC) systems are usually not efficient enough to provide adequate separation [*1,2*]; and while both normal and reversed phase liquid chromatography have sufficient efficiency for separation [*3,4*], the results are not readily characterized, and a separate operation such as IR or MS must be performed for confirmation. Because of the labile nature of these compounds, packed column gas chromatographic techniques have not been very successful [*5*]. Among the advantages of modern capillary column gas chromatography (GC) are the inertness of the system and the ability to generate a large number of theoretical plates rather quickly in a short column [*6*]. In addition, compound characterization by reten-

Received for publication 12 March 1983; revised manuscript received 9 April 1983; accepted for publication 12 April 1983.

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tion indices [7] and direct capillary GC/MS can be effectively utilized. These factors combine to make capillary gas chromatographic separation of LSD and LAMPA quite feasible and, in fact, rather easy on a routine basis.

Equipment

The instrument used in this work was a Hewlett/Packard Model 5880, Level IV gas chromatograph with capillary injector in the split mode at a split ratio of 40:1. Two features that contributed to the success of the chromatography were a fused silica injection port liner illustrated in Fig. 1, and the positioning of the column to within 1 mm of the jet tip as shown in Fig. 2.

The columns were J & W Scientific fused silica columns 0.25-mm inner diameter with a 0.25-μm film of SE-30. GC/MS work was done with the same columns using a Finnegan Model 4000 mass spectrometer. Standards of LSD and LAMPA were obtained from Applied Science.

Procedure

The cleanup procedure for street drug samples of suspected LSD was that which has been routinely used in this laboratory for a number of years. The sample, dissolved in 3 mL of 0.25*N* sulfuric acid and with sodium bicarbonate to give a pH of 8, was thoroughly mixed with 5 g of Celite 545 AW. This was packed in a 20- by 85-mm glass column and the drug was eluted with 50 mL of chloroform. The chloroform eluant was extracted with 2 mL of 1% tartaric acid which was then made basic with sodium bicarbonate and extracted with 50 μL of chloroform in a 5-mL centrifuge tube. This extract was sufficiently clean and of sufficient quantity for TLC, micro-color test, fluorometry, capillary GC, and capillary GC/MS analyses.

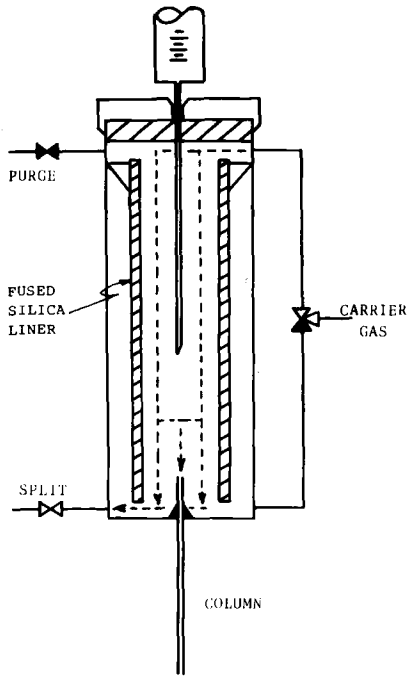


FIG. 1—*Schematic of capillary injection port.*

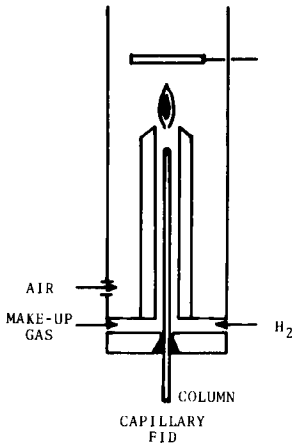


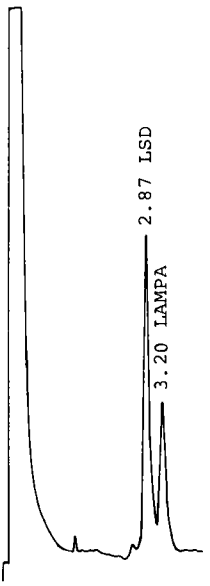
FIG. 2—*Positioning of the column to within 1 mm of the jet tip.*

Results of Capillary Gas Chromatography

The objectives of this work were:

(1) separate and characterize LSD and LAMPA by capillary gas chromatography and
(2) determine the effects of carrier gas and column conditions on the efficiency of the separation.

