SWGDRUG

Criteria for Identification of Synthetic Drugs

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Methods of Analysis/Synthetic Drug Identification

- SWGDRUG Recommendations PART IIIB Applies
- Validation to Ensure Specificity
- Use Appropriate Analytical Scheme(s)
- Account for any Limitations
- Accurately Report Results
- Reference Materials
- Tools and Resources

PART IIIB - Drug Identification

§ 1 Introduction

- … It is recognized that the correct identification of a drug or chemical depends on the use of an analytical scheme based on validated methods (see PART IV B - Validation) and the competence of the analyst.
- An appropriately constructed analytical scheme will result in, effectively, no uncertainty in reported identifications (see PART IV C - Uncertainty).
- SWGDRUG requires the use of multiple uncorrelated techniques (e.g., GC-Partition, TLC-Adsorption).
- It does not discourage the use of any particular method within an analytical scheme and it is accepted that unique requirements in different jurisdictions may dictate the practices followed by a particular laboratory.

Drug Identification

§ 2 Categorizing analytical techniques

Techniques for the analysis of drug samples are classified into three categories (see Table 1) based on their maximum potential discriminating power. However, the classification …

Table 1

Category A	***Category B***	***Category C***
Infrared Spectroscopy	*Capillary Electrophoresis*	*Color Tests*
Mass Spectrometry	*Gas Chromatography*	*Fluorescence Spectroscopy*
Nuclear Magnetic Resonance Spectroscopy	*Ion Mobility Spectrometry*	*Immunoassay*
Raman Spectroscopy	*Liquid Chromatography*	*Melting Point*
X-ray Diffractometry	*Microcrystalline Tests*	*Ultraviolet Spectroscopy*
	Pharmaceutical Identifiers	
	Thin Layer Chromatography	
	Cannabis only: *Macroscopic Examination* *Microscopic Examination*	

Drug Identification

§ 3 Identification criteria

- 3.1 When a validated Category A technique is incorporated into an analytical scheme, at least one other technique (from either Category A, B or C) shall be used.

- 3.2 When a Category A technique is not used, at least three different validated techniques shall be employed. Two of the three techniques shall be based on uncorrelated techniques from Category B.
  - Due to the variation of synthetic drugs and that they are not well known to the community, this analytical scheme is not recommended

Drug Identification

§ 3 Identification criteria

- 3.5 For the use of any method to be considered of value, test results shall be considered "positive." This addition is proposed: (i.e., it must meet the acceptance criteria defined in the method validation and operating protocol.) When possible, data from a test result should be compared to data generated from a reference material which has been analyzed under the same analytical conditions (see PART IV A Section 6.2). While "negative" test results provide useful information for ruling out the presence of a particular drug or drug class, these results have no value toward establishing the forensic identification of a drug.

Drug Identification

§ 3 Identification criteria

- 3.8 The chosen analytical scheme shall demonstrate the identity of the specific drug present and shall preclude a false positive identification and minimize false negatives. Where a scheme has limitations, this shall be reflected in the final interpretation (see PART IVC - Uncertainty).

