THE APPLICATION OF CIRCULAR DICHROISM IN THE IDENTIFICATION OF AMPHETAMINE AND TRYPTAMINE AND THEIR RELATED COMPOUNDS

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THE APPLICATION OF CIRCULAR DICHROISM IN THE IDENTIFICATION OF AMPHETAMINE AND TRYPTAMINE AND THEIR RELATED COMPOUNDS

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PREFACE

The main objective is to develop a system using circular dichroism for the identification of members of the amphetamine and tryptamine families . A variety of solvents were used in order to optimize the spectra in the ultraviolet region. Two main types of solvents: isotropic and anisotropic will be employed and comparisons of their spectra will be made.

The author wishes to express her deepest gratitude to her research advisor and friend, Dr. Neil Purdie, for his understanding and patience. Appreciation is also expressed to my committee members, Dr. Paul Devlin and Dr. Harry Gearhart.

A special thanks to John Bowen, who developed the early stages of some of the techniques that were used in my research project. I would like to extend my gratitude to Dolores Behrens for the excellence and speed with which she typed the manuscript.

I would especially like to thank my parents, Robert and Nancy Head, who encouraged me to persevere and have given me their full support and love the last twenty-four years.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER I

INTRODUCTION

Drug abuse is not a new problem in today's society. However, the awareness of the average individual to the effects of the misuse of drugs is more prevalent. This awareness has created a revolution in the analytical search for economical and specific detection systems. Clinical and forensic laboratories face the everyday problem of isolating and identifying the presence of prescribed and illicit drugs for a wide variety of reasons.

Modern technology allows the analyst to choose from numerous techniques that are effective for identification of relatively pure substances. Some of the methods are: thin layer chromotography (TLC), gas-liquid chromotography (GLC), spectrofluorimetry, X-ray diffraction, isotope-labelling, radio immuno-assay, qualitative and quantitative color tests, ultraviolet, visible, infrared, mass, and atomic absorption spectrophotometry.[1-3] None of these methods can stand alone as the single technique capable of unambiguous recognition of an anonymous sample but they do tend to complement each other to insure a more positive identification.

One of the most useful identification methods has been the absorption of ultraviolet light. This technique can be satisfactorily used to identify particular groups of compounds whose members have a common and unique molecular structure. Distinction among members of the same

group is a more difficult problem. Illustrative of the two levels of distinction is the fact that amphetamine derivatives can be distinguished from tryptamine derivatives but amphetamine is not readily distinguishable from methamphetamine, nor tryptamine from LSD. To improve the latter distinction, spectra are modified by changing solvent or, in an aqueous media, by changing the pH.

Although many of these compounds are chiral, the optically active properties of drug molecules have not been exploited in analysis, apart from the simple measurement of the angle of rotation of linearly polarized light at the wavelength of the Na-D line. In this work we intend to explore the potential of using polarized light to modify the ultraviolet spectra of both optically active and inactive drug molecules for their easier recognition in analysis. In particular we are interested in the amphetamine and tryptamine groups.

Linearly or circularly polarized light could be used for this purpose. We have chosen the latter. The technique is referred to as circular dichroism (CD) spectropolarimetry. CD spectra contain more parameters than those found in conventional spectrophotometry. In the event that additional parameters are still needed, the spectra will be further modified by changing solvents or solvent conditions. Included in the exploratory search are isotropic solvents such as ethanol and water (acidic and basic) and an anisotropic solvent which is a cholesteric liquid crystal mixed solvent system.

CHAPTER II

CIRCULAR DICHROISM

In order to understand completely the work that has been accomplished, a short summary of the basic concepts of circular dichroism is appropriate. Circular dichroism (CD) can be defined as an electromagnetic measurement using polarized light.

Production of Circularly Polarized Light

Unpolarized light exists as an infinite number of oscillating electrical and magnetic fields which are orthogonal to each other and which propagate in all directions. Certain crystals and films have the property of polarizing the electrical vector of light in one preferred direction. This direction depends upon the nature and orientation of the polarizong element. The single vector is called plane (linearly) polarized light which moves in a sinusoidal manner with time.

Plane polarized light[4-5] actually consists of two circularly polarized components, Figure 1, which move in phase with each other but in opposite directions. An optical device called a quarter-wave retarder can be used to divide the beam of plane polarized light into its circular component vectors. As will be seen in the next section, the effect on these two circularly polarized components by the transmitting medium produces the phenomena known as optical rotatory dispersion and circular dichroism from which it is possible to obtain

structural information about the medium.

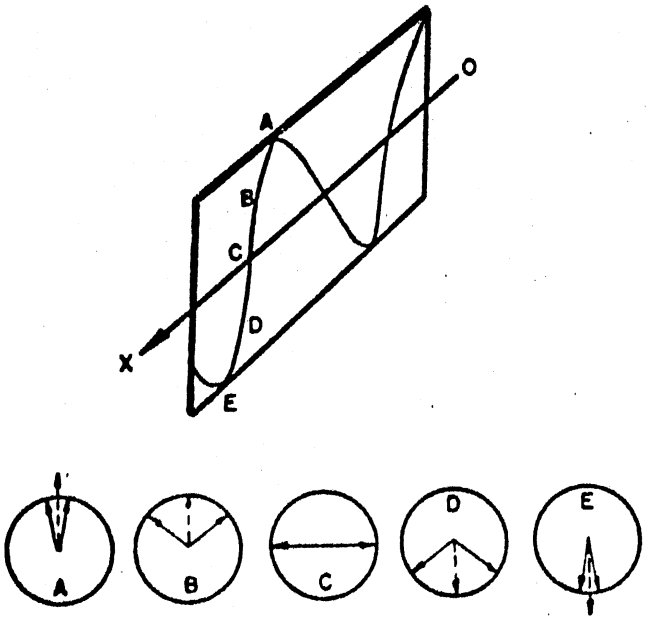

Figure 1. Production of Circularly Polarized Light

Origin of Circular Dichroism

In order to understand the origin of circular dichroism, one must first understand the meaning of an associated phenomenon known as Optical Rotatory Dispersion (ORD).[6-17] When an optically active medium (e.g. having a chiral center) is placed in the path of a plane polarized light beam, the circular components are altered relative to each other in terms of speed of propagation, which causes the two vectors to become out of phase. The two different speeds arise because the circular components experience two different refractive indices [$n_L \neq n_R$].

The phenomenon is known as circular birefringence. The linear resultant of the circularly polarized light beam when transmitted through the medium, is rotated by an angle, α, from its original direction.

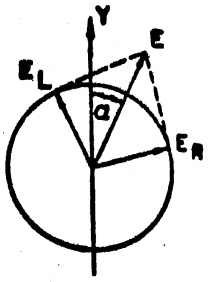

Figure 2. Optical Rotatory Dispersion

The degree of rotation can be measured using a simple polarimeter. In this instrument, a second polarizing element known as the analyzer is rotated until the plane polarized light beam is transmitted and detected visually. The polarimeter is usually operated at the wavelength of the Na-D line only and a single α_D value is obtained which is sensitive to the solvent medium, solution concentration, and temperature. For comparison among compounds, the values of α are usually normalized to one molar concentration. Optical rotation is obviously severely restricted in application because only one parameter can be used for identification, namely α_D.

In ORD, values for the specific rotation, $[\alpha]$, are measured throughout the ultraviolet and visible spectral region. A normalized ORD

spectrum is defined in terms of a molecular rotation which for solutions is given by the equation;

$$\text{molecular rotation: } [\phi] = \frac{[\alpha]MW}{100} \tag{1}$$

where

$$[\alpha] : \text{specific rotation} = \frac{\alpha}{\ell \cdot c} \tag{2}$$

α = angle of rotation of the plane of polarized light, degrees

ℓ = cell length, dm

c = concentration, g/ml

By measuring α at many wavelengths, the identification procedures are improved.

For a non-absorbing, but optically active, medium the molecular rotation is observed to increase in magnitude either positively or negatively as the wavelength is decreased and the resultant spectrum is referred to as a Plain ORD curve, Figure 3. If the medium absorbs energy in this region, an abnormal ORD spectrum is observed. Instead of a gradual increase in $[\phi]$, the values increase quickly to a sharp maximum (or minimum depending on the sign of rotation) which precedes a rapid change in direction over the next few wavelengths through a zero cross-over value to a minimum or trough. This behavior is typical of a single uncomplicated absorption process, and is referred to as a cotton effect. When the trough occurs at a shorter wavelength than the peak the cotton effect is negative, and vice versa. In more complicated

molecules where absorptions overlap, the entire inflection may not be observed. Components in broad absorption bands are sometimes separated by their opposite Cotton effects.

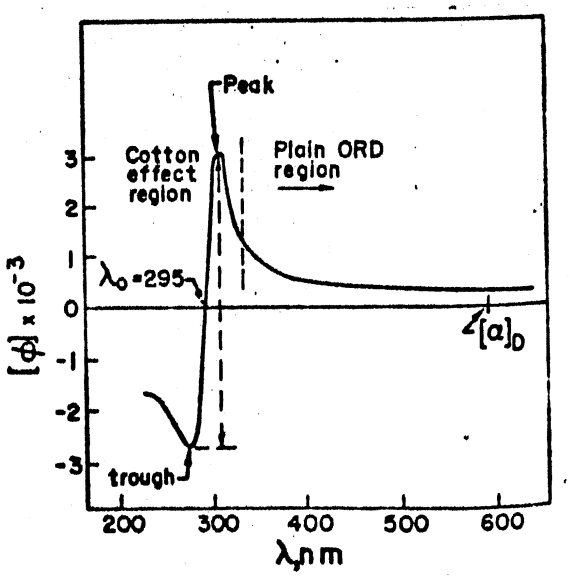

Figure 3. Typical ORD Curve

Besides creating an unusual change in molecular rotation with wavelength, an optically active medium might also absorb the circularly polarized components to different extents which would produce vectors of unequal length. Both components continue to move in a circular fashion and are out of phase, but the perimeter of their resultant vector sum is now an ellipse, Figure 4. The transmitted beam is referred to as elliptically polarized light.

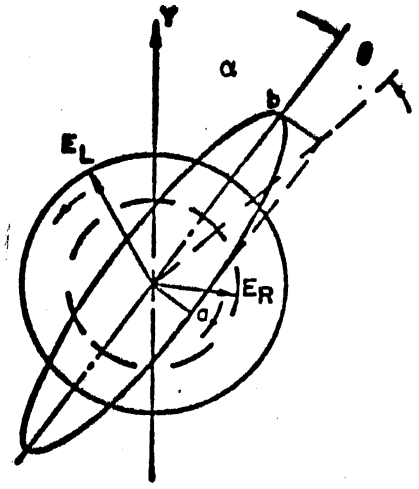

Figure 4. Elliptically Polarized Light

In Figure 4, α is the rotation of the major axis and θ is the ellipticity or the arctangent of the ratio of the minor to major axis. When the ellipticity is measured as a function of wavelength, the differences in absorbance have effectively been measured. This is the phenomenon of circular dichroism[6-17] which is defined as the difference in the molar absorptivity of the left- and right-hand components of circularly polarized light. CD can be expressed in terms of molecular ellipticity.

Molecular Ellipticity: $[\theta] = 3305\ (\varepsilon_L - \varepsilon_R)$ (3)

ε = molar extinction coefficient

$$[\theta] = \frac{\theta \cdot M}{100 \cdot \ell \cdot c} \qquad (4)$$

θ = angle of ellipticity, degrees

M = molecular weight

ℓ = cell length, dm

c = concentration, g/ml

It is apparent from Eq. (3) that a CD spectrum exhibits a Cotton effect only in the wavelength range of an absorption band, Figure 5. Multiple Cotton effects are common for CD as well as for ORD. The two phenomena are related to each other mathematically by the Kronig-Kramers equation.[9] Whereas ε_L and ε_R are either zero, or positive, their difference can be negative, zero, or positive. There is an added dimension to a CD spectrum compared to a conventional absorption spectrum in this change of sign. For a time CD was commonly used in the assignment of absorption bands to specific electronic transitions, to the study of molecular configurations, and most recently to analytical studies.

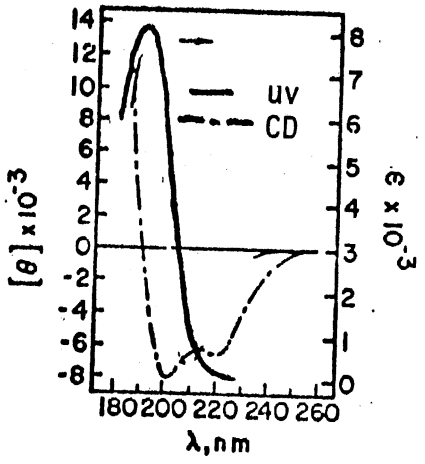

Figure 5. Typical CD Curve

CHAPTER III

SOLVENT SYSTEMS

Isotropic and Anisotropic Solvents

The solvents used in this technique can be divided into two general classes: isotropic and anisotropic. A solvent is said to be isotropic when its physical properties are the same in all directions. Water, ethanol, acids and base are prime examples of isotropic media. If the physical properties of a substance differ in different directions, the substance is anisotropic. Liquid crystals fall under the class of anisotropic solvents as do some single-crystals.

Liquid Crystals

A definition of liquid crystals[18-25] is somewhat of a contradiction in that it is a solid that behaves as a liquid as well as a liquid which resembles a solid. Generally, the term "liquid crystal" refers to the intermediate stages or mesophases between a solid and liquid. Over 5% of all organic compounds are capable of forming these thermodynamically metastable mesophases, which are more or less fluid yet have a long-range order. Combining liquid and solid properties in one phase produces a unique set of physical properties among which are temperature dependent and solvent sensitive color changes.

In 1888, an Austrian botanist, Freidrich Reinitzer,[25] prepared the compound cholesteryl benzoate. Upon heating the solid, he observed

it to melt to a turbid liquid at 146.6°C (liquid crystal) which became the anticipated clear liquid at 180.6°C. Lehmann[18] was the first to refer to the new intermediate stage as a liquid crystal. The French scientist, Friedel,[25] in 1922, studied the birefrigent* patterns created by the interaction of polarized light with the mesophases. He classified three types of temperature sensitive or thermotropic liquid crystals: nematic, cholesteric, smectic.

