Hair Proficiency Testing

Jeri D. Ropero-Miller, Ph.D., D-ABFT

Drug Testing Advisory Board
Open Session
July 15, 2013
Objectives

- Design and Results of SAMHSA’s Hair Pilot Proficiency Testing (PT) Program, 2000 to 2007
- Design and Results of Society of Hair Testing (SoHT) Hair Proficiency Testing Program, 1995-present
- Design of Other Hair Proficiency Testing Programs
Background
SAMHSA’s Pilot Hair PT Program

● April 1997, DTAB meeting discussed drug testing in alternate matrices
● Duration: 2000-2007
● Up to 13 laboratories participated
● 23 cycles of samples tested
  • Cycles 1-8: Method Optimization
  • Cycles 9-11: Same samples over 6 months
  • Cycles 12-23: Inter- and intra-lab variability
Background
SAMHSA’s Pilot Hair PT Program

- Important finding was a large variation in reported results
- 2006-2007 PT samples evaluated laboratory variability and sample stability
Design of Pilot PT Program 2007

- 6 or 7 participant laboratories
- Laboratories investigated accuracy and precision
- Single PT production
  - Decontaminated hair samples in PT production
  - Authentic hair fortified with drug analytes or known drug users
  - Stored ambient temperature prior to shipments
Design of Pilot PT Program (Continued)

- Three sample sets sent to each lab over 1 year (0, 3, 6, 12 months)
- Concentrations were 1.5 to 3 times the 2004 proposed confirmatory test cutoffs
- Laboratories quantitated each analyte 5 times in multiple batches
Design of Pilot PT Program (Continued)

- Set 1 (pg/mg):
  - THCA (0.82)*
  - THCA (0.15)
  - AMP/MAMP/MDA/MDEA/MDMA (900)
  - AMP/MAMP/MDA/MDEA/MDMA (450)

- Set 2 (pg/mg):
  - COC (5770)/BE (2547)/CE (54)/ NCOC (85)*
  - COC (750)/NCOC (75)/CE (75)
  - COC (1500)
  - BE (75)

- *Drug User Hair
Design of Pilot PT Program (Continued)

● Set 3 (pg/mg):
  • 6-AM (1411)/MOR (630)/COD (183)*
  • 6-AM/MOR/COD (600)
  • 6-AM/MOR/COD (300)
  • PCP (450)

● No decontamination
● Confirmatory testing only

● *Drug User Hair
**NLCP Pilot Hair PT — Methods**

- Deuterated internal standards
- Not all analytes reported by all labs
- Powdering vs. Strands
- Pre-Treatment Techniques
  - None/DI H2O/Solvent (MeOH/MeCl₂/Acetone)
- Digestion Techniques
  - None/Acidic/Basic/Enzymatic
- Extraction Techniques
  - L/L and SPE
Confirmatory Testing

Quantitation and identification by a confirmatory mass spectrometry method

PT result reports with group statistics sent to each lab for self-assessment
# Laboratory Pilot Hair PT Results: 2007

<table>
<thead>
<tr>
<th>(pg/mg)</th>
<th>AMP (450)</th>
<th>MAMP (450)</th>
<th>MDA (450)</th>
<th>MDMA (450)</th>
<th>MDEA (450)</th>
<th>THCA (0.15)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 months (Cycle 1)</td>
<td>397</td>
<td>411</td>
<td>421</td>
<td>316</td>
<td>278</td>
<td>0.17</td>
</tr>
<tr>
<td>3 months (Cycle 2)</td>
<td>463</td>
<td>493</td>
<td>460</td>
<td>397</td>
<td>432</td>
<td>0.16</td>
</tr>
<tr>
<td>6 months (Cycle 3)</td>
<td>436</td>
<td>469</td>
<td>467</td>
<td>399</td>
<td>398</td>
<td>0.15</td>
</tr>
<tr>
<td>12 months (Cycle 4)</td>
<td>449</td>
<td>481</td>
<td>455</td>
<td>408</td>
<td>412</td>
<td>0.13</td>
</tr>
<tr>
<td><strong>Mean ± SD</strong></td>
<td><strong>436 ± 29</strong></td>
<td><strong>464 ± 36</strong></td>
<td><strong>451 ± 20</strong></td>
<td><strong>380 ± 43</strong></td>
<td><strong>380 ± 69</strong></td>
<td><strong>0.15 ± 0.02</strong></td>
</tr>
</tbody>
</table>
## 2007 Pilot Hair PT Results: AMPS

