Forensic Chemistry Section

Fire Debris Analysis Gas Chromatograph Mass Spectrometer Policy

1. Scope

This document outlines the policies for maintaining and operating the GC/MS.

2. Autotune

2.1 An autotune will be performed every week that the instrument is in use, or prior to using if the instrument has been out of service for more than one week.

2.2 A printout of the autotune will be stored for future reference.

2.3 The autotune will be checked for the following:

- Number of peaks: should be below 150
- Air components should be less than 15% of 69 peak
- Peak widths of 69, 219, and 502: 0.60 Da ± 0.2 Da
- Mass 69 should have a relative abundance of 100%, isotope ratio of 0.7-1.3
- Mass 219 should have a relative abundance of >35%, isotope ratio of 3.7-5.2
- Mass 502 should have a relative abundance of >1.5%, isotope ratio of 7.9-13.2

2.4 Each of these parameters can vary slightly outside of acceptable limits, however if multiple parameters are outside of limits, the examiner should explore further to determine if maintenance is necessary.

2.5 The examiner may choose to re-run the autotune to improve the tune.

2.6 The following is a list of symptoms and possible causes:

Large number of peaks: air leak; source needs cleaned
Mass assignments out of specification: source needs cleaned
Fronting on peaks: source needs cleaned
EMV increases: filament needs replaced
Air components > 15%: air leak

2.7 The examiner will evaluate the autotune and determine if maintenance needs to be performed on the instrument prior to use. If maintenance does need to be performed, the instrument will be removed from service until the maintenance is complete and the instrument tunes appropriately.

2.8 An autotune will be performed after any service on the detector or if the detector has been vented to ensure that the detector is operating properly prior to use in casework.

2.9 The autotune will be printed and stored within the laboratory. Archived autotune records will be stored electronically in Paradigm.

3. Instrumental Set-up

3.1 Only Agilent compatible vials with crimp tops will be used.

3.2 All vials will be barcoded prior to analysis.

3.3 Manual injections will be 0.5 mL of headspace sample.

3.4 Liquid injections will be 1 uL of each liquid sample extract, standard or blank.

3.5 The solvent wash vials will be filled with appropriate solvents. One vial will contain pentane or other appropriate non-polar solvent and another vial will contain methanol or other appropriate polar solvent.

3.6 An Rtx-5MS Capillary Column - 5% Diphenyl, 95% Dimethlypolysiloxane, 30m x 0.2 mm ID x 0.25µm, or equivalent, will be used for fire debris analysis.

3.6.1 Upon installation, the capillary column will be conditioned per manufacturer's recommendations. A resolution check mix will be analyzed prior to utilizing the column in forensic casework. This may be done separately or at the beginning of a sample sequence.

4. Methods

4.1 The "ACS Method" will generally be used to evaluate samples extracted by Activated Charcoal Strip. This method may be used to evaluate samples from a solvent extraction.

4.2 Ignitable liquid library samples will be evaluated using the "ACS Method". When necessary, library samples will be evaluated using the other methods.

4.3 The "Lite Method" will be used to evaluate samples from a simple headspace extraction.

4.4 The "HACS Method" may be used to evaluate samples from a solvent extraction.

4.5 The "Heavy Method" will be used to evaluate waxes, oils and other heavy products that cannot be resolved using the "ACS Method".

4.6 The "ACSSIM Method" will be used at the analyst's discretion if increased sensitivity is desired.

4.7 The examiner will note the instrumental method used.

4.8 Upon changing columns, or other routine maintenance, slight adjustments to the method may be necessary. These adjustments will be identified using a version number system. Old version methods will be maintained in the computer. The electronic versions of the methods will be backed-up on the laboratory network.

5. Quality Control

5.1 The examiner will refer to the instrument manuals for instructions whenever necessary.

5.2 A known standard (Resolution Check Mix) will be run each day that the instrument is in use. If the sequence will be running for more than a 24 hour period, the examiner will include a resolution check mix at approximately 24 hour intervals.

5.2.1 If a resolution check mix is not performed prior to running samples on the instrument, the examiner may choose to re-run the sequence or to run a resolution check mix at the end of the sequence.

5.2.2 Resolution check mix will be run after maintenance prior to processing case samples.

5.3 The resolution check mix stock solution will consist of the following:

Tetrachloroethylene	1 ml	2-ethyltoluene	1 ml
Hexane	1 ml	3-ethyltoluene	1 ml
Octane	1 ml	Toluene	1 ml
Decane	1 ml	1,2,4 Trimethylbenzene	1 ml
Dodecane	1 ml	p-Xylene	1 ml
Tetradecane	1 ml	Naphthalene	~ 1g
Hexadecane	1 ml	Ethylbenzene	1 ml
Octadecane	~ 1g	Cyclohexane	1 ml
Eicosane	~ 1g	Isooctane	1 ml
	1 ml	Cyclopentane	1 ml

5.4 The resolution check mix stock solution will be diluted approximately 2 ul / ml in Dichloromethane with Diphenylmethane for daily performance checks.

5.5 The data from the resolution check mix is acceptable when the following occur:

- Tetrachloroethylene peak is above 6 minutes
- 1,2,4 Trimethylbenzene and decane peaks are resolved
- Naphthalene and dodecane peaks are resolved
- Diphenylmethane peak is above 18 minutes

5.6 If the resolution check mix produces unacceptable data, the instrument will be removed from service until maintenance is performed.

5.6.1 If the resolution check mix was part of a sequence, the entire sequence will be re-run.

5.7 The resolution check mix data will be stored electronically.

5.8 A solvent blank will be run before and after all casework samples.

5.9 Data will be stored on the laboratory network drive, which is backed-up daily.

5.10 If an examiner requires further instructions, hardware instruction manuals are located within the laboratory near the instrument. Software instructions are located electronically within the software.

6. Maintenance

6.1 If the GC/MS is covered by an Agilent Service Contract, the instrument will undergo a biennial preventative maintenance visit by an Agilent approved technician. As previously mentioned, a resolution check mix is run through the instrument prior to running any case samples. Any issues with this check mix or with the weekly autotune are addressed through in-house maintenance and repair.

6.2 The instrument is maintained in the secure access portion of the laboratory. Should the instrument require storage or transportation, the laboratory will contact the manufacturer for guidance.

6.3 The maintenance is performed as needed. However, a suggested maintenance schedule is as follows:

Monthly
- Check foreline pump oil
- Autotune

Quarterly
- Replace split vent trap
- Replace inlet liner

Semi-annually
- Replace foreline pump oil

- Clean ion source
- Replace gold seal

Annually

- Replace oxygen trap
- Replace hydrocarbon trap
- Replace moisture trap

General maintenance to be performed as needed:

- Replace Merlin Microseal
- Activate filament
- Replace filament
- Install column
- Replace syringe
- Replace diffusion pump oil