Drug Identification

§ 4 Comment

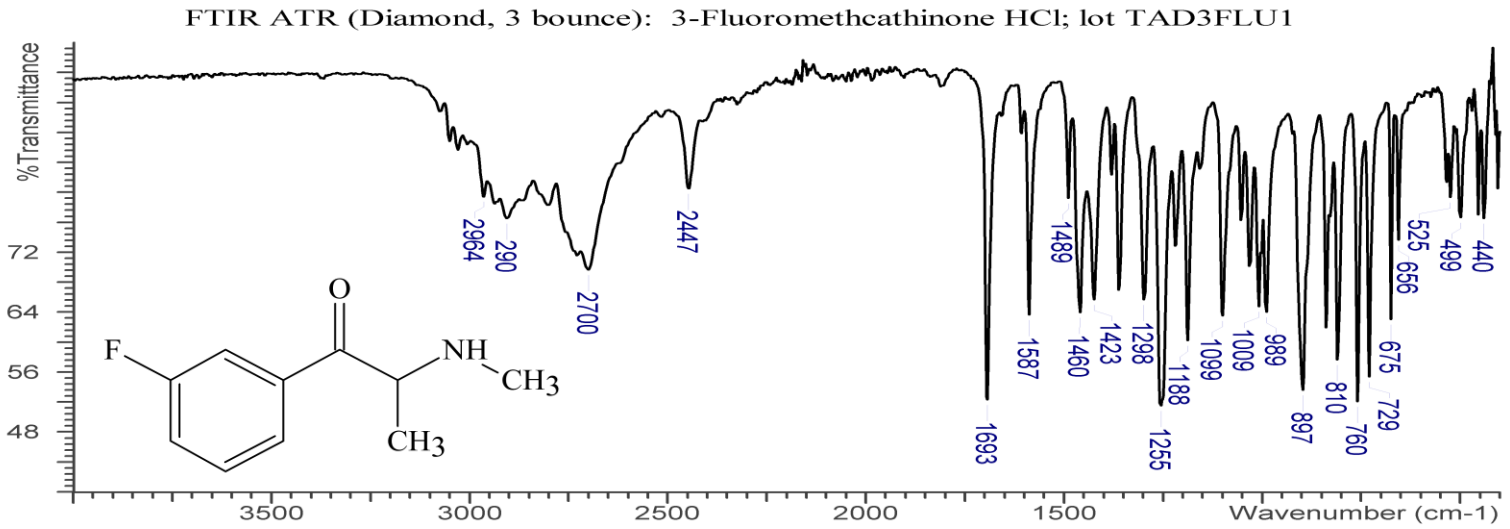
These recommendations are minimum standards for the forensic identification of commonly seized drugs. However, it should be recognized that they may not be sufficient for the identification of all drugs in all circumstances. Within these recommendations, it is up to the individual laboratory's management to determine which combination of analytical techniques best satisfies the requirements of its jurisdiction.