During the 1920-1930 period, many of the physical properties (e.g. viscosity reflectivity, dielectric properties) of liquid crystals were investigated. Only with the advent of more sophisticated experimental tools have the complex molecular structures been amenable to study. With this new knowledge, chemists, physicists, engineers, biologists and medical scientist now use liquid crystals and exploit their unique properties in many areas of interdisciplinary technology and research.

At present liquid crystals are divided into two categories: lyotropic and thermotropic. Lyotropic liquid crystals are composed of two or more components one of which is an amphiphilic molecule and the other a polar solvent such as water. They occur widely in nature, particularly in living systems. Thermotropic liquid crystals are temperature controlled. Thermotropic liquid crystals can be adequately divided into two major categories: smectic and nematic, from a structural viewpoint. The third category defined by Friedel, cholesteric liquid crystals, turns out to be no more than a twisted nematic mesophase. An interesting property of both thermotropic and lyotropic liquid crystals is that

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*Birefringent: different speeds due to the inequality between refractive index of left- and right-hand circularly polarized light.

both exhibit polymorphism, i.e. more than one kind of liquid crystalline phase exists.

Smectic liquid crystals have a two-dimensional structure as seen in Figure 6. The molecules have a high degree of order creating high viscosity and a high surface tension smectic phase. Smectic liquid crystal behave optically as uniaxial crystals. The less organized nematic mesophases are arranged with their long axes parallel while being free to move relative to one another in the direction of one of their axes. (Figure 6a).

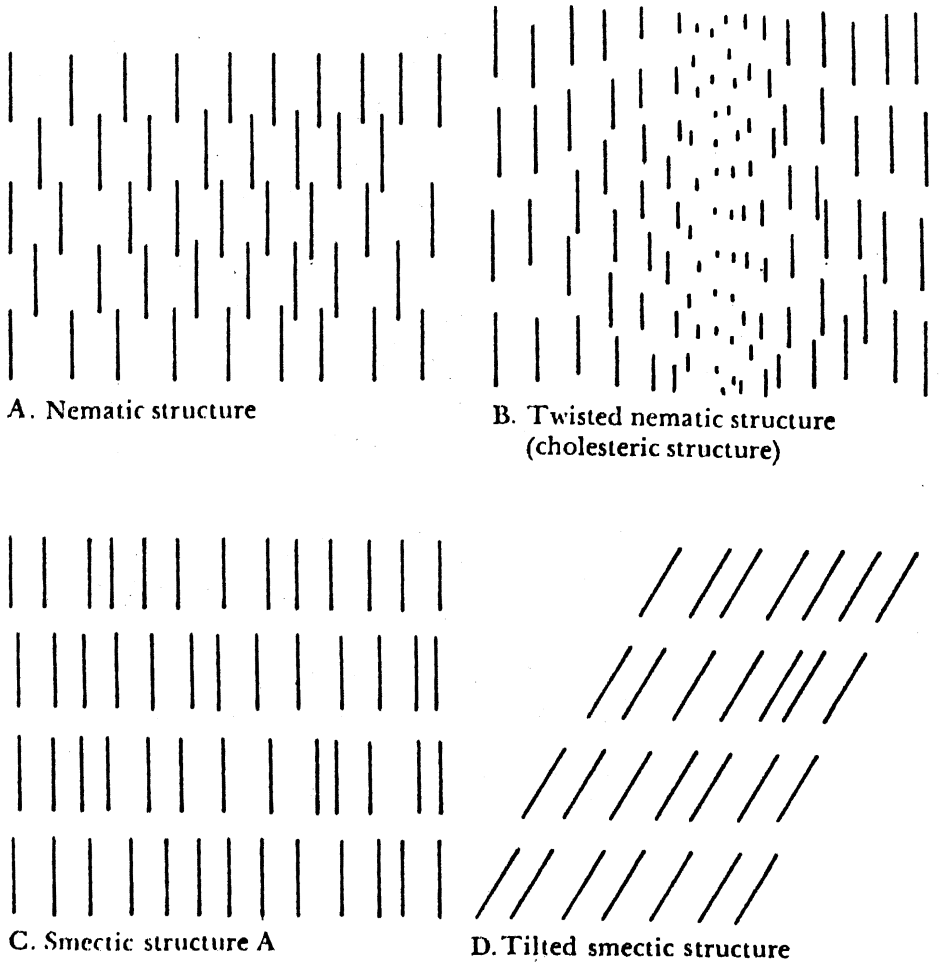
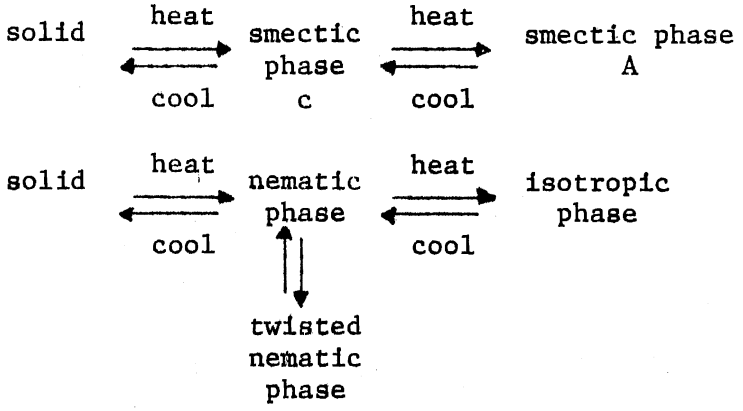


Figure 6. Types of Thermotropic Liquid Crystals

A good illustration of a nematic liquid crystal can be represented by pencils in a box. They can roll around each other but they remain parallel to each other. This spontaneous order of molecules leads to a fluid less fiscous than smectic. The preferred direction of the long axes usually varies from point to point throughout the medium but a homogeneously aligned species is optically uniaxial and strongly birefringent. This optical tendency gives rise to the colors exhibited by both smectic and nematic liquid crystals under the polarizing microscope.

A type of organized nematic crystal derived from esters of cholesterol are known as cholesteric liquid crystals. The change from one phase to another is a thermodynamically first-order and reversible process for all thermotropic liquid crystals.



There is no change in entropy between the nematic and the twisted nematic (cholesteric) phase. The structure of cholesteric liquid crystals, Figure 6B "consists of a nematic structure on which is superimposed a screw axis normal to the preferred direction."[20]

Chirality in the molecules causes a change in the molecular orientation

and a macrohelicoidal structure is produced, which can be either left- or right-handed, Figure 7. These molecules need not be derivatives of cholesterol but can be steroids or organic compounds which are capable of exhibiting the twisted nematic mesophase.

The following properties of these cholesteric mesophases are important in their use as an anisotropic* solvent.

1. Cholesteric mesophases have a so-called pitch which is the distance between two planes of identical molecular alignment.
2. The pitch of a cholesteric liquid is strongly thermally dependent.
3. The true melting point of all mixtures of components with opposite twisting sense lies well above room temperature making them easily used in the metastable state.
4. Cholesteric mesophases can become induced nematic mesophase by strong electric or magnetic fields.

Liquid Crystals as Solvents

Only thermotropic liquid crystals have been used as solvents and of these only nematic[26-28] and cholesteric[29-32] compounds. Both exhibit anisotropic behavior which is a valuable modification to have in a solvent, but solubility is limited to non-polar solutes. In some instances the solute at a sufficiently high mole fraction can alter

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*Anisotropic species are usually more highly ordered than the structurally-random isotropic species having preferred directions upon which physical properties depend on the direction in which they are measured.

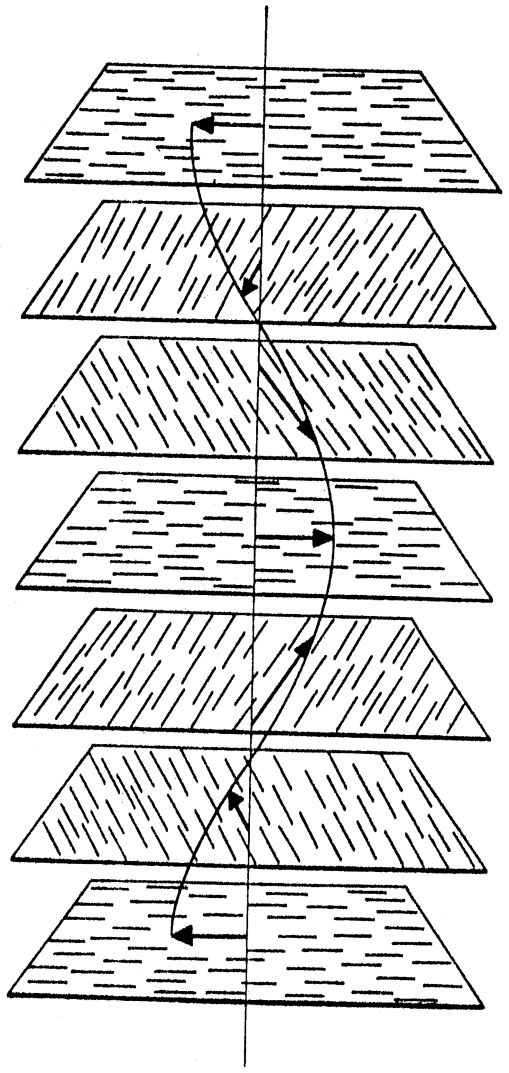


Figure 7. Schematic View of a Cholesteric Liquid Crystal

the structural properties of the solvent causing it to collapse to isotropic or to convert from one form to another. The helicoidal pitch in cholesterics is often changed by adding solutes.

In studies which involve polarized light, the nematics are best disposed to investigation using linearly polarized light. The cholesterics interact differently with right and left circularly polarized light making them better suited to CD studies. Whatever the solvent system, a necessary prerequisite is its transparency to visible-ultraviolet light.

One unique property of the cholesteric solvent media is their ability to arrange solute molecules in a helicoidal pattern. On illuminating the solution with circularly polarized light both ORD and CD spectra of the solute can be obtained, whether the solute is chiral or not. Where the solute is achiral, the phenomenon has been referred to as Liquid Crystal Induced Circular Dichroism (LCICD). Where the solute is inherently chiral, reference is made to the phenomenon as chiral amplification. The two effects should not be considered to be mutually exclusive for there is evidence for LCICD in chiral systems.

CHAPTER IV

ULTRAVIOLET SPECTROSCOPY

Amphetamines and tryptamines have as their parent compound, benzene. In the ultraviolet region, benzene is known to possess three electronic transitions[33] which correspond to three absorption bands as described in Table I. When the parent compound is substituted, the intensity changes and a shift in the wavelengths of the absorption bands occur.[34-35]

TABLE I

ULTRAVIOLET TRANSITIONS OF BENZENE

Transition	Symmetry Assignment	λ(nm)	Intensity
$\pi \longrightarrow \pi^*$	^1L_B	260	weak
$\pi \longrightarrow \pi^*$	^1L_A	202	strong
$\pi \longrightarrow \pi^*$	$^1B_{a,b}$	185	very strong

The amphetamine class of drugs[36-37] consists of monoalkylamino-benzene derivatives, Figure 8, with saturated bonds extending at least two carbon atoms from the point of ring attachment. Three types of transitions appear: $\pi \longrightarrow \pi^*$ at 257 nm which is the ^1L_B or "benzenoid band" and $\pi \longrightarrow \pi^*$ at 205 nm for the ^1L_A transition. The maximum absorption peak occurs below 200 nm due to the $^1B_{a,b}$ $\pi \longrightarrow \pi^*$ transition. However, we are only concerned about the wavelength region above 220 nm and below 350 nm. In this region the maximum absorption peak at 257 nm contains several shoulders which are lower in intensity.

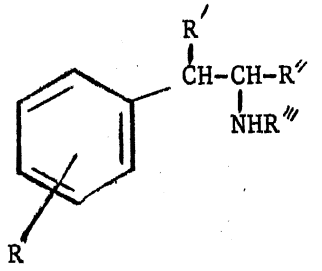


Figure 8. General Structure of the Amphetamine Class

The members of the tryptamine family are all derivatives of indole, Figure 9. Again the three transitions are observed as in benzene. A large maximum occurs below 200 nm which is of no interest to our study. A strong ^1L_A transition has been extended to a wavelength region at 216-226 nm. A much weaker band, the ^1L_B occurs near 280 nm and is known to increase in fine structure in less polar solvents.

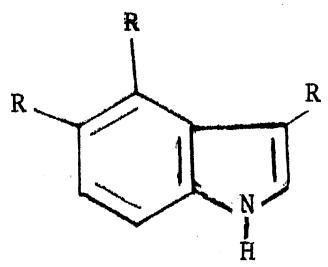


Figure 9. General Structure of Tryptamine Family

In Figure 10, a typical absorption spectrum[38-39] for a member of the amphetamine and a member of the tryptamine groups is represented. The dotted line in each spectrum trace the absorption under decinormal acidic conditions and the solid line represents absorptions under decinormal basic conditions. Both compounds absorb off scale at the lower end of the spectrum in the acidic medium. They are shown again at a second more dilute concentration to demonstrate all of the bands above 200 nm. Both spectra resemble each other in shape, but their absorption bands for the ^1L_B transition occur at different wavelengths allowing distinction to be easily obtainable between the two classes: amphetamines and tryptamines.

However, once we move into each family the use of ultraviolet spectroscopy for the identification of various members becomes limited. Modification of the spectra by the application of circular dichroism has been studied for both the amphetamine and tryptamine groups. Gottarelli and Samori[40] studied the solvent effects on the circular dichroism of α-phenethylamines. Smith and coworkers[41-42] have attempted to determine the sign of the ^1L_B Cotton effect of substituted optically

active amines such as norephedrine and phenethylamines and the ^1L_B Cotton effects of 1-substituted indans[43] which are parent compounds of the tryptamines. Several studies on the induced circular dichroism of the 2-benzoylbenzoic acid-amphetamine system[44-46] with regards to various ethers as the solvent were investigated.

Liquid Crystal Induced Circular Dichroism (LCICD) bridges two distinct fields: electromagnetic measurements using polarized light and cholesteric liquid crystals. As of now no work has been published linking the optically active solvent with either the amphetamines or tryptamines. The properties of liquid crystals have been discussed previously as well as the concept of circular dichroism. It is now appropriate to combine the two.

