Name of Procedure:

Hewlett-Packard/Agilent GC interfaced to the Hewlett-Packard/Agilent 5973 series MSD for Toxicology Analysis

Suggested Uses:

The gas chromatograph / quadrupole mass selective detector / data system is used to qualitate and quantitate compounds present in items of evidence.

The gas chromatograph separates mixtures of compounds and the mass spectra of the compounds of interest are examined. The mass spectrum of a compound is compared to reference spectra for confirmation. If necessary, mass spectral libraries can be searched through computer-based matching software to aid in identifying unknown compounds.

Apparatus Used to Perform Procedure:

Hewlett-Packard/Agilent Gas Chromatograph (GC)
Hewlett-Packard/Agilent 5973 series Mass Selective Detector (MSD)
Hewlett-Packard/Agilent Automatic Liquid Sampler
PC with HP Analytical MSD Productivity ChemStation Software, or equivalent
Output Device
Methanol
Ethyl Acetate
Sample vials and caps
crimper tool
10Φl syringe
DB-5 column (or other appropriate column)
Helium Gas
Perfluorotributylamine [PFTBA]

Calibration of the Hewlett Packard 5973 GC/MSD/DS:

1. The GC-MS system is kept on at all times.
2. Calibration is performed daily when in use with the Autotune program, using the Autotune option.
3. This procedure uses Perfluorotributylamine (PFTBA) as a tuning standard and the resulting data file is kept in a notebook near each instrument.

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4. Compare this tune file to previous ones and address any major variations which may indicate instrument problems. The tune file should conform to the manufacturer's requirements.

Procedure:

These procedures do not cover every aspect of the instrument. The operator of the instrument should consult the manual(s) for the instrument.

A. Sample Preparation (suggested):

1. Solid Phase Extraction residues: reconstitute with the appropriate solvent or derivatizing agent and transfer to injection vial.
2. 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.

B. GC/MS Methods (The listed methods are for specific applications. Other methods may be developed and used as needed.)

1. ACIDFS.M
   Initial Temperature 90 ^{o}C hold for 1 minute
   90 ^{o}C - 125 ^{o}C @ 40 ^{o}C/minute hold for 1.00 minute
   125 ^{o}C - 285 ^{o}C @ 17 ^{o}C/minute hold for 7.71 minutes
   Total time of run: 20.00 minutes

2. AUTO.M
   Initial Temperature 70 ^{o}C hold for 1.00 minute
   70 ^{o}C - 125 ^{o}C @ 40 ^{o}C/minute hold for 1.00 minute
   125 ^{o}C - 285 ^{o}C @ 17 ^{o}C/minute hold for 12.00 minutes
   Total time of run: 24.79 minutes

3. BENZOFS.M
   Initial Temperature 120 ^{o}C hold for 1 minute
   120 ^{o}C - 210 ^{o}C @ 30 ^{o}C/minute hold for 2.00 minutes
   210 ^{o}C - 300 ^{o}C @ 40 ^{o}C/minute hold for 11.75 minutes
   Total time of run: 20.00 minutes

4. CANSIM.M

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Initial Temperature 150 ^{o}C hold for 1 minute
150 ^{o}C - 235 ^{o}C @ 50 ^{o}C/minute hold for 2.00 minutes
235 ^{o}C - 300 ^{o}C @ 15 ^{o}C/minute hold for 5.97 minutes
Total time of run: 15.00 minutes

5. GHBBLOOD, GHBURINE, and GHBFS
   Initial temperature - 60 ^{o}C hold for 4.00 minutes
   60 ^{o}C - 180 ^{o}C @ 15 ^{o}C/minute hold for 0.00 minute
   180 ^{o}C - 250 ^{o}C @ 35 ^{o}C/minute hold for 4.00 minute
   Total time of run: 18.00 minutes
   GHBFS - full scan from 50-350 amu
   GHBBLOOD and GHBURINE - scan from 230-245 amu

6. 70METH
   Initial temperature - 70 ^{o}C hold for 1.00 minute
   70 ^{o}C - 125 ^{o}C @ 40 ^{o}C/minute hold for 1.00 minute
   125 ^{o}C - 285 ^{o}C @ 17 ^{o}C/minute hold for 12.00 minutes
   Total time of run: 24.79 minutes

C. Injection of Sample:

1. Obtain a chromatogram of a blank solvent injection prior to the analysis of the sample.
2. Dilute the sample with the appropriate solvent, if needed, before injecting the sample.
3. After the data system has collected the data, examine the chromatogram and spectra for the peaks of interest, print all necessary data and spectra.
4. The syringe must 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.

D. Reporting:

The requirements for analyte identification using the GC/MS system are described in the Toxicology Criteria for Identification of Analytes.

E. Activity Log:

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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:

1. Avoid syringe punctures of hand and fingers.
2. Use extreme caution handling organic solvents to avoid contact with skin and eyes.
3. Use extreme caution dismantling/installing/transporting compressed gas cylinders.
4. Caution: Gas Chromatograph and Mass Spectrometer may be extremely hot.

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.

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