Figure 3 shows a chromatogram of the 245°C isothermal separation of LSD and LAMPA on a 3.5-m column with helium as carrier gas at 44-cm/s linear velocity. Column efficiencies of over



L - 3.5m
He - 44cm/sec
Iso - 245°C

	LSD	LAMPA
k	22.9	25.6
N	8186	8851
N/m	2339	2529
Ne	7220	7686
Ne/m	2063	2196

0.25mm ID FUSED SILICA; 0.25um FILM, SE-30
40:1 SPLIT RATIO

A/S-2

FIG. 3—*Chromatogram of the 245°C isothermal separation of LSD and LAMPA on a 3.5-m column with helium as carrier gas at 44-cm/s linear velocity.*

2300 theoretical plates/m (*N*) and over 2000 effective theoretical plates/m (*Ne*) were obtained. This separation was achieved in less than 3.5 min. While baseline resolution was not obtained, it should be pointed out that the carrier gas linear velocity was nearly double the optimum velocity. To operate at a more nearly optimum linear velocity and keep analyses times short, a linear velocity of 27 cm/s was used with a shorter, 1-m column. Figure 4 shows the slightly better resolution in about the same time under these conditions, but at a lower temperature, 210°C.

In an attempt to obtain better efficiency without sacrificing analysis time, the carrier gas was changed to hydrogen at a linear velocity of 75 cm/s with a 5-m column operated isothermally at 240°C. Figure 5 shows the much better resolution that resulted, although the carrier velocity is nearly twice the optimum. The mass spectra of LSD and LAMPA from a capillary GC/MS run using a Finnegan Model 4000 system are shown in Figs. 6 and 7, respectively.

In the basic drug screen normally used by the University of Tennessee Toxicology Laboratory, a 15-m SE-30 capillary column is used in a programmed mode. Since this column is generally in place, and because we have developed a retention index data base for the system, it was desirable to investigate the use of the longer column and temperature programming for

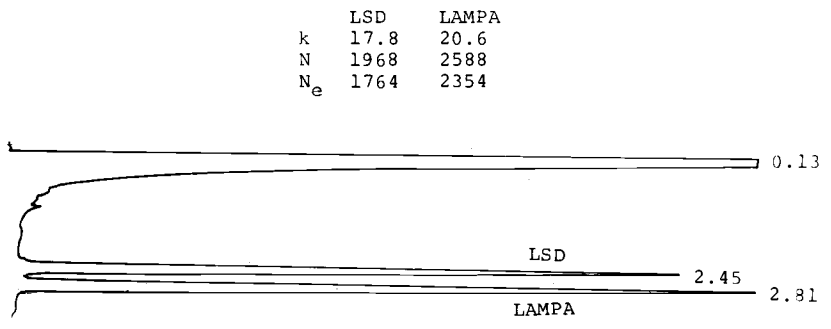


FIG. 4—*Chromatogram of the 210°C isothermal separation of LSD and LAMPA on a 1-m column with helium as carrier gas at 27-cm/s linear velocity.*

