Edward A. Julian,[1] *Ph.D.*

Microcrystalline Identification of Drugs of Abuse: The Psychedelic Amphetamines

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ABSTRACT: The optical crystallographic or microcrystalline properties of the diliturate derivatives (5-nitrobarbituric acid) of the most used psychedelic amphetamine drugs have been determined. The crystallographic properties, especially the orthographic projection drawings and photomicrography of the crystals and other optical crystallographic data, such as the indices of refraction, extinction angles, and optical orientations serve to characterize and identify this group of drug chemicals. These data can be used with ultraviolet-infrared (UV-IR) spectroscopy, thin-layer chromatography and gas-liquid chromatography/mass spectrometry (GLC/MS) data for the identification and confirmation of psychedelic amphetamine drugs.

KEYWORDS: toxicology, drug abuse, amphetamines, psychedelic amphetamines, optical crystallographic properties, chemical microscopy, photomicrographs, drug identification, confirmation, diliturate derivatives

The synthesis and illicit use of psychedelic amphetamines have increased considerably over the last few years and have resulted in the preparation of many new hallucinogenic substances, such as methylenedioxyamphetamine (MDA) and its cogeners. Because of their psychotropic properties and their high demand in illegitimate markets, various ring and chain-substituted amphetamine-like derivatives have been synthesized, often in clandestine chemical laboratories [*1*]. These illicitly synthesized psychedelic compounds tend to stay one step ahead of Schedule I classification as administered by the Drug Enforcement Administration (DEA) and, subsequently, have been utilized as "recreational" drugs [*2,3*].

Recurring reports in the literature on the use of these drugs indicate a need for analytical techniques for the identification of these amphetamine-like compounds [*4,5*]. At present, thin-layer chromatography (TLC) methods [*6–8*], gas-liquid chromatography (GLC) [*3,9*], and spectral methods [*3,10,11*] have been reported.

Chemical microscopy or optical crystallographic methods, which tend to identify but also confirm the results of other methods used [*12*], have been minimally reported in the literature [*13*]. The chemical microscopic or optical crystallographic method provides a visual confirmation of other analytical techniques utilized for the identification of these amphetamine derivatives [*14*].

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[1]Professor, School of Pharmacy, University of Wyoming, Laramie, WY.

Experimental Procedure

The methods used, the instrumentation, the preparation of crystalline derivatives, and the photomicrography employed were basically the same as those in previously reported publications [*12,14*].

The three-dimensional optical crystallographic data for the psychedelic amphetamine dilliturates were determined using standard methods found in the literature [*15,16*]. Crushed specimens in some cases were necessary to obtain refractive indices on uncentered crystal faces.

In this study, the "2 V" values were determined by using the "nomogram method" of Hartshorne and Stuart (Ref *16*, pp. 391–394). Briefly, the nomogram was utilized as a base upon which the three known refractive indices were used to determine the fourth variable, V ($V = \cos^2 \theta$).

The optical crystallographic and microcrystalline properties of the dilliturate salts of the psychedelic amphetamines are listed in Table 1, and descriptions of the individual crystals are found in Table 2. Individual crystal morphologies for each drug are represented by the drawings (Fig. 1) and the photomicrographs (Fig. 2). These data serve to characterize the crystalline psychedelic amphetamine dilliturates.

Results and Discussion

Significant Properties

The optical properties of the dilituric acid derivatives of selected psychedelic amphetamines are listed in Table 1. Many of the crystalline derivatives are so flattened that they tend to assume a common front face orientation on a microscope slide. In Table 1, optical orientations designated as acute, obtuse, or optic normal indicate that a centered interference figure is observed when the crystal is examined on a microscope slide. The descriptive term "inclined" describes an optical orientation figure which is not "centered," and thus, these optical orientations do not show exact refractive indices in one or both vibration planes of the crystal. Extinction angles, the angle between any specified crystallographic direction and either of the two polarized planes of the microscope, are measured from the long axis on the front face of each crystal. In some specimens, accurate refractive indices can only be determined in "crushed" crystalline material in which "centered" figures have been identified.