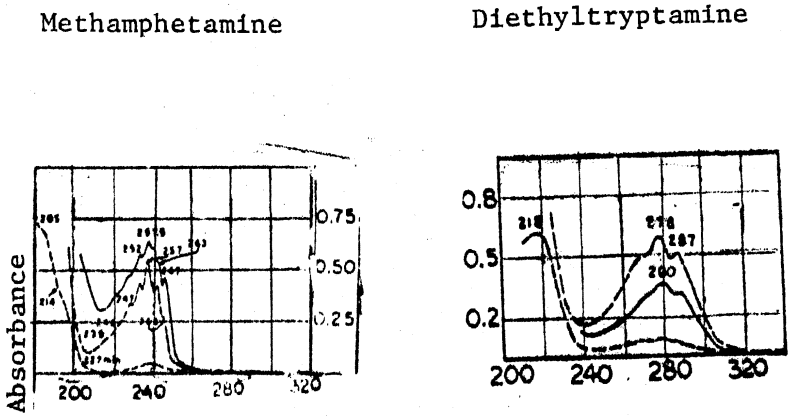


Figure 10. Ultraviolet Spectra of Methamphetamine and Diethyltryptamine

The phenomenon of LCICD was first extensively discussed by Saeva and coworkers[46-52] where an optically inactive molecule demonstrated an ellipticity when dissolved in cholesteric liquid crystals. The signal is primarily due to the cooperative alignment of the solute with the chiral structure of the cholesteric solvents. The magnitude and sign of the LCICD depends upon the solute concentration, molecular symmetry, electronic and magnetic properties as well as the physical characteristics of the liquid-crystalline solvent. Sackmann, et al.[53-56] reported that the circular dichroism observed in a cholesteric solution is caused by the helical arrangement of the solute molecules rather than solute-solvent interactions causing an induced optically active solute.

Many applications of LCICD[57-58] have been developed towards the identification of organic molecules. Snatzke and coworkers[59-60] studied the Cotton effects of substituted benzene derivatives as did Saeva.[61] Many of the concepts and techniques on LCICD were extremely useful. It can be hoped that with further experimentation and understanding that LCICD will become a standard circular dichroism method.

CHAPTER V

EXPERIMENTAL PROCEDURES

Two general classes of compounds were analyzed consisting of drugs and their related compounds. Table II lists the members of the amphetamine and tryptamine groups used in this study.

Three methods were used to modify the ultraviolet spectrum of each compound: circular dichroism in ethanol (ECD), acid/base circular dichroism (ACD)/(BCD), liquid crystal circular dichroism (LCICD). Due to problems of solubility, not all of the compounds were capable of being used in each step.

Preparation of Drug Free Bases from Their Salts

Amphetamine, methamphetamine, and mescaline were obtained as sulfate or hydrochloride salts. In order to eliminate the problem of very low solubility in the liquid crystal solvent, it was necessary to separate the salt from the drug producing the drug in its free base form.

The salts were dissolved in 50.0 ml of H_2O and 200.0 ml of 1.0 M $NaHCO_3$ and the free base drug was extracted three times with 50.0 ml of reagent grade $CHCl_3$. The free bases were collected at the appropriate boiling point by fractional distillation d-amphetamine, d-and dl-methamphetamine, and mescaline were all prepared in this manner and used in the free base form in all of the analyses.

TABLE II

LIST OF COMPOUNDS AND STRUCTURES

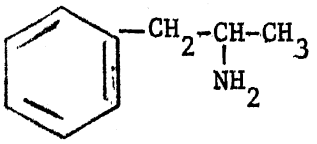
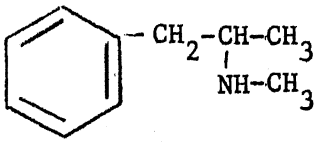
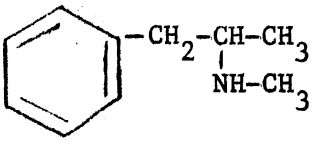
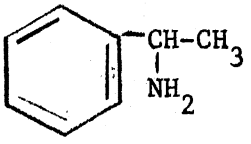
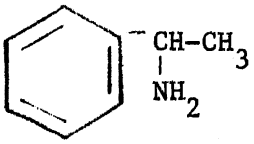
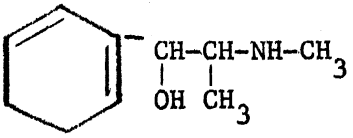
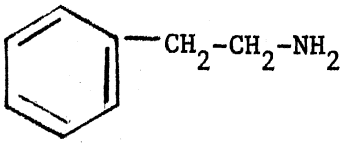
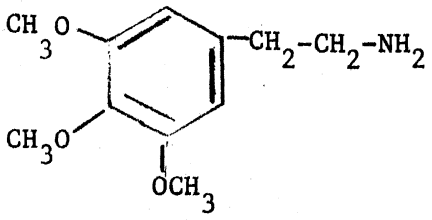
Amphetamine Group		Molecular Weight
d-Amphetamine*		135.21
d-Methamphetamine*		149.24
dl-Methamphetamine*		149.24
(+)-(α)-phenethylamine		121.88
(-)-(α)-phenethylamine		121.88
Ephedrine		165.24

TABLE II (Continued)

Amphetamine Group		Molecular Weight
β-phenethylamine		121.88
Mescaline*		211.25

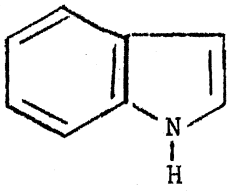
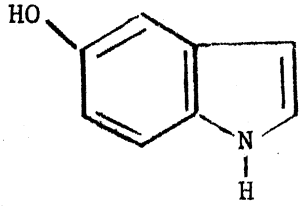
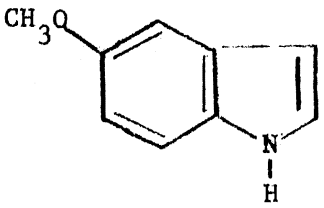
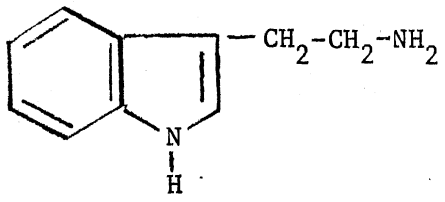
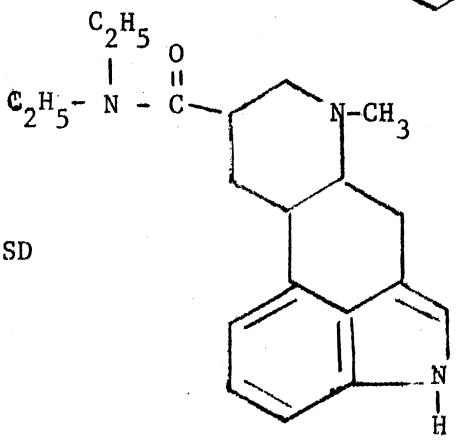
Tryptamine Group		Molecular Weight
Indole		117.14
5-Hydroxy Indole		133.15
5-Methoxy Indole		147.18

TABLE II (Continued)

Tryptamine Group	Molecular Weight
Tryptamine 	160.21
*d-LSD 	323.42

*Controlled Substance

Ultraviolet Absorption Spectroscopy

The absorption spectra for the amphetamine and tryptamine groups were obtained using a Cary 14 spectrophotometer. Each compound was weighed on an analytical balance with an accuracy of $\pm$ 0.0001 g and dissolved in absolute ethanol. Matched 1 cm quartz cells filled with absolute ethanol were used to obtain a baseline in the ultraviolet region from 350 nm to 200 nm. The absorbance spectra of the compounds were obtained with respect to the reference solvent. The molar extinction coefficients, ε, were calculated using the Beer-Lambert equation and, where possible were compared to literature values. Agreement was good in all cases.

Ethanol Circular Dichroism (ECD)

The first step in modifying the ultraviolet spectrum is to use polarized light. A Cary 61 spectropolarimeter, Figure 11, was used to measure the ellipticities of the compounds in ethanol in a 1 cm quartz cell versus absolute ethanol in the reference cell. No CD spectra were obtainable for achiral molecules or racemic mixtures.

Acid Circular Dichroism (ACD) and Base Circular Dichroism (BCD)

Since the ultraviolet absorption spectral data previously reported for the two classes of drugs were of compounds dissolved in acid and base media, a logical modification was to obtain the CD spectra for the compounds in the same aqueous environments. Table III lists the members of the amphetamine family which were selected for the ACD and BCD

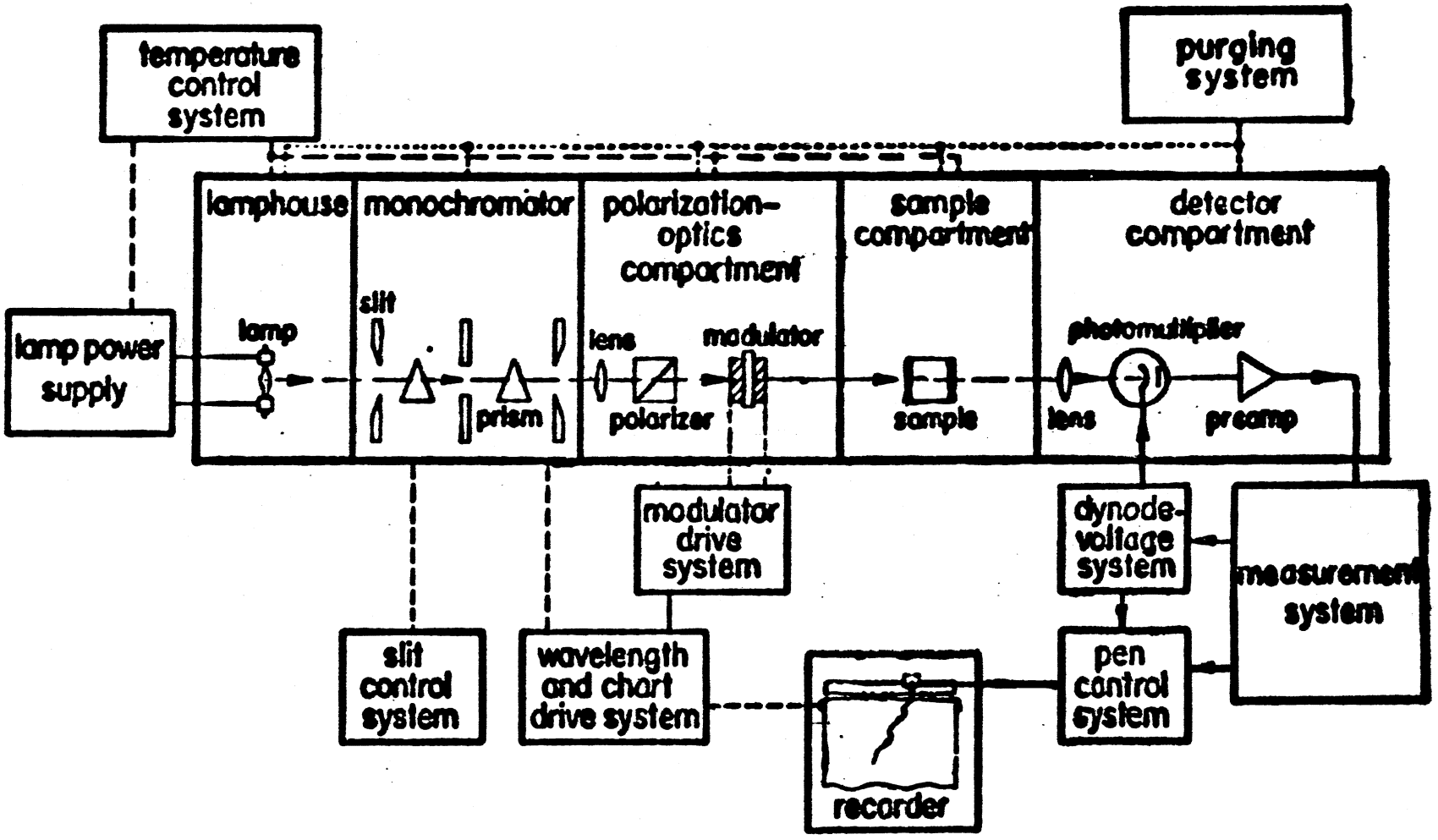


Figure 11. Cary 61 Spectrophotometer

studies based on their ease of solubility in both acid and base.

TABLE III

COMPOUNDS ANALYZED USING ACID CD AND BASE CD

d - Amphetamine
(+)-(α) - phenethylamine
(-)-(α) - phenethylamine
d - Methamphetamine
Ephedrine
d-LSD

Each compound was weighed on the analytical balance to $\pm$ 0.0001 g and dissolved in either 1.0M HCl or in a solution buffered to pH 10. The ellipticity was measured against the reference solvent of acid or base in the wavelength region of 350-230 nm.

Liquid Crystal Sample Preparation

A mixture of two liquid crystal materials with structures of opposite helical sense were used, i.e. cholesteryl nonanoate left-handed helix and cholesteryl chloride-right-handed helix. A mixture which is 38% by weight cholesteryl chloride and 62% by weight cholesteryl nonanoate produces a left-hand helix as the liquid crystal solvent.

A stock solution of liquid crystal is made by dissolving 1.5 g of liquid crystal mixture in 2.0 ml of dichloromethane and 23.0 ml of spectroquality acetone. Each accurately weighed compound of approximately 5.0 mg was dissolved in 5.0 ml of liquid crystal stock solution. The acetone-dichloromethane was removed by slow evaporation and stirring the mixture in a water bath. The process usually takes 10-15 minutes.

The special cell is shown in the exploded diagram, Figure 12. It consists of two metal cell holders, an upper and lower quartz disc, two o-rings, and plastic spacers. Self-adhesive cellophane tape is fastened to one quartz disc and with the center cut out acts as the actual solution container with a pathlength of 1.274×10^{-4} dm.

Each disc is cleaned first with $CHCl_3$ in order to exclude any liquid crystal material from previous samples. Two 100 µl pipets are allowed to warm so that when filled with liquid crystal the temperature will be in excess of the cholesteric-isotropic transition temperature. Each cell is allowed to warm 45-60 seconds on a hot plate to maintain the samples in the isotropic state when added. A sample size of 20 µl is dispensed in the center of the taped cell. The clear quartz disc is placed on top and along with the o-ring and spacer is pressed for 5-10 seconds. The cell lid is tightened completely. This technique must be followed rigorously in order to achieve sample cells with no air pockets or striations due to premature or inhomogeneous cooling of the liquid crystal.

