History for
the GC/MS SOP

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

Approval

Technical Leader _David Sincerbeaux_ Date: 3-24-03

QA/QC Manager Rick Groff _Rick Groff_ Date: 3-17-03

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Gas Chromatograph Mass Spectrometer Standard Operating Procedure

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 spectral and retention time data.

2.0.0 Scope

The purpose of this SOP is to layout the basic daily tune, calibration requirements, scheduled periodic maintenance, and data interpretation necessary to perform quality analysis using a GC/MS.

3.0.0 Equipment, Reagents, and Methods

3.1.0 A GC/MS and corresponding analytical software.

3.2.1 Reagent grade, or better, organic solvents

3.2.2 Standards of the analytes of interest. Standard solutions may be prepared in-house or 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 Capillary column and data acquisition methods sufficient to separate the analytes of interest.

4.0.0 Mass Spectrometer Tune

4.1.0 Frequency

4.1.1 Using Hewlett-Packard software and instrumentation an AUTOTUNE or STANDARD SPECTRA TUNE 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 are encountered in the QUICKTUNE.

4.1.2 Using Hewlett-Packard software and instrumentation, a successful MS QUICKTUNE, or AUTOTUNE, will be run on a daily basis. 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 QUICKTUNE run before the sequence can continue.

4.2.0 Definition of a Successful Tune (using PFTBA)
Using HP-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 0.5. This may vary depending on the instrument.
- 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 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 Calibration

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 will 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 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.

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, do not need to 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.
6.1.3 Check paper in printer.
6.1.4 Check syringe. Clean or replace if necessary.

6.2.0 Weekly (consumables)
6.2.1 Replace autosampler rinse solutions.
6.2.2 Replace injector septa if necessary.

6.3.0 Monthly
6.3.1 Run a column efficiency standard (GROB, Npiso, etc.) and compare to previous months runs, making sure the same type sample mix is analyzed using the same data acquisition method. Retention times should be within +/- 0.04 minutes. A printout is kept in the maintenance logbook.

6.3.2 Check injection liner and O-ring. Replace if necessary.

6.4.0 Quarterly (if needed).
6.4.1 Change precolumn (if installed).
6.4.2 Check rough pump oil. Fill if needed.

6.5.0 Semi-annual.
6.5.1 Vacuum interior and exterior.
6.5.2 Replace solvent trap.

7.0.0 Non-scheduled Maintenance

7.1.0 Replace or trim column as needed. After a column has been replaced or trimmed the column efficiency standard will be run.
7.2.0 Clean MSD when needed. Consult with manufacturers manual 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. 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 windows are determined using the method described in "EPA SW846, method 8000B, section 7.6, Revision 2, December 1996 ". A copy of this method is included in the ISP Controlled Substances SOP manual.
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.

8.2.3 The ratios of the relative abundances of the major ions, from the sample, should be similar to those of the standard.

8.2.4 Major spurious ions in a sample must be accounted for. Possible sources of spurious ions can include background, coeluting compounds etc.

9.0.0 Blanks

The purpose of instrument blanks is to reduce the effects of carry-over between samples.

9.1.0 Frequency. An instrument blank will be run after the daily standard(s) and between all samples.

9.2.0 Definition. A blank 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 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.

9.4.0 Documentation. Only the documentation used to reach the conclusion need be kept in the case-file.

2

Gas Chromatograph Mass Spectrometer Standard Operating Procedure

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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 spectral and retention time data.

2.0.0 Scope

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3.2.1 Reagent grade, or better, organic solvents

3.2.2 Standards of the analytes of interest. Standard solutions may be prepared in-house or 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 Capillary column and data acquisition methods sufficient to separate the analytes of interest.

4.0.0 Mass Spectrometer Tune

4.1.0 Frequency

4.1.1 Using Hewlett-Packard software and instrumentation an AUTOTUNE or STANDARD SPECTRA TUNE 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 are encountered in the QUICKTUNE.

4.1.2 Using Hewlett-Packard 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 QUICKTUNE run before the sequence can continue.

4.2.0 Definition of a Successful Tune (using PFTBA)
Using HP-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 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 will 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.

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, do not need to 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.

