



Authentication of Reference Materials

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Topics

- Reference Material Sources
- Authentication Process
- Monographs
- Contact Information



Where do DEA Reference Materials come from?

- Purchased from commercial sources
- Synthesized in-house



Authentication of Reference Materials

Why??

1. Verify structure of the Reference Material (RM)
2. Determine the purity of the RM



Purity Determination

- Quantitative Nuclear Magnetic Resonance (NMR)
 - Use a NIST traceable internal standard
 - Multiple analyses in order to establish an uncertainty value
 - Material must be $\geq 98.0\%$ for quantitative reference materials & $\geq 90.0\%$ for qualitative reference materials



Purity Determination

- Chromatography
 - Gas Chromatography/Flame Ionization Detector (GC/FID)
 - Liquid Chromatography/Photodiode Array Detector (UPLC/PDA)
 - Capillary Electrophoresis (CE)
- Differential Scanning Calorimetry
 - Also provides Melting Point data
- Thermogravimetric Analysis (TGA)
 - Water of Hydration



Structural Confirmation

- Required:
 - NMR
 - Gas Chromatography/Mass Spectrometry (GC/MS)
 - Infrared Spectroscopy (FTIR)
- As needed:
 - Polarimetry/CE for isomer determination
 - Microcrystalline tests/Optical crystallography

