DIVISION OF NARCOTIC DRUGS
Vienna

RECOMMENDED METHODS FOR TESTING LYSERGIDE (LSD)

MANUAL FOR USE BY NATIONAL NARCOTICS LABORATORIES

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ST/NAR/17

CONTENTS

INTRODUCTION

Background

Over the past few years there has been a considerable increase in the number of substances newly put under international control. At the same time, seized quantities of drugs already under control have also shown an alarming and unprecedented increase in certain regions. This new situation, involving an increase both in the frequency and volume of seizures, presents a challenge not only to national law enforcement authorities, but also to the technical and scientific staff of forensic laboratories.

Analysts have to be able to deal with more substances and preparations and to use faster, more accurate and more specific methods of identification and analysis. In addition, the international character of drug trafficking requires the timely exchange of analytical data between laboratories and law enforcement authorities both on the national and the international levels.

The Commission on Narcotic Drugs, at its tenth special session in February, 1988, reviewed the technical and scientific assistance programme of the Division of Narcotic Drugs with special emphasis on the development of laboratory methodologies. It noted with satisfaction that the harmonization of laboratory methods and the programme on establishment of recommended methods on testing for national forensic laboratories was pursued vigorously and many such methods had already been developed and published.

In emphasizing the importance of the expert group meetings organized by the Division on various scientific and technical aspects of drug control and the high practical value for national law enforcement and laboratory services of the technical manuals as the outcome of the expert meetings, the Commission strongly recommended that such meetings and the publication of laboratory manuals continue on a regular basis.

Purpose of the manual

In response to the Commission's request, a group of eleven experts was convened in June 1988 in Ottawa, Canada, by the Division of Narcotic Drugs in co-operation and with the financial support of the Government of Canada through UNFDAC. The present manual published by the United Nations Division of Narcotic Drugs reflects the conclusions of the group of experts and has been designed to provide assistance to national authorities by describing recommended methods to be used in forensic laboratories for the identification and analysis of LSD. The manual may also serve as a guide to national authorities in assessing existing methods used within their own Government and university laboratories.

This manual is one in a series of similar publications dealing with the identification and analysis of various groups of drugs under international control; it was preceeded by manuals on heroin (ST/NAR/6), cocaine (ST/NAR/7), cannabis (ST/NAR/8), amphetamine and methamphetamine (ST/NAR/9), opium/crude morphine (ST/NAR/11), ring-substituted amphetamine derivatives (ST/NAR/12), methaqualone/mecloqualone (ST/NAR/15) and benzodiazepines (ST/NAR/16).

These manuals suggest approaches that may help the forensic analyst to select a technique appropriate to the sample currently being examined. The analyst may then choose to follow any of the methods described in the manual, as each method can be expected to produce reliable analytical information with respect to the samples to which they are applied. Each method has been used for a number of years in reputable forensic laboratories and has been published in the scientific literature. In identifying these methods, the expert group was aware that many other useful and acceptable methods produce worthwhile analysis and information for the forensic analyst, and that a number of other acceptable options are recorded in the forensic scientific literature.

Use of the manual

Few methods are perfect, least of all in forensic drug analysis where the materials under examination are very likely to show significant variation both in their physical form and chemical composition. The choice of methodology and approach to analysis remain within the control of the analyst working within his or her own country. The analyst alone has seen the suspect material and can best judge the correct approach to the problem at hand. Furthermore the choice of methods may necessarily depend on the availability of reference materials and of instrumentation.

Not all the methods listed need to be applied to all samples suspected to contain LSD. Requirements may vary, for example, as a result of local trends in samples encountered, facilities available, and the standard of proof acceptable in the prosecution system within which the analyst works.

In order to establish the identity of any controlled drug, it is suggested that the criteria should be at least two independent analytical parameters. The selection of these parameters in any particular case would take into account the drug involved and the laboratory resources available to the analyst. For example, two uncorrelated TLC systems would count as two parameters. Uncorrelated TLC systems in this context means that either the solvent systems or the coating on the plates are completely different. When possible, three entirely different analytical techniques should be used, for example: colour test, chromatography (TLC, GLC or HPLC) and spectroscopy (IR or UV). The actual choice of parameters is left to the discretion of the chemist.