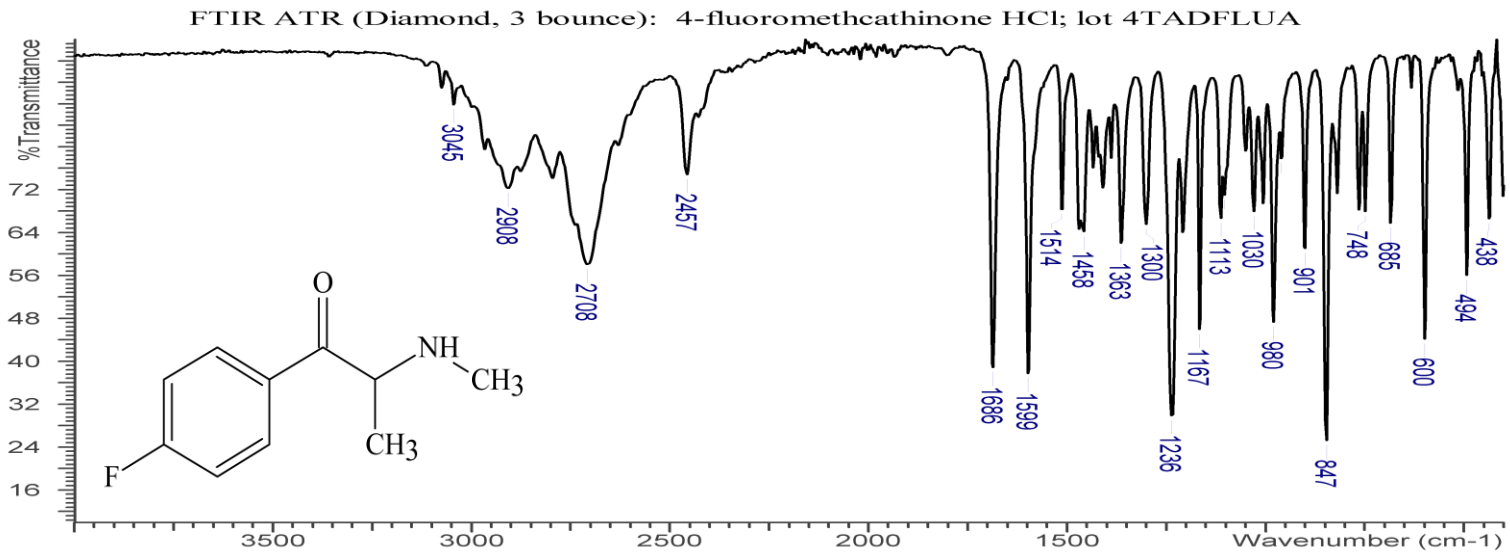
Validation of Analytical Scheme

- Must choose analytical scheme wisely

- PART IVB 1.2 An analytical scheme shall be comprised of validated methods that are appropriate for the analyte.
  - IVB.1.2.1 The combinations of methods chosen for a particular analytical scheme shall identify the specific drug of interest, preclude a false positive and minimize false negatives.