Both cells are filled with liquid crystal and allowed to warm in thermo-jacketted holders in the Cary 61 at a temperature of $40^{o}C$ for five minutes. The baseline is adjusted with both cells filled

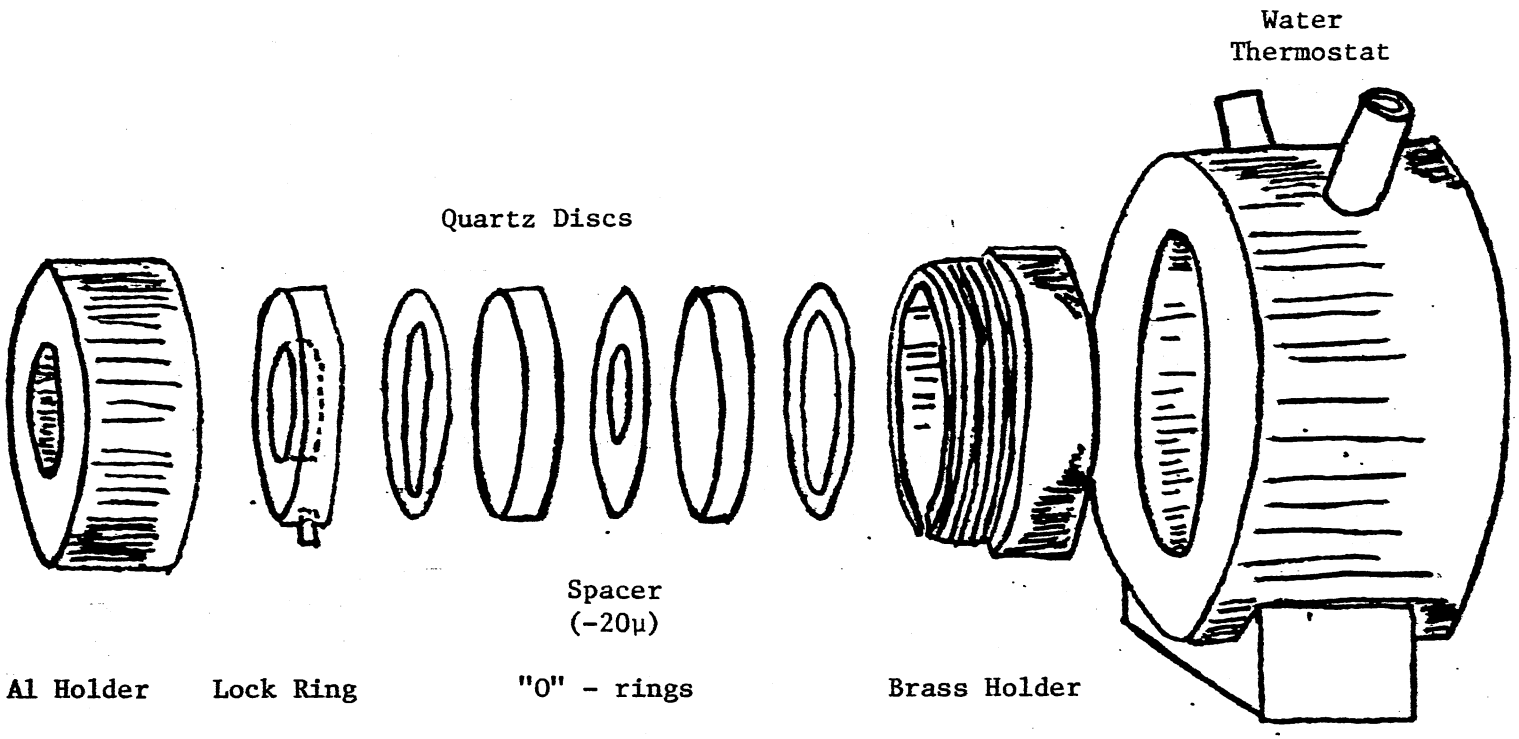


Figure 12. Circular Dichroism Thermostatted Cell

with only the liquid crystal solvent. The sample cell is cleaned as described previously and a 20 µl sample of drug dissolved in the liquid crystal mixture is added and the cell is reassembled. Circular dichroism spectra are obtained in the usual way. Spectra were obtained for all compounds, chiral or achiral.

CHAPTER VI

RESULTS OF DATA

Ultraviolet Spectroscopy

In order to compare the absorption spectra obtained using the Cary 14 spectrophotometer, the data must be normalized, by dividing the experimental absorbances by the molar concentration. Tables IV and V are a collection of the ultraviolet data obtained for the two classes of compounds. Figures 13 and 14 are representative of the normalized spectra.

Ethanol Circular Dichroism, Acid Circular Dichroism and Base Circular Dichroism

A normalization of the ECD, ACD and BCD spectra required that the experimental ellipticities be corrected for a concentration of 1.0 g/ml and a full range of 1.0 units. Five optically active members of the amphetamine family were used in the modification techniques employing three isotropic media: ethanol, acid and base. Tables VI, VII and VIII list the maximum ellipticities. Figures 15 and 16 show the normalized spectra for the optically active compounds belonging to the amphetamine group.

Spectra for the only member of the tryptamine group that is both optically active and soluble in the isotropic media, d-LSD,

TABLE IV

SUMMARY OF ULTRAVIOLET DATA IN ETHANOL AMPHETAMINE GROUP

	Compound	λ(nm)
(A)	d-Amphetamine	268, 264, 258* ($\varepsilon = 18.0 \times 10^4$), 253, 247
(B)	d-Methamphetamine	268, 264, 261, 258* ($\varepsilon = 19.6 \times 10^4$), 253, 248
(C)	dl-Methamphetamine	268, 264, 261, 258, 257* ($\varepsilon = 22.8 \times 10^4$) 253, 248
(D)	β-Phenethylamine	268, 264, 261, 258* ($\varepsilon = 20.4 \times 10^4$), 253, 247
(E)	(+)-(α)-Phenethylamine	264, 258* ($\varepsilon = 18.4 \times 10^4$), 252, 248
(F)	(-)-(α)-Phenethylamine	264, 258* ($\varepsilon = 16.1 \times 10^4$), 253, 247
(G)	Ephedrine	268, 264, 258* ($\varepsilon = 15.5 \times 10^4$), 253
(H)	Mescaline	270* ($\varepsilon = 39.4 \times 10^3$)

*λ^{max}

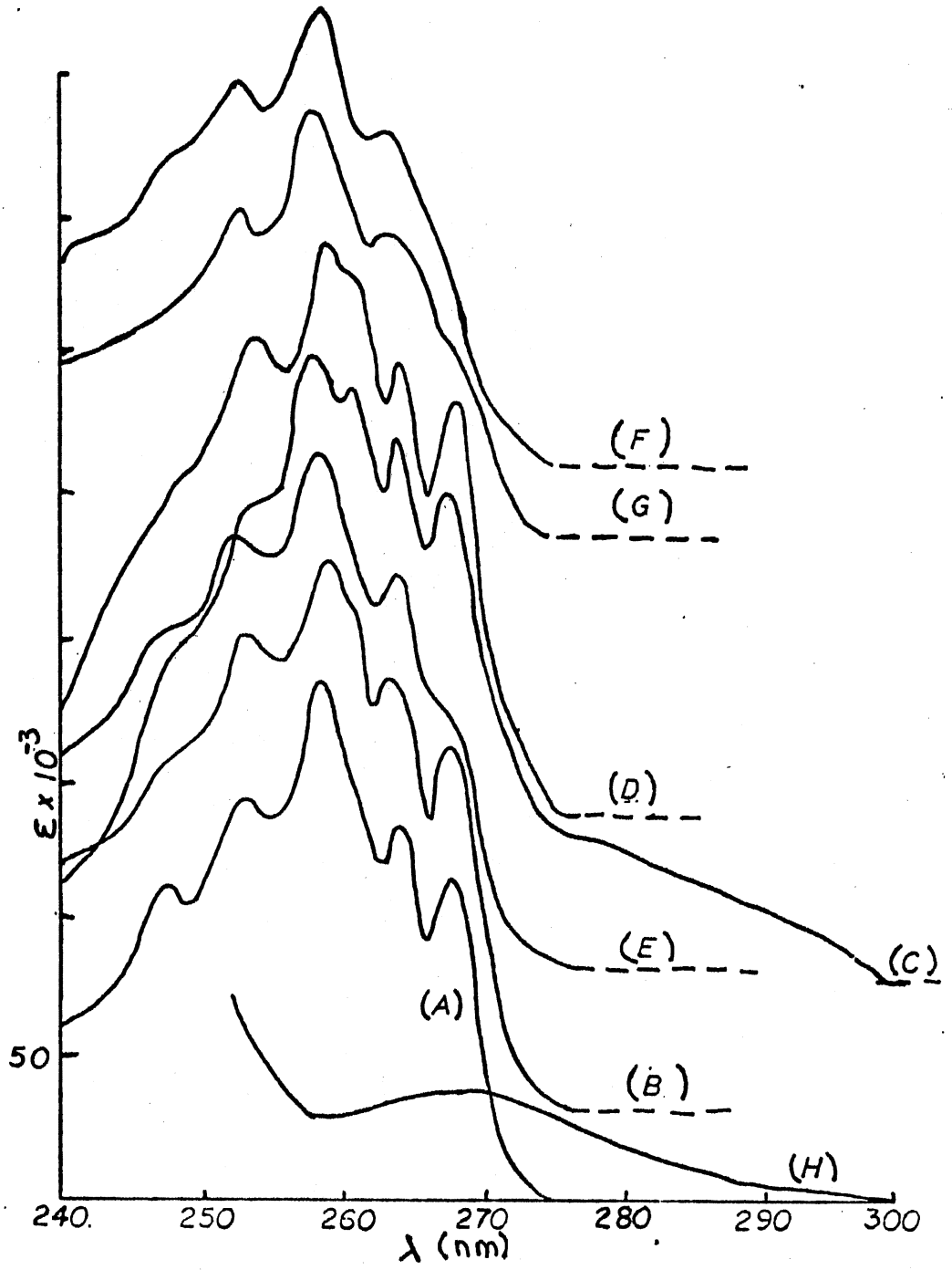


Figure 13. Amphetamine Group Ultraviolet Spectra; the Dotted Lines Represent the baseline; Each Section on the y-Axis Represents 50 ($\varepsilon \times 10^{-3}$)

TABLE V

SUMMARY OF ULTRAVIOLET DATA IN ETHANOL TRYPTAMINE GROUP

Compound	λ(nm)
(I) Indole	288, 280, 273* (ε = 1060)
(J) 5-Hydroxy Indole	298, 271* (ε = 8630)
(K) 5-Methoxy Indole	306, 294, 280, 276, 268* (ε = 6300), 265
(L) Tryptamine	291, 283* (ε = 5220)
(M) d-LSD	310* (ε = 1400)

*λ^{max}

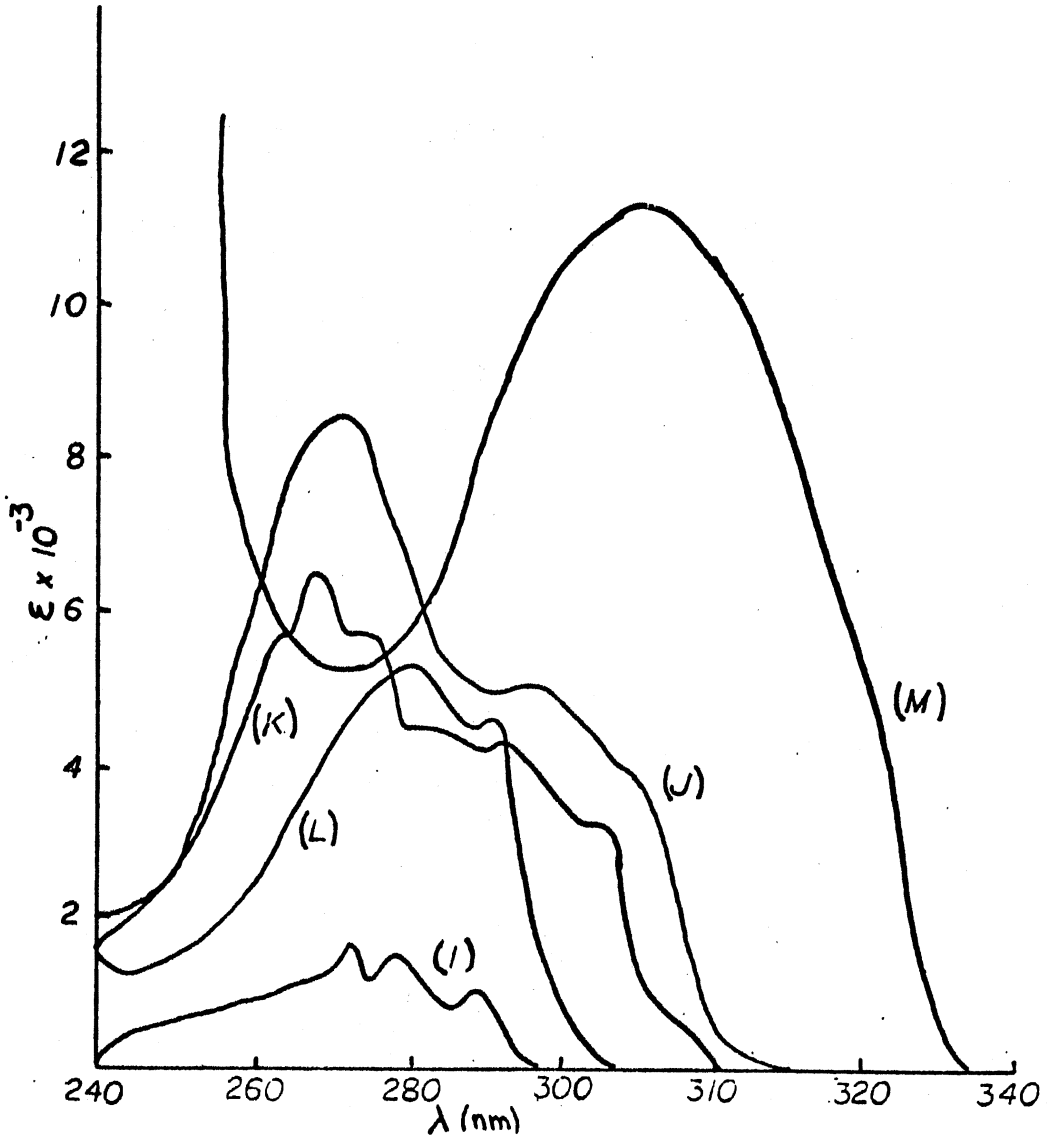


Figure 14. Tryptamine Group Ultraviolet Spectra

TABLE VI

ECD MAXIMUM: AMPHETAMINE GROUP

	λ(nm)	θ	[θ]
(A)	d-Amphetamine		
	266	+497	+67,267
	260	+709	+9,528
	253	+544	+7,359
(B)	d-Methamphetamine		
	265.5	+132	+24,210
	260	+1808	+26,974
	255	+1372	+20,472
(C)	Ephedrine		
	266.5	+4460	+73,704
	260	+5640	+93,182
	255	+4270	+70,541
(D)	(+)-(α)-Phenethylamine		
	266	-6600	-80,400
	260	-9500	-115,800
	255	-7800	-95,100
	240	-5400	-65,800
(E)	(-)-(α)-Phenethylamine		
	266	+8600	+104,800
	260	+10,500	+128,000
	255	+9,700	+118,200
	248	+5,400	+65,800