<table>
<thead>
<tr>
<th>Lab</th>
<th>Cycle 12</th>
<th>Cycle 15</th>
<th>Cycle 18</th>
<th>Cycle 21</th>
</tr>
</thead>
<tbody>
<tr>
<td>E</td>
<td>564</td>
<td>950</td>
<td>1023</td>
<td>866</td>
</tr>
<tr>
<td>G</td>
<td>926</td>
<td>885</td>
<td>939</td>
<td>836</td>
</tr>
<tr>
<td>L</td>
<td>597</td>
<td>612</td>
<td>781</td>
<td>946</td>
</tr>
<tr>
<td>M</td>
<td>1084</td>
<td>1148</td>
<td>1134</td>
<td>1054</td>
</tr>
<tr>
<td>Q</td>
<td>1097</td>
<td>529</td>
<td>1081</td>
<td>970</td>
</tr>
</tbody>
</table>
## 2007 Pilot Hair PT Results: COC

<table>
<thead>
<tr>
<th>Lab</th>
<th>Cycle 13</th>
<th>Cycle 16</th>
<th>Cycle 19</th>
<th>Cycle 22</th>
</tr>
</thead>
<tbody>
<tr>
<td>E</td>
<td>632</td>
<td>608.4</td>
<td>667.4</td>
<td>735.4</td>
</tr>
<tr>
<td>G</td>
<td>634</td>
<td>603</td>
<td>639</td>
<td>582.6</td>
</tr>
<tr>
<td>L</td>
<td>527.2</td>
<td>591.6</td>
<td>531.8</td>
<td>479.8</td>
</tr>
<tr>
<td>M</td>
<td>720.4</td>
<td>682.8</td>
<td>693.2</td>
<td>599.6</td>
</tr>
</tbody>
</table>
# 2007 Pilot Hair PT Results: THCA

<table>
<thead>
<tr>
<th>Lab</th>
<th>Cycle 12</th>
<th>Cycle 15</th>
<th>Cycle 18</th>
<th>Cycle 21</th>
</tr>
</thead>
<tbody>
<tr>
<td>G</td>
<td>0.13</td>
<td>0.11</td>
<td>0.11</td>
<td>0.09</td>
</tr>
<tr>
<td>M</td>
<td>0.17</td>
<td>0.26</td>
<td>0.18</td>
<td>0.15</td>
</tr>
<tr>
<td>Q</td>
<td>0.21</td>
<td>0.12</td>
<td>0.17</td>
<td>0.16</td>
</tr>
</tbody>
</table>
# System Variance Over 1 Year by Cycle

