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Gas Chromatograph Mass Spectrometer Analytical Method

1.0.0 Background

The gas chromatograph mass spectrometer (GC/MS) is an analytical instrument that separates and identifies a wide variety of organic compounds based on their mass spectra and retention time data.

2.0.0 Scope

The purpose of this Analytical Method is to layout the basic daily tune, scheduled periodic maintenance, sample preparation, and data interpretation necessary to perform quality analysis using a GC/MS. This method is limited to those compounds that produce adequate spectra and chromatography using the instruments owned and operated by the Idaho State Police.

3.0.0 Equipment and Reagents

3.1.0 Equipment

3.1.1 A GC/MS and corresponding analytical software.

3.1.2 Capillary column and data acquisition methods sufficient to separate the analytes of interest.

3.2.0 Reagents

3.2.1 ACS grade, or better, organic solvents.

3.2.2 Standards of the analytes of interest. Standard solutions may be prepared in-house or purchased from a commercial source. They can contain a single analyte or a mixture but all must be authenticated before use in casework.

3.2.3 Sodium carbonate and bicarbonate.

4.0.0 Mass Spectrometer Tune

4.1.0 Frequency

4.1.1 Using Hewlett-Packard/Agilent software and instrumentation an AUTOTUNE will be run after every major maintenance procedure, i.e. source cleaning or column change. They will also be run whenever a drift from expected values is encountered in the QUICKTUNE, see 4.2.

- 4.1.2 Using Hewlett-Packard/Agilent software and instrumentation, a successful MS QUICKTUNE or AUTOTUNE, will be run each day that the instrument is used. A day is defined as a twenty-four (24) hour period starting at the time of the tune. If a sequence of samples will run longer than twenty-four hours then it must be interrupted and a successful AUTOTUNE/ QUICKTUNE, and daily standard run before the sequence can continue.
- 4.2.0 Definition of a Successful Tune (using PFTBA)
Using Chemstation software the following parameters should be met.
- 4.2.1 Mass assignments within +/- 0.2 AMU of 69, 219, and 502
- 4.2.2 Peak widths (PW) should be within 0.1 AMU of 0.55.
- 4.2.3 The relative abundances should show 69 as the base peak, although it might switch with the 219 peak. Under no circumstances should the base peak be anything other than 69 or 219. The relative abundances should be anything greater than 30% for 219, anything higher than 1% for 502.
- 4.2.4 The Isotope mass assignments should be approximately 1 AMU greater than the parent peak and the ratios should be 0.5-1.5% for mass 70, 2-8% for mass 220, and 5-15% for mass 503.
- 4.2.5 The presence of mass 18 (water) and/or 28 (nitrogen) indicate an air leak into the system. If either mass is above 10% relative abundance then maintenance to repair an air leak is required. The exception to this rule is, one to four hours following the pump down of the system or the refilling of the calibration vial; there may be residual air in the system.
- 4.3.0 The QUICKTUNE, STANDARD SPECTRA TUNE, and AUTOTUNE printouts shall be initialed by a drug analyst and kept in a logbook.

5.0.0 GC/MS Quality Assurance

- 5.1.0 For each GC/MS, a standard containing at least one controlled substance will be analyzed on each day that samples are to be run. This standard should be run before any casework is analyzed. If for any reason this standard fails, change of retention time, MS scan etc., then the samples analyzed after the previous standard and before the failed standard are to be considered suspect (for the failed analyte). It will be left to the analyst's discretion whether or not the failure of the standard is germane to each sample and whether the affected samples need to be reanalyzed. The failure of the standard due to instrument failure should be noted in the logbook, along with whatever maintenance that was performed to remedy the situation.
- 5.2.0 To confirm any substance, there must be a standard of that substance analyzed within twenty-four hours of the sample run.
- 5.3.0 Sample extracts are not to be concentrated to less than approximately 250ul, the volume of a vial insert.

6.0.0 General Scheduled Maintenance

All non-consumable items that are repaired or replaced must be entered into the maintenance logbook. Entries into the logbook should include any symptoms of problems along with the status of the system after the repair has been completed.

6.1.0 Daily (consumables). These items are needed to operate the GC/MS system but their replacement, or repair, does not need to be entered into the maintenance logbook.

6.1.1 Perform Autotune

6.1.2 Check and fill solvent rinse vial on autosampler, empty waste solvent vials.

6.2.0 Monthly and after maintenance run a column efficiency standard mix consisting of phentermine, methamphetamine, cocaine, testosterone propionate, morphine and alprazolam and compare to previous month's runs. The data acquisition method must clearly separate each of these compounds. Retention times should be within +/- 0.04 minutes. A printout is kept in the maintenance logbook. To establish a baseline for training and information purposes this mix will be run on each GC/MS method and the printouts kept with the printed methods. The exceptions to this are any non controlled substances (blood inhalants, etc.) and meth quant methods. The mix does not need to be rerun on other specifically named methods that are of limited scope unless there is reason to believe that performed maintenance would result in a significant change of retention times, i.e. a different or drastically shortened column.