Figure 1 shows orthographic projection drawings of the crystals, presenting front, side, and top views. These drawings supplement the data found in Table 1, as do the photomicrographs found in Fig. 2. An asterisk in the drawings indicates the higher index value on views with uncentered or inclined orientations. For side and top views, the crystals were rolled in Canada balsam. Dashed lines indicate the two vibration directions of each crystal along which refractive indices may be measured.

Descriptions of Crystal Derivatives

Additional descriptions of some of the individual psychedelic amphetamine dilliturates will facilitate identification of them. Dilituric acid, the crystallizing agent, rarely precipitates out of the reaction solution in the concentration utilized; however, dilituric acid crystals appear as small square to rectangular shaped tablets with a low alpha index of refraction ($n = 1.388$) running parallel to the length of the crystal and show a low extinction angle (9°) on the top face.

Photomicrographs of each crystal are found in Fig. 2 and represent the crystals as

TABLE 1—*Optical crystallographic properties of psychedelic amphetamines.*

Drug Dilturate[a]	System[b]	Habit	Refractive Index			2 V(c) (Nomogram), deg	Optical Orientation	Optic Sign	Extinction Angle, deg
			Alpha	Beta	Gamma				
4-PMA	O	lath	1.492	1.668	1.736	58	obtuse	(−)	parallel = 0
MDA	M	lath	1.472	1.690	1.747	48	obtuse	(−)	7 front
3-MMDA	M	thin platelet	1.450	1.725	1.732	16	optic normal	(−)	18 front
DOM/STP	O	needle	1.440	1.710	1.732	28	acute	(−)	parallel = 0
DOB	T	thin platelet	1.481	1.712	1.748	42	inclined obtuse	(−)	30 front
TMA-1	T	thin platelet	1.446	1.728	1.740	18	inclined obtuse	(−)	42 front
Mescaline	O	thin platelet	1.547	1.683	1.697	33	optic normal	(−)	parallel = 0
MDMA	M	small prism	1.585	1.635	1.735	64	acute	(+)	39 front
MDEA	M	thin platelet	1.465	1.644	1.683	45	inclined obtuse	(−)	24 side

[a]The author acknowledges and expresses his appreciation for the following gifts of psychedelic amphetamines from the National Institute on Drug Abuse (NIDA), Rockville, MD: 3,4-methylenedioxyamphetamine (MDA); 2-methoxy-4,5-methylenedioxyamphetamine (2-MMDA); 3-methoxy-4,5-methylenedioxyamphetamine (3-MMDA); 4-methyl-2,5-dimethoxyamphetamine (DOM/"STP"); 4-bromo-2,5-dimethoxyamphetamine (DOB); 4-methoxyamphetamine (4-PMA); 3,4,5-trimethoxyamphetamine (TMA-1); 2,4,5-trimethoxyamphetamine (TMA-2); 3,4-methylenedioxymethamphetamine (MDMA); 3,4-methylenedioxyethamphetamine (MDEA) (= MDE); and mescaline (3,4,5-trimethoxyphenethylamine as the acid salts).

[b]O = orthorhombic; M = monoclinic; T = triclinic crystal systems.

[c]Determined by the method of Hartshorne and Stuart (Ref *16*, pp. 391–394).

TABLE 2—*Dilituarate derivatives psychedelic amphetamines.*

Amphetamine	Description
	PLATELETS
Mescaline	very thin "curved" tablets; 1st- to 2nd-order polarization colors; 4-sided, rectangular crystals; parallel extinction
3-MMDA	very thin tablets; 1st- to 2nd-order polarization colors; 8-sided, rectangular crystals; 18° extinction angle
MDEA	very thin tablets; 1st- to 2nd-order polarization colors; 6-sided, rectangular crystals; parallel extinction
TMA-1	very thin tablets; 1st- to 2nd-order polarization colors; 2 V = 18°; 6-sided, rectangular crystals; 42° front face extinction
DOB	very thin lath-like platelets; 1st- to 2nd-order colors; 2 V = 42°; 6-sided, rectangular crystals; 30° front face extinction
	ACICULAR (NEEDLE)
DOM/STP	stellate conformation; twinned on 010
	LATH TYPES
MDA	curved, needle-like lath crystals; 7° extinction angle on front face
4-PMA	lath-like crystal; orthorhombic classification with parallel extinction on front face
	PRISMATIC
MDMA	small prisms; the only crystal with a positive (+) optic sign

