Drug Chemistry Section
Drug Chemistry Procedure Manual
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Modification of H-3
Prepared by: A. Joncich
Approved by: Deena Koontz
Supercedes: September 1, 1996

Name of Procedure:

Varian Saturn 3 GC/MS/DS Mass Spectrometer

Suggested Uses:

The ion trap detector/gas chromatograph/data system is used to identify compounds (controlled and non-controlled) present in items of evidence in the field of Forensic Chemistry. It is utilized to identify nanogram levels of substances that cannot be easily identified with other conventional methods. This procedure produces a mass spectra of the compound and often provides the molecular weight. The gas chromatograph (GC) is used to separate mixtures into individual compounds represented by peaks on the ion chromatograph (TIC). A mass spectra of each peak is examined and identification is attempted. The mass spectra and the GC retention time of a compound is compared to a standard spectra and retention time from a library to make an identification of a compound.

Apparatus Used to Perform Procedure:

High purity solvent (methanol, chloroform, or ethyl acetate)
Sample vial (clean/new) with screw top or septum seal (silanized or unsilanized)
5 or 10μL syringe
DB-5 column or equivalent, 30 meter, 0.25μm film thickness, 0.25mm ID
UHP Helium Carrier Gas
Mass spectrometer - Varian Saturn 3
Gas chromatograph: Varian Star 3400cx
Data station: PC 486/66 or better
Pump: Alcatel - 2004A or equivalent.
Software for data acquisition: Varian Saturn version 5.0 or equivalent
Saturn GC/MS Operator's Manual, Part Number 03-914353-40:4
Saturn GC/MS Reference Manual, Part Number 03-914354-40:3
Perfluorotributylamine [FC-43]
Bis(pentafluorophenyl)-phenylphosphine [DFTPP]

Calibration of the Mass Spectrometer:

1. The Saturn 3 must be tuned on a regular basis. Using the following checks, verify that they are within acceptable ranges according to the manual or operator training:

    a. Integrator Zero

b. RF Modulator Response
c. Set EM Voltage
d. Set Filament Emission
e. Set Cal Gas
f. Set AGC target value
g. Perform automatic Mass Calibration
h. Check air and water levels

2. Standards used for Calibration:

   a. Perfluorotributylamine [FC-43]
   b. Bis(pentafluorophenyl)-phenylphosphine [DFTPP]

A regular calibration report will contain the following:

a. Spectra of FC-43
b. Ratio Tune Report of FC-43
c. Instrumental settings for the mass spectrometer

Application of Procedure on Evidence:

These procedures do not cover every aspect of the instrument used. The operator of the instrument should read the manual for the instrument before using this procedure.

1. Sample Preparation (suggested):

   a. Solid Phase Extraction residues: reconstitute with the appropriate solvent or derivatizing agent and transfer to injection vial.
   b. Tablets:
      1. Alprazolam, lorazepam, diazepam, etc.: add a several drops of solvent to an intact (not crushed) tablet(s).
      2. Coated tables: remove coating before adding several drops of solvent to the remaining intact tablet(s).
   c. Suspected dry LSD: one (1) square or microdot per vial “dry” (no solvent).
   d. Syringes: Wash with methanol and extract if necessary (if excessive quantities of blood or other liquids are present in syringe then an extraction is required.
   e. Alkyl Nitrites: Place approximately 3 drops in a headspace vial and seal.
   f. Other volatile compounds: Place 3-5 drops in a headspace vial and seal.

2. Analysis / Data Acquisition Programs:[SUGGESTED]

    a. Common Drugs: 100° for 1min, 10°per min. to 280°, 280° for 21 min. Ion Control - on, scan masses 50-450, fil. delay - 5 min.
    b. Low MW and early eluting drugs: 100° for 1min, 10°per min. to 280°, 280° for 21 min., Ion Control - on, scan masses 40-250, fil. delay - 3.4 min.
    c. SIS (Selected Ion Storage) may be used to identify compounds at low concentrations as long as ion ratios are used for comparison to standards. Less than or equal to 20% variation in ion ratios is acceptable for confirmation purposes.

    Note: Some sample combinations may require deviation to these temperature programs [operator discretion].

3. Analyze Sample:

    a. Inject an organic solvent blank to obtain a solvent blank chromatogram prior to the analysis of the sample.
    b. Inject the sample.
    c. After the data system has collected the data, observe the spectra for the peaks of interest, print/plot the library search, print/plot the spectra and the chromatogram.

4. Reporting:

    The requirements for drug/chemical identification using the GC/MS system are the approximate relative retention time for the column and method used, and a reasonable comparison between a standard and the identified drug/chemical's mass spectra.

5. Activity Log:

    A log of all injections and maintenance will be kept. The log will include the date, sample identification, initials of operator, GC/MS method used, and comments.

<u>Safety Concerns</u>:

a. Avoid syringe punctures of hand and fingers.
b. Use extreme caution handling organic solvents to avoid contact with skin and eyes.

c. Use extreme caution handling compressed gas cylinders.
d. Avoid electrical shock and hot surfaces during maintenance and repair.

Comments:

Due to the various nature of the many possible compounds analyzed, it is impractical to address every possible set of conditions used to analyze them via the GC/MS.

Literature References:

Moffat, Jackson, Moss and Widdop, **Clarke's Isolation and Identification of Drugs**, 2nd edition, Vol. 1, 1986.

Pfleger, Maurer and Weber, **Mass Spectral and GC Data of Drugs, Poisons, Pesticides, Pollutants**, 2nd edition, Vols. 1-3, 1992.

The Merck Index; Merck and Co. Inc.; 11th. Ed.,1989.

Mills, McCurdy and Wall, **Instrumental Data for Drug Analysis**, Vols. 1-5, 1993.