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Barbiturate Differentiation by Chemical Microscopy

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BARBITURATE DIFFERENTIATION BY CHEMICAL MICROSCOPY

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Of the crystal tests currently in use for the identification of narcotics, it would appear from the literature that many of them were developed as a result of research in toxicology, where they have been largely supplanted by spectrophotometric and other advanced methods. Such tests as have been developed, however, retain their reliability and utility for the qualitative analysis of confiscated drugs, and are included in standard texts within this field. Additional information is accessible to the worker, in bulletins and manuals dealing specifically with the narcotic problem, and the criminalist who is generally familiar with the subject of chemical microscopy has no difficulty in finding suitable tests for these materials.

With respect to the "dangerous drugs," however, a somewhat different situation exists. Many of the drugs currently on the market (and encountered in the drug traffic) are not narcotics, but are nevertheless controlled by various drug laws. Possession of hypnotic and stimulant drugs without a prescription may be a violation, and the criminalist is often called upon to identify an unknown pill, capsule, or powdered material of this type. Of these, the barbiturates are perhaps the most common. An examination of the usual texts in criminalistics, however, reveals little in the way of new information or tests for such materials. For the most part they include little more than the standard color reactions, melting point data, and related information adapted from earlier works. While considerable work has been done on the barbiturates with paper chromatography, and the spectrophotometer, these methods are not well suited to the rapid identification and differentiation of individual barbiturates. Although spectrophotometric, chromatographic, or melting point and color-test methods may identify the barbiturates, such procedures cannot compare with the simplicity of the usual crystal tests for the narcotics.

It is true that crystal tests have been developed for a number of the barbiturates, but a limited review of the literature fails to indicate any general scheme or procedure applicable to the barbiturates as a group, and capable of differentiating between them. Believing the need for such a procedure to exist, this writer began in February, 1960, an investigation into certain crystal reactions which seemed best suited to the solution of the problem. As a result of work performed, a simple procedure has been worked out which has thus far proved most encouraging, and which has permitted the identification and differentiation of most of the barbiturates tested. Research on this project resulted in the formulation of a new silver reagent which is particularly well suited to barbiturate testing, and which represents the most useful reagent of the series.

Basically, the recommended procedure consists of the following tests.

First apply Koppanyi's test.[1] If positive, utilize remaining tests.

(1) Dissolve a small amount of the material in 2% potassium hydroxide solution (KOH), then add syrupy phosphoric acid to precipitate the drug.

(2) Dissolve a small amount of the material in concentrated sulfuric acid, then add water to precipitate.

(3) Test with Wagenaar's Reagent (5% aqueous copper sulfate plus sufficient ethylenediamine to give a purple liquid).

(4) Test with author's reagent (10% aqueous silver nitrate to which is added 15% ethylenediamine by volume).

Tests (1) through (4) are performed on a microscope slide, using drop-quantities of the reagents.[2]

[1] Drug on filter paper, add drop of 1% cobaltous acetate in methanol and follow with 5% isopropylamine in methanol. Pinkish violet for positive reaction.

[2] Tests included here were selected primarily on the basis of capacity to differentiate between the greatest

In applying these tests, while purified (extracted or sublimed) drugs will often give more well-defined results, shavings from pills and tablets have proved generally satisfactory throughout. Starch, lactose, and other materials (including amphetamine) which may be present, seem not appreciably to interfere with the reactions. Where pure drugs are used, the material is best ground to a powder before testing; otherwise the crystal form of the original compound may confuse the determination.

A few comments on these tests are in order. First, one should keep in mind that materials other than barbiturates (hydantoins, for example) may react to the Koppanyi test, and to the crystal tests. Secondly, tests (1) and (2) are useful for testing many compounds where no standard tests are known, and reactions obtained here may be due to materials other than barbiturates. Finally, some organic compounds may react with or be precipitated by the ethylenediamine of tests (3) and (4). For these and other reasons it is essential that known standards be tested for comparison purposes when using these tests for the barbiturates.

