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Identification of Drug Tablet and Capsule Evidence as to Source

The identification of the manufacturing source or common source of drug tablets and capsules in illicit channels provides intelligence on the diversion of commercially made products. It also provides information on the source and distribution pattern of counterfeit and other illicitly made products. Identification is made by comparison of the punch marks and formulation of unknown samples with those of authentic samples from known manufacturers [1,2]. This technique, in the Drug Enforcement Administration (DEA), has come to be known as the "ballistics" method of drug identification.

Tablet Identification

Tablets suspected of coming either from commercial manufacturers or from clandestine sources are submitted for a variety of reasons to the Drug Enforcement Administration's Special Testing and Research Laboratory for examinations. Here they are compared with tablets in a reference collection, which consists of more than 7000 authentic samples. Samples of commercially made controlled drugs are collected by DEA agents from manufacturers, together with batch records which, among other facts, contain information as to the formulation and type of punches used. In the laboratory these authentic samples are catalogued and arranged for ready reference and comparison in groups having related physical characteristics. Since an authentic sample usually cannot be obtained from a clandestine laboratory, the first exhibit received from a previously unknown set of punches becomes the reference sample.

Tablets are made by mixing the active ingredient with a combination of excipients, including diluents, binders, lubricants, disintegrators, colors, and flavors. Formulations vary considerably in ingredients from one manufacturer to another. For example, the sugar used may be sucrose, lactose, or dextrose, or combinations of these; any of a number of starches may be used, such as corn, wheat, potato, arrowroot, etc; and, in addition, calcium sulfate, calcium phosphate, or calcium carbonate, or other excipients, may be used.

The mixture is prepared for the tablet press, where the tablets are compressed between two punches in a die. The simplest type of tablet press is the single-station machine, having only one pair of punches, and capable of punching 100 tablets per minute. Most manufacturers who produce tablets in large quantities use rotary presses with 8, 16, or more pairs of punches.

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Many types of punches are used in tablet manufacture. In outline they (and the matching die cavity) may be round, oval, oblong, square, triangular, pentagonal, or other shape, and may be obtained in several sizes. The faces of the punches may be flat, concave, or convex; they may be unscored, single-scored, or double-scored; they may have an embossed or engraved trademark or design; they may have edges that produce a flat bevel, round bevel, or no bevel at all. Further, each of these types includes variations, such as the degree of curvature of the concave or convex faces, bevel angle, groove or score angle, or width and depth of groove.

Any deviation of the punch faces from a smooth flat surface will be shown in reverse on the surface of the tablets. Punches, even when new, may show characteristic microscopic marks which can be used to identify them. As the punches are used, they develop characteristic shapes, marks, or imperfections which are pressed into the tablets, and serve to positively identify a tablet with a given manufacturer's punch. These imperfections may be brought about by uneven wear in normal use or by carelessness in use and storage of the punches. Rough handling of the punches may cause characteristic scratches, pits, or ridges on the faces, which will be matched in reverse on the tablets. Sometimes punches are carelessly reworked, and the marks of the grinding tool or file are left on the punches and show up on the tablets as straight or variously curved striations.

If a single-station press is used, each tablet will have identical microscopic punch marks. In a multiple punch set, each punch has its own set of punch marks, so that a batch of tablets from a multiple station tablet press will have 8, 16, or more sets of punch marks. Evidence as to the number of punches used for making tablets of clandestine origin is helpful in determining the type of tableting and associated equipment, and hence the probable size and output of the clandestine laboratory.

The first step in tablet identification is the examination of the tablets with respect to the "gross punch marks." This examination includes the measurement of tablet diameter and thickness and the determination of weight, tablet shape, color, type of score marks, bevel, surface contour, presence of embossed or engraved monogram, etc.

The second step is the examination of the tablet surfaces under a stereoscopic microscope to detect minute punch marks. Usually a magnification of $\times 10$ is sufficient. The next step in the process of tablet identification is measuring the groove angle and the width and depth of the groove. This is done by mounting a tablet so that a silhouette of the tablet can be projected and the groove angle measured with a protractor. The tablet can also be mounted with the groove vertical under a microscope equipped with a cross-hair ocular and a rotating stage calibrated in degrees.

