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# APPENDICES

# APPENDIX 28

# Section 4: 1613 Data Analysis & Reporting

Paradigm Analytical Labs - Standard Operating Procedure

| Last Revised By: | Asst. Lab Director: | QA Officer: |
|---|---|---|
| W.M. Larkins | C.K. Cornwell | Greg Dickinson |

## Purpose

To describe the processes used in operating the HRGC/HRMS system, as well as the procedures followed in the generation, interpretation and review of laboratory data for Method 1613.

## Summary

This SOP details how to analyze and report samples by EPA Method 1613. HRGC/HRMS is used to detect and quantitate PCDD/Fs. Samples arrive at the MS lab having been extracted and fractionated using procedures in Section 3. Analyses are grouped into 12-hour sequences that include analyses of samples and standards mixtures. Upon completion of the sequence, the analyst reviews the data associated with both standards and samples in order to confirm the validity of the sequence and to determine any potential need for re-analysis or re-extraction. The analyst generates quantitation reports and chromatograms using sophisticated software. These reports are used to generate forms that summarize the results of the analysis.

## 4.1 Operation of HRGC/HRMS

### 4.1.1 Equipment

- HP6890 GC, Micromass Autospec Ultima high resolution mass spectrometer, vortex mixer, 10-100 uL pipette

### 4.1.2 Procedure

- Recall the GC temperature/pressure/flow program.
- Recall the MS experiment (see Table 1).
- Perform any necessary maintenance.
- Tune the MS resolution to 100 ppm at 5% height.
- Acquire location data to calibrate the MS and print a copy of function one MS resolution.
- Inject the window defining/GC resolution/continuing calibration mix (RETCON). Evaluate descriptor-switching times for accuracy. If any window defining peaks have shifted outside the descriptor windows, adjust the switching times before injecting any samples. This injection is also used to verify that there is less than or equal to 25% peak to valley for the two close eluters of 2,3,7,8-TCDD. Print a copy of the GC resolution check. If the valleys are within specifications, proceed to calibrate or verify a previous calibration. If not, further investigation and/or maintenance may be required. Re-inject this solution after maintenance to check for improvement.
- Now that the GC/MS resolution and descriptor switching times have been verified, a series of five initial calibration standards may be injected and reviewed for method requirements. If an initial calibration already exists, a RETCON may be analyzed to verify continuing calibration. If the curve or the RETCON passes method requirements, sample analysis may begin.
- Reconstitution of a sample is accomplished by adding nonane containing the injection standards, capping the vial, and mixing well with a vortex mixer.
- Samples are injected under conditions identical to those used to establish calibration.
- A “back-end” print out of the MS resolution must be performed.
- The calibration data from a sequence is filed in a folder cabinet under the day it was analyzed and includes the all GC/MS resolution checks, window verification, valley verification, front end Retcons, run logs and window defining mix (WDM) retention time sheets.
- Each sample hardcopy should include the quant report, totals pages, deviations, chromatograms, and report forms.
- Columns: DB-225, 30 m, id 0.25 mm, 0.25 μm; DB-5MS, 60 m, id 0.25 mm, 0.25 μm.