It will be noted that test (1) is essentially the same as that often used for pentobarbital, viz., dissolving the material in dilute ammonia and precipitating with acetic acid. Tests (1) and (2), however, have the advantage that they may be allowed to stand uncovered for hours, if necessary, without evaporating. Materials which do not give crystals in these reactions at first (secobarbital, e.g.) will often yield large crystals after a few hours time. Further, while crystal forms obtained with tests (1) and (2) are often alike, for a given barbiturate, they are at times quite different so that the two provide a double check as to the nature of the drug.

Composition of the silver reagent is critical. A 5% or a 20% silver concentration generally gives entirely different crystal forms. The 10% solution appears to have the greatest capacity to differentiate between the barbiturates, and was selected on that basis.

number of barbiturates. Supplementary reagents may be utilized should these fail to distinguish between two or more materials. Experiments indicate that a number of metallic-ethylenediamine reagents are potentially useful for this work (Pt for amytal; Tl for butisol, etc.) Thiobarbiturates have not been fully tested with this scheme or with the supplementary reagents, though it is noted that Mosidal gives excellent crystals with both Zn and Ni in ethylenediamine, either of which is better than the copper or silver reagents, for this compound.

Crystals formed in all of these tests are examined at a magnification of 100×. The use of a polarized-light microscope[3] is strongly recommended for examination of crystals formed in tests (1), (2), and (4). Tests (1) and (2) are particularly inclined to form oily mists or globules which may obscure the colorless crystals obtained, and gel-like precipitates of test (4) will in some instances interfere with the examination, should ordinary illumination be utilized exclusively.

Technique is particularly important in performing these crystal tests, and experimental testing of known materials is essential. Notation of the best technique or procedure should be made with respect to each of the barbiturates tested, and it will be found helpful to make supplementary notes as to whether crystals form rapidly or slowly, whether they form immediately or come out of an oily mist, etc.

Accompanying this material is a chart which illustrates the basic crystal forms, and conditions of precipitation, of a number of the barbiturates tested by the author. Sketches shown in the chart are provided only as a guide to the most "representative" crystal form obtained in each instance. With some of the barbiturates (especially in tests with the silver reagent), numerous additional crystal shapes are to be found, and the change in form of the crystals after standing may be important.

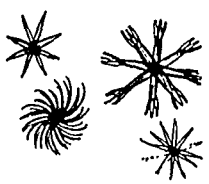
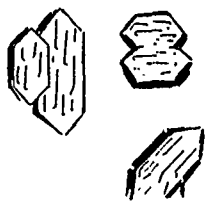
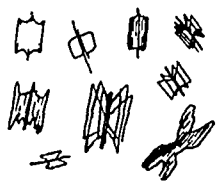
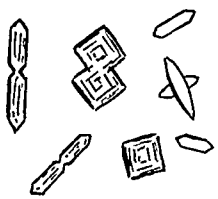
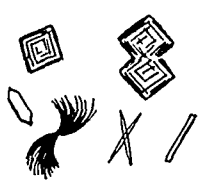
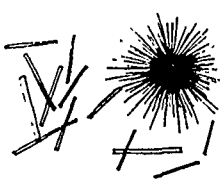
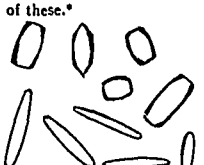
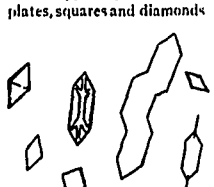
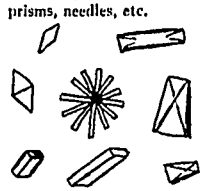
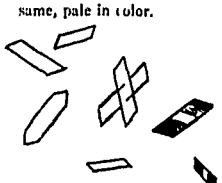
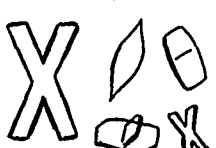
As to techniques of applying these tests, the following should be mentioned:

(a) In tests (1) and (2), test that amount of material which, when the precipitating reagent is added, will give a visible oily mist or crystal precipitate, but not so much that a dense cloudy precipitate is produced. If too small an amount of the drug is tested, crystals may not form, and if too much is added, they may be precipitated as excessively small or atypical forms. Use no cover glass on preparations.