The final step in the process is the identification of ingredients by means of optical-crystallographic methods [2-4], and the study of their microscopic characteristics. Each crystalline ingredient has its own set of peculiar and specific optical properties which serve to identify the compound, even in a tablet mixture. These properties include refractive indices, extinction angle, sign of elongation, optic sign, axial angle, etc. These may be determined for each crystalline substance present by mounting portions of the crushed tablet in a suitable series of calibrated refractive index liquids and examining them under a polarizing microscope with polarized light. Tables and files of optical data are then consulted to determine the identity of the crystalline compounds present [1, 2, 5-8].

In many cases, the optical-crystallographic method of identifying crystalline ingredients is supplemented by the use of microcrystal tests [1, 2, 5, 9, 10]. This technique is particularly useful when the amount of active ingredient is so small that it is difficult to characterize or detect by direct observation under a microscope. X-ray diffraction and other instrumental techniques also are frequently used to supplement microscopic methods.

The results of the microscopic analysis of the unknown tablets are then compared to the results of a similar analysis of authentic tablets from the suspected manufacturing source and to the manufacturer's batch record. When an authentic sample is found which is identical to the unknown tablets, the manufacturing source is established.

Capsule Identification

Capsule identification proceeds in somewhat the same way. It is less often successful than tablet identification, however, since there are fewer external characteristics that can be determined and there are usually fewer ingredients used. The capsule size, shape, color, and weight are determined, and any characteristic markings or locking features are observed. Some manufacturers use capsule shells with distinctive shapes, such as bullet-shaped capsules (Eli Lilly & Co., Indianapolis, Ind.) and "taper-end" (truncated) capsules (Smith, Kline & French Laboratories, Philadelphia, Pa.). In the case of timed distintegration (TD) capsules, the external characteristics of the pellets are determined, such as glossiness, color, size, shape, mottling of color patterns, lumpiness of surface, etc. All of these characteristics, as well as identity and microscopic habits of ingredients, are used in the identification procedure.

Tracing Drugs to Point of Diversion

The ballistics method of tablet identification is often used in tracing tablets back to the point of diversion from legitimate commerce. In one investigation, amphetamine tablets which were appearing in large quantities in illegal channels were identified as the product of Manufacturer A. Batch sheets from that firm showed that these tablets were made for Distributor B. Examination of the distributor's shipping records showed that unusually large sales were made to Obesity Clinic C. Investigation by DEA special agents showed that these amphetamine tablets were being sold illegally by the medical practitioner who was operating the clinic. Shipping records of the distributor documented the sales of tremendous quantities of amphetamine tablets to this clinic.

In another recent case, the punch marks of unknown amphetamine tablets could not be matched to those of any tablets in the reference collection. The formulation, however, (containing anhydrous calcium sulfate, lactose, and cornstarch) was known to be used by only two drug manufacturers in the United States, one of whom was located in a city near the place where the unknown tablets were being sold illegally. Investigation at this manufacturer's plant revealed that a new set of punches was being used, which produced tablets matching the unknown tablets. Further investigation showed that the tablets in question were being stolen by an employee of the manufacturer and sold on the street.

Clandestinely Manufactured Drugs

The same laboratory techniques are used to detect counterfeit tablets. The suspected tablet is compared both as to external characteristics and identity of ingredients to the authentic samples of the genuine product, and to the data furnished by the legitimate manufacturer regarding the punches and formulation used. Counterfeit³ or imitation

³The Controlled Substances Act, Sec. 102 [7] states: "The term counterfeit substance means a controlled substance which, or the container or labeling of which, without authorization, bears the trademark, trade name, or other identifying mark, imprint, number, device, or any likeness thereof, of a manufacturer, distributor, or dispenser other than the person or persons who in fact manufactured, distributed, or dispensed such substance and which thereby falsely purports or is represented to be the product of, or to have been distributed by, such other manufacturer, distributor, or dispenser."

drug products are detected by their deviations from the genuine in external characteristics or combination of ingredients. For example, one counterfeit was detected because the arrangement of letters in the monogram engraved on one face differed slightly from that of the genuine product. Also, the counterfeit tablets contained sucrose and cornstarch, while the genuine tablets contained lactose and potato starch as excipients. Another counterfeit was detected when it was found to have a much larger groove angle than the genuine and was discovered to have been made on a single-station press, rather than on the multiple-station press used to produce the genuine.