6.3.0 Quarterly, if possible.

6.3.1 Check the oil level of the rough pump. Fill if needed and note in logbook. This can only be done if the pump is not running.

6.4.0 Annual.

6.4.1 Replace solvent trap and pump oil if so equipped. Should be done when other maintenance is performed, approximately once a year.

7.0.0 Non-scheduled Maintenance

All non-scheduled maintenance is to be performed on an "as needed" basis as indicated from failure of the autotune, poor chromatography, and or other indications of a system failure. All of these types of repairs will be noted in the maintenance log.

7.1.0 Replace or trim column. After a column has been replaced or trimmed the column efficiency standard will be run.

7.2.0 Clean MSD, replace filaments, gold seal, and injection liner, when needed. Consult with manufacturer's manual or software for cleaning procedure.

7.3.0 Replace electron multiplier if, after repeated cleaning of the source, the mv readings remain at or above 3000.

7.4.0 Replace any part, or system

of parts, as necessary.

8.0.0 Data Interpretation

8.1.0 Retention time.

- 8.1.1 A sample's retention time will be considered acceptable if a mass spectral scan of the analyte is within +/- 0.04 min of a matching scan from a known standard. Retention time window was determined using the method described in "EPA SW846, method 8000B, section 7.6, Revision 2, December 1996".
- 8.1.2 The instrumentation and data acquisition parameters must be sufficient to maintain a 0.1 minute retention time difference between analytes of interest that produce similar mass spectra.
- 8.1.3 The analyte of interest's peak shape must be acceptable, i.e. limited tailing or fronting.

8.2.0 Mass spectral interpretation. For the purpose of drug identification, analysis of mass spectra is one of pattern recognition. A great deal of the interpretation is dependent on each analyst's opinion as to what constitutes a match. All comparisons for the purpose of confirmation are made between analytical standards, not library searches, and the sample spectra. The determination of what constitutes a minor peak, and its relative significance, shall be left up to the individual analyst. The following are the minimum requirements to determine a match.

- 8.2.1 Identification of the molecular (parent) ion, if normally present. * Note* Some compounds do not have molecular ions in their mass spectra.
- 8.2.2 Presence of the correct base ion. Exception, for cocaine the base ion of the sample does not have to match that of the standard but does have to be present in significant abundance.
- 8.2.3 The ratios of the relative abundances of the major ions, from the sample, should be similar to those of the standard.

9.0.0 Blanks

The purpose of instrument blanks is to check for carry-over between samples, while an extraction blank (negative control) checks the level of contamination of all solvents etc. used in preparing the sample. It is acceptable to use an extraction blank as the instrument blank. For the purposes of this section an internal standard is not considered an analyte of interest.

- 9.1.0 Frequency. An instrument blank will be run after the daily standard(s) and immediately before each sample(s). An extraction blank is to be prepared and run daily.
- 9.2.0 Interpretation. A blank run is considered blank if the analyte(s) of interest would not be identified using the above criteria from 8.0.
- 9.3.0 If a blank has an identifiable analyte of interest then the blank will be rerun or

replaced until the analyte of interest cannot be identified. The sample(s) immediately following the suspect blank(s) will be re-extracted and reanalyzed after an acceptable blank has been generated.

10.0.0 Documentation.

Only the documentation used to reach the conclusion need be kept in the case-file. These include chromatograms of sample(s), standard(s), library search results, and blank(s).

12.0.0 History

Revision #	Issue or review date	History	Author or Reviewer
0	4/1/01	Original Issue	D.C. Sincerbeaux
1	8/27/02	Add #	D.C. Sincerbeaux
2	1/10/03	Add sec 9	D.C. Sincerbeaux
3	9/13/05	Changed 9.0.0, 9.1.0, 9.2.0 and 9.4.0 became 10.0.0	D.C. Sincerbeaux
4	6/30/06	Changed 6.0.0, 6.4.1, dropped 6.2.0, 6.3.2, 6.5.0. Changed 7.0.0 and 7.2.0	D.C. Sincerbeaux
5	1/12/07	Added new sec #10, 12, 5.3 Added pg #s, changed name, sec 3, dropped 6.1.3 &4, and minor word changes throughout	D.C. Sincerbeaux
6	7/3/2007	Add 8.1.2	D.C. Sincerbeaux
7	6/22/10	Add 8.1.3. Changed 8.2.2, 10.3.1, 2.0.0, delete 3.1.3	

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D.C. Sincerbeaux
Changed 5.0.0, 6.2.0, 6.4.1 Dropped section 10.
D.C. Sincerbeaux

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