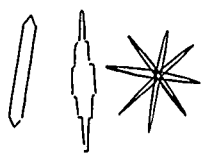
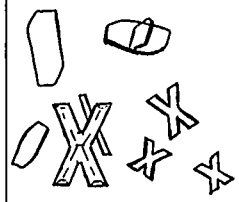
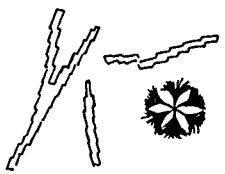
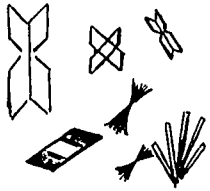
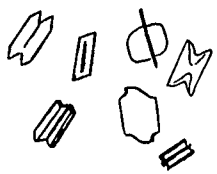
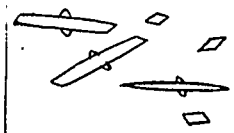
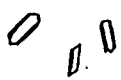
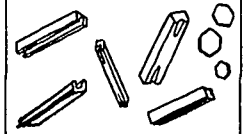
(b) In test (3), add a fair quantity of the dry powdered material to one edge of the reagent drop. If crystals do not form within 15 minutes, scratch the slide slightly or repeat using a larger amount of the drug. Cover glass may be used, though preferably left uncovered.

(c) In test (4) add generous quantity of the dry

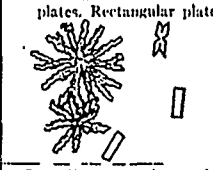
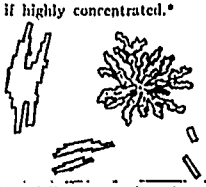
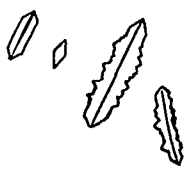
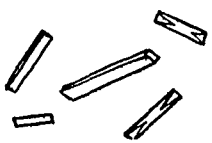
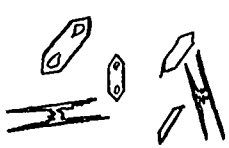
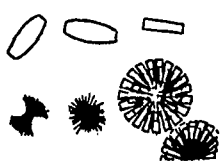
[3] See Technical Note. Volume 51, No. 4, page 489, of this Journal.

Barbiturate	Potassium Hydroxide & Phosphoric Acid (1)	Sulfuric Acid & Water (2)	Wagenaar's Reagent (3)	Davis' Silver Reagent (4)	Remarks on Silver Test (4)
A. ALPHENAL (Allyl phenyl barbituric acid)	Oily mist slowly gives long hex plates and aggregates of same. Branching groups of plates. Scratch slide to precipitate. 	Oily mist. Quick forming *Large* thorny radiates. Ends may branch or become brushy. If concentrated, dense curve-armed rosettes instead. 	With solid drug, amorphous ppt. With solution of drug, immediate large, thick hexagonal tablets with "etched" surfaces.* 	Amorphous ppt. with solid. With solution of drug, a cloudy ppt. and rapid-forming thin plates as "paddle-wheels," etc. Many become "etched" with parallel lines.* 	Forms variable with concentration. Use no cover glass on preparations. Large variety of shapes out of the silver reagent—all thin except for some spindle-shaped aggregates of needles which may accompany the plate forms. Some large radiates of spindle-shaped crystals may form. Note—Where solution of drug is utilized, the solvent is 15% ethylenediamine in water. Note—Sketches limited to the most "characteristic" or significant forms.
B. AMOBARBITAL ("Amytal"—Lilly)	Oily mist or globs from which quickly separate long hex plates, or square forms, often as twins. Etched.* 	As with KOH and phosphoric; plus curve-armed floating "frost" or ferny radiates. 	Quick-forming coarse needles or rods, isolated or in aggregates. Some fine needle-radiates. 	Use solid drug. Becomes gummy, but scratch slide or allow to stand with cover glass. Often *Slow* to form. Gives hex-ended spindles or "pillows." Some aggregates of these.* 	With silver reagent, often advisable to use concentrated drug, and cover with cover glass. To precipitate, lift corner of cover glass now and then and replace immediately. May take an hour or more at times for crystals to form.
C. APROBARBITAL ("Alurate", "Numal")	Oily mist or droplets. Scratch slide to ppt. Crystals are hex plates, squares and diamonds 	Use generous amount of drug. Oily mist from which form diamonds, rectangular prisms, needles, etc. 	Very slow forming. Scratch slide to get hex plates or parallelograms. Rosettes of same, pale in color. 	Crystals form rapidly, especially if scratch slide to initiate. Large X's and plates in various shapes.* 	X's and plates formed are quite similar to those of Butisol but are usually distinguishable. Let droplet dry and add more silver reagent; crystals will break down into small prisms. All crystals are large and thick in the initial silver test.