they appeared suspended in water on a microscope slide. First- and second-order polarization colors characteristic of very thin, tabular platelets are found in mescaline, 3-methoxy-4,5-methylenedioxyamphetamine (3-MMDA), 3,4-methylenedioxyethamphetamine (MDEA), 3,4,5-trimethoxyamphetamine (TMA-1), and 4-bromo-2,5-dimethoxyamphetamine (DOB) samples. Notable optic axial angles (= 2 V) are found in 3,4-methylenedioxymethamphetamine (MDMA) (= Ecstasy) = 64°; TMA-1 = 18°, and 3-MMDA = 16°. The only crystal with a positive optic sign is MDMA.

Two psychedelic amphetamine drug chemicals did not form crystalline derivatives for study. These compounds are 2-methoxy-4,5-methylenedioxyamphetamine (2-MMDA) and 2,4,5-trimethoxyamphetamine (TMA-2).

Other descriptions of the crystals used to characterize them are found in Tables 1 and 2 and in Figs. 1 and 2.

Summary and Conclusions

A procedure for the preparation of the dilituric acid (5-nitrobarbituric acid) derivatives of some psychedelic amphetamines suitable for optical crystallographic and microcrystalline study has been presented. The optical crystallographic properties of the nine crystalline derivatives studied serve as a means of identification and confirmation of these psychedelic drug chemicals.

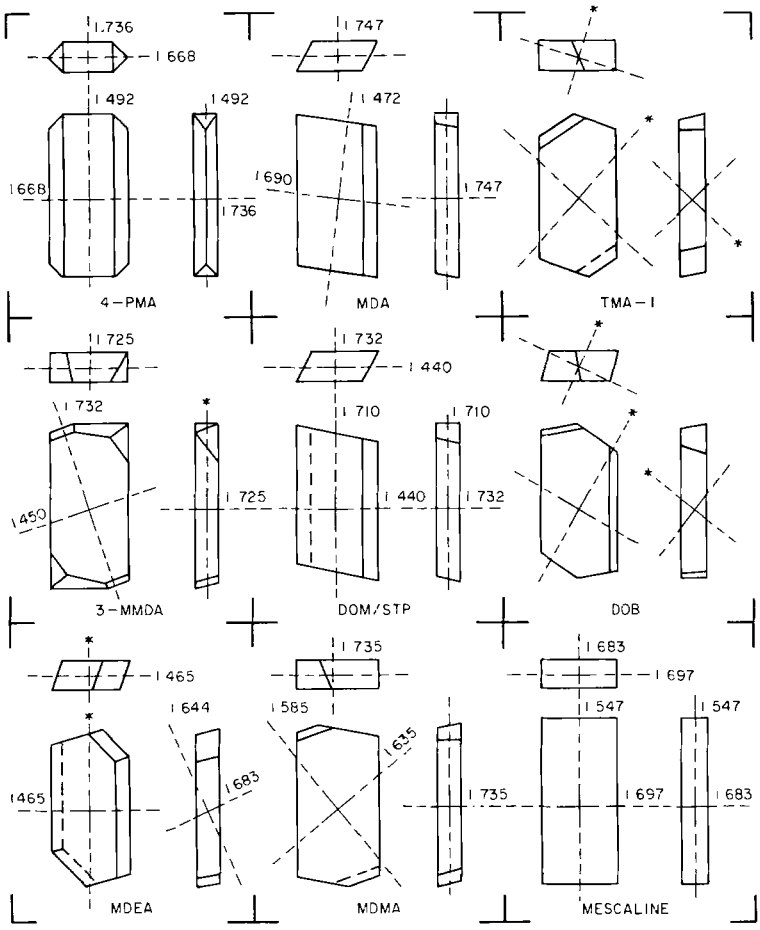
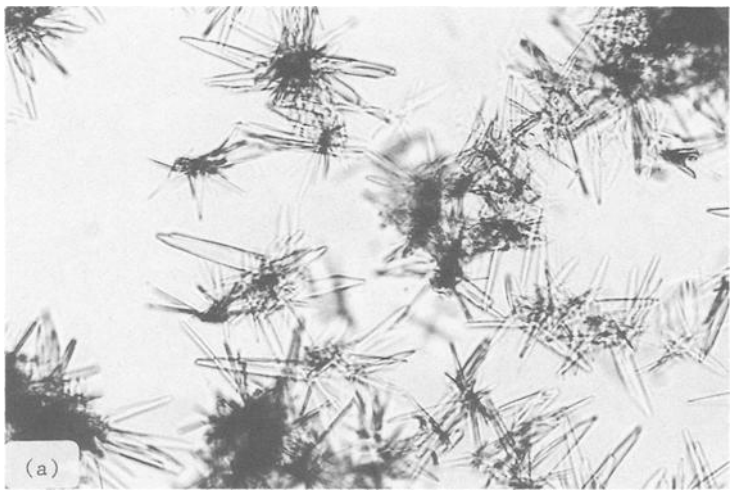