Products of clandestine laboratories, whether they are counterfeits or imitations of legitimate drugs or illegal hallucinogens such as LSD, PCP, etc, may sometimes be recognized because of their crude or unusual appearance (see Figs. 1-8). Often they are made with a single-station press, resulting in each tablet of a batch having identical microscopic punch marks. Tablets with unusual shapes resembling space capsules (Fig. 6), pentagons (Fig. 8), mushrooms (Fig. 7), pumpkin seeds, etc, have often been encountered from these laboratories. Frequently the fillers, or diluents, used are readily available materials, such as dried skim milk or various forms of calcium carbonate.

Sometimes caffeine dosage forms, closely resembling well-known brands of amphetamines in color and shape, are encountered. These may even have the name or monogram of the legitimate manufacturer engraved or imprinted on the tablet or capsule.

Drugs of clandestine origin are compared in the DEA laboratory with reference samples of similar type which have been retained in the tablet library as prototypes from earlier investigations, in order to determine whether they come from a common manufacturing source. In cases where tablet punches are seized from clandestine laboratories, microscopic marks on the punch faces are compared with punch marks on tablets suspected to have been made with that set of punches. If possible, placebo or blank tablets are made with these punches, using microcrystalline cellulose or other suitable granulations. If sufficient pressure is used, smooth, glossy tablets can be produced that are a replica of the punch surface. These placebo tablets are more readily compared with the suspect tablets than are the punches themselves.

Compilation and Use of the Data

To make the data derived from the ballistics examinations of use to both the enforcement officer and the intelligence analyst, they must be compiled into some meaningful form. This is done by the Investigative Services Section of the Scientific Services Division in the Office of Research and Technology at DEA headquarters. All reports of ballistics examinations are sent to that section, where a trained scientific intelligence coordinator evaluates them.

In all instances, the laboratory chemist supplies the key for compilation. For example, the results of examination may show that the unknown has unique characteristics that have not been seen before; therefore, the exhibit becomes a new ballistics prototype in the library of the laboratory. In other cases, the expert states that the tablets are identical to a prototype examined in the past. Using these and similar statements from the report, the professional at DEA headquarters keys the information to a set of tables. Each table consists of all known submissions from seizures and purchases of tablets that have been made with a common set of punches. Thus, each table is an abstract of all that is known in the scientific area about that source. It is also an extremely valuable source of leads to investigative files, where additional information is contained.

If the scientific intelligence coordinator agrees that the tablets in question belong on a particular table, all of the major facts about the particular submission are entered into the computer. This generates a printout of the updated table, which is returned to the submitting office with any other known information about the event. In all special cases,

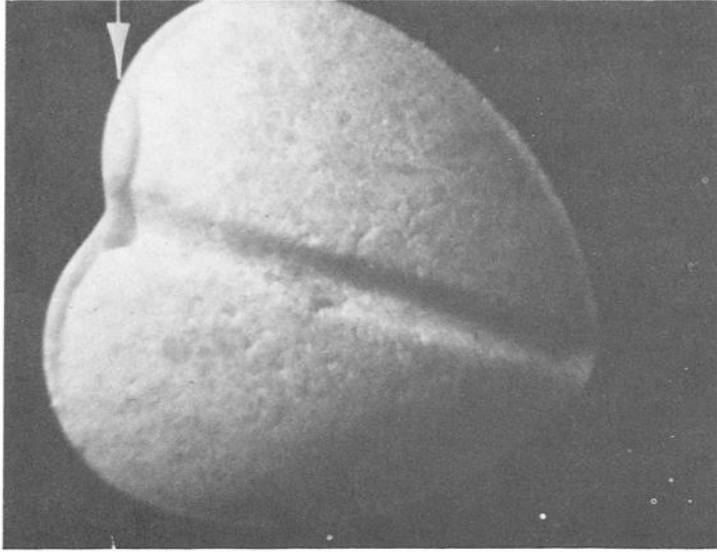


FIG. 2—Amphetamine sulfate "mini-heart" showing lump at edge ($\times 10$).