6.1.3 Check paper in printer.

6.1.4 Check syringe. Clean or replace if necessary.

6.2.0 Weekly

6.2.1 Replace autosampler rinse solutions.

6.3.0 Monthly

6.3.1 Run a column efficiency standard (GROB, NP ISO, etc.) and compare to previous months runs, making sure the same type sample mix is analyzed using the same data acquisition method. Retention times should be within +/- 0.04 minutes. A printout is kept in the maintenance logbook.

6.3.2 Check injection liner and O-ring. Replace if necessary.

6.4.0 Quarterly .

6.4.1 Check rough pump oil. Fill if needed.

6.5.0 Semi-annual.

6.5.1 Vacuum interior and exterior.

6.6.0 Annual.

6.6.1 Replace solvent trap, if the part is available, and replace pump oil. Should be done when other maintenance is performed but not to exceed one year.

7.0.0 Non-scheduled Maintenance

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

7.2.0 Clean MSD, replace filaments, gold seal when needed. Consult with manufacturers manual 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. 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 windows are determined using the method described in "EPA SW846, method 8000B, section 7.6, Revision 2, December 1996 ". A copy of this method is included in the ISP Controlled Substances SOP manual.

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.

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 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).

History for the GC/MS SOP

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

Approval

Technical Leader ______________ Date: 9-12-05
David Sincerbeaux

QA/QC Manager Richard D Groff Date: 9-13-2005
Rick Groff

#3

Fourier Transform Infrared Spectrometer Standard Operating Procedure

1.0.0 Background

The Fourier Transform Infrared Spectrometer (FTIR) is an analytical instrument that is used to identify compounds based on their infrared absorption properties. The advantages of using FTIR are that it can differentiate stereo isomers and it is f
sample can be analyzed in less than five minutes. The main disadv
order to produce a high quality result; a sample with a r
needed.

Date issued
8-27-02

Date Replaced
7-1-2006

2.0.0 Scope

This SOP will describe the routine maintenance
perform quality analysis using a FTIR.

3.0.0 Equipment and Reagents

3.1.0 A FTIR and corresponding analytical softwa
3.2.0 IR grade potassium bromide (KBr). Should b
3.3.0 Hydraulic or other press for making KBr winc
3.4.0 Any other sample introduction equipment, i.e.

4.0.0 Routine Maintenance

4.1.0 Aside from the normal cleaning of the outside of ncluding the sample chamber, a thorough cleaning of the interi e instrument should be done on an annual basis. Vacuum or spray away dust from interior of instrument making sure not to touch any of the mirrors.

4.2.0 Background spectra will be collected before any samples are run. Background spectra should be run once every hour when performing batch analysis.

4.3.0 Monthly calibration check. Using the manufacture's procedures, a calibration check of the instrument's performance is done using polystyrene film. This procedure will be performed monthly and after any maintenance. All printouts generated are initialed by the analyst and kept in the maintenance logbook. If the calibration does not pass and /or there is any other symptoms of system failure then consult the manufacture. All maintenance is recorded in a logbook.

5.0.0 Standard Library Preparation

In order to confirm the presence of an analyte in a sample, the scan of the sample must match that of a known standard. It is not acceptable to confirm on the basis of a match from a commercially produced library (Georgia State etc.).

5.1.0 Production of valid standard library.

A pure sample of a standard is prepared and analyzed using the same procedures that will be used with an unknown. Once a scan has been produced it can then be stored in an internal library. A match made from this library is acceptable to use for confirmation. Libraries should be made up of all available standards including various salt forms and isomers (d & dl etc.). These standard scans can be produced and entered into the library as they are encountered in casework.

5.2.0 Sample preparation methods are covered under the appropriate analytical method SOP's.

6.0.0 Identification Criteria

If a sample's FTIR spectra matches a spectra of a standard that was prepared the same as the sample, and the second test, if ran, is positive, then the compound is confirmed.

6.1.0 Standard spectra are prepared from authenticated standards and then stored internally for each FTIR instrument, at each laboratory.

6.2.0 FTIR spectra are considered matched if the peaks of the standard are present in the sample, in location, shape, and relative intensities. Any extra major peaks in the sample must be explainable.

History for the FTIR SOP

Revision #	Issue or review date	History	Author or Reviewer
0	4/1/01	Original Issue	D.C. Sincerbeaux
1	8/27/02	Add Section 6 and #	D.C. Sincerbeaux

Approval

Technical Leader ________ Date: 8-27-02
David Sincerbeaux

QA/QC Manager Rick Groff ________ Date: 8-27-02
Rick Groff