FIG. 1—*Psychedelic amphetamine dilituratcs.*



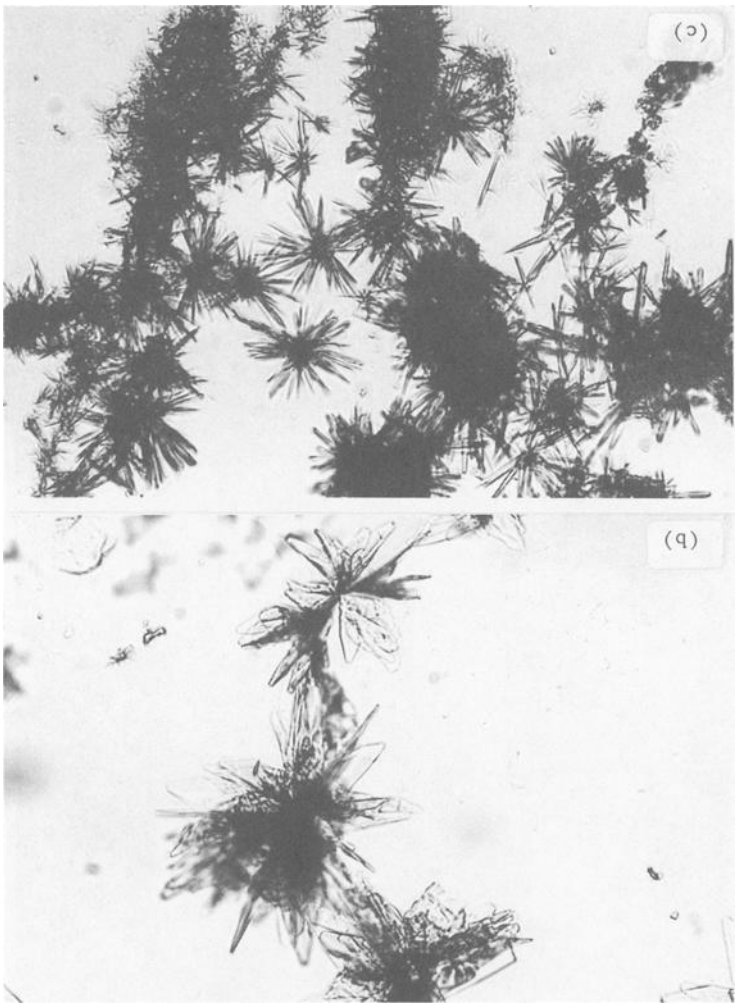


FIG. 2—*Continued:* (b) *3-MMDA (6 V/1 s);* (c) *STP (8 V/0.5 s).*

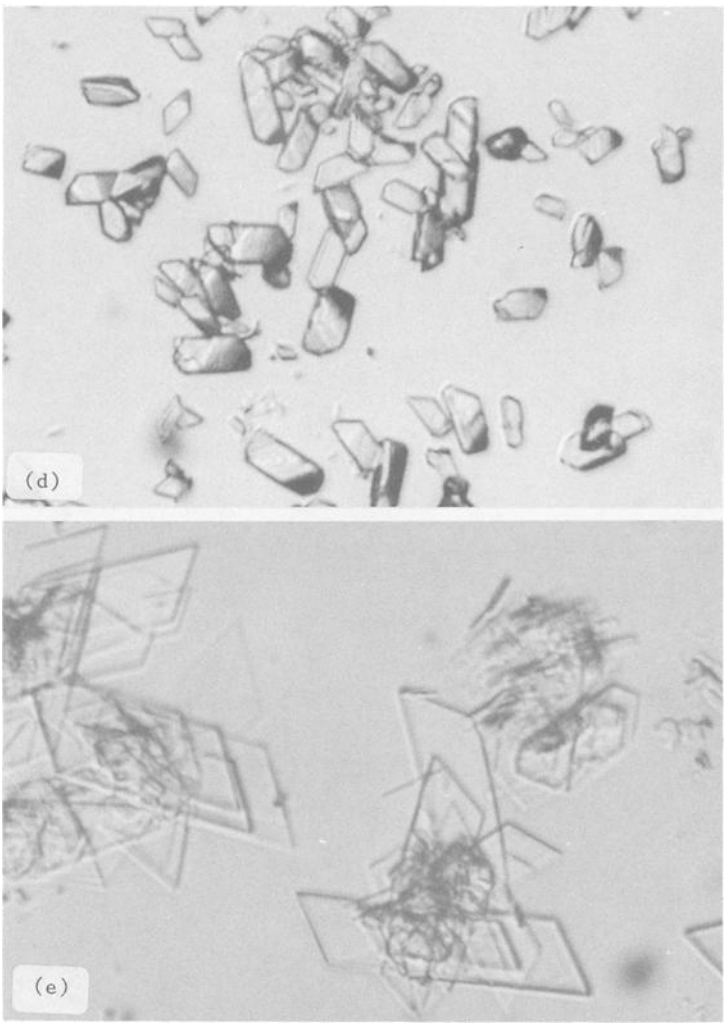


FIG. 2—*Continued:* (d) *MDMA (Ecstasy), small prisms (6 V/0.5 s);* (e) *MDEA (EVA) and MDE, thin platelets (6 V/0.5 s).*