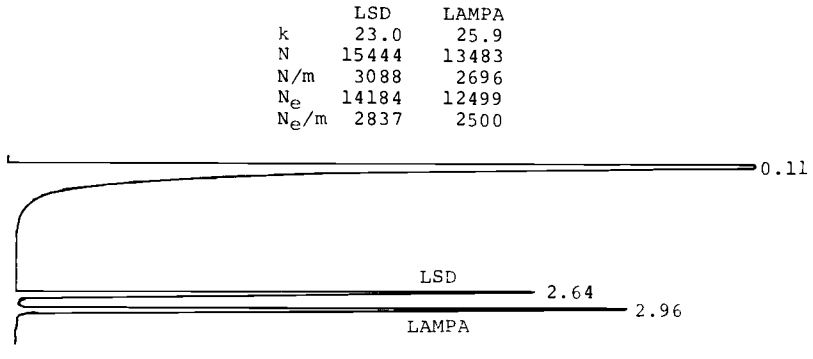


FIG. 5—*Chromatogram of the 240°C isothermal separation of LSD and LAMPA on a 5-m column with hydrogen as carrier gas at 75-cm/s linear velocity.*

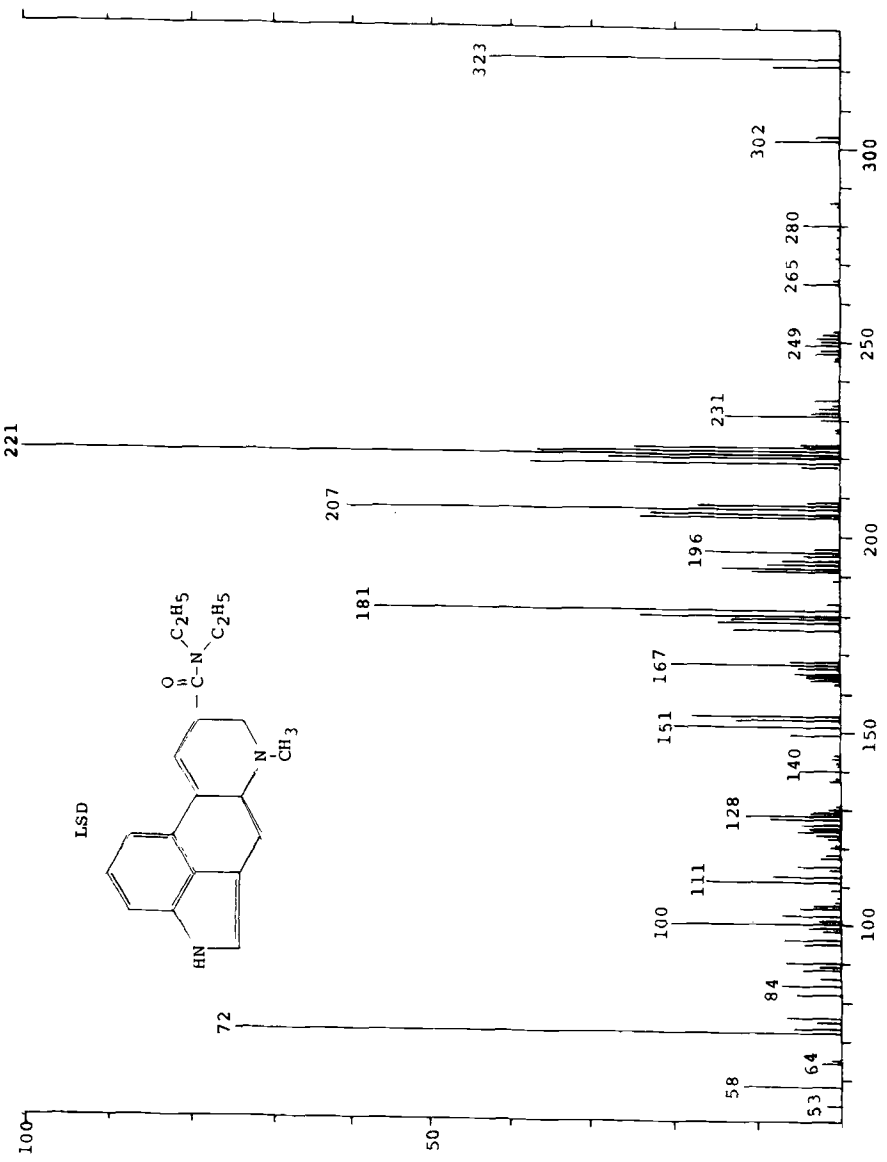


FIG. 6—*Mass spectra of LSD from a capillary GC/MS run using a Finnegan Model 4000 system.*