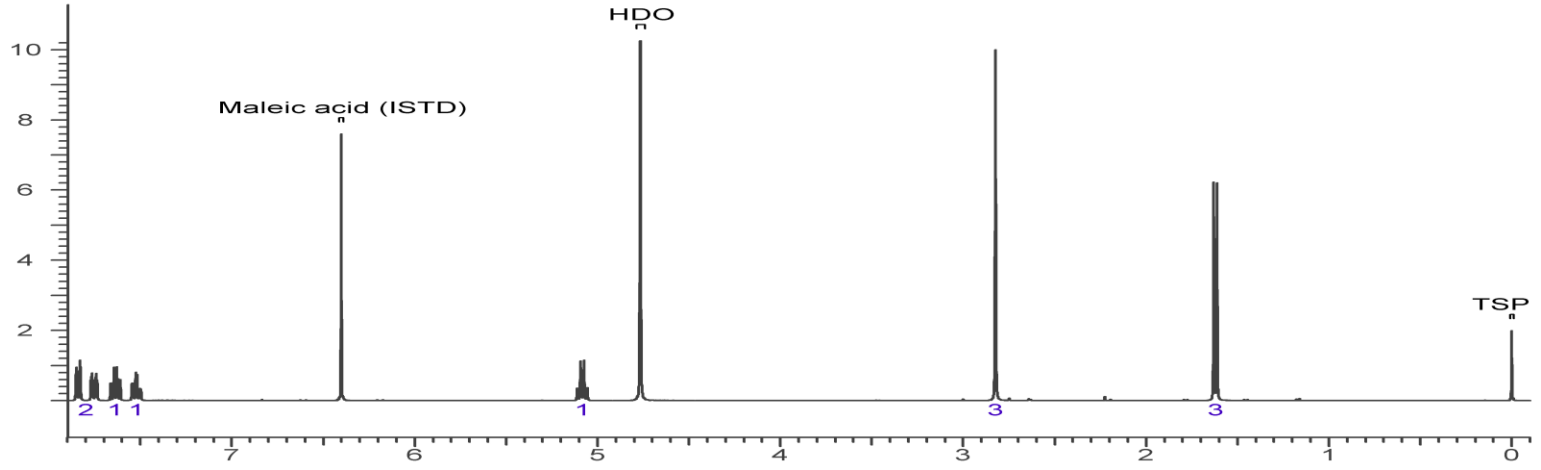
Analytical Scheme - FTIR



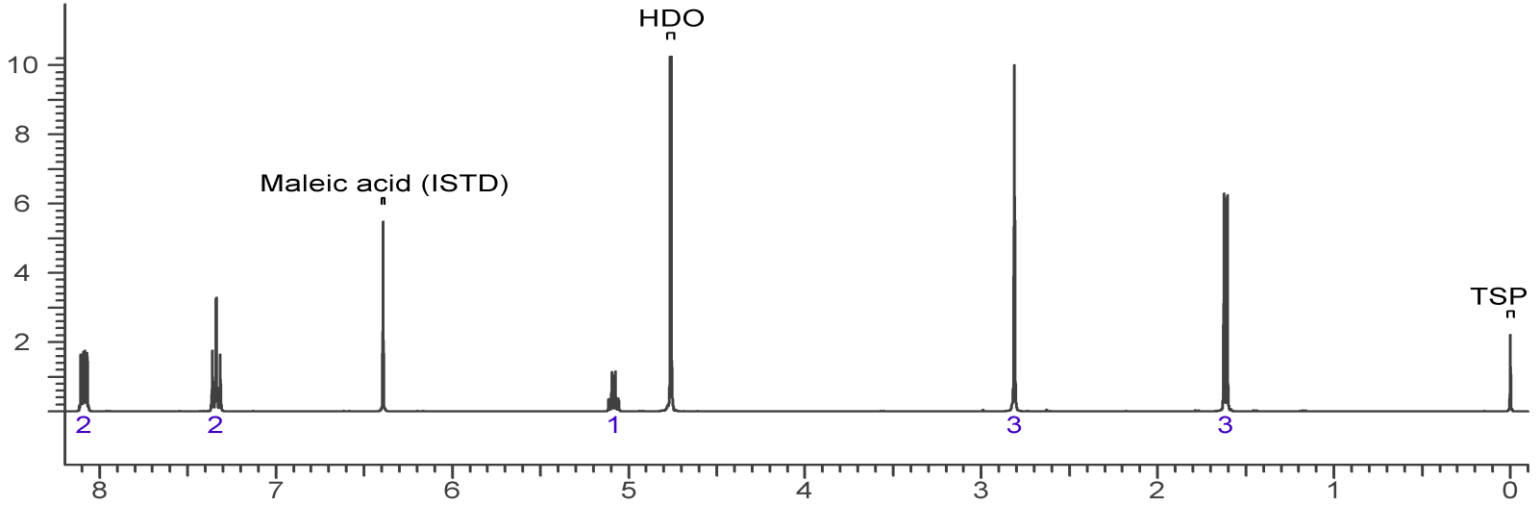


Analytical Scheme - NMR

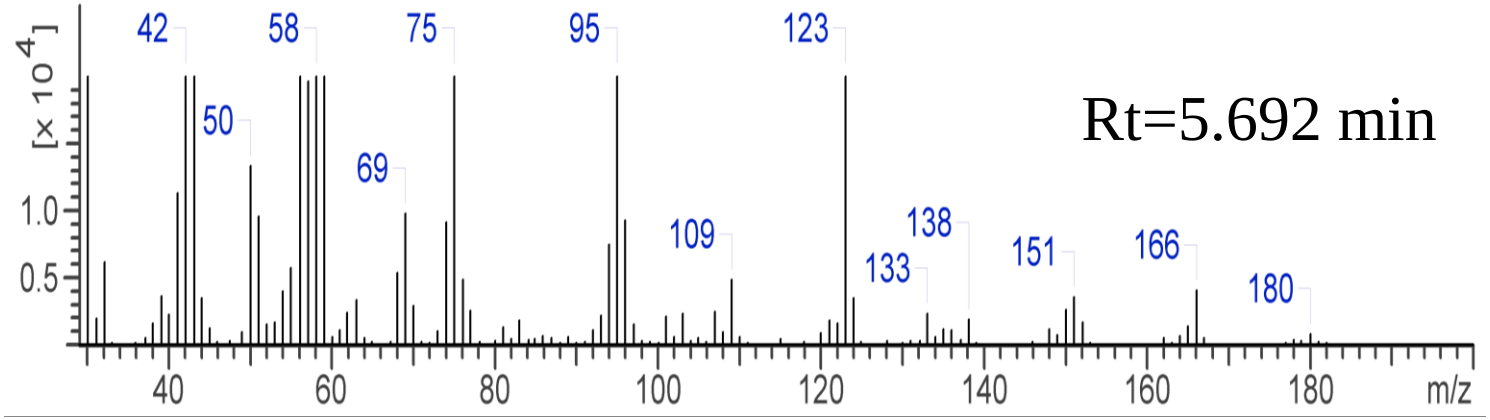
^{1}H NMR: 3-Fluoromethcathinone HCl; lot TAD3FLU1; D_2O, 400 MHz