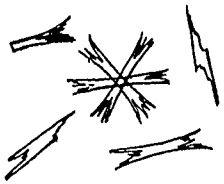
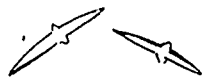
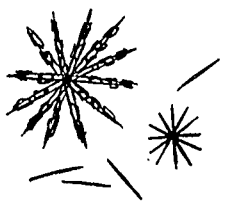
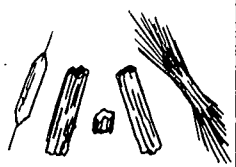
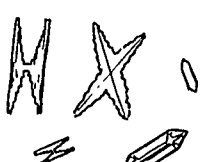
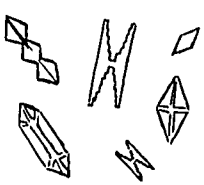
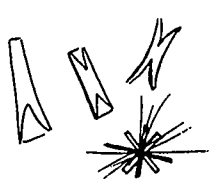
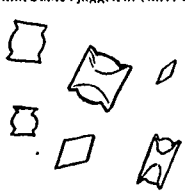
* Indicates best test(s).

Barbiturate	Potassium Hydroxide & Phosphoric Acid (1)	Sulfuric Acid & Water (2)	Wagenaar's Reagent (3)	Davis' Silver Reagent (4)	Remarks on Silver Test (4)
D. BUTABARITAL ("Butisol")	Immediate crystals as *Long* hex needles or prisms. Some rosettes or radiates of spindles. 	Immediate ppt. needles and spindles singly and in aggregates. May have brushy tips. Some minute rosettes. 	Slow forming. Scratch slide to initiate. Plates, diamond and hex forms. Rods and brushy spindles. 	Large thick X's and plates. X's forming twins and multiples. Plates with single tabular side-arm.* 	May be advisable to use cover glass on concentrated preparation and allow to stand until crystals form around particles of the drug regions. See Aprobarbital for similar. Butisol X's do not break down into small prisms though they may become granular. X's have sharper corners than those from Aprobarbital as a rule.
E. CYCLOPAL	Oily mist quickly gives ppt. of jagged-edged blades singly and in radiates. Some brushy rosettes.* 	Discolors acid, forms gel-like scum. No crystals within 15 minutes.	Difficult to dissolve. Stir and scratch slide. Rods and needles in radiates. Some thick prisms in aggregates. 	Variable with concentration from plates to tabular forms singly or as rosettes.* 	With silver reagent, advisable to use solution of drug rather than solid. Larger crystal forms. Crystals resemble other barbs, such as Seconal, Talbutal, Dial, Ipral, Nostal. With very concentrated solutions, aggregates of thick plates with fringes of small plates as arms.
F. DIAL (Di-allyl barbituric acid)	Use generous amount of drug. Scratch slide to ppt. crystals. Thin to thick hex plates singly and in aggregates. 	Use generous amount of drug. Scratch slide. Crystals are diamonds or long slant-ended rods with knob in middle. 	Apparently no reaction. If allowed to stand may get thick rods or rhombic prisms of questionable value. 	Dissolve drug in solvent to test. Immediate large square-ended rods with hollow ends. Singles and radiates, some hex plates.* 	With silver reagent, may be necessary to scratch slide to initiate crystal formation, but if scratched too much all crystals will precipitate at once in minute forms difficult to evaluate. Otherwise, very large rods.