Attention is also drawn to the vital importance of the availability of textbooks on drugs of abuse and analytical techniques. Furthermore, the analyst must continually keep abreast of current trends in analysis, consistently following current analytical and forensic science literature. Analysts should refer to these and to previous manuals in this series for general descriptions of the analytical techniques included in this manual.

It is equally important that the latest information on changes in drugs available in the illicit traffic be quickly disseminated. This may often need to be done prior to publication in specialized periodicals dealing with forensic and other chemical analyses, since these publications are available to the forensic community some two to three years after the changes become known. The value of frequently published national reports on the latest information on such changes in drugs and on work being undertaken and analytical results obtained within individual laboratories cannot be over-emphasized.

The Division of Narcotic Drugs would welcome observations on the contents and usefulness of the present manual. Comments and suggestions may be addressed to:

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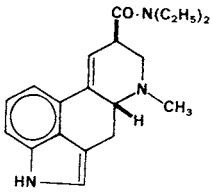
I. DESCRIPTION OF THE PURE COMPOUND

LSD

d-lysergic acid diethylamide
N,N-diethyl-d-lysergamide
Lysergide

Scheduled under the "Convention on Psychotropic Substances 1971"

LSD — Schedule I



$C_{20}H_{25}N_3O$
M.Wt. = 323.4

m.pt. = 80-85°C (decomp.)

LSD tartrate

$(C_{20}H_{25}N_3O)_2C_4H_6O_6.2CH_3OH$
M.Wt. = 860.9

m.pt. = 198-200°C (solvated)
(crystallized from methanol)

Solubilities	Base	Tartrate
Water	v. sl. soluble	soluble
Methanol	soluble	soluble

II. ILLICIT PRODUCTION OF LSD

LSD is one of the most potent hallucinogenic substances known. Its properties were first discovered in the 1930's and for many years it was occasionally used experimentally in the treatment of mental disorders. There has been no licit use for LSD in over 20 years and LSD products encountered today on the illicit market are produced only in clandestine laboratories.

LSD can be produced by several different methods, the majority of which use lysergic acid as the starting material. Lysergic acid itself is also produced in clandestine laboratories using, most commonly, ergometrine or ergotamine tartrate as starting material. Other ergot alkaloids may be substituted for these although they are not believed to be used frequently. It is not known which synthetic method is most commonly employed by clandestine laboratory operations.

LSD synthesis from lysergic acid

There are three reported methods for producing LSD using lysergic acid as the precursor. The first involves treatment of lysergic acid with lithium hydroxide to form lithium lysergate which is then reacted with a sulfur-trioxide dimethylformamide complex and diethylamine to form the crude LSD product.

The second method employs the reaction of lysergic acid with N,N-carbonyldiimidazole followed by treatment with diethylamine. The last involves the reaction of lysergic acid with trifluoroacetic anhydride and treating the resulting mixed anhydrides with diethylamine.

LSD synthesis from ergot alkaloids

In this method an ergot alkaloid or a mixture of ergot alkaloids are used as the starting material. The alkaloid(s) is treated with hydrazine hydrate to form lysergic acid hydrazide. Using sodium nitrite, the hydrazide is converted to the azide which is then reacted with diethylamine to form the finished product.

All of the methods described for the synthesis of LSD produce a crude product which contains large amounts of iso-LSD and other by-products. Removal of these impurities is generally accomplished by chromatographing the crude product on an alumina column or by a series of partitioning between weak organic acids and weak bases with a suitable organic solvent. Tartaric acid, sodium bicarbonate and methylene chloride are examples of the types of chemicals that have been used. Also, because of the instability of LSD base, the tartrate salt is generally produced.

