

Name of Procedure:

Quality Assurance of Reference Standards

Suggested Uses:

Verify and document primary and secondary reference standards.

Apparatus Used to Perform Procedure:

(See Technical Procedures F-01 Infrared Spectrophotometry and H-06 Gas Chromatograph/Mass Spectrometer, as needed.)

Reference Standard material

Application of Procedure on Primary Standard:

- A. Any material to be used as a Primary Reference Standard (PRS) must be tested to confirm its identity using either Infrared Spectroscopy and/or Mass Spectroscopy, whichever is appropriate for the application.
- B. Once confirmed, an FTIR scan and a mass spectrum of the Primary Reference Standard will be added to the Section instrumental libraries.
- C. All data collected and/or received with the PRS will be filed in a series of notebooks titled "Certified Standards" stored in the Section Library.
- D. The certifying chemist will initial and date when received on the PRS container.
- E. File the Material Safety Data Sheet (MSDS) in the Certified Standards notebook.
- F. The Certificate of Analysis is filed in the Certified Standards notebook.

If the Certificate of Analysis is not included in the PRS shipment, refer to the manufacturer's internet site to obtain this certificate.

- G. Obtain an **FTIR** scan of the compound.
 - 1. Label the scan with the name, Manufacturer ID Number, and Lot Number.

(Do not print instrument information/date/time stamp on this scan.)

2. Compare the FTIR scan obtained to a known literature reference in order to verify that the compound's identity is correct.
3. File a hard copy of the FTIR scan in Certified Standards notebook.
4. Scan the hard copy to make an electronic version – label the file with the compound name &/or abbreviation.
5. Add the electronic version to the current Section Certified Standards FTIR spectra library.
6. Amend the current Section Certified Standard FTIR search library to include this scan – rename the search library with a new number to show that it is an updated version.
7. Distribute the electronic version of the spectra and the updated search library to all section infrareds. (Delete old copies of the search library.)

G. Obtain a **GC/MS** of the compound

1. Label the file with the name of compound or abbreviation.
(Make sure the appropriate temperature program is used!)
2. Add to the *CertDrugs* library per the following steps:
 - a. Select "New Scan". Truncate any unwanted high masses by the following:
 - Tools
 - Options
 - On Command Line box type "MSCLIP,325:650" execute, or enter the lower # as needed
 - Type "Draw" and hit enter
 - b. Once a good scan is obtained – enter it into the library:
 - Spectrum
 - Edit Library
 - Add New Entry (*Edit Existing entries from here as well.*)
 - Type in the Name with Manufacturer's identifier and lot number
 - Molecular formula
(Print Merck entry and add to Certified Standards notebook for this information.)
 - Add the Chemical Abstracts Service (CAS) number, if available
 - Insure the box is checked at the bottom for "Add to Search".
 - c. Print a hard copy of the new library entry; file in the Certified Standards notebook.
3. Distribute the new version of the search library to all the MS instruments in the

section with the exception of those used in toxicology.

4. File a hard copy of the blank, Total Ion Current (TIC) chromatogram, and the corresponding Mass Spectra with search hit in the Certified Standards notebook.
- H. Obtain a final gross weight of reference standard before it goes into the vault/freezer and record it into the logbook in the vault.
- I. Assign a locator number for the new reference standard.
 1. Place the locator number on Reference Standard container.
 2. Place the locator number in the Drug Chemistry Standards Vault with an identifier on the drawer.
 3. Fill out the Vault logbook entry with the requested information, and add a comment identifying a new addition to the inventory.
- J. Store in the vault or freezer/refrigerator located in the toxicology unit, with desiccant if needed, according to manufacturer's directions.

Application of Procedure on Secondary Standard:

- A. Any material used as a Secondary Reference Standard (SRS) must be tested to confirm its identity using either Infrared Spectroscopy and/or Mass Spectroscopy, whichever is appropriate for the application.
- B. The spectral data, known as standard reference materials, are to be compared to reference literature for confirmation.
- C. The standard reference materials must be labeled with the certifying chemist's initials and the unique identification number referenced below, and stored in notebook(s) titled "Secondary Standard Reference Materials," and stored in the Section Library.
- D. The container containing the SRS should be marked with a unique identification number, the certifying chemist's initials and the date the standard was placed in the container.

The unique identification number will be made up in the following order:

The certifying chemist's initials, name or abbreviation of the substance, and the date the certifying data was collected. (Example: JDHeroin20080806.)

Safety Concerns:

Avoid accidental ingestion of powders or liquids.
Some standards require refrigeration.

Literature References:

Keller, Roger, **The Sigma Library of FT-IR Spectra**, Edition 1, Vol. 1 and 2, Sigma Chemical Company, Inc., 1986.

Mills, III, Terry and Roberson, Conrad J., **Instrumental Data for Drug Analysis**, 2nd Ed., Vols. 1-5, CRC Press, Inc., 1993.

Moffat, A. C. Ed., **Clarke's Isolation and Identification of Drugs**, 2nd. Ed., The Pharmaceutical Press, 1986.

Pouchert, Charles J., **The Aldrich Library of Infrared Spectra**, Aldrich Chemical Company, 1981.

Silverstein, R. M. And Brassler, Clayton G., and Terence C. Morrill, **Spectrometric Identification of Organic Compounds**, New York, Wiley, 1991.