

Name of Procedure:


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

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
Computer Printer
Methanol
Ethyl Acetate
Chloroform
Sample vials and caps
crimper tool
10  syringe
DB-5 column (or other appropriate column)
Helium Gas
Perfluorotributylamine [FC-43]

Calibration of the Hewlett Packard 5973 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

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. Tablets:
 - a. Alprazolam, lorazepam, diazepam, etc.: add a several drops of solvent to an intact (not crushed) tablet(s).
 - b. Coated tablets: remove coating before adding several drops of solvent to the remaining intact tablet(s).
3. Suspected dry LSD: one (1) square or microdot per vial "dry" (no solvent).
4. 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).
5. Alkyl Nitrites: Place approximately 3 drops in a headspace vial and seal.
6. Other volatile compounds: Place 3-5 drops in a headspace vial and seal.

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

1. ACIDDRUG.M and ACIDFS.M
Initial Temperature 90 °C hold for 1 minute
90 °C - 125 °C @ 40 °C/minute hold for 1.00 minute
125 °C - 285 °C @ 17 °C/minute hold for 7.71 minutes
Total time of run: 20.00 minutes
ACIDFS.M - Full scan
ACIDDRUG.M - SIM
Nominal masses of ions monitored
Butalbital - 168*, 153, 141
Pentobarbital - 156*, 141, 157
Phenobarbital - 204*, 117, 232
Secobarbital - 168*, 153, 195
Hexobarbital (internal Standard) - 221*, 157, 156
* - Quantitation ion

2. AUTO.M and AUTOMAN.M

Initial Temperature 70 °C hold for 1.00 minute
70 °C - 125 °C @ 40 °C/minute hold for 1.00 minute
125 °C - 285 °C @ 17 °C/minute hold for 12.00 minutes
Total time of run: 24.79 minutes
AUTO.M - utilizes 7683 Automatic Liquid Sampler
AUTOMAN.M - manual injection
Full scan

3. AMPH METH.M

Initial temperature - 60 °C hold for 2.00 minutes
60 °C - 225 °C @ 20 °C/minute hold for 4.00 minutes
225 °C - 260 °C @ 70 °C/minute hold for 6.25 minutes
Total time of run: 21.00 minutes
Nominal masses of ions monitored
Amphetamine (HFBA derivative) - 240*, 118, 91
Methamphetamine (HFBA derivative) - 254*, 210, 118
Amphetamine -d₅ (HFBA derivative) (Internal standard) - 244*, 123, 92
Methamphetamine -d₁₁ (HFBA derivative) (Internal standard) - 260*, 213, 123
* - Quantitation ion

4. BENZODAI.M and BENZOFS.M

Initial Temperature 120 °C hold for 1 minute
120 °C - 210 °C @ 30 °C/minute hold for 2.00 minutes
210 °C - 300 °C @ 40 °C/minute hold for 11.75 minutes
Total time of run: 20.00 minutes
BENZOFS.M - Full scan
BENZODAI.M - SIM
Nominal masses of ions monitored
Alprazolam - 308*, 279, 204
Diazepam - 256*, 283, 221
Midazolam - 325*, 312, 310
Nordiazepam (TMS) - 341*, 342, 343
Prazepam (Internal Standard) - 269*, 241, 324
Oxazepam-d₅ (TMS) (Internal Standard) - 435*, 434, 318
* - Quantitation ion

5. CANSIM.M

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

Nominal masses of ions monitored
THC (TMS) - 371*, 303, 386
THC-COOH (TMS) - 371*, 473, 488
THC-d₃ (TMS) (Internal Standard) - 374*, 306, 389
THC-COOH-d₃ (TMS) (Internal Standard) - 374*, 476, 491
* - Quantitation ion

6. COCAINE.M and COCAINFS.M
Initial Temperature 100 °C hold for 0.00 minute
100 °C - 140 °C @ 40 °C/minute hold for 1.00 minute
140 °C - 280 °C @ 20 °C/minute hold for 6.00 minutes
Total time of run: 15.00 minutes
COCAINFS.M - Full scan
COCAINE.M - SIM
Nominal masses of ions monitored
Ecgonine Methyl Ester (TMS) - 96*, 182, 271
Cocaine - 182*, 198, 303
Benzoylecgonine (TMS) - 240*, 256, 361
Cocaine-d₃ (Internal Standard) - 185*, 201, 306
Benzoylecgonine-d₃ (TMS) (Internal Standard) - 243*, 259, 364
* - Quantitation ion
7. GHBBLOOD, GHBURINE, and GHBFS
Initial temperature - 60 °C hold for 4.00 minutes
60 °C - 180 °C @ 15 °C/minute hold for 0.00 minute
180 °C - 250 °C @ 35 °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
8. MEOH.M
Initial temperature - 90 °C hold for 1.00 minute
90 °C - 125 °C @ 40 °C/minute hold for 1.00 minute
125 °C - 285 °C @ 17 °C/minute hold for 8.00 minutes
Total time of run: 20.29 minutes
Full Scan
9. OPIATE.M and OPIATEFS.M
Initial temperature - 100 °C hold for 1.00 minute
100 °C - 250 °C @ 25 °C/minute hold for 2.00 minutes
250 °C - 290 °C @ 10 °C/minute hold for 0.50 minute
290 °C - 325 °C @ 25 °C/minute hold for 3.10 minutes

Total time of run 18.00 minutes
OPIATEFS.M - full scan
OPIATE.M - SIM
Nominal masses of ions monitored
Hydrocodone - 299*, 242, 214
Codeine (Propyl derivative) - 355*, 282, 229
Oxycodone (Propyl derivative) - 371*, 314, 298
6-Acetylmorphine (Propyl derivative) - 327*, 268, 383
Morphine (Propyl derivative) - 341*, 268, 397
Codeine-d₃ (Propyl derivative) (Internal standard) - 358*, 285, 232
Morphine-d₃ (Propyl derivative) (Internal standard) - 344*, 271, 400
* - Quantitation ion

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 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.

E. 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:

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.

Kitchen, Telepchak, and August, "**An Automated Solid Phase Extraction Method for Thebaine, 6-Acetylmorphine and Other Opiates in Urine**"; United Chemical Technologies, Inc.

"Improving Ion Mass Ratio Performance at Low Concentrations in Methamphetamine GC-MS Assay through Internal Standard Selection," **Journal of Analytical Toxicology**, Vol. 20, November/December 1996, pp. 592-595.