<table>
<thead>
<tr>
<th>Cycle</th>
<th>AMP 900</th>
<th>AMP 450</th>
<th>MAMP 900</th>
<th>MAMP 450</th>
<th>MDA 900</th>
<th>MDA 450</th>
<th>MDMA 900</th>
<th>MDMA 450</th>
<th>MDEA 900</th>
<th>MDEA 450</th>
<th>THCA 0.82</th>
<th>THCA 0.15</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 months (Cycle 1)</td>
<td>31.3</td>
<td>38.9</td>
<td>30.8</td>
<td>40.1</td>
<td>31.4</td>
<td>42.2</td>
<td>33.0</td>
<td>39.3</td>
<td>35.7</td>
<td>36.2</td>
<td>28.0</td>
<td>28.2</td>
</tr>
<tr>
<td>3 months (Cycle 2)</td>
<td>29.3</td>
<td>21.9</td>
<td>29.1</td>
<td>22.6</td>
<td>28.3</td>
<td>21.7</td>
<td>31.8</td>
<td>24.0</td>
<td>24.1</td>
<td>18.8</td>
<td>29.8</td>
<td>45.5</td>
</tr>
<tr>
<td>6 months (Cycle 3)</td>
<td>13.6</td>
<td>20.3</td>
<td>15.6</td>
<td>22.0</td>
<td>15.8</td>
<td>18.2</td>
<td>16.3</td>
<td>19.7</td>
<td>16.7</td>
<td>22.1</td>
<td>33.0</td>
<td>24.2</td>
</tr>
<tr>
<td>12 months (Cycle 4)</td>
<td>11.5</td>
<td>11.6</td>
<td>11.0</td>
<td>13.7</td>
<td>12.7</td>
<td>11.6</td>
<td>13.9</td>
<td>15.0</td>
<td>7.7</td>
<td>14.2</td>
<td>21.7</td>
<td>24.6</td>
</tr>
<tr>
<td>Urine (2004-2005)*</td>
<td>7.4</td>
<td>7.4</td>
<td>8.5</td>
<td>8.5</td>
<td>4.6</td>
<td>4.6</td>
<td>7.1</td>
<td>7.1</td>
<td>7.6</td>
<td>7.6</td>
<td>10.3</td>
<td>10.3</td>
</tr>
</tbody>
</table>
## System Variance Over 1 Year by Cycle

<table>
<thead>
<tr>
<th>PT Program</th>
<th>6AM</th>
<th>COD</th>
<th>MOR</th>
<th>PCP</th>
<th>THC / THCA</th>
<th>AMP</th>
<th>MAMP</th>
<th>BE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Urine NLCP Maintenance PTs (2006)</td>
<td>8.3</td>
<td>7.4</td>
<td>7.7</td>
<td>7.7</td>
<td>10.7</td>
<td>7.7</td>
<td>7.4</td>
<td>6.4</td>
</tr>
<tr>
<td>Hair Pilot PTs (2006-2007)</td>
<td>14.5</td>
<td>23.1</td>
<td>29.7</td>
<td>20.1</td>
<td>32.2</td>
<td>28.1</td>
<td>29.3</td>
<td>29</td>
</tr>
<tr>
<td>Oral Fluid Pilot PTs (2006-2007)</td>
<td>11.1</td>
<td>7.9</td>
<td>15.8</td>
<td>11.8</td>
<td>23.9</td>
<td>13.3</td>
<td>11.4</td>
<td>14.4</td>
</tr>
</tbody>
</table>
Summary 2006 Pilot PT Program

● Stable material for all drugs over 1 year
● Results for the system of participating hair-testing laboratories are not currently consistent
● For some analytes, there was increased variability in results after 1 year, illustrative of the laboratory challenge of maintaining performance
● Material performed well for all analytical methods
SOCIETY OF HAIR TESTING HAIR PROFICIENCY PROGRAM
Background
Society of Hair Testing (SoHT)

● “…development of proficiency tests (PTs), so that all laboratories which perform hair analysis can produce comparable results, or at least detect the same compounds” (Forens Sci Intl, 2003)

● Voluntary Program

● PT Rounds
  • 18-36 participating laboratories
  • 2008, 2009, 2010 (EtG Special PT)
  • Qualitative and Quantitative results
Design of SoHT PT Program - 2001

- Goals
  - Accuracy of Detection and Quantification
  - Performance of Extraction Methods
- 15 Participating Labs & 3 Reference Labs
- Samples
  - Short Hair Segments
  - Drug User Hair- COC, BE, MDMA, MDEA, THC
  - Drug User Hair- COC, BE, 6AM, MOR, COD, DHC
  - Drug Free Hair
Results for SoHT Hair PT Program - 2001

- Not all analytes reported by all labs
  - Amphetamines - 8
  - Cannabis - 9
- Qualitative results
  - False Negatives - 1 (BE, 6AM, MOR), 3 (COD)
  - False Positive - 1 (COD, 6AM)
- Quantitative results
  - Mean inter-laboratory %CVs — 41.7% to 71% except THC (93.1%)
  - Extraction procedures — source of variation
Design of SoHT PT Program - 2011