This is done by precipitating the salt from a methanol solution of LSD base using a solution of tartaric acid in methanol as the precipitating reagent. Further details of the synthesis of LSD are contained in ST/NAR/10 (Clandestine Manufacture of Substances under International Control).

Lysergic acid synthesis

The most common method for producing lysergic acid in clandestine laboratories is the conversion of ergometrine or ergotamine tartrate to lysergic acid. This is accomplished by refluxing the ergot alkaloid with potassium hydroxide and hydrazine in an alcohol/water medium. Alternatively, lysergic acid can be produced by extracting lysergamide from Morning Glory or Hawaiian Baby Woodrose seeds and treating the purified extract of lysergamide in the same manner as described for ergotamine.

Lysergic acid can also be produced by fermentation of cultures of Claviceps purpurea or Aspergillus clavatus or through a multi-step process beginning with methyl-6-methylnicotinoate.

III. PHYSICAL APPEARANCE OF ILLICIT LSD PRODUCTS

When LSD was first introduced into the illicit market in the 1960's, it was common to apply it to a variety of substrates by adding a drop of an LSD solution to an absorbent material. Among the substrates commonly used were sugar cubes, blotter or other absorbent paper and pharmacologically inert powders which were then used to fill empty gelatin capsules. Another common dosage form was termed "window panes" or "pyramids" in which the LSD was incorporated into a gelatin matrix and the solidified gelatin cut into small squares. The most common dosage forms, however, were tablets of various sizes, shapes and colours.

The content of the tablets was extremely variable, ranging from 20 to 500 microgrammes of LSD due to the difficulty of obtaining a homogenous powder for tabletting. Thus, even though LSD tablets continued to be the predominant dosage form in the 1970's, the number of types of tablets decreased, being limited to those laboratories which were able to produce a more uniform product. One type of tablet in particular, the "microdot", became prevalent and consisted of round tablets, approximately 1.6 mm in diameter, containing reasonably uniform dosage of about 100 microgrammes of LSD per tablet.

In the 1980's, paper dosage forms became much more common. However, unlike earlier paper forms, where the LSD was dropped onto the paper and which are still frequently encountered in several countries, the new paper dosage forms are produced by soaking preprinted paper in a solution of LSD, thereby ensuring a more uniform product. Typically, these sheets are perforated into squares of approximately 5 mm^2 in size each containing a typical dose of 30-50 microgrammes of LSD. A variety of designs have been encountered on these sheets, ranging from abstract art to cartoon figures. Papers onto which LSD has been spotted are still frequently encountered in several countries.

At the present time, the great majority of the types of LSD dosage forms found in the illicit market are either paper dosage units, small tablets similar to the "microdots" and gelatin forms. The content of these forms is generally about 50 microgrammes of LSD. Nevertheless, due to the ease with which LSD solutions can be applied to a variety of substrates, other forms should not be discounted.

IV. THE ANALYSIS OF MATERIALS CONTAINING LSD

Because of the extremely high potency of LSD, it is essential to exercise good laboratory practice when analyzing materials containing LSD to prevent accidental ingestion or absorption of LSD by the analyst. Care must be taken during all stages of the analysis from handling the sample when it is received to final storage of the material after analysis. Particular care should also be taken during all testing procedures.

A. Sampling

The principal reason for a sampling procedure is to produce a correct and meaningful chemical analysis. Because most methods - qualitative and quantitative - used in forensic science laboratories for the examination of drugs require very small aliquots of material, it is vital that these small aliquots be entirely representative of the bulk from which they have been drawn. Sampling should be undertaken to conform to the principles of analytical chemistry, as laid down, for example, in national pharmacopoeias or by such organizations as the Association of Official Analytical Chemists.

There may be situations where, for legal reasons, the normal rules of sampling and homogenization cannot be followed if, for example, the analyst wishes to preserve some part of an exhibit as visual evidence. Alternatively, it may be necessary to perform separate assays on two powder items, rather than combining the powders prior to a single assay being performed on the mixture, because each has been separately exhibited by the seizing officer, and the legal system within which the analyst works requires individual results on every exhibit which is to be taken before the courts.