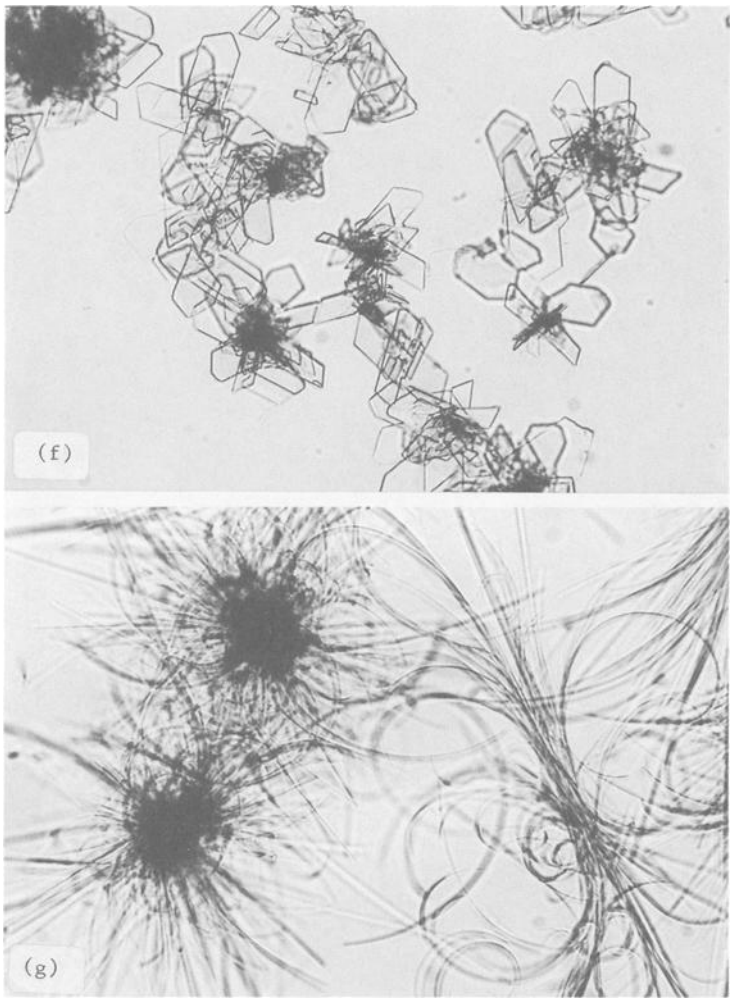


FIG. 2—*Continued:* (f) *TMA-1 (7 V/0.5 s);* (g) *DOB (6 V/1 s).*

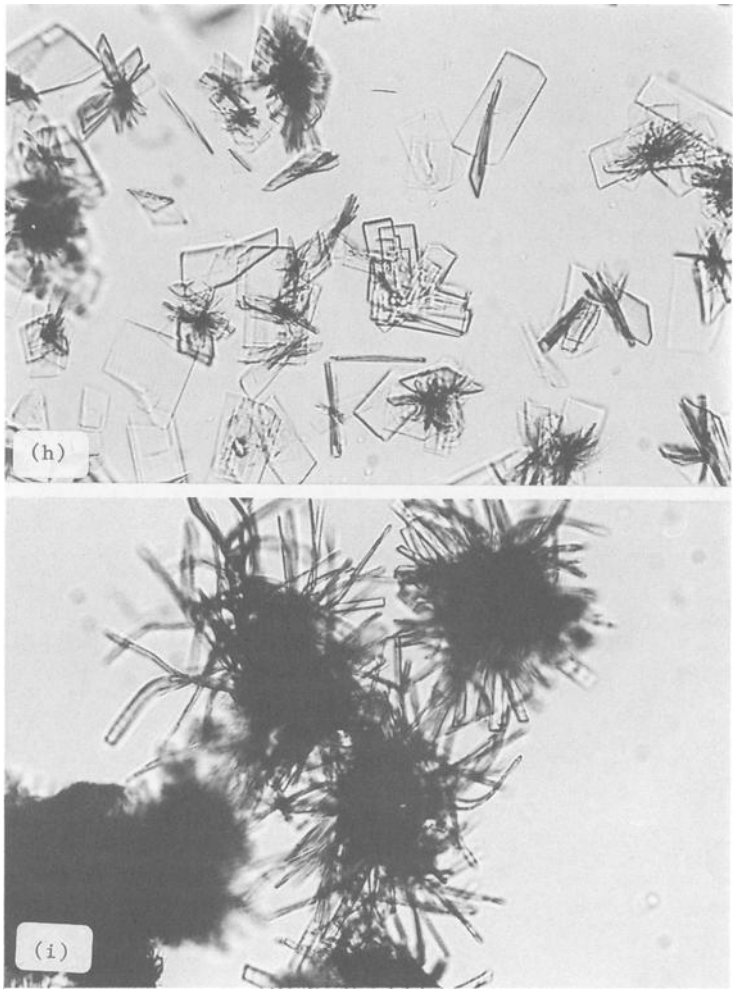


FIG. 2—*Continued:* (h) *mescaline, thin platelets (6 V/0.5 s);* (i) *4-PMA (7 V/0.5 s).*

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Address requests for reprints or additional information to
Edward A. Julian, Ph.D.
University of Wyoming
School of Pharmacy
Box 3375
Laramie, WY 82071-3375