## Name of Procedure:

Mass Spectrometer
Hewlett-Packard 5890 Series II GC interfaced to the Hewlett-Packard 5970 MSD

## Suggested Uses:

The quadrupole mass selective detector /gas chromatograph/ data system is used to identify compounds (controlled and noncontrolled) 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 spectrum of each peak is examined and identification is attempted. Standard mass spectra and case mass spectra are printed and become part of the case file.

## Apparatus Used to Perform Procedure:

Hewlett-Packard 5890 Series II gas chromatograph (GC)
Hewlett-Packard 5970B Mass Selective Detector (MSD)
Hewlett-Packard Automatic Sampler and Controller
PC type data system with HP G1034C, or equivalent, software installed
Printer and printer paper for plotting spectra and library search
High purity solvent (methanol, chloroform, or ethyl acetate)
Sample vial (clean/new) with screw top or septum seal (silanized or unsilanized)
10Φl syringe
DB-5 column (or equivalent), 30 meter, 0.25Φm film thickness, 0.25mm ID
Septa 11-mm low bleed
UHP Helium Carrier Gas
Hewlett-Packard 5890 Series II Operating Manual, Manual Part No. 05890-90260
Hewlett-Packard HP 5970B MSD Hardware Manual, Publication Number 05970-90049
Hewlett-Packard HP 7673 Auto Sampler Operating Manual, Part No. 07673-90185
Hewlett-Packard HP G1034C MS ChemStation User□s Guide (DOS Series)
Perfluorotributylamine [FC-43]

## Calibration of the Hewlett Packard 5970B GC/MSD/DS:

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

For each instrument, a set of standard drugs will be run at least monthly to collect retention time and mass spectral data. If maintenance is performed that may affect retention times, the standards will be re-run to reflect new retention times. If the retention times shift greater than 2% between standard drug runs, the instrument will be evaluated and determined to be in compliance before casework is performed.

## 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 of Samples:

    a. Temperature Programs of GC: [ SUGGESTED ]

1. Used for the majority of drugs and metabolites analyzed such as cocaine (and metabolites), barbiturates, benzodiazepines (and metabolites), and opiates in medium-high amounts (as measured by immunoassay).

   $100^{E}$ - $280^{E}$ @ $10^{E}$/min, time: 40 minutes; Injector purge delay - 0.40 minute

2. Used for the majority of drugs and metabolites analyzed such as cocaine (and metabolites), barbiturates, benzodiazepines (and metabolites), and opiates in low-trace amounts (as measured by immunoassay).

   $100^{E}$ - $280^{E}$ @ $10^{E}$/min, time: 40 minutes; Injector purge delay - 1.00 minute

3. Used for cannabinoids and metabolites of cannabinoids (delta-9-tetrahydrocannabinol and 11-nor-delta-9-tetrahydrocannabinol-9-carboxylic acid).

   Level 1: $150^{E}$ - 235E @ $50^{E}$/min
   Level 2: $235^{E}$ - $300^{E}$ @ $30^{E}$/min, time: 20 minutes

4. Used for the headspace analysis of alkyl nitrites and other volatile compounds.

   $35^{E}$ Isothermal, time: 10 minutes

5. Used to analyze the heptafluorobutyric derivatives of phenethylamines (amphetamine, methamphetamine, phentermine, ephedrine, etc.)

   Level 1: $60^{E}$ - $225^{E}$ @ $20^{E}$/min
   Level 2: $225^{E}$ - $260^{E}$ @ $20^{E}$/min, time: 21 minutes

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

b. Injection of Sample:

1. Inject an organic solvent blank to obtain a solvent blank chromatogram prior to the analysis of the sample.

2. Inject the sample.

3. 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.

3. Reporting:

   The requirements for drug/chemical identification using the GC/MS system are the approximate retention time for the column and method used, and a reasonable comparison between a standard and the identified drug/chemical□s mass spectra. In Drug Chemistry, if the retention time is being used as a confirmatory test for identification, a standard of the drug must be run to show retention times are consistent within + or - 2%. The standard data must be included in the case file.

4. 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.

**Safety Concerns:**

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 dismantling/installing/transporting compressed gas cylinders.
d. Avoid electrical shock and hot surfaces during maintenance and repair.

**Literature References:**

Moffat, Jackson, Moss and **Widdop, Clarke□s Isolation and Identification of Drugs**; 2nd Ed., Vol. 1, 1986.

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

Telepchak, Long, and Moore, ◘**Determination of Delta-9-Tetrahydrocannabinol (THC) and its Metabolite 11-Nor-Delta-9-Tetrahydrocannabinol -9- Carboxylic Acid (THCA) in Whole Blood**◘; United Chemical Technologies, Inc.

◘Distinguishing Sympathomimetic Amines from Amphetamine and Methamphetamine in Urine by Gas Chromatography/Mass Spectrometry◘, **Journal of Analytical Toxicology**; Vol. 16 January-February, 1992, pp 19-27.

◘Analysis of Alkyl Nitrites by Capillary Gas Chromatography-Mass Spectrometry◘, **Journal of the Forensic Science Society**; Vol. 28, No. 3, 1988, pp 185-190.

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