To preserve valuable resources and time, forensic analysts should seek, on all possible occasions, to use an approved sampling system and thereby reduce the number of quantitative determinations needed. To facilitate such an approach, the forensic analyst may need to discuss individual situations with both seizing officers and the legal personnel with whom he works.

As mentioned in Section III most LSD exhibits are either in paper, tablet or gelatin form. Powders are generally not encountered. The sampling procedure provided is applicable to these three forms. For the purposes of the sampling plan, one sheet of paper subdivided into smaller dosage units should be considered as one "container".

As stated in Section II, no licit LSD products are produced and therefore quality control may be regarded as non-existent. Wide variations may be suspected in each dosage form, although in most instances, some of the active constituent will be present in each. Some screening of individual units or containers is, therefore, necessary.

(a) Single container

Determine the total number of dosage units and the average weight per dosage unit (du).

For sample sizes up to 10 du -- screen all dosage units.

For sample sizes from 11 du to 27 du -- randomly select and screen 3/4 of all dosage units, rounding upward to the next higher integer.

For sample sizes from 28 du -- randomly select and screen 1/2 of all dosage units rounding upward to the next higher integer and selecting a minimum of 21 du and a maximum of 50 du.

Based on the results of the screening tests, proceed as follows:

1. If all dosage units appear to be identical, form a composite of screened dosage units by grinding, sieving through a 20-mesh sieve and thoroughly mixing in the case of tablets, or simply combining the units in the case of papers and gelatin dosage forms. Perform the analysis on the composite;

2. If the sample contains two dosage forms, subdivide the sample. If necessary, screen additional dosage units until both subsamples contain material for analysis, then form two composites and analyze;

3. If more than two dosage forms are present, the strategy is to make a composite of the most abundant dosage form, then to screen additional units until a sample of the same size is formed that contains only the less abundant dosage forms. This procedure is repeated until a composite is formed for each dosage form or until the sample is exhausted;

The percentage of dosage units containing LSD may be estimated by using the percent of units found to contain that substance out of the total number of units which were randomly selected and screened.

(b) Multiple containers

The analyst should examine the contents of all containers by eye to determine if one or more containers contain material different to that of the majority of packages. The simplest indicator is the physical appearance of the sample. If one or more containers obviously differ in content, these should be segregated and subjected to separate analysis.

Determine the square root of the total number of containers in each group. Randomly select a number of containers equivalent to the square root, rounded to the next highest integer.

From each of the selected containers, randomly select a number of dosage units equivalent to the square root of the total number of dosage units divided by the square root of the number of packages, rounded to the next higher integer.

Screen each unit using a presumptive test and/or TLC.

Based on the results of the screening test, proceed as follows:

1. If all screened units appear the same, combine screened units from all containers and form a composite as described above for single containers;

2. If all screened units do not appear the same, each container should be treated as a separate exhibit or entity. Thus for each container, proceed according to the direction above for a single container.

B. Extraction techniques

1. For presumptive testing or the qualitative analysis of LSD using chromatographic procedures, simple extraction of the LSD from its matrix with methanol will often suffice.

METHOD

Mix the test sample for 30 seconds with a small amount of methanol sufficient to obtain a solution of approximately 1 microgramme LSD in 1 ml. After filtration, the extract can be used directly.

2. For quantitative determinations or where impurities are present which may interfere with the simplified methanol extraction method, the following procedure is recommended:

METHOD

Dissolve or suspend the representative sample in 15 ml of 1% tartaric acid solution in a separatory funnel. Extract three times with an equal volume of chloroform and discard the chloroform layers. Make the aqueous layer basic with 1N sodium bicarbonate and extract the LSD base three times with 15 ml chloroform. Combine these chloroform extracts and filter through glass wool. Bring the filtered extract to a suitable, known volume either through dilution or evaporation under a stream of nitrogen.

