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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.

3.2.4 N-Tridecane internal standard. Use the ratio of 1.3ml to 1L chloroform.

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

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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. The exception to this is if the sequence of a methamphetamine quantitation run lasts longer than 24 hours.

4.2.0 Definition of a Successful Tune (using PFTBA)

Using Chemstation/Masshunter 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), 28 (nitrogen), and or 32 (oxygen) may indicate an air leak into the system. If any of these masses are above 10% relative abundance then maintenance to the instrumental system might be required. Although elevated levels indicate an issue, by themselves, they do not affect analysis other than possibly an elevated background noise level. Prolonged exposure to oxygen or water can lead to a decrease in the life of a column and thus should be addressed through maintenance.

4.3.0 The QUICKTUNE 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 run. If for any reason this standard fails, change of retention time, MS scan, weak or no response etc., then a determination of the cause of the failure and corrective actions must be undertaken. Samples may need to be reanalyzed. Consultation with the discipline leader may be necessary. 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, regardless of when the tune(s) were run.

6.0.0 General Scheduled Maintenance

All non-consumable items that are repaired or replaced must be entered into the maintenance logbook. Entrees 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 If so equipped, 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, solvent line, and pump oil if so equipped. Should be done when other maintenance is performed, approximately once a year.
- 6.4.2 Vacuum dust from electronics and fans.

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 chromotagraphy, 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 2500.

7.4.0 Replace any part, or system of parts, as necessary.

7.5.0 Clean injector guides

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. Some compounds do not chomatograph well, i.e. stanozolol.

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, some compounds have several ions that depending on spectral shifting may change base ions, cocaine is an example of this. In these cases 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 a blank is to check for carry-over between samples, and to verify the lack of contamination of the solvents.

9.1.0 Frequency. A blank will be run immediately before each sample.

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, one subsequently identified in the sample, 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 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	D.C. Sincerbeaux
8	7/2/12	Changed 5.0.0, 6.2.0, 6.4.1 Dropped section 10.	D.C. Sincerbeaux
9	2/25/13	Changed 4.1.2, 4.2.5, 6.3.0, 7.3.0, 8.2.2	D.C. Sincerbeaux
10	12/1/14	Changed 4.2.5, 4.3.0, 5.1.0, 5.2.0, 6.4.1, 9.0.0, 9.1.0, 9.3.0 Added 3.2.4, 6.4.2, 7.5.0 deleted 5.3.0	D.C. Sincerbeaux