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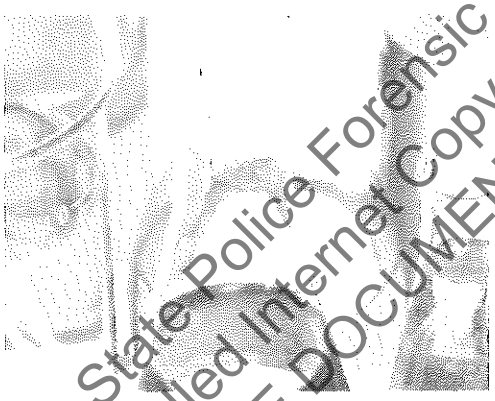

Discipline/Name of Document: Toxicology
5.10 Authentication of Reference Materials – Urine and Blood Toxicology

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APPROVED BY: Corinna C Owsley
Quality Manager

5/7/07
Date Signed

Section Five
Quality Assurance

5.10 Authentication of Reference Materials - Urine and Blood Toxicology

5.10.1 BACKGROUND

The quality assurance measures applied towards analysis promote confidence in results. This analytical method was created so that the shared requirements did not have to be included in every toxicology discipline analytical method.

5.10.2 SCOPE

This analytical method addresses qualitative and quantitative authentication of reference material used to verify analysis performance by thin layer chromatography (TLC) and gas chromatography with a nitrogen phosphorus or mass selective detective. Reference materials include both analytical reference standards and commercially obtained matrix controls.

5.10.3 EQUIPMENT AND SUPPLIES

5.10.3.1 Tube Rocker (Fisher Selentific or equivalent)
5.10.3.2 Laboratory Centrifuge (Fisher Marathon or equivalent)
5.10.3.3 Waterbath
5.10.3.4 Drybath
5.10.3.5 Evaporative Concentrator (Zymark TurboVap or equivalent) equipped with nitrogen tank.
5.10.3.6 Glassware
Refer to appropriate analytical method for extraction glassware.
GC/MS ALS vials
GC/MS vial microinsert
5.10.3.7 Gas Chromatograph equipped with a mass selective detector (and a low bleed (5%-Diphenyl-95%-Dimethylsiloxane co-polymer) capillary column or comparable.

5.10.4 REAGENTS

5.10.4.1 Refer to appropriate analytical method for solution preparation instructions.

5.10.5 REFERENCE MATERIAL AUTHENTICATION

5.10.5.1 General
5.10.5.1.1 Appropriate authentication must be documented for reference materials prior to official use.

5.10.5.1.2 Reference materials applied for qualitative purposes must have their chemical identity well established.

5.10.5.1.3 The manufacturer of reference standards used for quantitative purposes must utilize balances calibrated with weights traceable to National Institute of Standards and Technology (NIST) standards. The certificate of analysis must be consulted to verify compliance with this requirement.

5.10.5.1.4 When a standard or control contains more than one constituent, only the compound(s) of interest need be authenticated.

5.10.5.1.5 When possible, reference materials used for quantitation must be analyzed against existing reference materials.

5.10.5.1.6 Whenever possible, source of reference material used to prepare matrix controls must differ from that used to prepare a quantitative response curve. If different vendors are not available, the standards and controls must be prepared separately.

5.10.5.1.7 For qualitative authentication, evaluate a single GC-MSD analysis obtained in full scan mode.

5.10.5.1.8 For quantitative authentication, a minimum of three determinations, in a single analysis run, must be evaluated.

5.10.5.1.9 For quantitative determinations, utilize analytical method and GC-MSD conditions optimized for the analyte under evaluation.

5.10.5.1.10 Whenever possible, the GC-MSD data must be compared to a previous lot of reference material.

5.10.5.1.11 The mean quantitative concentration must fall within 20% of the target value listed on *Certificate of Analysis* for standards or *Package Insert* for matrix controls. The precision between replicates must be ≤ 5%.

5.10.5.1.12 Certificate of Analysis (COA) for all standards and package inserts for commercially obtained matrix controls, will be stored centrally in the laboratory performing the authentication.