3. For those instances where LSD must be separated from iso-LSD, the following reference is provided:

J. Assoc. Off. Anal. Chem., 50, 1967, pp. 1362-1366.

C. Presumptive tests

It must be stressed that positive results to the tests contained in this Section are only presumptive indications of the possible presence of LSD. All ergot alkaloids, many of which are legitimate pharmaceutical products and not subject to national or international control, will give similar results with these tests. Additionally, components of the wide variety of matrices in which LSD is incorporated may also result in the sample giving false positives or negatives. It is therefore mandatory for analysts to confirm such results by the use of alternative techniques.

1. Fluorescence

METHOD

Observe the original sample dosage form under long wavelength UV light. Alternatively, place a drop of the methanol extract described in Section B onto filter paper and allow to dry. Observe the spot under long wavelength UV light. In both cases the presence of LSD is indicated by a blue fluorescence. The detection limit of this method is less than 1 ug.

2. Colour test

Ehrlich Reagent

Dissolve 1 g of para-dimethylamine benzaldehyde in 10 ml methanol, then add 10 ml of concentrated orthophosphoric acid (S.G. ca. 1.75).

METHOD

Place a small amount of the sample or two drops of a methanol extract of the sample in a depression of a spot plate and add two drops of Ehrlich reagent. A blue to purple colour indicates the presence of LSD. The detection limit of this test is approximately 1 ug.

3. Crystal tests

The use of crystal tests is not recommended for the presumptive identification of LSD.

D. Thin layer chromatography

PLATES

Activated silica gel G on glass backed plates; the coating (0.25 mm thickness) contains an additive which fluoresces at 254 nm.

DEVELOPING SOLVENTS

SYSTEM A:	Chloroform	90
	Methanol	10
SYSTEM B:	Chloroform	20
	Acetone	80

Preparation of solutions to be applied to the TLC plates

Sample: Extract the material using either of the methods outlined in Section IV B and prepare a solution containing the equivalent of approximately 1 mg/ml

Standard solutions: All made at a concentration of 1 mg/ml in methanol.

Apply 2 to 3 ul of the sample and standard solutions to the plate.

VISUALIZATION

Air-dry the plates prior to visualization at room temperature.

Spray reagent

Ehrlich reagent: Prepare as in Section IV C 2, above.

METHODS

Observe the plate under UV light at 254 nm. LSD will absorb the light and appear as a dark spot on the fluorescent background. Then observe the plate under UV light at 365 nm. A fluorescent spot on a dark background is given by LSD. Finally, spray the plate with Ehrlich reagent. LSD gives a blue-purple colour with this reagent.

RESULTS

R_f x 100 values:

Compound	Developing System	
	A	B
Lysergic acid	0	0
Ergometrine	10	14
Ergotamine	39	67
LSD	48	60
Ergocristine	62	84
Ergocornine	62	84

Reference: J. Chromatogr. Sci., 12, 1974, pp. 265-266.

REMARK

In addition to the results reported above for ergot alkaloids, system A has been shown to separate LSD from its analogs with the exception of the methyl-propyl analog (J. Assoc. Off. Anal. Chem. 56, 1973, pp. 88-99).

E. Gas liquid chromatography

1. Packed column technique

(a) Without derivatization

The use of the packed column technique for the analysis of underivatized LSD is not recommended.

(b) With derivatization

Operating conditions:	
Detector:	FID
Column:	3 ft, 2.4 mm ID glass
Packing:	3% SE-30 on 80-100 mesh Chromosorb W
Carrier gas:	Nitrogen at 30 ml/min
Column temperature:	250°C
Injector/detector temperature:	275°C
Internal standard:	n-alkanes in chloroform
Derivatizing agent:	N,O-bis-trimethylsilylacetamide (BSA)

METHOD

The standard solution of LSD is prepared to a concentration of approximately 1 mg/ml thusly: the LSD standard is dissolved in a minimum volume of methanol to which 1 ml of the internal standard solution is added. Dilute the mixture to the appropriate volume with chloroform such that the final concentration of the internal standard should be approximately equal to that of the LSD.