● Goals
  • Accuracy of Detection and Quantification

● 36 Participating Labs & 3 Reference Labs

● Samples
  • Short Hair Segments
  • Drug User Hair- COC, BE, 6AM, MOR, COD, THC
  • Drug User Hair- COC, BE, MAMP, THC
  • Drug Free Hair
Results for SoHT Hair PT Program - 2011

• Z-score (Z-score = (x - \chi) / IQR
  • Satisfactory (>2.0)
  • Questionable (2 < Z-score \leq 3)
  • Unsatisfactory (\geq3.0)

• Quantitative results - Sample 1
  • 6-AM: 2 Not analyzed; 1 questionable
  • MOR: 1 Not analyzed; 1 questionable
  • COD: 13 Not analyzed or Neg; 1 unsatisfactory
  • THC: 10 Not analyzed or Neg; 1 unsatisfactory
Results for SoHT Hair PT Program - 2011

- Quantitative results -
  - COC: 1 Not analyzed; 2 questionable
  - BE: 4 Not analyzed
  - MAMP: 7 Not analyzed or Neg; 1 questionable

- Mean inter-laboratory %CVs — 34% to 55% except COD (306%)
Comparison of Hair PT Programs

● Worldwide 5 Hair PT Programs
● Professional and governmental programs
● Hair PT Programs
  • GTFCh - German Society of Toxicological and Forensic Chemistry (late 1990s to Present)
  • HAIRVEQ - Germany and Spain (2004, 2006)
  • NLCP - National Laboratory Certification Program (2000-2007)
  • SFTA - French Society of Analytical Toxicology (1992-1999)
  • SoHT - Society of Hair Testing (1995 to Present)
# Comparison of Hair PT Programs

<table>
<thead>
<tr>
<th>Hair PT Program</th>
<th>Hair Type</th>
<th>PT Type</th>
<th>Analytes</th>
<th>Conc.</th>
<th>Washing</th>
<th>Preparation</th>
</tr>
</thead>
<tbody>
<tr>
<td>GTFCH</td>
<td>Authentic</td>
<td>Spiked; Drug User</td>
<td>All but PCP; +THC (addition of further analytes possible)</td>
<td>≥ SAMHSA</td>
<td>Not Reported</td>
<td>Lab Choice</td>
</tr>
<tr>
<td>HAIRVEQ</td>
<td>Authentic</td>
<td>Drug User</td>
<td>opiates, cocaine, MDMA, and methadone</td>
<td>≥ SAMHSA</td>
<td>Not Reported</td>
<td>Lab Choice; Standardized</td>
</tr>
<tr>
<td>SFTA</td>
<td>Authentic</td>
<td>Drug User</td>
<td>All but PCP; +CBD</td>
<td>≥ SAMHSA</td>
<td>Step in PT Production</td>
<td>Standardized</td>
</tr>
<tr>
<td>SoHT</td>
<td>Authentic</td>
<td>Drug User</td>
<td>All but PCP; +THC</td>
<td>≥ SAMHSA</td>
<td>Not Reported</td>
<td>Lab Choice</td>
</tr>
<tr>
<td>NLCP</td>
<td>Authentic</td>
<td>Spiked; Drug User</td>
<td>AMP/MAMP MDA/MDMA/MDEA COC/BE COD/MOR/6AM THCA PCP</td>
<td>AMP: 200</td>
<td>Step in PT Production</td>
<td>Lab Choice</td>
</tr>
</tbody>
</table>
Conclusions

- Performance Testing is a necessary quality control measure
- Results for the systems of participating hair-testing laboratories are not currently consistent
- Inter-laboratory and Intra-laboratory results remain higher than other hair testing matrices
- PT materials in authentic hair samples are stable
- Laboratory preparation and analytical treatment of hair contribute to the variability of PT results
References

- Society of Hair Testing. [http://www.soht.org](http://www.soht.org)