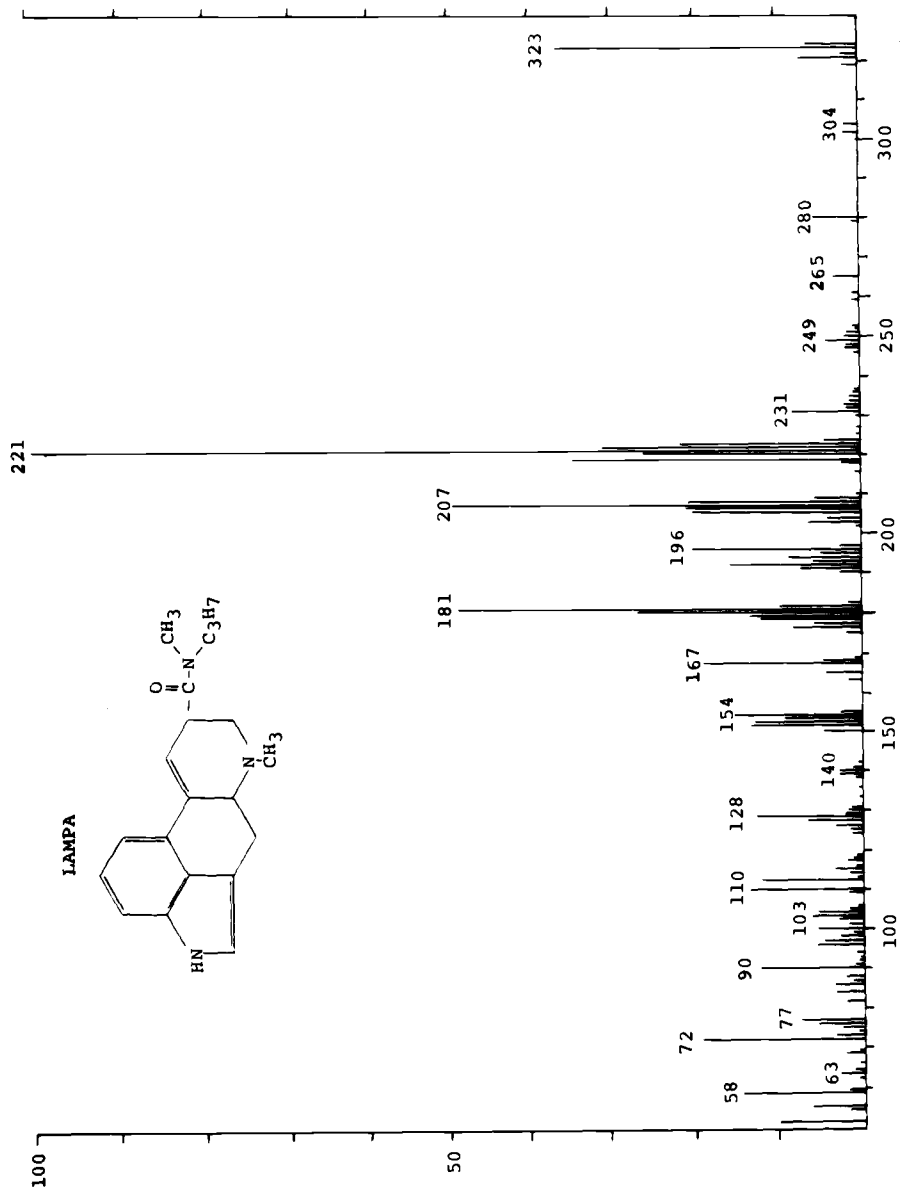


FIG. 7—*Mass spectra of LAMPA from a capillary GC/MS run using a Finnegan Model 4000 system.*