5.10.5.2 Reference Standard Authentication

5.10.5.2.1 Reference standards are used for both qualitative and quantitative purposes.

5.10.5.2.2 Whenever possible, qualitative authentication is done by comparing the instrumental data obtained through the instrumental analysis of the new standard with data from a peer reviewed scientific journal, reference standard compendium, instrumental data and/or library searches in conjunction with the data provided on the COA, when available.

5.10.5.2.2.1 Comparison must result in no significant differences.

5.10.5.2.3 When comparison to a journal, compendium or other document, is not an option, mass spectral interpretation may be used in conjunction with the COA. This would apply in cases where instrumental data for a drug metabolite is not yet published but a structurally similar compound is available to assist with interpretation.

5.10.5.2.4 For the quantitative authentication of the concentration of a component of a reference standard, evaluate gas chromatography-mass spectrometry (GC-MS) data in conjunction with the certificate of analysis (COA) provided by the manufacturer.

5.10.5.2.5 Deuterated internal standards may also be evaluated in SIM mode prior to use.

5.10.5.3 Matrix Control Authentication

5.10.5.3.1 Matrix controls are analyzed in parallel with casework samples to demonstrate that a procedure performed as intended.

5.10.5.3.2 Matrix controls serve to verify the accuracy of a response curve.

5.10.5.3.3 Matrix controls may be prepared with authenticated reference standards and appropriate matrix or obtained through a vendor.

5.10.5.3.4 When possible, the qualitative identity of component(s) in a commercially obtained matrix control must be based on the package insert or certificate of analysis. If the analyst is unfamiliar with the MS of the component, reference materials must be consulted as described in 5.10.5.2.2.

5.10.5.3.5 The matrix control must be extracted as described in the appropriate analytical method. For controls containing a mixture of analytes such as Toxi-Control 19, a reference mixture can be prepared from authenticated drug standards.

5.10.5.3.6 To authenticate the qualitative presence of components when the manufacturer does not provide a certificate of analysis or package insert and another lot of the control is in use, a new lot of a commercially obtained matrix control is compared to the existing lot.

5.10.5.3.7 To authenticate the concentration of a component of a commercially obtained matrix control for quantitative applications, evaluate gas chromatography-mass spectrometry (GC-MS) data in conjunction with the package insert provided by the manufacturer.

5.10.5.3.8 Controls in use prior to the issue date of this analytical method revision can be used until consumed.

5.10.5.4 Authentication Documentation

5.10.5.4.1 A coversheet providing the information necessary for authentication will be prepared and placed with the MSD data. The coversheet for qualitative validation must, at a minimum, list the lot number, vendor, date of analysis, analyst, and mode of authentication. For

quantitative authentication, the coversheet must include an evaluation of quantitative data.

5.10.5.4.2 Coversheet and GC-MSD data must be initialed and stored centrally in a designated location.

5.10.5.4.3 The stock container for the standard or control may be designated as "authenticated" after the authenticity of the standard has been validated.

5.10.5.4.4 Unopened stock reference material must be stored in a designated area until official use.

5.10.5.4.5 It is the responsibility of each analyst to verify that each standard or control used has been properly authenticated.

5.10.6 REFERENCES

5.10.6.1 Wu Chen, N.B. Cody, J.T., Garriott, J.C., Foltz, R.L., et al., *Report of the Ad Hoc Committee on Forensic GC/MS: Recommended guidelines for forensic GC/MS procedures in toxicology laboratory associated with offices of medical examiners and/or coroners*, J. Foren. Sci, 236 (35): 236-242, 1990.

5.10.6.2 Goldberger, B.A., Huestis, M.A., Wilkins, D.G., *Commonly practiced quality control and quality assurance procedures for gas chromatograph/mass spectrometry analysis in forensic urine drug-testing laboratories*, For Sci Review, 9(2): 60-79, 1997.

5.10.6.3 SOFT/AAFS Forensic Toxicology Laboratory Guidelines, 2002.

Revision History

Section Five
Quality Assurance

5.10 Authentication of Reference

Revision	Issue Date	History
0	05-24-2006	Original Issue – Split from analytical method 5.8. Clarifications of authentication process described.
1	05-07-2007	Reformat, updated QA language