Name of Procedure:

Mass Spectrometers
Hewlett-Packard 5890 Series II GC interfaced to the Hewlett-Packard 5970 MSD
Hewett-Packard 6890 GC PLUS interfaced to the Hewett-Packard 5973 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 spectra of each peak is examined and identification is attempted. The mass spectra of the compound is compared to a standard spectra from the GBI Spectra Library,User Library (condensed library of commonly encountered compounds) or the NIST Library for the comparison of the spectra for confirmation and library search. The library confirmation and mass spectra are printed and become part of the case file.

Apparatus Needed to Perform Procedure:

Hewlett-Packard 5890 Series II and 6890 PLUS gas chromatographs (GC)
Hewlett-Packard 5970B and 5973 Mass Selective Detectors (MSD)
Hewlett-Packard 7673 Automatic Sampler and Controller
PC type data system with HP G1034C software installed
Printer and printer paper for plotting spectra and library search
Ultra-high purity solvent (methanol or ethyl acetate)
Sample vial (clean/new) with screw top or septum seal (silanized or unsilanized)
10µl syringe
DB-5 column, 30 meter, 0.25µm 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]

Apparatus Needed to Perform Procedure (continued):

Bis(pentafluorophenyl)-phenylphosphine [DFTPP]
GC/MS Sample Control Log

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

Standards used for Calibration:

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

The calibration report will contain the following:

a. Spectra of FC-43 and DFTPP
b. Mass list of FC-43 and DFTPP
c. Ratio Tune Report of FC-43
d. Instrumental settings for the mass spectrometer

Note: The calibration report is maintained and filed by the GC/MS Coordinator as set forth in the Drug Chemistry Policy and Procedure Manual.

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.

3. Sample Preparation:

a. Tablets:
   1. Alprazolam, lorazepam, diazepam, etc.: submit “dry” (no solvent) and intact (not crushed).
b. Suspected LSD: one (1) square or microdot per vial “dry” (no solvent).
c. Syringes: Wash with methanol and extract if necessary (if liquids are present in syringe then an extraction is required.
d. Alkyl Nitrites: Place approximately 3 drops in a headspace vial and seal.
e. Other volatile compounds: Place 3-5 drops in a headspace vial and seal. Submit appropriate standards if available.

4. 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° - 280° @ 10°/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° - 280° @ 10°/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° - 235° @ 50°/min
   Level 2: 235° - 300° @ 30°/min, time: 20 minutes

4. Analysis of Samples (continued):

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

   35° Isothermal, time: 10 minutes

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

   Level 1: 60° - 225° @ 20°/min
   Level 2: 225° - 260° @ 20°/min, time: 21 minutes

Note: Some sample combinations may require some deviation to these temperature progra

b. Injection of Sample:

1. Inject approximately 1µl of organic solvent into the injection port to obtain a solvent blank spectra prior to the analysis of the submitted sample.
2. If the sample has been derivatized do not add solvent; otherwise, add 2-4 drops of an appropriate organic solvent to the vial containing the item submitted for analysis.
3. Draw approximately 1µl of the solution into the syringe, inject the solvent into the injection port and start the preselected temperature program for the 5890 gas chromatograph by pressing the start button on the gas chromatograph.
4. 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.
5. <u>It is essential that the solvent dispensing tip does not contact the lip or other portion of the sample vial and contaminate the solvent. The syringe will be flushed at least 10 times with clean solvent between injections to insure the sample integrity between injections and that no sample transfer is made between sample vials.</u>

5. Data Acquisition:

Each peak in the chromatogram is examined. The mass spectra of the compound(s) present is identified and compared to one or more of the spectral libraries in the data system for confirmation. The mass spectra and the library search results are printed. The total ion chromatograph is printed. The resulting printouts are returned to the submitting chemist. The printed data will become part of the case file.

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.

Hewlett-Packard 5890 Series II and 6890 Gas Chromatograph Operating Manual; Hewlett-Packard; 3rd. Ed., 1990.

HP 7673 Automatic Sampler Operating & Installation Manual; Hewlett-Packard; 1st Ed., 1989.

HP 5970B and 5973 Mass Selective Detector Hardware Manual; Hewlett-Packard, 1988.

MS ChemStation User's Guide; Hewlett-Packard, 1st. Ed., 1993.

Literature References (continued):

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

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

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