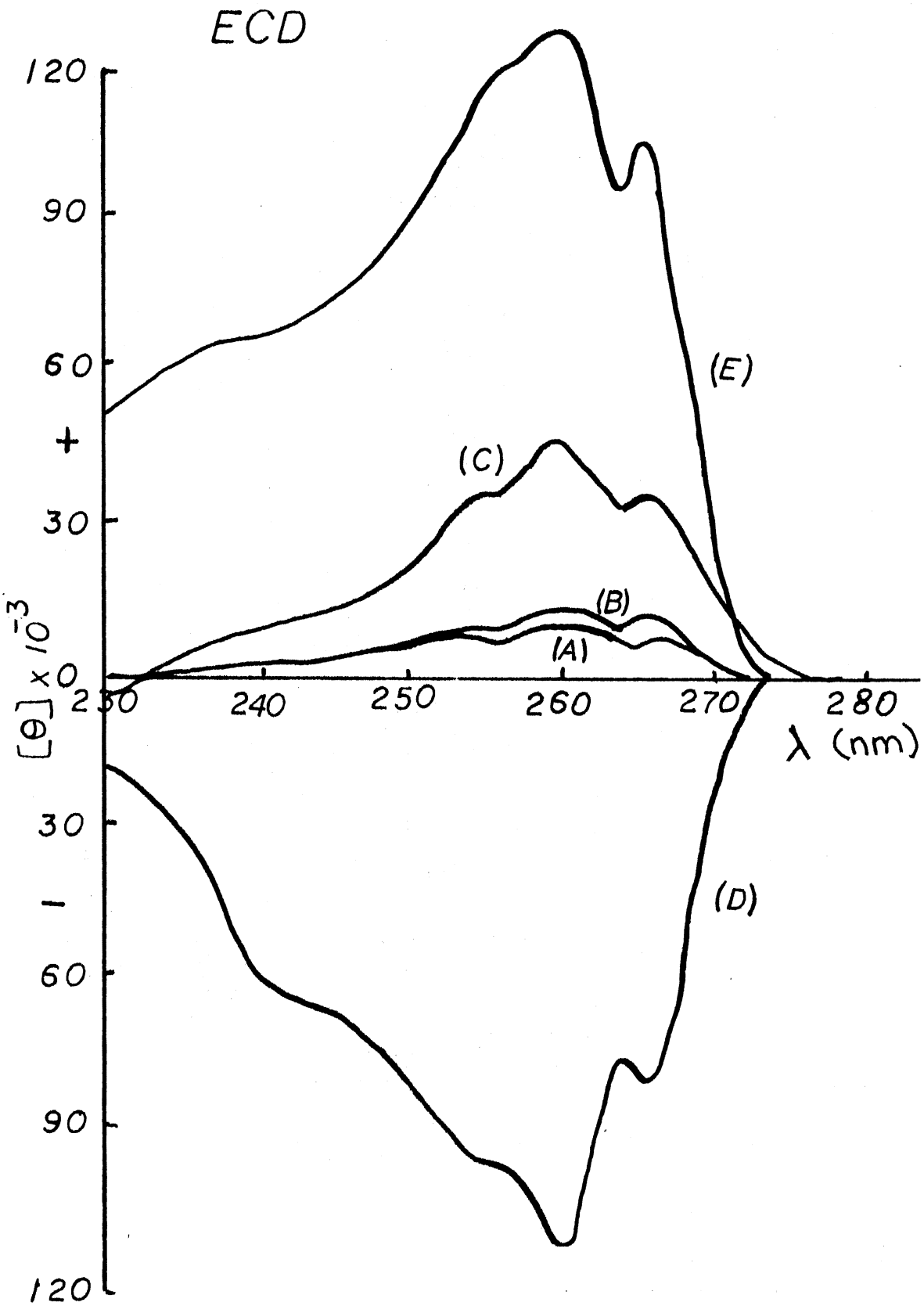


Figure 15. Amphetamine Group ECD Spectra

TABLE VII

ACD MAXIMA: AMPHETAMINE GROUP

	λ(nm)	θ	$[\theta] \times 10^{-3}$
(A)	d-Amphetamine		
	263	+310	+4.21
	257	+384	+5.19
	255	+208	+2.80
	233	-32	-.43
(B)	d-Methamphetamine		
	265	+594	+8.84
	257	+640	+9.56
	254	+448	+6.69
(C)	Ephedrine		
	264	+2166	+35.8
	259	+2740	+45.3
	253	+2102	+34.75
(D)	(+)-(α)-Phenethylamine		
	264	-1120	-13.65
	258	-1546	-18.0
	254	-1172	-14.0
(E)	(-)-(α)-Phenethylamine		
	264	+1333	+16.2
	258	+1812	+22.1
	254	+1494	+18.2

TABLE VIII

BCD MAXIMA: AMPHETAMINE GROUP

	λ(nm)	θ	$[\theta] \times 10^{-3}$
(A)	d-Amphetamine		
	263	+455	+6.15
	258	+496	+6.70
	254	+485	+5.87
	252	+424	+5.73
	247	+351	+4.75
(B)	d-Methamphetamine		
	264	+651	+9.71
	257	+992	+14.8
	253	+589	+8.79
	235	+155	+2.00
(C)	Ephedrine		
	265	+2585	+41.6
	259	+3282	+54.2
	254	+2585	+41.6
(D)	(+)-(α)-Phenlthylamine		
	265	-2294	-27.96
	259	-2986	-36.4
	254	-2293	-27.96
(E)	(-)-(α)-Phenethylamine		
	265	+2772	+33.8
	259	+3678	+44.8
	253	+2888	+34.5

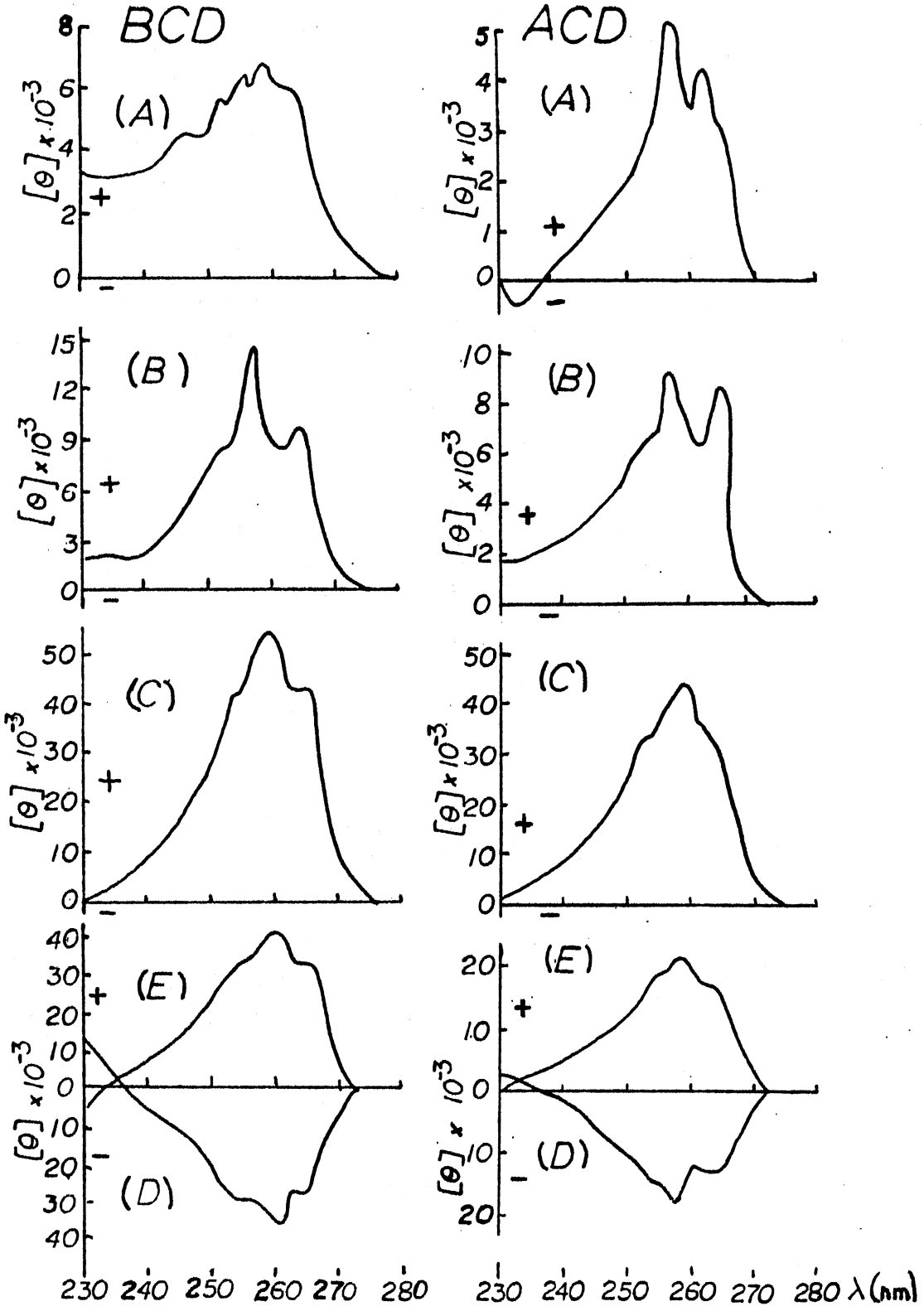


Figure 16. Amphetamine Group ACD and BCD Spectra

are shown in Figure 17 in ethanol, 0.1 N. NaOH, and 0.5 N. HCl. The maxima are collected in Table IX.

TABLE IX

ECD, ACD AND BCD: d_LSD

	λ(nm)	θ	$[\theta] \times 10^{-4}$
ECD			
	320	+1647	+5.3
	247	-2408	-7.9
	217	+4694	+15.2
ACD			
	325	+1012	+3.3
	247	-1518	-4.9
	222	+761	+2.5
BCD			
	320	+949	+3.1
	245	-1263	-4.1
	220	+2027	+6.6

Liquid Crystal Induced Circular Dichroism

The final technique involving liquid crystals as a solvent to obtain a circular dichroism also requires normalization for a full range of 1.0 units and a concentration 1.0 g/g. Tables X and XI list the ellipticities and molal ellipticities of their maxima. Figures 18-24 show the normalized spectra for the amphetamine and tryptamine group.

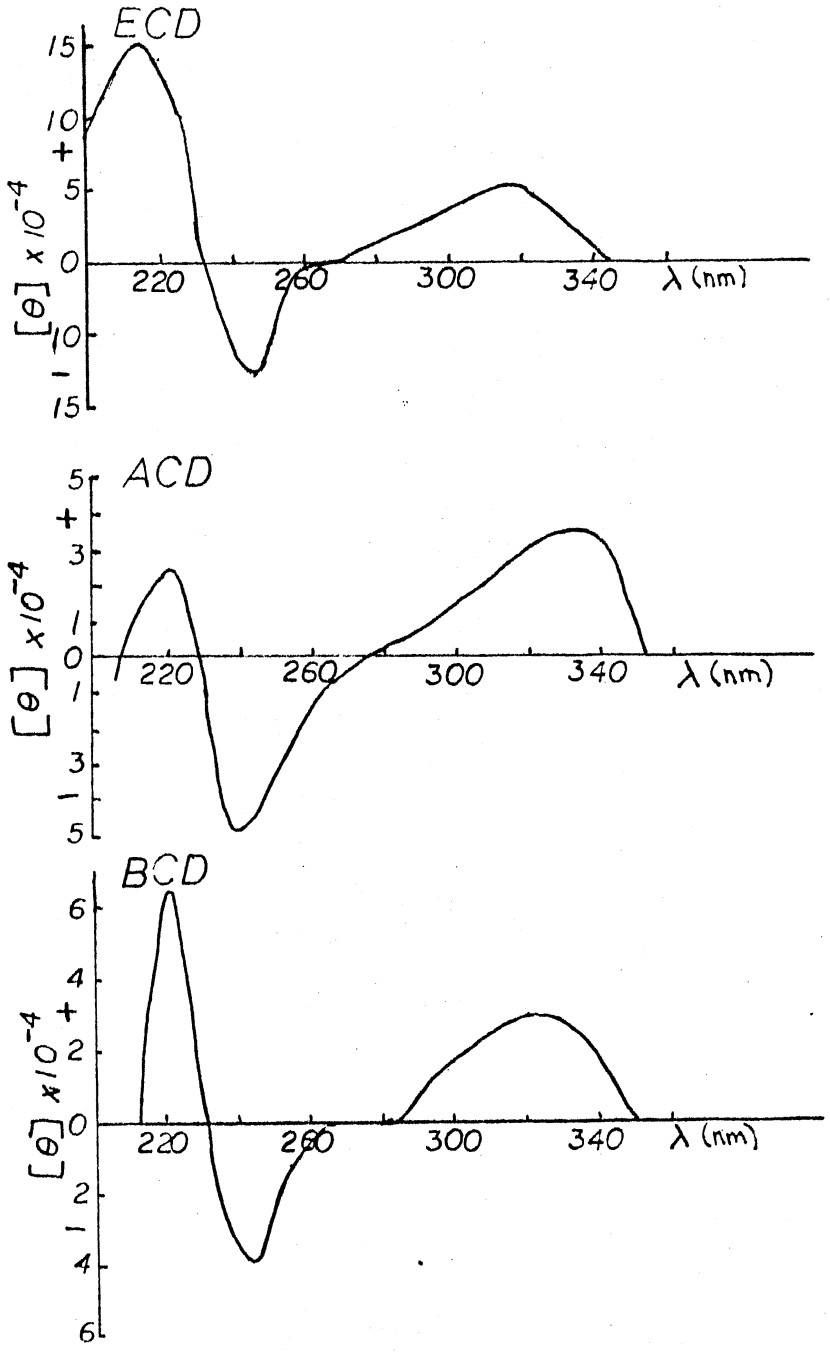


Figure 17. d-LSD, ECD, ACD, and BCD Spectra

TABLE X

LCICD MAXIMA: AMPHETAMINE GROUP

	λ(nm)	θ	(As Listed)
(A)	d-Amphetamine		$[\pi] \times 10^{-4}$
	266	-2.15	-2.28
	260	-3.31	-3.5
	253	-2.48	-2.6
	233	+3.47	+3.7
(B)	d-Methamphetamine		$[\pi] \times 10^{-4}$
	269	- .33	+.34
	266	-2.3	-2.7
	263	-3.3	-3.87
	245	-4.46	+5.22
	231	-9.91	-11.6
(C)	Ephedrine		$[\pi] \times 10^{-4}$
	265	-2.15	-2.79
	258	-2.87	-3.72
	246	+3.11	+4.03
(D)	(+)-(α)-Phenethylamine		$[\pi] \times 10^{-4}$
	257	-29.3	-28.0
	253	-31.8	-30.4
	246	-29.3	-28.0
	241	-24.4	-23.3
(E)	(-)-(α)-Phenethylamine		$[\pi] \times 10^{-4}$
	264	-11.2	-10.7
	257	-19.8	-18.9
	253	-18.6	-17.8
	250	-19.0	-18.2
(F)	Mescaline		$[\pi] \times 10^{-5}$
	270	-157.9	-26.2
(G)	dl-Methamphetamine		$[\pi] \times 10^{-4}$
	264	-7.34	-8.6
	258	-12.24	-14.34
	253	-8.04	-9.41
(H)	β-Phenethylamine		$[\pi] \times 10^{-4}$
	265	-4.54	-4.34
	260	-7.84	-7.50
	254	-3.72	-3.56