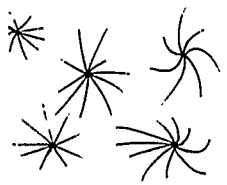
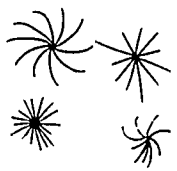
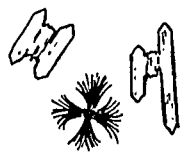
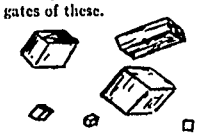
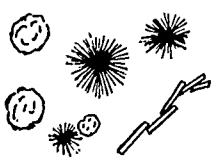
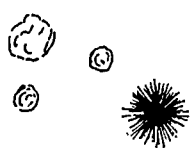
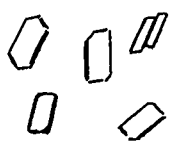
* Indicates best test(s).

Barbiturate	Potassium Hydroxide & Phosphoric Acid (1)	Sulfuric Acid & Water (2)	Wagenaar's Reagent (3)	Davis' Silver Reagent (4)	Remarks on Silver Test (4)
G. HEXOBARBITAL ("Evipal")	Immediate ppt. of oily mist and crystals. Crystals weedy floating masses of radiating plates. Rectangular plates.* 	Slowly gives crystals similar to test (1). Immediate crystals if highly concentrated.* 	Slow-forming crystals. Fine rods, some etched barrel-shaped plates. May not be a reaction product. 	No significant reaction. If using solid drug, forms amorphous mass. No crystals obtained.	Such crystals as are obtained in the ethylenediamine reagents appear due to the ethylenediamine rather than the metallic ions present.
H. PROBARBITAL ("Ipral")	Immediate ppt. of crystals as diamond and hex plates or long floating blades with jagged edges. 	Essentially same as those from test (1).* 	Slow forming. Scratch slide to ppt. Large rods and bars, slant, square or hex-ended. 	Use solid or a solution of the drug. Plates and tablets of various shapes grow large and thick. Distinctive forms.* 	Some of the crystals obtained with the silver reagent will resemble those with Seconal, Amytal, or other barbiturates, but many are obviously different from those drugs, especially when they have grown somewhat.
I. MEPHOBARBITAL ("Mebaral")	Oily mist quickly yields crystals of X's, "stag horns," etc., made up of small plates. Some mossy pads.* 	Slow to form crystals. Scratch slide. Gives small hex plates with hollow near ends. Big feathery X's *If* quite concentrated. 	No significant reaction.	Use cover glass as gives slow reaction. Burrs and needle radiates followed by barrel-shaped plates or perfect rosettes of overlapping plates.* 	Silver reagent is good but slow. Allow considerable time for growth of the plate rosettes. Some brushy-ended crosses may form. Barrel-shaped plates may resemble some Amytal crystals.

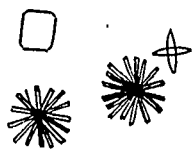
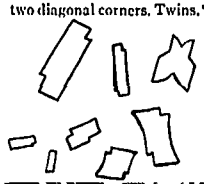
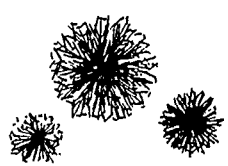
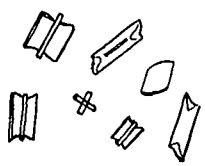
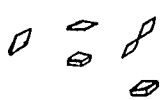
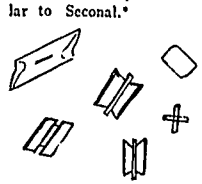
* Indicates best test(s).