FIG. 1—Amphetamine sulfate "mini-bennie" showing gouge at edge and tiny lumps in groove bottoms. ($\times 10$).

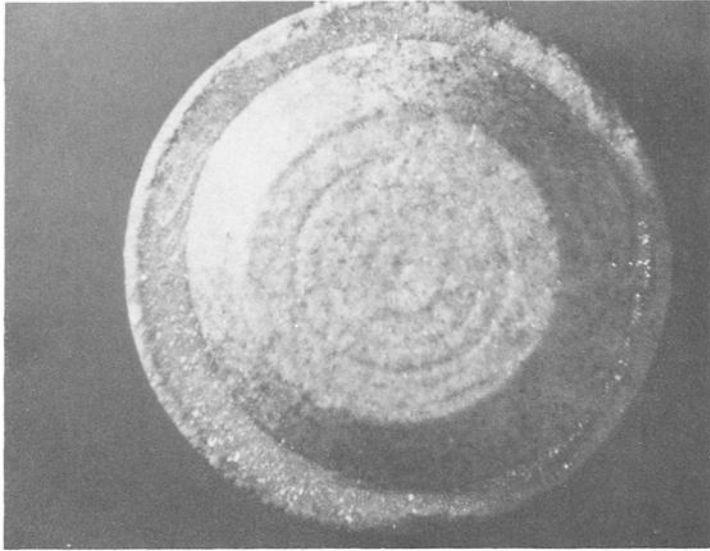


FIG. 4—LSD tablet containing brass particles and showing concentric punch marks ($\times 10$).



FIG. 3—LSD "peace pill" with many punch marks (lumps and ridges) on surface ($\times 10$).

a report is telephoned to the agent as soon as the table has been identified and additional pertinent information is collected.

Computerization of the ballistics data permits many different compilations to be routinely printed and distributed to enforcement and intelligence offices. For example, an alphabetical list of names of suspects and a listing by case file number are prepared. Frequency tables are printed by state and drug, which helps in the evaluation of distribution. Other listings can be produced routinely or on request. A special query capability is being programmed to permit interrogation by various characteristics. For example, an analyst may want a listing of all amphetamine tablets 15/64 in. in diameter encountered in the state of California. This will be readily done by the computer.

Detailed evaluation of the ballistics tables can reveal considerable information. For example, whether the tablets were made with a single- or multiple-punch set gives a rough measure as to the potential of the sources. The length of time the source has been known to DEA, and the number of encounters and their frequency, also provide a clue as to size of the operation. It also shows the spread of the market distribution (that is, intrastate, national, or international), as well as the development of that market in time. Suspects' or defendants' names are listed, as is the sales price of the drug. The latter permits market analysis, which helps to zero in on the probable geographical source. Evaluation of the potency data and kinds and relationships of the diluents permits an indication of the number of batches, which, with other information, also helps to indicate the size of the operation.

Packaging

Finally, among other information, packaging is an important source of data. Large numbers of tablets at the wholesale level may be packaged in a particular manner, while smaller quantities at the retail level may be packaged in another way.

Millions of "mini-bennie" amphetamine tablets have been smuggled into the United States at the Mexico-California border. These are usually packaged about 25,000 tablets in black plastic "pillows" or "kegs" (depending on their size and dimensions), sometimes in clear plastic, and are heat-sealed with one kind of heat-sealer at the bottom and another kind at the top. Later these tablets appear in various parts of the country sold in 1000-tablet amounts in small clear plastic bags. Occasionally, 1000 tablets appear in one of the large black plastic bags, obviously the last of the 25,000 tablets from that bag.