^{1}H NMR: 4-Fluoromethcathinone HCl; lot 4TADFLUA; D_2O, 400 MHz

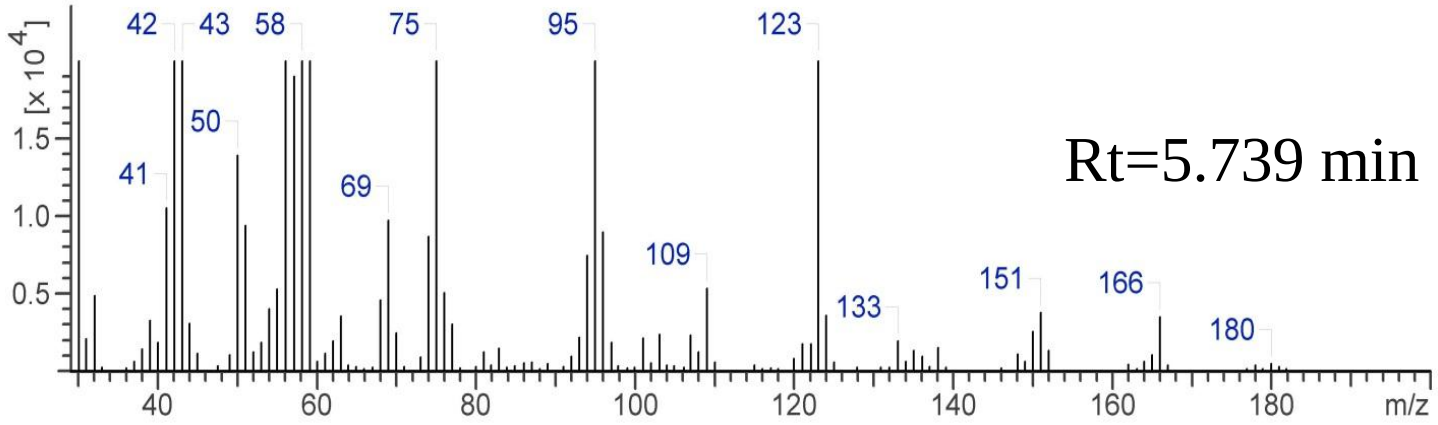


Analytical Scheme - GCMS



Rt=5.692 min

3-Fluoromethcathinone

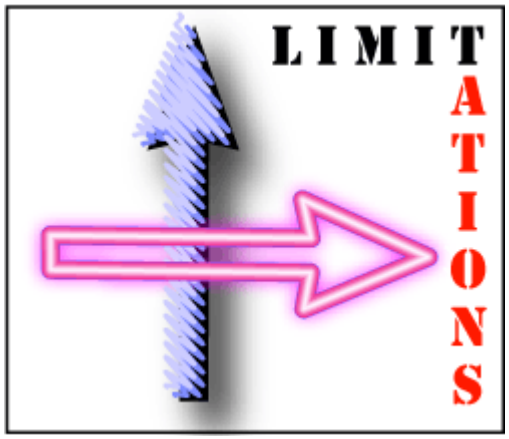


Rt=5.739 min

4-Fluoromethcathinone

Reporting

- **Limitations**
  - If you cannot determine the position of the Flurorine (3, 4 or 5), can you report Flurometh**c**athinone?
    - Depends on your laboratory policy and jurisdictional requirements
    - When doing so, include verbiage that indicates position is not known