Add internal standard solution to a portion of the sample extract described in IV B above. The concentrations of LSD and internal standard should be approximately equal to that of the standard solution.

Evaporate 0.5 ml of the standard solution to dryness under nitrogen in a stoppered tube. Add 0.5 ml of the derivatizing agent and heat at 100°C for 10 minutes. Treat 0.5 ml of the illicit LSD solution in the same manner.

Inject 1-2 ul into the gas chromatograph.

The content (%) of any component can be calculated using the general formula:

$$C_x\% = \frac{C_{r.\ std.}}{C_{sam.}} \times \frac{A_x / A_{int.std.\ in\ sam.\ chrom.}}{A_{r.st.}/A_{int.std.in\ std.\ chrom.}} \times 100$$

Where:

$C_x\%$ = content of component x in the sample (w/w %).

C_r std. = concentration of substance x in the standard reference solution (w/w %).

A_x = peak area for substance x during the sample chromatography.

$A_{int.\ std.\ in\ sam.\ chrom.}$ = peak area of the internal standard obtained during the sample chromatography.

$A_{int.\ std.\ in\ std.\ chrom.}$ = area of the internal standard obtained during the standard chromatography.

$C_{sam.}$ = concentration of the sample (w/v %).

Reference: J. Assoc. Off. Anal. Chem., Vol. 56 (No.1), 1973, pp. 88-99.

2. Capillary column technique

Operating conditions:	
Detector:	FID
Column:	BP1 fused silica
Film thickness:	0.25 um
Length	25 m, 0.22 mm ID
Carrier gas:	Nitrogen at 1 ml/min
Split ratio	20:1
Column temperature:	275°C
Injector/detector temperature:	300°C/325°C
Internal standard:	n-alkanes

METHOD

Prepare drug standard solutions and unknown sample solutions at a concentration of 1 mg/ml as described above. Inject 1 ul of the solutions into the gas chromatograph.

RESULTS

Compound	Retention Indices a/
Ergotamine	2410
Iso-lysergic acid	2947
Dihydroergotamine	2953
Ergometrine	2999
LSD	3130
N-methyl-N-propyl lysergamide	3175
Methylsergide	3300

a/ These values will vary depending upon laboratory conditions and other instrumental parameters.

Reference: J. Forensic Sci. 32, 1987, pp. 933-940.

For alternative GLC methods see:

J. Chromatogr. Sci., 12, 1974, 265-266.
J. Forensic Sci., 29, 1984, 291-298.

F. High performance liquid chromatography

1. Normal phase

Column:	125 mm by 4.9 mm ID.
Packing material:	Silica HPLC grade, 5 um diameter (Spherisorb S5W or equivalent).
Mobile phase:	A solution containing 1.17 g (0.01M) of ammonium perchlorate in 1000 ml of methanol. Adjust to pH 6.7 by adding 0.1M sodium hydroxide in methanol (ca. 1 ml).
Flow rate:	2.0 ml/min.
Detection:	UV at 313 nm; or Fluorescence, excitation at 308 nm, emission of 370-700 nm. (Fluorescence detection provides better selectivity and sensitivity than UV detection although for most forensic work UV detection is satisfactory).
Sample and standard solutions:	All materials are dissolved in methanol to give an approximate concentration of 1 mg/ml.
Injection volume:	1 - 5 ul by syringe or loop-injector.
Quantitation:	By peak area, external standard method.
Reference:	J. Chromatogr., 323, 1985, 191-225.

2. Reverse phase

METHOD 1

Column:	10 cm by 4.6 mm ID.
Packing material:	Octadecyl-silica HPLC grade 5 um (Spherisorb 5-ODS or equivalent).
Mobile phase:	A solution of 65% methanol and 35% 0.025M disodium hydrogen phosphate in water adjusted to pH 8.0 with 10% orthophosphoric acid.