In recent months both ephedrine and caffeine tablets having the same appearance as the "mini-bennie" amphetamines have been encountered. These are also packaged at wholesale in black plastic like the amphetamines. And, in the case of caffeine tablets from one set of punches, they commonly appear, particularly in one part of the country, with "mini-bennie" amphetamine tablets made with another set of punches. They may or may not be packaged together.

Using data similar to the foregoing, compilations were made of ballistics data and information in case files and elsewhere which indicated that a father, his son, and his daughter-in-law were involved. The ballistics compilations showed the family to be involved with sales of amphetamines and secobarbital listed on four tables. The end result was that one major distributor of drugs smuggled into the United States was put out of business.

Another case involved secobarbital capsules. Compilations were made of the various kinds of capsules being smuggled into the United States in large numbers. Using this ballistics information, plus information compiled from case files and elsewhere, DEA compliance officials working with Mexican officials located a modern encapsulating operation. The plant was seized and, as a result, there has been a marked reduction in the quantity of secobarbital capsules being smuggled into the United States.

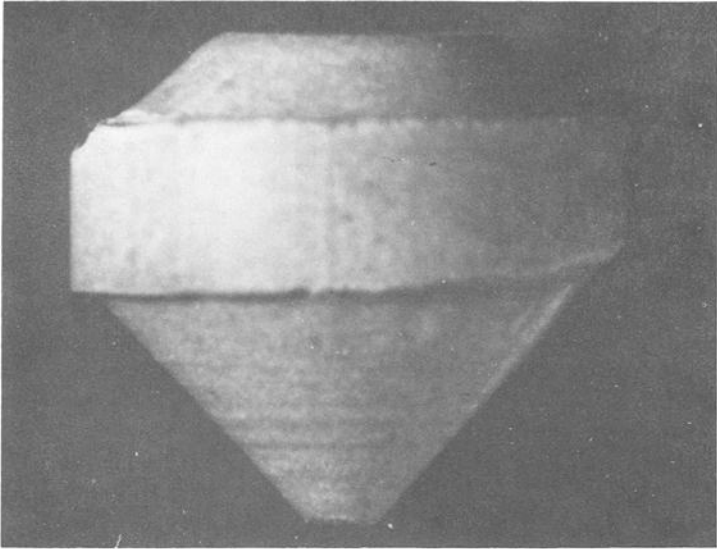


FIG. 6—LSD "space capsule" (X10).

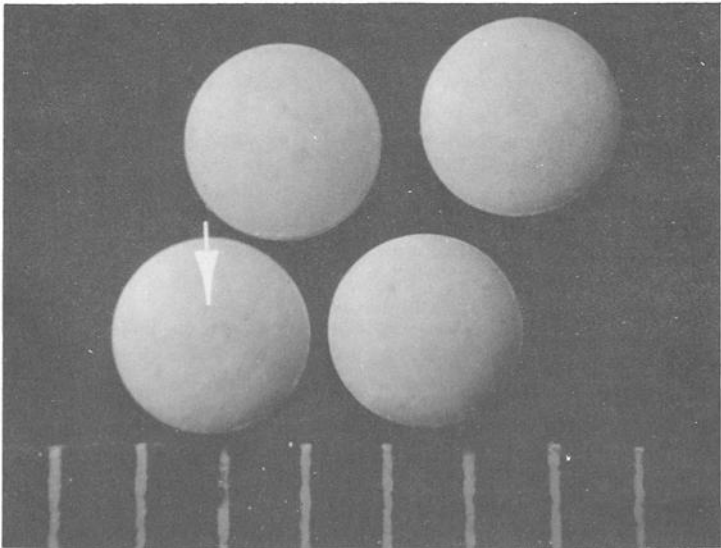


FIG. 5—LSD "micro-dois" showing dimple at center of face (X10).