Barbiturate	Potassium Hydroxide & Phosphoric Acid (1)	Sulfuric Acid & Water (2)	Wagenaar's Reagent (3)	Davis' Silver Reagent (1)	Remarks on Silver Test (1)
J. NARCONUMAL	Oily mist. Scratch slide to get *slow* forming fan-ended rods or plates or brushy radiates. 10 to 20 minutes. 	Oily mist. No crystals within 20 minutes, even if scratch slide.	Oily globs. If use solution of drug for test, may get blades at near-dryness. Question value. 	Use solid drug, with cover glass. Allow 30 minutes or more. From gummy masses may form small triangular plates singly or in circular rosettes.* 	If use a solution of the drug, will get "pillow-shaped" prisms, singly and in radiates. If these are allowed to dry and fresh silver reagent added, they will quickly change to the triangular plate form. (No cover glass.)
K. BUTETHAL ("Neonal")	Oily droplets. Later spindle-shaped needles or radiates of them. Jagged small plates on the needles.* 	Similar to (1), jagged-ended plates may get brushy ends. Slow forming from dilute solutions. 	Scratch slide to get square plates with rounded corners. Some prisms. 	Oily globs may form. Scratch slide once or twice to get striated or fluted rods which may grow "whiskers" to become brushy.* 	As with all of the crystals formed with the silver reagent, it is advisable to examine them over some period of time. Changes in form will then permit distinction between barbiturates which yield similar rods or shapes initially.
L. PROPALLYLONAL ("Nostal", "Noctal")	Mist and ppt. of crystals at same time. The crystals are jagged X or H shapes made up of smaller plates.* 	No appreciable mist or oily precipitate. The crystals form at once as X and H forms, plus diamonds or skeletal hex plates. 	Test a solution of the drug. Scratch slide. Rectangular rods or bars with flared ends. Some wispy needles. 	Use a solution of the drug, without cover glass. Slight oily mist followed by thin plates in varying shapes mostly with 2 sides parallel, and 2 sides jagged or convex.* 	A number of plates or tabular forms are obtained with the silver reagent, and growing quite large, paddle wheels, "pot-bellied squares" and rhombs being the most common.

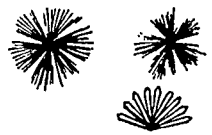
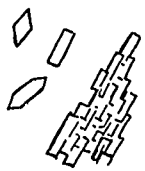
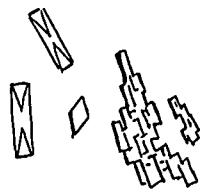
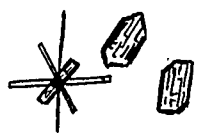
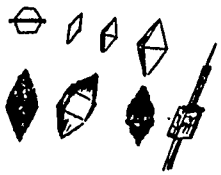
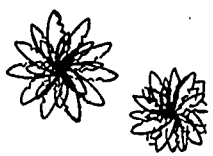
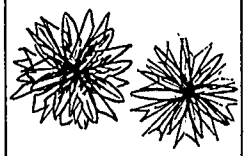
* Indicates best test(s).

Barbiturate	Potassium Hydroxide & Phosphoric Acid (1)	Sulfuric Acid & Water (2)	Wagenaar's Reagent (3)	Davis' Silver Reagent (4)	Remarks on Silver Test (4)
M. Hexethal ("Oital","Hebaral")	Heavy oily mist from which slowly separate radiates of wispy needles.* 	Similar to (1), straight or curve-armed radiates. 	Immediate crystals of hex plates in twins. Brushy radiates also. Variable. 	Oil drops or amorphous globs to gel-like. No significant crystals form for hours. Then possible rosettes of lenticular plates. 	Silver fails to give crystals within reasonable length of time. Given sufficient time and precipitation by scratching slide, crystals obtained somewhat similar to those given by phenobarbital.
N Pentobarbital ("Nembutal")	Dense mist or droplets slowly forming lens-shaped plates and/or weedy pads.* 	Essentially similar to test (1) crystals. Slow forming. 	Quick-forming coarse rods inclined to form bundles or barrel-shaped aggregates.* 	From granular ppt. slowly form near cubes or "calcite-like" prisms. Some aggregates of these. 	Silver reagent works with a solution of the drug provided it is quite concentrated. Scratch slide to precipitate. With solid drug, five or ten minutes may be required before crystals form.
O. Phenobarbital ("Luminal")	Granular mist quickly gives *small* burrs of minute needles, or "walnuts" brownish by transmitted light. Some branches of plates possible. 	Essentially identical to test (1) crystals. 	Almost immediate formation of thick, stubby rectangular or hex-ended crystals. Often in parallel twins.* 	With solid drug, minute rhombs. Dissolve drug in solvent for best results. Gives rhombic or blade-shaped "sled-runners" crystals singly or in rosettes.* 	Silver test is quite sensitive and crystals form at once from mixture of reagent with a solution of the drug in 15% ethylenediamine-water. Test-drop must be dilute for best results; otherwise small burrs are obtained throughout.