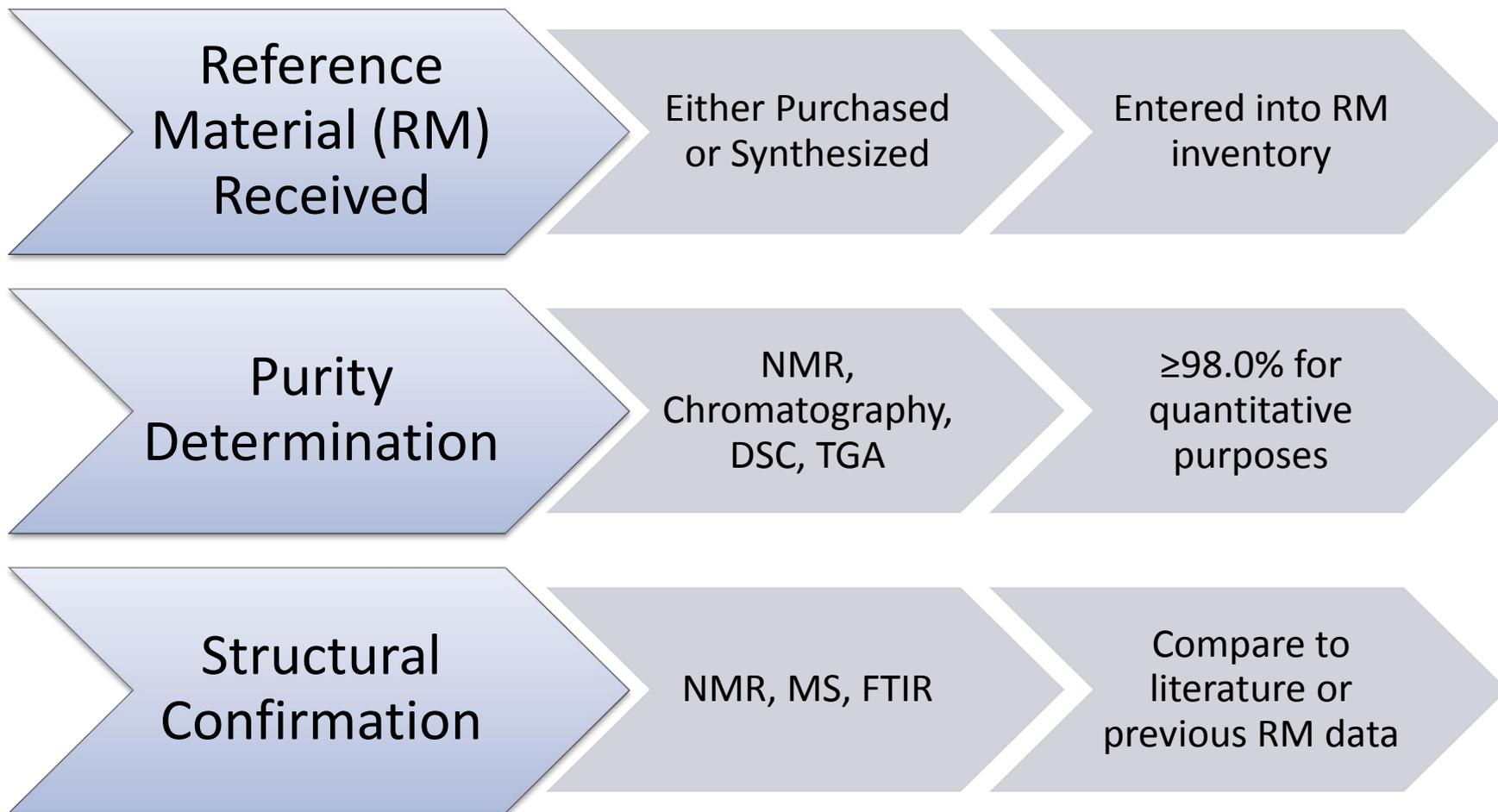


Structural Confirmation

- Data from identification techniques are compared to:
 - Instrumental Data of Drug Analysis (IDDA)
 - Clarke's Analysis of Drugs and Poisons
 - Peer reviewed articles
 - Previous lots of reference materials



Authentication Process Recap



Compare to literature or previous RM data?

What happens when there is no previous data for comparison?



Structural Elucidation

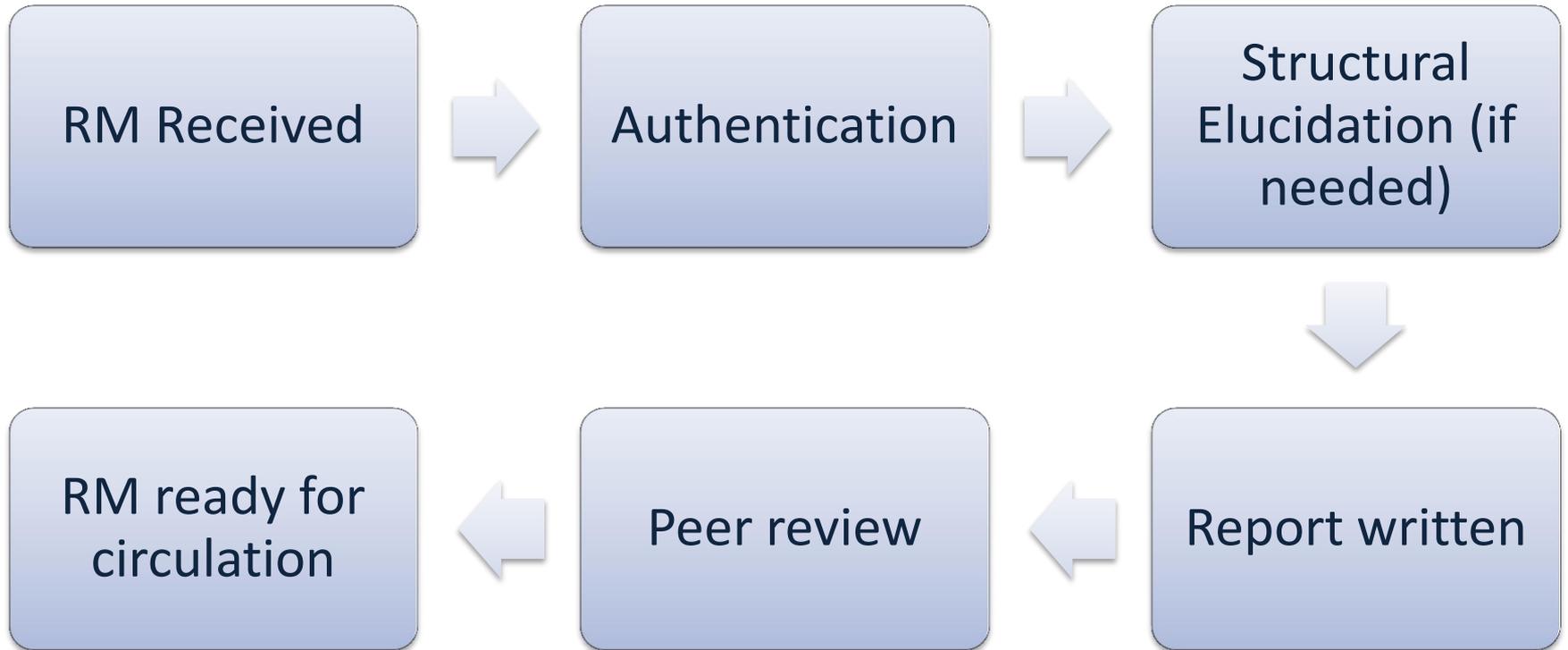


Structural Elucidation

- NMR Experimentation (1D & 2D)
 - Proton NMR (HNMR)
 - Carbon NMR (CNMR)
 - Correlation Spectroscopy (COSY)
 - Heteronuclear Single Quantum Coherence (HSQC)
 - Heteronuclear Multiple Bond Correlation (HMBC)
- MS Interpretation



Authentication Process Recap



How can DEA assist other forensic laboratories?