In another case, an amphetamine resin complex was shipped in bulk to Mexico where it was encapsulated, and then large quantities of black capsules containing the drug were smuggled into the United States. Ballistics examination showed a 10-percent difference in filled weight of the capsules made domestically compared to those made in Mexico. Also, the radius of the curve at the bottom of the letter "J" in the capsule logo had a different radius than that of the domestically made product. Compilations of ballistics data, together with information from the compliance investigation, proved the smuggling operation. As a result, the firm lost its license to export amphetamine.

Tablets and capsules are submitted to the Special Testing and Research Laboratory from foreign, federal, state, and local agencies for ballistics examination. This provides a large data base, a necessity for accurate evaluation of the data. Reports of results of examination are always sent to the submitting agency, with copies of the reports going to local DEA offices. This permits an exchange of information between the two enforcement offices.

In one instance, DEA was looking for a young lady who had jumped \$10,000 bail on a drug charge. Ballistics examination had been done on tablets sold by the defendant. A short time later, tablets were submitted for ballistics examination by a state police office. The suspect's name was the same. DEA's field office was notified and the defendant apprehended in a joint DEA-state action.

Although the laboratory identifies about four or five new ballistics prototypes each month (mostly involving LSD tablets), tableting machines and their punches are also frequently seized.

In one case our agents working with Mexican officials seized a 33-station tableting machine capable of making several thousand tablets per minute. Placebo "authentic" tablets were made, and examination showed that the tablets were a new ballistics prototype. We learned that the laboratory had only been in production about two weeks, and the machine had been adjusted to make only about 1100 tablets per minute. Shortly thereafter, the DEA laboratory identified tablets from that set of punches in submissions coming from a number of country-wide locations. In the next two months, ballistics submissions indicated that about 2 million tablets had been made on the machine before it was seized.

At present, DEA has compilations on over 200 sets of punches involved in clandestine drugs manufacture. Almost all of the punches have been used in the past two years. About 70 have been active—some quite active—in the past six months. Six of these are punches used in the production of "mini-bennie" amphetamines, and the remainder in producing LSD, PCP, etc. In the near future, all of the computerized ballistics tables and the various indices will be made available periodically to DEA regional offices and laboratories. Later, use of computer terminals or microfiche will be at the disposal of investigating agents and intelligence analysts.

We are exploring the possible use of computer-assisted identifications. This will permit a local laboratory to use its terminal to presumptively identify a tablet and learn something about the possible source. For example, this will involve having the DEA drug tablet reference library on computer-retrievable holograms, with light pens and split-screen cathode ray terminals (CRT) in the local laboratory. Half of the screen would show the unknown, with both the light pen and the terminal being used to input data. Computer "strikes" could then appear on the other half of the CRT screen, permitting comparison of characteristics by the searcher until he found a "match." For court testimony, of course, the tablets would have to be submitted for positive comparison with known tablets.

This, or a similar kind of system, if it works, would be of benefit to local police agencies and would bring new tablets to DEA's attention more quickly than at present. Conceivably the system, with modification, could be used to help the nation's poison control centers, thus using existing law enforcement data for additional humane

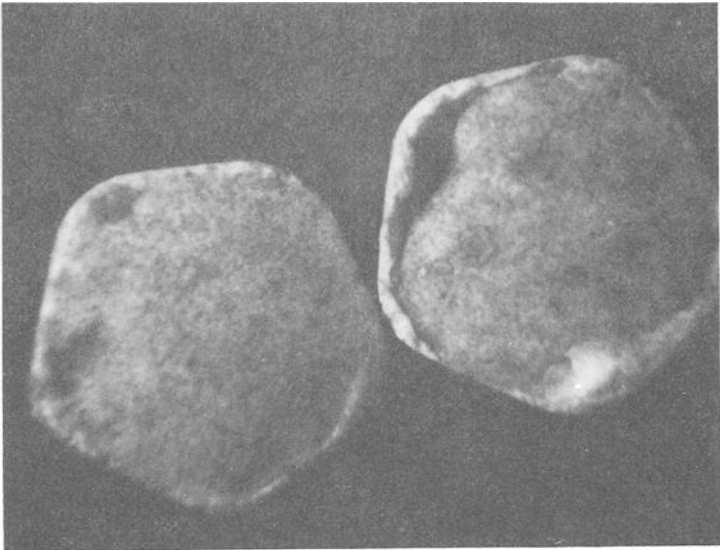


FIG. 8—LSD "pentagon" showing both faces (x10).