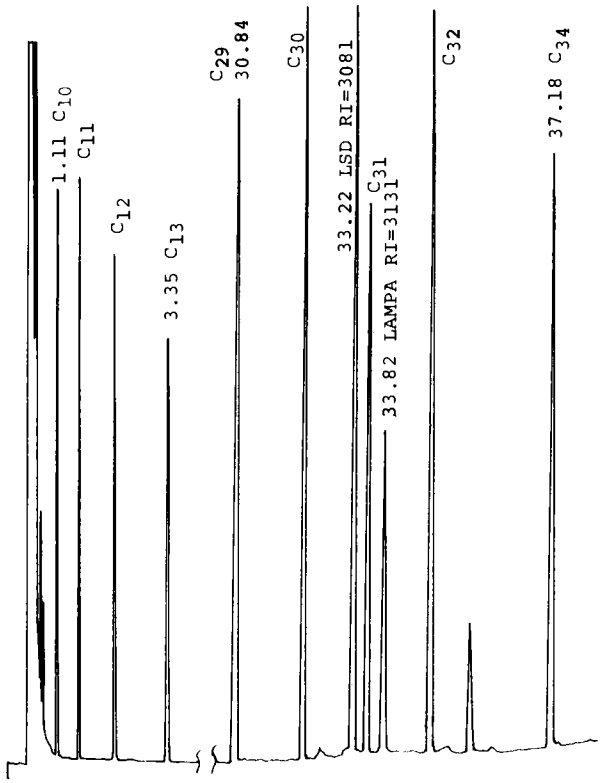


FIG. 8—*Capillary chromatography with a 15-m SE-30 capillary column using helium at a linear gas velocity of 44 cm/s and a temperature program from an initial temperature of 100°C to a final temperature of 295°C at a rate of 5°C/min.*

the LSD and LAMPA separation. Figure 8 illustrates the capillary chromatography with this column using helium at a linear gas velocity of 44 cm/s and a temperature program from an initial temperature of 100°C to a final temperature of 295°C at a rate of 5°C/min. As is evident, the two compounds are separated by 50 retention index units and have excellent shapes. The *n*-hydrocarbons from C-10 to C-34 are included for reference; and retention indices are calculated by linear interpolation between the two hydrocarbons bracketing the peak of interest. This allows the column to be used quite effectively for the separation and characterization of these compounds.

All of the development work was done using known LSD and LAMPA standards. To date, 30 to 50 street drug samples containing LSD have been analyzed using the procedure, with backup confirmation by mass spectrometry. In none of these would the presence of LSD have been incorrectly reported using only the capillary GC data; and as suspected, LAMPA has not been detected in any of them.

Discussion

It is possible to separate and characterize LSD and LAMPA quite efficiently using capillary column gas chromatography. Primary factors in handling these labile compounds by GC are

the use of the very inert fused silica injection port liner and column, and positioning of the column in the flame-ionization detector (FID) jet. This minimizes contact with active surfaces that promote degradation.

For fast isothermal chromatography a relatively short column, 5 m, with hydrogen carrier gas permits baseline separation in less than 3 min.

For better characterization a longer, 15-m column can be used with temperature programming. This can result in separation of the compounds by 50 retention index units in a system that has been demonstrated to be capable in routine use of reproducing retention indices to ± 2 index units over a period of usage greater than one year. The system also has been demonstrated to be quite effective for use in capillary GC/MS operation.

A sufficiently large number of street drug cases have been run to demonstrate the applicability to actual samples as well as standards. This system provides the drug chemist with the most powerful and definitive separation technique currently available for handling LSD and LAMPA.

Acknowledgment

The authors acknowledge the help and express their appreciation to Roger Martz of the FBI Laboratory Division, Washington, DC, in obtaining the mass spectral data.

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