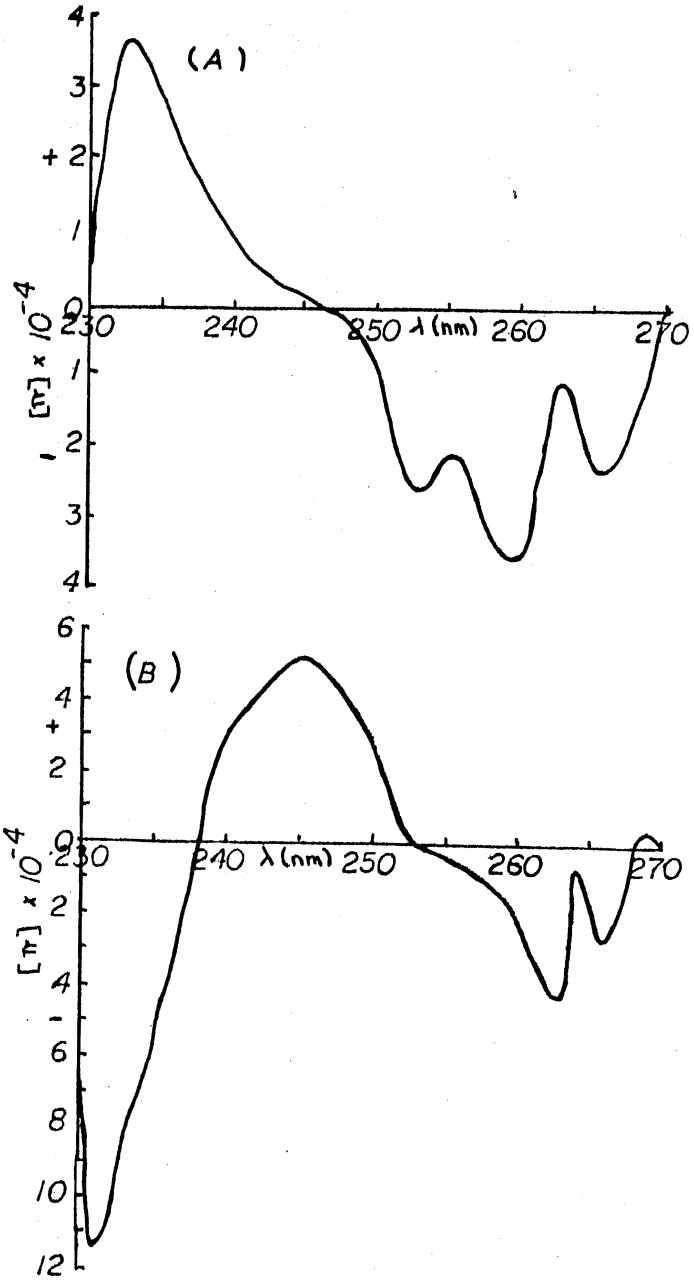


Figure 18. LCICD of (A)-d-Amphetamine and (B)-d-Methamphetamine

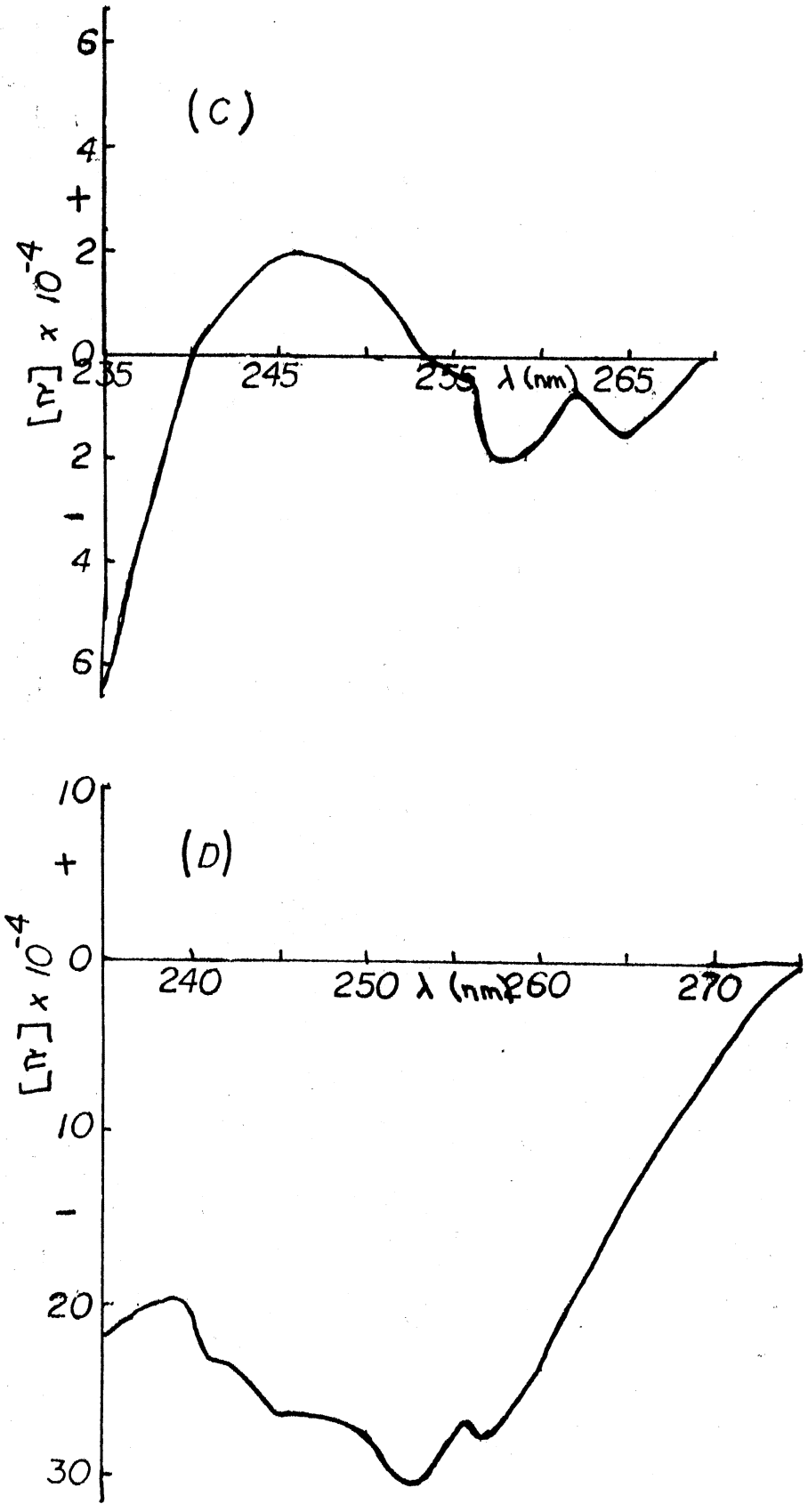


Figure 19. LCICD of (C)-Ephedrine and (D)-(+)-(α)-Phenethylamine

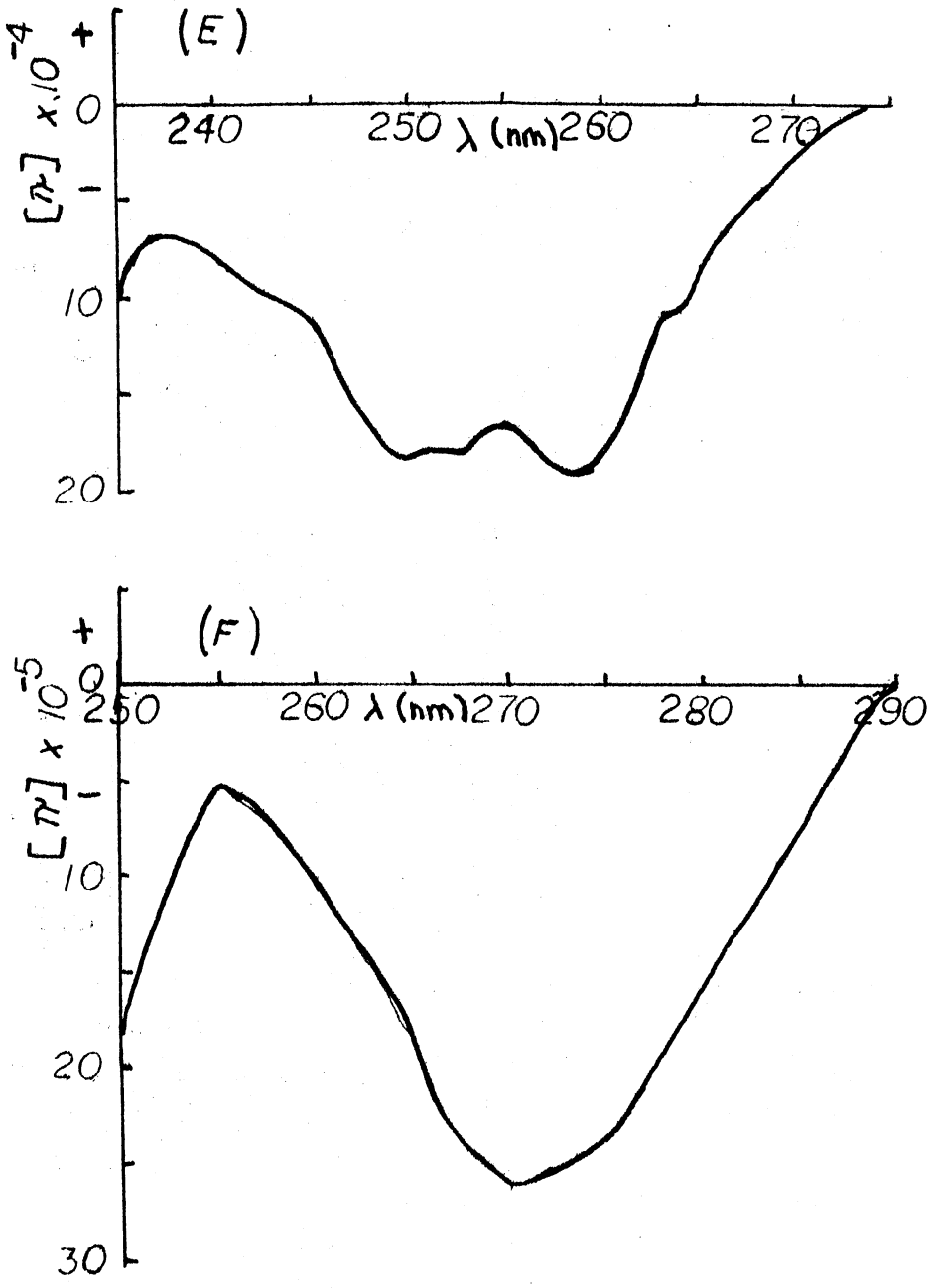


Figure 20. LCICD of (E)-(-)-(α)-Phenethylamine and (F)-Mescaline

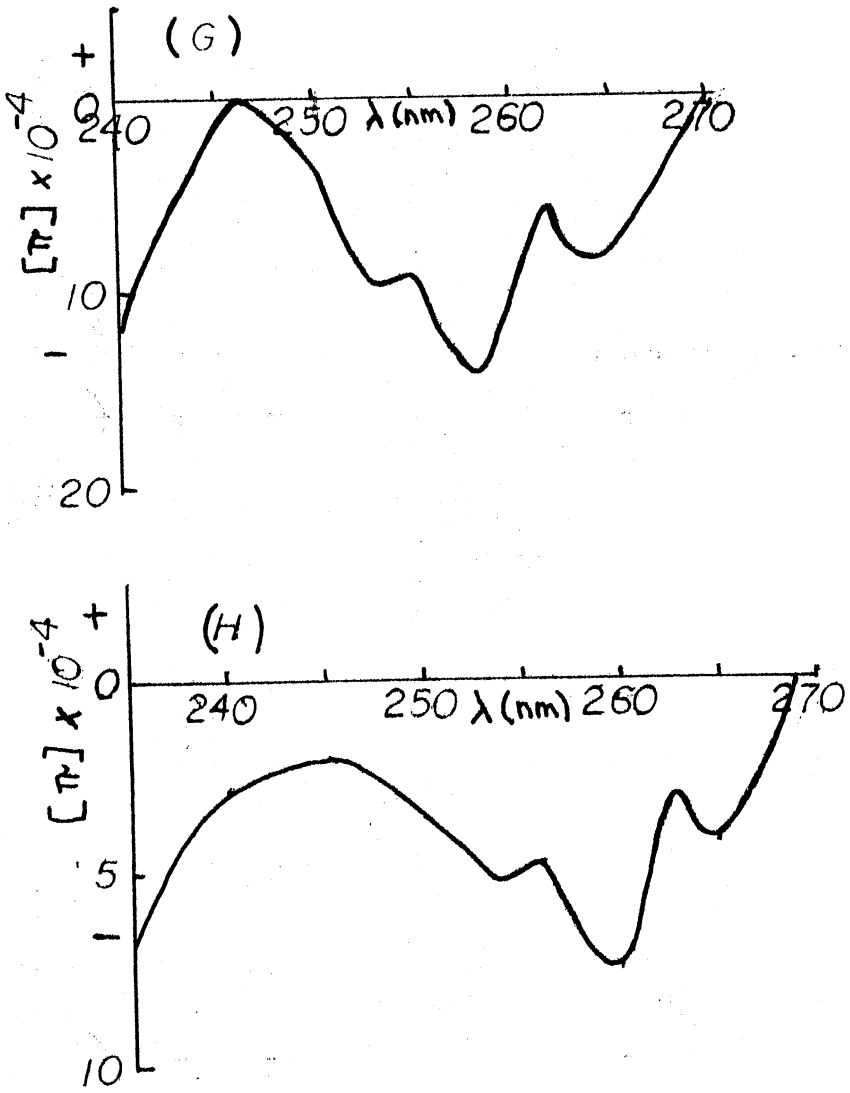


Figure 21. LCICD of (G)-d-Methamphetamine and (H)-β-Phenethylamine

TABLE XI

LCICD MAXIMA: TRYPTAMINE GROUP

	λ(nm)	θ	(As Listed)
(A)	Indole		$[\pi] \times 10^{-4}$
	288	-62.7	-57.7
	280	-78.2	-72.1
	272	-84.7	-77.9
	265	-81.6	-75.0
(B)	5-Methoxy Indole		$[\pi] \times 10^{-4}$
	308	-11.8	-13.6
	271	-236.6	-273.3
(C)	5-Hydroxy Indole		$[\pi] \times 10^{-5}$
	310	-2371	-24.8
	300	-3192	-33.4
	270	-8299	-86.7
(D)	Tryptamine		$[\pi] \times 10^{-4}$
	304	-.62	-.8
	292	-35.3	-44.4
	285	-35.9	-45.1
	277	-27.9	-35.1
(E)	d-LSD		$[\pi] \times 10^{-4}$
	310	-59.9	-152.1

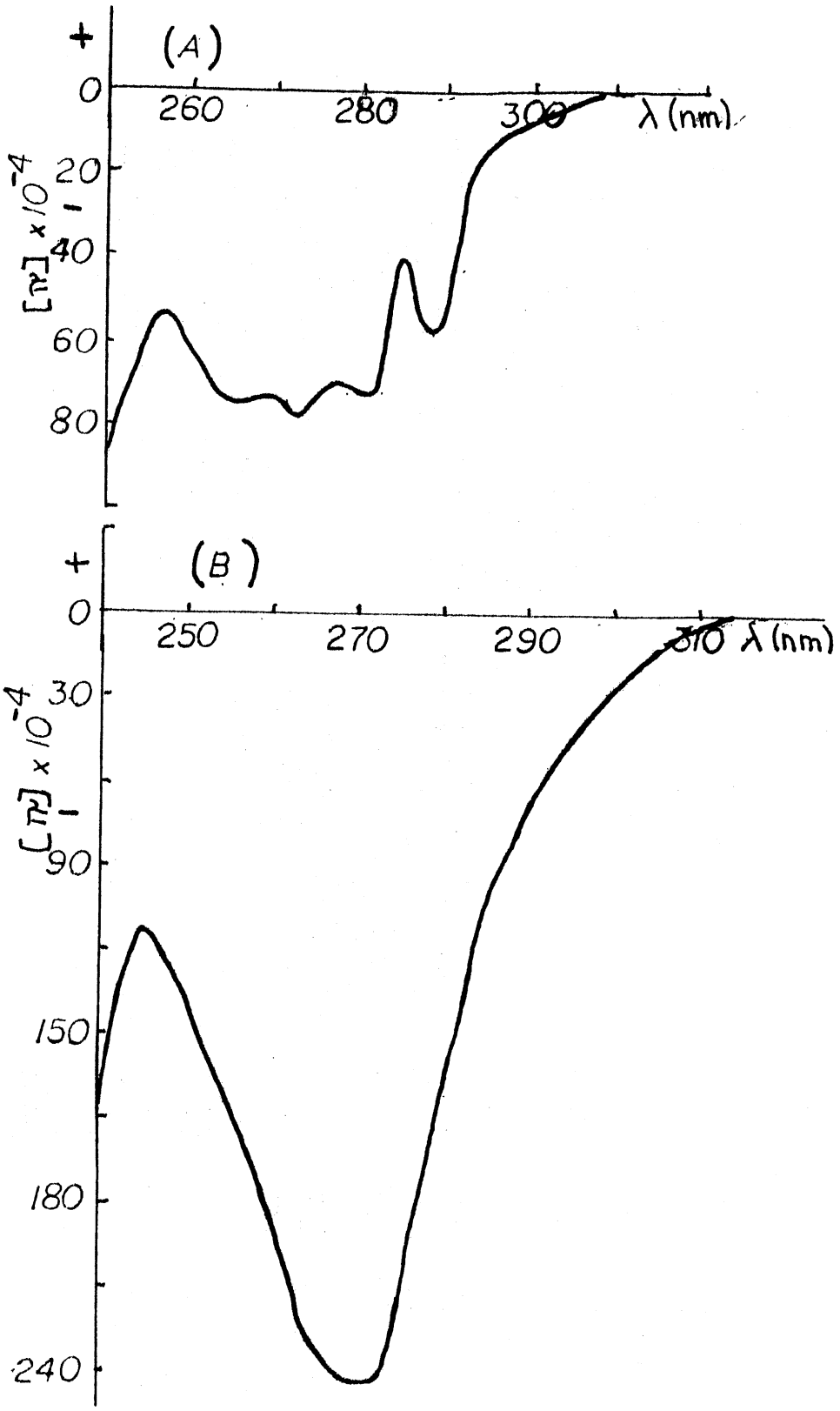


Figure 22. LCICD of (A)-Indole and (B)-5-Methoxy-Indole

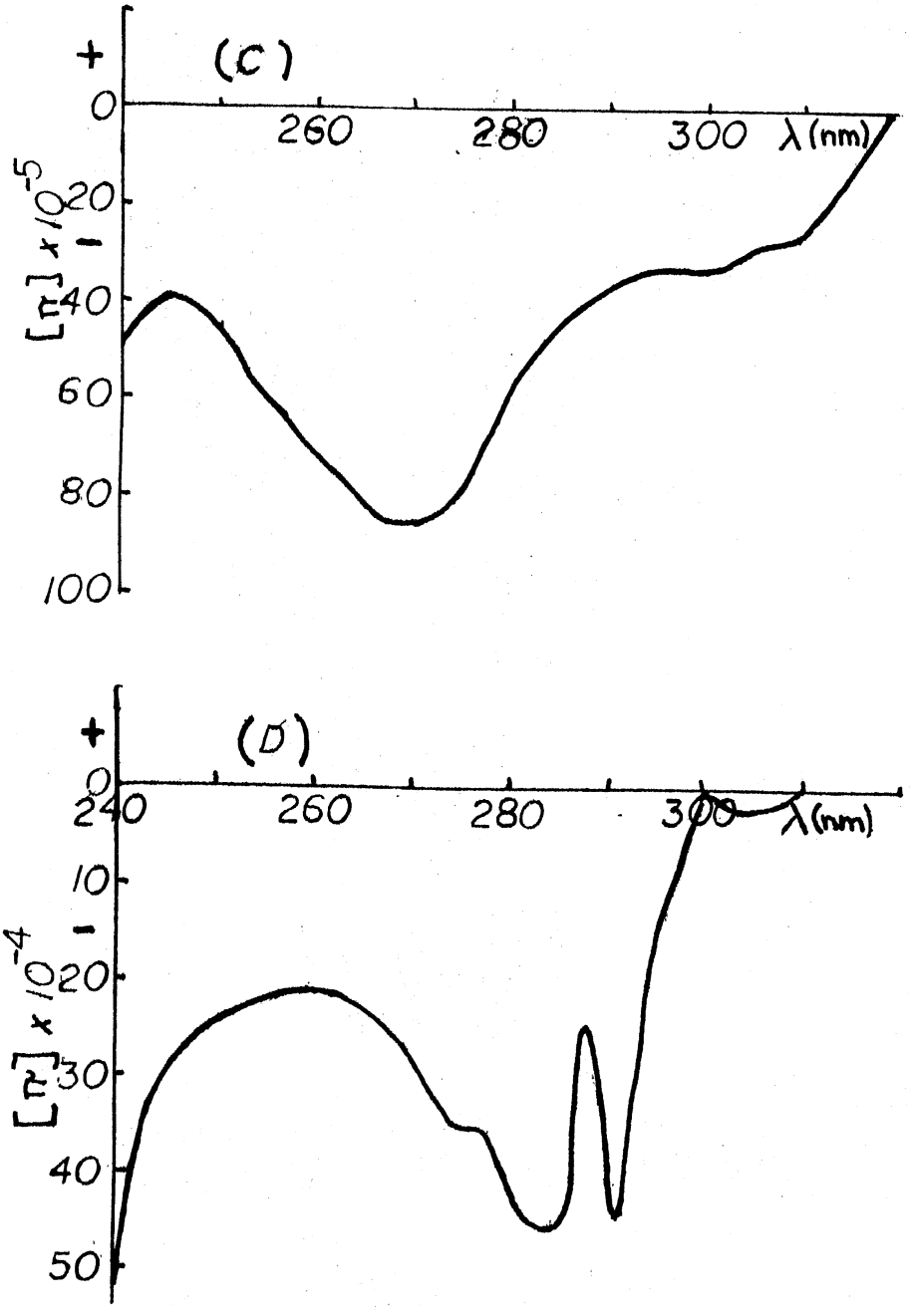


Figure 23. LCICD of (C)-5-Hydroxy Indole and (D)-Tryptamine

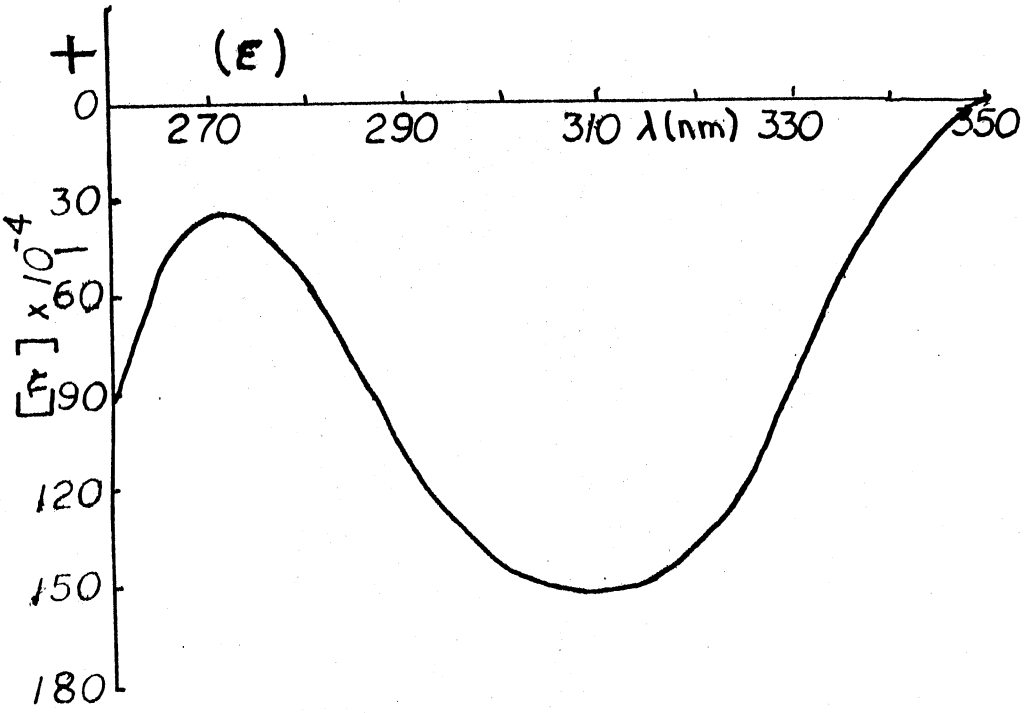


Figure 24. LCICD of (E)-d-LSD

CHAPTER VII

DISCUSSION AND CONCLUSIONS

Ultraviolet Absorption Results in Ethanol

The spectra for the amphetamine group result in a series of sharp absorptions concentrated in the wavelength region from 270-250 nm, as seen in Figure 13. For each compound the maximum absorption occurs at 258 nm. The only exception lies in the spectrum of d-mescaline where the structure is lost and the broad absorption band maximum has been shifted to 270 nm (Figure 13). This shift is probably related to the fact that d-mescaline has four sites of substitution on the benzene ring compared to the others which have, at the most, two sites of substitution. The similarity of the ultraviolet spectra for the amphetamine group clearly demonstrates the inability of the method to distinguish among compounds belonging to the same family.

The members of the tryptamine group have ultraviolet spectra (Figure 14) which are somewhat different from one another. Compared to the sharp absorption maxima of the amphetamine group, the tryptamine spectra are composed of broader bands. Generally these bands range from 310 to 240 nm and with the exception of tryptamine and d-LSD, the maximum lies around 270 nm (Figure 14). Though the tryptamine spectra appear to allow for greater distinction among various members than the amphetamine spectra; identification is still very limited.

Circular Dichroism Results Using Isotropic Solvents

Circular Dichroism provides the additional advantage of modifying the ultraviolet absorption spectrum of a compound. An absorption band due to an electronic transition can have a positive or negative ellipticity or have zero ellipticity if the right and left molar extinction coefficients of the right and left circular components are equal. One of the objectives is to find the best solvent system that will allow better distinction among members of the amphetamine and members of the tryptamine families.