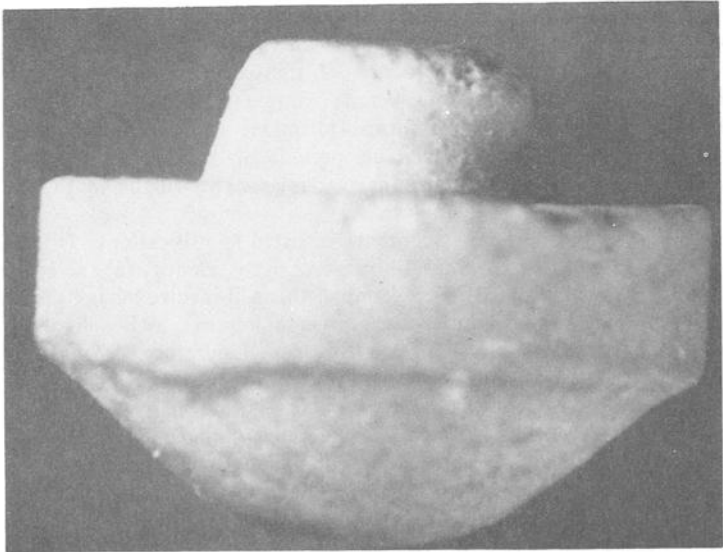


FIG. 7—LSD "mushroom" (x10).

purposes, plus making the cost-effectiveness ratio much more favorable for the taxpayers.

Conclusion

The ballistics program uses the classic toolmark examination with microchemical and macrochemical analysis of drug tablets and capsules to identify those from a common source. Computerization of the data provides a new tool for the enforcement officer and intelligence analyst in their efforts to stop the supply of illicit drugs.

References

- [1] Eisenberg, W. V. and Tillson, A. H., "Identification of Counterfeit Drugs, Particularly Barbiturates and Amphetamines by Microscopic, Chemical, and Instrumental Techniques," *Journal of Forensic Sciences*, JFSCA, Vol. 11, 1966, pp. 529-551.
- [2] *Microscopic-Analytical Methods in Food and Drug Control*, U.S. Department of Health, Education and Welfare, Food and Drug Administration, Washington, D.C., 1960, Chapters X and XI.
- [3] Bloss, F. D., *An Introduction to the Methods of Optical Crystallography*, Holt, Rinehart and Winston, New York, 1961.
- [4] Hartshorne, N. H. and Stuart, A., *Crystals and the Polarizing Microscope*, 4th ed., American Elsevier Publishing Co., New York, 1970.
- [5] *Official Methods of Analysis of the Association of Official Analytical Chemists*, 11th ed., Association of Official Analytical Chemists, Washington, D.C., 1970, pp. 708-721, 952-963.
- [6] Winchell, A. N., *The Optical Properties of Organic Compounds*, 2nd ed., Academic Press, New York, 1954.
- [7] Winchell, A. N., and Winchell, H., *The Microscopic Characters of Artificial Inorganic Solid Substances*, 3rd ed., Academic Press, New York, 1964.
- [8] Eisenberg, W. V. and Schulze, A. E. in *National Formulary*, 13th ed., American Pharmaceutical Association, Washington, D.C., First Supplement, 1970, pp. 1041-1056 and Third Supplement, 1972, p. 1109.
- [9] Fulton, C. C., *Encyclopedia of Microscopy*, G. L. Clark, Ed., Reinhold Publishing Corp., New York, 1961, pp. 65-72.
- [10] Fulton, C. C., *Modern Microcrystal Tests for Drugs*, John Wiley & Sons, New York, 1969.

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