Flow rate:	1.0 ml/min.
Detection:	UV at 280 nm; or Fluorescence: excitation at 320 nm, emission at 400 nm.
Sample and standard solutions	All materials are dissolved in methanol to give an approximate concentration of 0.5 to 1.0 mg per ml.
Injection volume:	1 - 5 ul by syringe or loop injector.
Quantitation:	By peak area, external standard method.
Reference:	J. Chromatogr., 150, 1978, pp. 73-84.

METHOD 2

Column:	30 cm by 1 mm ID.
Packing material:	Octadecyl-silica HPLC grade 10 um (LiChrosorb RP-18 or equivalent)
Mobile phase:	Water solution containing 1 g/l $(NH_4)_2CO_3$ 1 Acetonitril - Methanol 25 : 75 1
Flow rate:	2.0 ml/min.
Detection:	UV at 313 nm
Sample preparation:	A portion of the extract described in Section IV B equivalent to approximately 1 mg LSD is evaporated to dryness under nitrogen. The residue is dissolved in 1 ml of an internal standard solution containing 10 mg benzocaine in 100 ml acetonitrile.
Standard solutions:	Dissolve a sufficient amount of LSD standard in an internal solution containing 10 mg benzocaine in 100 ml acetonitrile to give a concentration of approximately 1 mg per ml.
Injection volume:	10 ul by syringe or loop injector
Quantitation:	Internal standard method using the equation provided in Section IV E.
Reference:	Arch. Krim. 164, 1979, pp. 25-30 (modified)

RESULTS

The capacity ratios (K'values) or retention times (relative to LSD) are as follows:

Compound	NORMAL PHASE a/	REVERSE PHASE a/ 1	2
LSD	0.7	1.00	1.00
d-Lysergamide	0.31	0.38	-
d-Lysergic acid	0.8	0.23	0.42
d-Lysergic acid monoethylamide	--*	0.52	
Ergocornine	0.4	1.39	
Ergocristine	0.25	2.31	
Ergocryptine	0.26	1.86	
Ergometrine	0.26	0.26	
Ergosine	0.25	1.22	
Ergotamine	0.29	1.57	
Benzocaine (internal standard)	--	--	0.74

* Not determined

a/ These values will vary depending upon laboratory conditions and other instrumental parameters.

For alternative HPLC systems see:

J. Forensic Sci. Soc., 19, 1979, p. 253.
J. Liquid Chromatogr., 7, 1981, pp. 357-374.
J. Forensic Sci., 32, 1987, pp. 933-940.

G. Spectroscopic techniques

In some countries, confirmation of identity by spectroscopic means is required. Theoretically, each substance has a unique infrared spectrum and this method would permit the unequivocal identification of LSD. With few exceptions, mass spectroscopy will also provide such identification. Because the LSD in samples received by forensic laboratories is invariably only a very small portion of the sample, it must be separated and isolated in a pure form prior to spectroscopic analysis. The extraction procedure outlined in Section IV B above is generally suitable for liberating the LSD from the rest of the material.

The following sections provide references dealing with spectroscopic techniques for those laboratories which require such confirmation.

A. UV/Fluorescence

Because other ergot alkaloids and LSD analogs yield similar results, these methods are not specific for the analysis of LSD and therefore are not recommended.

B. Infrared Spectroscopy

For a description of the standard methods (halide disk, microhalide, nujol mull and thin-film techniques) see previous manuals in the series.

References:

1. J. Assoc. Off. Anal. Chem., 50, 1967, pp. 1362-1366.
2. J. Assoc. Off. Anal. Chem., 56, 1973, pp. 88-99.
3. Bull. Narc., 19, 1967, pp. 39-45.

C. Mass Spectroscopy

References:

1. J. Assoc. Off. Anal. Chem., 51, 1968, pp. 164-175.
2. J. Assoc. Off. Anal. Chem., 56, 1973, pp. 88-99.