Using an isotropic medium (absolute ethanol, acid, or base) was a logical progression because a direct comparison of absorptions of unpolarized and circularly polarized light could be made along with taking into account the solvent effect. Optical activity in the amphetamine group is due to the chiral center(s) located on the alkyl chains which extend from the benzene ring. The only members of the group which are optically inactive are the compounds mescaline and β-phenethylamine The compound dl-methamphetamine is a racemate and shows no ellipticity.

The ECD spectra of d-amphetamine and d-methamphetamine show a shift in wavelength towards the red with a maximum ellipticity at 260 nm (Figure 15). Both compounds have three positive ellipticities in the same wavelength region. The two compounds can only be distinguished from each other by the difference in magnitude of their molar ellipticities as seen in Table VI. The spectrum for ephedrine also resembles that for d-amphetamine and d-methamphetamine in both position of the band maxima and the sign, but the molar ellipticity coefficients

are much greater (Figure 15 and Table VI). For the (α)-phenethylamines, the maxima are also shifted upward with one exception. That at the shortest wavelength is shifted lower. The magnitudes of the four ellipticities are greater than the other three compounds so far discussed (Figure 15 and Table VI). The band maxima, like those for the other optically active members of the amphetamine series, is at 260 nm. A series of positive bands are observed for (-)-(α)-phenethylamine and a negative set for (+)-(α)-phenethylamine, but each peak is approximately the same magnitude.

ECD in comparison with ultraviolet absorption is less successful in distinguishing among the amphetamines. While molar parameters are useful comparisons for pure compounds, they are of little value in qualitative identification. Further more, the fact that no ECD is obtainable for achiral compounds automatically eliminates them from possible identification.

The optically inactive tryptamine group, with the exception of the chiral molecule d-LSD, exhibit no circular dichroism in ethanol. D-LSD is therefore readily identified; but it was just as readily identified by its ultraviolet absorption spectrum in comparison to other members of the tryptamine family. Nevertheless in a more complete study of more members of this group and the ergot alkaloids, ECD may prove to be of great value. The method should not be considered a failure at this time when only one chiral compound has been included in the study.

In summary, the ECD spectra are only slightly modified from ultraviolet absorption spectra and not sufficiently enough to add to the level of distinction until more chiral compounds can be included in the study.

The pH-dependence of ultraviolet spectra for the amphetamines and some tryptamines are well documented. The dependences observed among members of each group are similar, and distinction is not possible. Unfortunately the same appears to be true of the ACD and BCD spectra, with the singular exception of d-LSD. The ACD spectrum of d-LSD shows the three characteristic peaks (Figure 16) observed in the ECD spectrum. However the magnitudes of the ellipticities are smaller. Moving to a basic environment increases the size of the ellipticity maximum at 220 nm by a factor of three (Table VII and Table VIII), but the relative magnitude of the other two peaks remains constant.

This result is quite different from other work being done concurrently in this laboratory with cocaine and the opiates. There the ACD and BCD changes with pH are large and individual for a number of derivatives. A very important property which contributes to their distinction appears to be that the molecular structures are rigid. All of the amphetamines are flexible, and only d-LSD of the tryptamines is a multiple ring derivative. Better analytical success should be obtained with the ergot alkaloids which have a tryptamine parent structure and are polycyclic.

Circular Dichroism Results Using Liquid Crystals

By virtue of the property of an anisotropic solvent to induce chirality, all of the compounds in both the amphetamine and tryptamine groups can be included in a LCICD study. For the optically active compounds, the signal has been increased or amplified by a factor of

ten with respect to the circular dichroism of the compounds in an isotropic medium (Figures 18-21 and Table X). It must be remembered that the helical sense of the cholesteryl chloride/cholesteryl nonanoate liquid crystal solvent is to the right and has a negative ellipticity.

The LClCD spectrum for d-amphetamine has four ellipticity maxima, but the first three bands above 247 nm are negative in sign (Figure 18). At 270 nm, the LClCD crosses the baseline to the positive side for the 233 nm band. The LClCD of d-methamphetamine shows three positive ellipticity at 269, 266, and 263 nm and has negative broad peak at 245 nm (Figure 18). The CD spectrum again crosses over at 238 nm to a sharp band maximum at 233 nm. Direct comparison of the LClCD of d-amphetamine and d-methamphetamine clearly indicates that an anisotropic solvent such as a liquid crystal system is capable of distinguishing between two very similar compounds.

Ephedrine (Figure 19) dissolved in liquid crystals produces a spectrum similar in shape to d-methamphetamine, but peaks are at different wavelengths and have smaller magnitudes.

The most dramatic change occurs in the circular dichroism of the (α)-phenethylamines (Figures 19-20). For the optical enantiomers in an isotropic solvent, the CD spectra are mirror images of each other. In liquid crystals, the helical sense of the solvent predominates and induces only all negative CD spectra for both enantiomers. The (+)-(α)-phenethylamine LClCD has twice the molar ellipticity at the maxima. This reduces the powers of distinction between enantiomers dissolved in anisotropic solvents in comparison to isotropic solvents where distinction is easily made. The phenethylamine spectra differ

dramatically from the other optically active amphetamine members confirming the success of LClCD as a more qualitative technique.

The optically inactive members of the amphetamine group (Figures 20 and 21) exhibit an induced negative CD. Mescaline produces an induced CD in liquid crystals that has one large, broad, negative band maximizing at 270 nm. The racemate dl-methamphetamine shows an induced CD that is completely negative except at 246 nm where the CD is zero. The LClCD of β-phenethylamine resembles dl-methamphetamine upon first glance but has lower intensities and never has an ellipticity of zero.

It becomes apparent that the LClCD spectra is capable of distinguishing between compounds of similar structure belonging to the amphetamine group. But the degree of distinction and the reproducibility has not been fully investigated. The methamphetamine compounds, d- and dl-, illustrate an excellent example where the separation and identification of the same compound which differ only in their optical activity, is possible. The employment of the LClCD technique is not as applicable to the identification of (+)-(α)- and (-)-(α)-phenethylamines due to the anisotropic nature of the solvent. Selectivity of the solute is an important factor when using liquid crystals.

The LClCD of the tryptamine group show the most variety in spectra and the negative sign of the ellipticities is consistent with the anisotropic solvent signal. The parent compounds, indole and tryptamine, produce signals that are relatively small in magnitude. Substitution on the indole ring at the five position changes the LClCD by decreasing the number of bands and increasing their magnitude in

contrast to indole. The LClCD method generates different spectra for 5-hydroxy indole and 5-methoxy indole which provides another facet to the technique as a method for qualitative distinction. The LClCD of d-LSD demonstrates that for a more complex chiral molecular system, the CD is not induced greatly. Since only one chiral compound was looked at belonging to the tryptamine family, it is not possible to evaluate the distinction capabilities of CD compared to other solvent systems.

The members of the amphetamine group are flexible molecules compared to the tryptamine group. Further investigation may demonstrate, as was found for d-LSD, that chances for greater distinction lies in isotropic solvents for chiral molecularly rigid molecules.

The major purpose of this project dealt with the qualitative aspects of the analysis of drugs and their related compounds. An illicit mixture of d-amphetamine and lactose would show only the presence of the former in both the ultraviolet absorption and circular dichroism in an isotropic solvent since lactose is non-absorbing and achiral. An absorbing, chiral entity such as a dyestuff added to the illicit mixture could interfere in the signal obtained by d-amphetamine in an isotropic solvent. By using LClCD and depending on the molecular structure of the dyestuff, one might be able to obtain the characteristic d-amphetamine induced CD.

Though this set of techniques is still in the exploratory stages, the advantages of the various solvents and the use of circular dichroism does show promise in the qualitative as well as quantitative analysis

of drugs and their related compounds. LC1CD is not yet ready for incorporation as a routine qualitative technique due to the lack of consistent experimental data. However, this study has served as a basis for greater understanding of the properties of similar compounds and the use of circular dichroism.

BIBLIOGRAPHY

1. Berman, E., "Analysis of Drugs of Abuse", Heyden Press, London, 1977.

2. Mulé, S. J., J. of Chem. Sci., 10, 275 (1972).

3. Mulé, S. J., J. of Chem. Sci., 12, 245 (1974).

4. Abu-Shumays, A. and Duffield, J. J., Anal. Chem., 38, 29A (1966).

5. Foss, J. G., J. of Chem. Ed., 40, 592 (1963).

6. Crabbé, P., "Optical Rotatory Dispersion and Circular Dichroism in Chemistry and Bio-Chemistry", Academic Press, London and New York, 1960.

7. Djerassi, C., "Optical Rotatory Dispersion", McGraw-Hill, New York, (1960).

8. Djerassi, C., Wolf, H., Bunnenberg, E., J. of Chem. Soc. (London), 84, 4552 (1962).

9. Lambert, J. B., Shurvell, H. F, Verbit, L., Cooks, R. G., Stout, G. H., "Organic Structural Analysis", MacMillan Publishing Co., Inc., New York, N.Y., 1976.

10. Murphy, W. S., J. of Chem. Ed., 52, 774 (1975).

11. Norden, B., Acta Chemica Scand., 26, 2 (1972).

12. Schellman, J. A., Chem. Rev., 75, 323 (1975).

13. Scopes, P. M., "Annual Reports on the Progress of Chemistry, Section B-Organic Chemistry", Vol. II, 35 (1974).

14. Scopes, P. M., "Annual Reports on the Progress of Chemistry, Section B-Organic Chemistry", Vol. II, 84 (1972).

15. Snatzke, G., "Optical Rotatory Dispersion and Circular Dichroism in Organic Chemistry", Heyden and Son Limited, London, 1967.

16. Velleez, L. and Legrand, M., Bull. Soc. Chim. France, 1785 (1970).

17. Woody, R. W., J. of Polym. Sci.: Macromolecular Reviews, 12, 181 (1971).

18. Brown, G. H., Amer. Sci., 60, 64 (1972).

19. Chandrasekhar, S., "Liquid Crystals", Cambridge University Press, 1977.

20. Chandrasekhar, S., Madhusudana, N.V., Appl. Spectr., 6, 189 (1972).

21. DeVries, H. L., Acta Cryst., 4, 219 (1951).

22. Fergasen, J. L., Mol. Cryst., 1, 293 (1966).

23. Fergason, J. C., Goldberg, N. N., Nadalin, R. J., Mol. Cryst., 1, 309 (1966).

24. Steinrasser, R. and Pohl, L., Angew Chem. Inter., 12, 617 (1973).

25. Verbet, L., J. of Chem. Ed., 49, 36 (1972).

26. Buckingham, A. D., Ceasar, G. P., Dunn, M. B., Chem. Phys. Letters, 3, 540 (1969).

27. Stegemeyer, H. and Marnusch, K.-J., Chem. Phys. Letters, 6, 5 (1970).

28. Same as above, 8, 425 (1971).

29. Norbonne, J. M., Weiss, R. G., J. of Amer. Chem. Soc., 101, 402 (1979).

30. Sakamoto, K., Yoshida, R., Hatano, M., Tachibana, T., J. of Amer. Chem. Soc., 100, 6898 (1978).

31. Saupe, A., Mol. Crys. Liq. Cry., 16, 87 (1972).

32. Saupe, A., and Englert, G., Phys. Rev. Letters, 11, 462 (1963).

33. Stevenson, P. E., J. of Chem. Ed., 234, (1962).

34. Sklar, A. L., J. Chem. Phys., 10, 135 (1942).

35. Petruska, J. J., J. Chem. Phys. 34, 1111 (1961).

36. Siek, T. J., J. of Forensic Chem., 19, 193 (1974).

37. Siek, T. J., J. of Forensic Chem., 19, 18 (1973).

38. Sunshine, Irving, "Handbook of Analytical Toxicology", Chemical Rubber Company, Cleveland, Ohio, 1969.

39. "An Encyclopedia of Chemicals and Drugs" in The Merck Index, 9th ed., Martha Windholtz, ed., Merck and Company, Inc., Rahway, N. N.J., 1976.

40. Gottarelli, G. and Samori, B., J. Chem. Soc. (B), 2418 (1971).

41. Smith, H. E., Burrow, E. P., Chen, F., J. Amer. Chem. Soc., 100, 3714 (1978).

42. Smith, J. E., Burrow, E. P., Chem. F., J. Org. Chem. 40, 1562 (1975).

43. Smith, H. E., Padilla, B. G., Neergaard, J. R., Chen, F., J. Amer. Chem. Soc., 100, 6035 (1978).

44. Takenaka, S., Kondo, K., Tokura, N., J. Chem. Soc., Perkin II, 1749 (1975).

45. Tokura, N., Nagai, T., Takenaka, S., Oshima, T., J. Chem. Soc., Perkin II, 337 (1974).

46. Takenaka, S., Kondo, K., Tokura, N., J. Chem. Soc. Perkin II, 1520 (1975).

47. Saeva, F. D., J. of Amer. Chem. Soc., 93, 5928 (1971).

48. Saeva, F. D., J. of Amer. Chem. Soc., 94, 5135 (1972).

49. Saeva, F. D., Mol. Crys. Lia. Crys., 18, 375 (1972).

50. Saeva, F. D., J. of Amer. Chem. Soc., 95, 7656 (1973).

51. Saeva, F. D., J. of Amer. Chem. Soc., 95, 7882 (1973).

52. Saeva, F. D., Mol. Crys. Liq. Crys., 23, 171 (1973).

53. Saeva, F. D., J. of Amer. Chem. Soc., 98, 2709 (1976).

54. Sackmann, E., and Voss, J., J. Chem. Phys. Letters, 14, 2528 (1972).

55. Sackmann, E., Krebs, P., Bega, H. U., Voss, J., and Mohwald, H., Mol, Crys. Liq. Crys., 24, 283 (1973).

56. Sackmann, E. and Mohwald, H., J. Chem. Phys., 58, 5407 (1973).

57. Sackmann, E., Krebs, P. Rega, H. U., Voss, J. and Mohwald, H., Mol. Crys. Liq. Crys., 24, 238 (1974).

58. Buckingham, A. D., Ceasar, G. P., and Dunn, M. B., Chem. Phys. Letters, 3, 540 (1969).

59. Snatzke, G. and Ho, P. C., Tetrahedron, 27, 3645 (1971).

60. Snatzke, G., Kajtár, M. and Wernerzamojska, F., Tetrahedron, 28, 281 (1972).

61. Saeva, F. D., Sharpe, P. E., and Alin, G. R., J. of Amer. Chem. Soc., 95, 7660 (1973).

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