

DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL		
Procedure B-10	Polarized Light Microscopy Volatility test using Gold Chloride in dilute Phosphoric Acid with 10% Sodium Hydroxide	
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**Name of Procedure:**

Polarized Light Microscopy  
Volatility test using Gold Chloride in dilute Phosphoric Acid with 10% Sodium Hydroxide

**Suggested Uses:**

Microcrystalline test for d-(or l-), and dl-amphetamine, and d-(or l-), and dl-methamphetamine.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Polarized Light Microscope  
Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Spatula  
Microscope slides  
Weighing paper  
Graduated cylinder  
Glass stirring rods  
Glass beakers  
Reagent bottles  
Gold chloride 1.0 gram ampule  
Concentrated phosphoric acid  
Distilled water  
Spot plate  
Sodium hydroxide  
d-amphetamine standard  
l-amphetamine standard  
d-methamphetamine standard  
l-methamphetamine standard

**Formula for Preparing Reagent:**

**For gold chloride in phosphoric acid:**

1. Measure out 20 milliliters of distilled water.

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**Formula for Preparing Reagent (continued):**

2. Measure out 10 milliliters of concentrated phosphoric acid.
3. Pour the 10 milliliters of acid into the water and stir.
4. Pour 20 milliliters of the diluted phosphoric acid solution into a beaker.
5. Add 1.0 gram of gold chloride to the 20 milliliter solution in the beaker.
6. Stir until dissolved.
7. Pour in a reagent bottle.
8. Properly label reagent bottle.

**For 10% Sodium Hydroxide:**

1. Weigh out 2.5 grams of sodium hydroxide and place in a graduated cylinder.
2. Add enough distilled water to bring the total volume to 25 milliliters.
3. Stir until dissolved.
4. Pour the solution into a reagent bottle.
5. Properly label reagent bottle.

**Quality Control Check:**

Check the reagents with a known standard of d-methamphetamine using the application procedure listed below.

**Expiration Date of Chemical Reagent:**

The reagents can be used until depletion provided they are stored in airtight reagent bottles.

**Application of Procedure on Evidence:**

**For unknown sample only:**

1. Place a small portion of the unknown substance in a clean well of the spot plate.
2. Add 1 drop of the 10% sodium hydroxide solution to the well and stir briefly.
3. Place 1 drop of the gold chloride/phosphoric acid solution on a microscope slide.
4. Invert the slide over the well with the 10% sodium hydroxide solution.
5. Let stand for a few minutes.
6. Reinvert the slide and view any crystal formation using the polarized light microscope.
7. Record results.

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### **Application of Procedure on Evidence (continued):**

#### **For unknown sample mixed with the known d- or l- isomer of the suspected compound:**

1. Take a small portion of the unknown and mix thoroughly with an equal portion of the d- or l- isomer of the known compound.
2. Place a small portion of the mixture in a clean well of the spot plate.
3. Add 1 drop of the 10% sodium hydroxide solution to the well and stir briefly.
4. Place 1 drop of the gold chloride/phosphoric acid solution on a microscope slide.
5. Invert the slide over the well with the 10% sodium hydroxide solution.
6. Let stand for a few minutes.
7. Reinvert slide and view any crystal formation using the polarizing light microscope.
8. If the crystal pattern that forms is different from the crystal pattern of just the unknown and is also identical with the known dl-racemic mixture, then the optical isomer of the unknown substance will be opposite the optical isomer of the known compound used.
9. Record results.

### **Safety Concerns:**

Always wear eye protection, gloves, and a laboratory coat when preparing this reagent.

Eye protection and a laboratory coat should be worn when using this reagent for the microcrystalline test.

Always dispose of used microscope slides in a broken glass container.

Sodium hydroxide is caustic.

Phosphoric acid is a strong oxidizing agent and it is corrosive.

### **Literature References:**

Fulton, Charles C., **Modern Microcrystalline Test for Drugs**, New York: Wiley-Interscience a Division of John Wiley & Sons, 1969.

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Chichilo, Peter, Paul A. Clifford, William Herwitz, and Helen Reynolds, **Official Methods of Analysis of the Association of Official Agriculture Chemist**, 10th edition, Washington: AOAC, 1965.

**Literature References (continued):**

Teer, Charles B., “**Modification of the Microcrystalline Test for d-Amphetamine,**” **BNDD Laboratory Notes**, June 8, 1970, No. 5.

Developed for use with amphetamine and methamphetamine by Chemist J.R. Daniel of the North Carolina State Bureau of Investigation, in use in the laboratory since 1980.