* Indicates best test(s).

Barbiturate	Potassium Hydroxide & Phosphoric Acid (1)	Sulfuric Acid & Water (2)	Wagenaar's Reagent (3)	Davis' Silver Reagent (4)	Remarks on Silver Test (4)
P. SANDOPTAL (Allyl isobutyl barbituric acid)	Oily mist gives quick-forming thin squares or skeletal squares. Grow into 4-armed "snow-flakes," etc. Feathery masses float on top. 	Oily mist soon gives squares, 4-pointed stars and radiates of long rods. 	Best dissolve drug in solvent first. Slow-forming thick diamond-like hex forms. Some branching rods. 	Immediate small-to-large rhombs or squarish plates, almost always notched on two diagonal corners. Twins.* 	Crystals with the silver reagent grow quite large (scratch slide to bring down if using drug in solvent), relatively thick, and notched forms are characteristic. Some "butterfly" shapes may resemble Seconal or Talbutal, etc.
O. SECOBARBITAL ("Seconal")	Oily mist. No significant crystals form. Small (minute) needles possible after 15 minutes. (Large rods after two or three hours.)	Oily mist gives oil droplets or drops which *may* crystallize internally to give "sycamore ball" crystals. (Smaller than phenobarb burrs.) 	Oily globs which gradually give "lace doily" crystal forms around edges. Very filmy appearing, or feathery.* 	Oily globs which yield thick "butterfly" or "paddle-wheel" shapes. Large plates also. Grow large in time.* 	Two or three other barbiturates give similar crystals with the silver reagent, though they can be distinguished by remaining tests. When cover glass is used, plate forms predominate. May form immediately or be delayed up to an hour, depending on concentration, conditions, etc.
R. TALBUTAL ("Lotusate")	Oily mist from which slowly separate diamond-shaped plates and "boxes." 	Results similar to test (1). Crystals small to indistinct. Very slow to form. 	Wispy radiates of fine needles out of oily drops. Slow to form. 	Crystals form quickly as "paddle-wheels" and plates similar to Seconal.* 	Seconal crystals essentially identical with silver reagent but Seconal preceded by oily globs, while Talbutal appears not to be.

* Indicates best test(s).

Barbiturate	Potassium Hydroxide & Phosphoric Acid (1)	Sulfuric Acid & Water (2)	Wagenaar's Reagent (3)	Davis' Silver Reagent (4)	Remarks on Silver Test (4)
S. TUINAL (Mixture of Amobarbital and Secobarbital)	Oily mist soon gives brushy-ended radiates or "S"-shaped feather-ended formations.* 	Oily droplets slowly give plates and discs or rectangles having rounded corners. Twins of some. Some brushy types. 	Oily globs give radiates of fine needles. Rarely single needles as with Amytal. Not filmy like Seconal. Some blades. 	Slow forming. Use generous amount of drug. Crystals thick to thin plates, rods, prisms, etc. Some may resemble Seconal or Amytal, but generally blending into Pentobarb types. 	Some of the crystals obtained with the silver reagent will resemble component barbiturates, or may instead resemble another barbiturate altogether. Amytal, when present with another barbiturate, seems to cause "S"-shaped formations in tests (1) and (2) in a number of cases.
T. BARBITAL ("Veronal")	Immediate formation of crystals as diamond, square hex, or rectangular plates, often overlapped shingle-fashion, in large aggregates. 	Essentially same as in test (1). Use fairly generous amount of drug.* 	Slow forming. Scratch slide to ppt. Long rods or thick striated prisms. Some resemble Phenobarb, but for etching or striated surfaces. 	Powdered drug on reagent and examine as added. Smooth thick diamonds, grow larger and become *rough*. Some rods and prisms, often intersecting.* 	With the silver reagent it is necessary to watch formation of crystals, as they change from smooth to granular or rough diamonds quite rapidly at times. Diamonds are thick, like pyramids, base to base. May eventually degenerate to small prisms or rod forms.
U. VINBARBITAL ("Delvinal")	Oily mist quickly gives beautiful rosettes of filmy blades with rough edges. Flower-like.* 	Similar to test (1) results. Filmy bladed rosettes.* 	Slow-forming lenticular plates or rhombic plates along edges. May not be a reaction product. 	Rods and plates. Plates have notch both ends. One side grows faster than other giving "war-hatchet" shapes. May grow very large and become laminar. 	Silver reagent gives various forms, but the "war hatchet" shape is seldom noted in tests for other barbiturates with this reagent. Rods may not be distinctive, though. May be very slow to form or require slide to be scratched before they will form.

