Chapter 10: Technical Procedure for Microcrystalline Tests

1. Purpose / Scope

This procedure provides instruction for the performance of microcrystalline tests in the Drug Chemistry Unit of the Wake County Bureau of Forensic Services.

2. Definitions

2.1. Quality control check - Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.

2.2. Reference Material - Material sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties.

3. Equipment, Materials and Reagents

3.1. Equipment

3.1.1. Nikon Eclipse E400 Pol polarizing microscope equipped with 10X eyepiece and 10X objective to produce magnification of 100X
3.1.2. Balance

3.2. Materials

3.2.1. Beakers or other glass vessels
3.2.2. Graduated cylinder
3.2.3. Glass stirring rod
3.2.4. Reagent bottle(s)
3.2.5. Microscope slides
3.2.6. Spatula
3.2.7. Weigh boats or other weigh vessels
3.2.8. Water

3.3. Reference Material

3.3.1. Cocaine

3.4. Reagents

3.4.1. Gold chloride trihydrate, ACS grade

4. Standards and Controls

4.1. Reagents shall be prepared, labeled and stored in accordance with the Drug Chemistry Unit Technical Procedure for Quality Assurance.

4.2. Perform positive and negative quality control checks on all use containers of microcrystalline test reagents prior to use for analysis. The quality control checks must have acceptable results prior to use of the reagent for analysis.

4.3. Perform negative quality control checks (NQCC) according to the procedure with no sample present.

4.3.1. Acceptable result is no crystal formation, i.e., Negative.

4.3.2. If crystals do form, steps will be taken until no crystals are formed. These steps may include retesting with a new microscope slide, re-cleaning any utensils used, or making a new reagent.

4.4. Perform positive quality control checks (PQCC) according to the using the specified reference material.

4.4.1. Refer to each microcrystalline test for acceptable results.

4.4.1.1. If acceptable results are not observed, steps will be taken until acceptable results are obtained. These steps may include retesting with a new microscope slide, re-cleaning any utensils used, or making a new reagent.

4.4.1.2. Record any observations and the results of the positive quality control check in the prepared reagent log.

5. Operation of the Polarizing Microscope

5.1. Refer to the Drug Chemistry Unit Technical Procedure for General Laboratory Equipment.

5.1.1. Turn on the light source.

5.1.2. Place the specimen slide on the stage.

5.1.3. Adjust the desired light intensity with the control lever.

5.1.4. Make sure the field diaphragm is open to the edge of the field view.

5.1.5. Focus with the coarse and fine adjustments for the desired objective.

5.1.6. Move the microscope slide around to view the entire specimen, adjusting the focus accordingly.

5.1.7. Push the filter in to view the specimen with polars crossed, or pull it out to view with uncrossed polars.

6. Procedure

6.1. Place a small portion of the sample, a few particles of material, on a microscope slide and add a drop of the microcrystalline reagent and mix with the sample.

6.2. Samples that have evaporated to dryness shall not be used for evaluation of crystals.

6.3. Observe any crystals on the polarizing microscope under non-polarized and/or polarized light.

6.3.1. If no crystals are observed record in the case file.

6.4. If the crystal observed is that of a reference material used for a PQCC recorded by the Drug Chemist performing the microcrystalline test then the reference material need not be analyzed contemporaneously with the sample.

6.4.1. A positive comparison occurs when the observed sample crystal is morphologically the same as the observed reference material crystal.

6.4.1.1. Record a description of the crystals observed for the sample and the reference material, if applicable, in the case file.

6.4.2. If the observed sample crystal and the observed reference material crystal are not morphologically the same, then the comparison is negative.

6.4.2.1. Record a description of the crystals observed for the sample and the reference material, if applicable, in the case file.

7. Microcrystalline Reagents

7.1. Gold Chloride in 20 % Acetic Acid

7.1.1. This reagent is used for cocaine.

7.1.2. Selected characteristic results:

7.1.2.1. Cocaine - cross-shaped crystals.

7.1.3. Preparation

7.1.3.1. 2% Gold Chloride (w/v) in 20 % Acetic Acid (v/v)

7.1.3.1.1. Add 10 milliliters glacial acetic acid to 40 milliliters of water, mix.

7.1.3.1.2. Dissolve 1.0 gram of gold chloride in the 50 milliliters of 20 % acetic acid, with stirring.

7.1.3.1.3. Storage: Closed container

7.1.3.1.4. Expiration: Stock container: Three years
Use container: One year

7.1.3.1.5. Lot number: Eight-digit format year/month/day/AuCl/Initials of preparer.

Example: 2010123AuClXXX

7.1.3.1.6. PQCC

7.1.3.1.6.1. Reference material: Cocaine
7.1.3.1.6.2. Acceptable result: Cross-shaped crystals

8. Limitations

8.1. Diluents and adulterants may interfere with crystal formation.

9. Safety – NA

10. References

10.1. *Nikon Polarizing Microscope Eclipse E400Pol Instructions*, Nikon Inc, Melville, NY, M216E 98.8.VF.1.

10.2. Clarke, E.G.C., and R.G. Todd, eds. *Isolation and Identification of Drugs*. 1st Edition. London: Pharmaceutical Press, 1969: 135-141, 801.

10.3. Allen, A. C., Copper, D. A., Kiser, W. O., Cottrell, R. C., "The Cocaine Diastereoisomers," *Journal of Forensic Sciences*, Vol. 26, No.1, Jan. 1981, pp. 12–26.

10.4. Smith, F.P., ed. *Handbook of Forensic Drug Analysis.* Boston, Massachusetts: Elsevier Academic Press, 2005: 238.

11. Records

11.1. Reagent log

Document Revision History		
Revision Date	Prepared By	Revision
11/7/2024	A. Abernethy	Document revised to reflect the agency name change from Raleigh/Wake City-County Bureau of Identification to Wake County Bureau of Forensic Services, effective December 1, 2024. Changed header and revision history format. No change to procedure content.