Reference Materials - Current

§ 6.2 Verification of drug reference materials

6.2.1 The identity of certified reference materials shall be verified prior to their first use.

6.2.2 The identity of uncertified reference materials shall be authenticated prior to use by methods such as mixed melting point determination, Mass Spectrometry, Infrared Spectroscopy, or Nuclear Magnetic Resonance Spectroscopy.

6.2.3 Verification shall be performed on each new lot of drug reference material.

6.2.4 All verification testing shall be documented. The documentation shall include the name of the individual who performed the verification, date of verification, verification test data and reference used in verification.

Reference Materials - Proposed

- Reference materials and reference data are critical to demonstrating the validity of quantitative and qualitative test results.
- Acceptance criteria order of preference
  - 1. Comparison to data obtained from a suitable drug reference material analyzed under the same analytical conditions as the test/case sample…
  - 2. Comparisons to external reference data may be used where a reference material is unavailable…
    - ✓ Veracity of data shall be assessed…
  - 3. When neither reference materials nor external reference data are available structural elucidation techniques may be employed…
    - ✓ Interpretations by competent analysts…

Reference Materials - Proposed

- Assessment of reference materials
  - ISO/IEC 17025 specifies that reference materials shall, where possible, be traceable to SI units of measurement, or to certified reference materials (CRM). For seized drugs this requirement is difficult to fulfill because the concept of traceability for drug standards is not internationally established and CRM's for drug analysis are not readily available or affordable.
    - Note: A certificate does not necessarily define a material as a CRM.
  - Fit for purpose for qualitative work requires an assessment of chemical identity (structure), stability, matrix, and homogeneity.
  - For quantitative work, it is necessary to assess the purity and its associated uncertainty of measurement in addition to the parameters in Section 6.2.3.

SWGDRUG MS Library

SWGDRUG Mass Spectral Library:
SWGDRUG has compiled a mass spectral library from a variety of sources, containing drugs and drug-related compounds. All spectra were collected using electron impact mass spectrometry systems. This library is available for download from this website.

DISCLAIMER: Although SWGDRUG makes an effort to review the accuracy of spectra prior to entry, this library should only be used as an analytical tool. SWGDRUG recommends the use of traceable reference materials to support identifications of drugs (Part IV B – Quality Assurance Section 2.3)

The SWGDRUG library is supported by the NIST MSSEARCH program, which is available on-line at no charge (see below). Additionally, the library was converted to Agilent Technologies format. Lastly, two raw data formats are included below depending upon your desired application. Click on the appropriate link below to download the compressed file and follow the instructions below.

SWGDRUG MS Library Version 1.8 (April 9, 2013):

NIST Format

Drug Monographs

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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A B C D E F G H I J K L M N O P Q R S T U V W X Y Z

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

Drug Monographs

- **Purpose:**
  - Reference material verification
  - Limited methodology
  - Limitations
- **Peer reviewed** (structural elucidation)
- **Availability:**
  - www.swgdrug.org
  - November 2012
  - monographs uploaded weekly
- **Prioritized based on community needs**

Current SWGDRUG Projects

- **Reference Materials Sub-committee**
- **SWGDRUG Recommendations 6.1 (DRAFT)**
  - Assessment of reference materials
  - **Issues:** availability, companies, structural elucidation, etc.
  - Public comments: were due March 29, 2013
- **Analogues Sub-committee**
- **DRAFT** document
  - Addressing current issues regarding conclusions and opinions on analogues and structural class identifications
  - Public comments: Due May 3, 2013

THANK YOU!

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