* Indicates best test(s).

powdered material directly to the reagent drop. If crystals do not form at once, cover glass may be put on the drop.[4] With some barbiturates (phenobarbital, especially), it is better to first dissolve a small amount of the barbiturate in 15% ethylenediamine-water solution and run this into a drop of the silver reagent. In other instances (secobarbital, butabarbital) it may be well to make a thick paste of the pill-scrapings in ethylenediamine-water and run the drop of silver reagent into that. If a gum or jelly-like precipitate forms, it may be scratched *slightly* to precipitate the crystals.

Initial tests with the silver reagent will likely prove discouraging, as in some instances a matter of 30 minutes to an hour may be required before crystals are found. With practice and development of proper techniques, however, crystals are generally obtainable within three minutes or less. When test conditions are proper, these crystals grow very large.

Most laboratories performing tests of this type will have on hand a collection of barbiturates in pill and capsule form for initial comparison against any confiscated evidence specimens. Such comparison should, of course, precede any chemical testing, and will often indicate immediately which barbiturate (if any) is most likely present. Where the specimen is believed to have two or more barbiturates present, one should not expect the crystal reactions obtained to match any of the component barbiturates. Combinations of these drugs will usually give crystal forms entirely different from the pure forms of any one of them. Combinations are best treated as if they were a single "new" barbiturate, and compared accordingly.

[4] If cover glass is used, maintain a thick test drop by propping up one side of the cover glass with a fragment of another one.

REFERENCES

1. BAMFORD; POISONS, THEIR ISOLATION AND IDENTIFICATION; Blakiston; [Group reactions, crystal forms, schematic color tests]
2. METHODS OF ANALYSIS; Association of Official Agricultural Chemists, Washington, D. C. [Crystal tests]
3. METHODS OF ANALYSIS—Laboratory Manual of the Alcohol and Tobacco Tax Division Laboratory, U. S. Treasury Department, Internal Revenue Service. [Solubilities, melting points, crystal reactions and tests, structural formulas, etc.]
4. FULTON, CHARLES C.; AN IODINE-KI IDENTIFICATION TEST FOR BARBITURATES; Division of Microbiology, Food & Drug Administration, Department of Health, Education and Welfare, Washington, D. C. 1958. [Reagent 5 gms. Iodine, 80 gms. KI, water to make 100 ml. Use 1 part of this by volume, with 2 parts syrupy phosphoric acid—crystal reactions with a number of the barbiturates.]
5. PENPRASE AND BILES; The Use of Microscopic and X-Ray Diffraction Methods for the Identification of Barbituric Acid Derivatives; JOURNAL OF THE AMERICAN PHARMACEUTICAL ASSOCIATION, Scientific Edition, Volume XLV, No. 9, Sept. 1956. Related paper same publication, Vol. XLVII, No. 7, July 1958. [Crystal test procedures and photomicrographs.]
6. BULLETIN ON NARCOTICS; United Nations publication; Volume IX, No. 1, January–March, 1957. [Article by Levi; chemical aspects, structural formulae, synonyms, etc. List of references to related data.]