- Provide spectra
 - MS & IR data
- Provide drug monographs for SWGDRUG





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Welcome to the Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Website

What's New:

- 1) See the new proposed SWGDRUG Recommendations on Analogues and Structural Class Determinations. We are currently seeking public comment until May 3, 2013. SWGDRUG invites you to take part in the recommendation process, please take a moment to visit the [pending documents](#) section of our website, view the proposed changes and provide your feedback.
- 2) West Virginia University is conducting a survey to better understand the status and needs of the seized drug analysis community. If you would like to participate in this survey, please visit their website <http://forensictoxicology.eberly.wvu.edu/survey>
- 3) [Supplemental Document SD-4](#), Examples of Measurement Uncertainty for Purity Determinations
- 4) Searchable Mass Spectral Library [Version 1.7](#) dated December 31, 2012
- 5) [Drug Monographs](#) (being updated weekly)
- 6) NIST and DEA have partnered to host a free workshop in Gaithersburg, MD on April 30 to May 1, 2013, to explore emerging trends in the analysis of synthetic cannabinoids, substituted cathinones, and novel hallucinogens. For more information, please visit the [Emerging Trends in Synthetic Drugs Workshop](#) event page.

SWGDRUG's Mission:

The mission of SWGDRUG is to recommend minimum standards for the forensic examination of seized drugs and to seek their international acceptance.

Suggestions/Comments:

We hope that you find our website easy to navigate and encourage you to forward any suggestions or comments to the SWGDRUG facilitator at: swgdrug@hotmail.com

Last Update April 2013

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Monographs:

The following monographs contain detailed information and analytical data for reference materials which have been analyzed, verified, and authenticated by the Drug Enforcement Administration Special Testing and Research Laboratory. These monographs may be used for the verification of acquired reference materials and for the identification of drug materials (subject to laboratory policy). Monographs are being uploaded as they are peer reviewed and approved for publication.

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Common Name	Other Names	Date
A796.260	{1-[2-(morpholin-4-yl)ethyl]-1H-indol-3-yl}(2,2,3,3-tetramethylcyclopropyl)methanone	12-28-12
4AcODMT	4-AcO-DMT; psilacetin; O-acetylpsilocin	03-18-13
AKB-48	APINACA; N-adamantyl-1-pentylindazole-3-carboxamide; N-adamantyl-1-pentyl-1H-indazole-3-carboxamide	12-07-12
5-Fluoro-AKB-48	5F-APINACA; APINACA 5-Fluoropentyl analog; 5F-AKB-48	02-08-13



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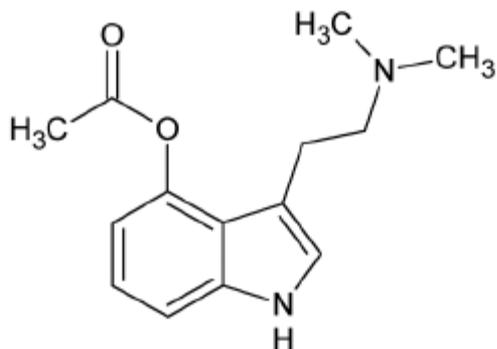
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5-Fluoro-AKB-48	5F-APINACA; APINACA 5-Fluoropentyl analog; 5F-AKB-48	02-08-13





1. GENERAL INFORMATION

IUPAC Name: 3-[2-(Dimethylamino)ethyl]-1H-indol-4-yl acetate

CFR: Not Scheduled (3/2013)

CAS #: 92292-84-7

Synonyms: 4-AcO-DMT; psilacetin; *O*-acetylpsilocin

Source: DEA Reference Material Collection

Appearance: Off-white powder (oxalate)

Kovat's Index: Pending

UV_{max}: Not Determined

2. CHEMICAL AND PHYSICAL DATA

2.1 CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Base	C ₁₄ H ₁₈ N ₂ O ₂	246	Not Determined
Oxalate	C ₁₄ H ₁₈ N ₂ O ₂ ·C ₂ H ₂ O ₄	336	144.2

SWGDRUG MONOGRAPHS

Contain:

- ❖ General Information
- ❖ Chemical and Physical Data
- ❖ Additional Resources
 - Links to websites
 - Article references
- ❖ Qualitative Data
 - Spectra for NMR, MS, & IR
 - Sample preparation and Instrumental conditions used
 - Any analytical observations during data collection



Contact Information

Questions regarding:

- Reference Materials:
 - DEALabRefMaterials@usdoj.gov
- SWGDRUG Monographs:
 - swgdrug@hotmail.com



Thank You