Table 1: Mass Descriptors used for Selected Ion Recording HRMS

| Function (#) | Channel (#) | Mass (amu) | Dwell Time (ms) | I.C. Delay (ms) |
|---|---|---|---|---|
| 1 | 1 | 303.9016 | 100 | 20 |
| 1 | 2 | 305.8987 | 100 | 10 |
| 1 | 3 | 315.9419 | 40 | 10 |
| 1 | 4 | 316.9824 | 20 | 10 |
| 1 | 5 | 316.9824 | (Lock) | 50 |
| 1 | 6 | 317.9389 | 40 | 10 |
| 1 | 7 | 319.8965 | 100 | 10 |
| 1 | 8 | 321.8936 | 100 | 10 |
| 1 | 9 | 327.8847 | 40 | 10 |
| 1 | 10 | 331.9368 | 40 | 10 |
| 1 | 11 | 333.9339 | 40 | 10 |
| 1 | 12 | 375.8364 | 30 | 20 |
| 2 | 1 | 339.8597 | 100 | 20 |
| 2 | 2 | 341.8568 | 100 | 10 |
| 2 | 3 | 351.9000 | 40 | 10 |
| 2 | 4 | 353.8970 | 40 | 10 |
| 2 | 5 | 355.8546 | 100 | 10 |
| 2 | 6 | 357.8517 | 100 | 10 |
| 2 | 7 | 366.9792 | 20 | 10 |
| 2 | 8 | 366.9792 | (Lock) | 50 |
| 2 | 9 | 367.8949 | 40 | 10 |
| 2 | 10 | 369.8919 | 40 | 10 |
| 2 | 11 | 409.7974 | 30 | 20 |
| 3 | 1 | 373.8207 | 100 | 20 |
| 3 | 2 | 375.8178 | 100 | 10 |
| 3 | 3 | 380.9760 | 20 | 10 |
| 3 | 4 | 380.9760 | (Lock) | 50 |
| 3 | 5 | 383.8639 | 40 | 10 |
| 3 | 6 | 385.8610 | 40 | 10 |
| 3 | 7 | 389.8156 | 100 | 10 |
| 3 | 8 | 391.8127 | 100 | 10 |
| 3 | 9 | 401.8559 | 40 | 10 |
| 3 | 10 | 403.8530 | 40 | 10 |
| 3 | 11 | 445.7555 | 30 | 20 |
| 4 | 1 | 407.7818 | 100 | 20 |
| 4 | 2 | 409.7788 | 100 | 10 |
| 4 | 3 | 417.8253 | 40 | 10 |
| 4 | 4 | 419.8220 | 40 | 10 |
| 4 | 5 | 423.7767 | 100 | 10 |
| 4 | 6 | 425.7737 | 100 | 10 |
| 4 | 7 | 430.9728 | 20 | 10 |
| 4 | 8 | 430.9728 | (Lock) | 50 |
| 4 | 9 | 435.8169 | 40 | 10 |
| 4 | 10 | 437.8140 | 40 | 10 |
| 4 | 11 | 479.7165 | 30 | 20 |
| 5 | 1 | 441.7427 | 100 | 20 |
| 5 | 2 | 443.7398 | 100 | 10 |
| 5 | 3 | 454.9728 | 20 | 10 |
| 5 | 4 | 454.9728 | (Lock) | 50 |
| 5 | 5 | 457.7377 | 100 | 10 |
| 5 | 6 | 459.7348 | 100 | 10 |
| 5 | 7 | 469.7780 | 40 | 10 |
| 5 | 8 | 471.7750 | 40 | 10 |
| 5 | 9 | 513.6775 | 30 | 20 |

## 4.2 Data Generation, Interpretation and Review

Paradigm Analytical Labs defines a batch of samples as no more than 20 samples processed within a 12-hour shift. One LMB and one OPR are processed per analytical batch, following the same procedures as the field samples. Generally, soil is replaced by salt ($Na_2SO_4$), effluent by deionized water and biological tissues by vegetable oil. An invalid LMB or OPR requires a re-extraction of the affected samples.

### 4.2.1 Quality Assurance/Quality control

On an annual schedule, the laboratory shall perform Method Detection Limit studies (MDLs) for each matrix analyzed. Additionally, the laboratory shall perform and MDL study for each extraction method utilized per matrix. All MDL studies will be conducted following the guidelines set forth in 40 CFR, Part 136, appendix B and must be lower than one-third the regulatory compliance level or one third the Minimum Levels (ML) set forth in Table 2 of the reference method.

### 4.2.2 Initial Calibrations

The percent relative standard deviations for the mean response factors from the seventeen unlabeled standards must not exceed +/- 20%. The percent relative standard deviations from the labeled standards (i. e. extraction standards, cleanup standards and sampling standards) must not exceed +/- 35%. The signal to noise ratio for all signals present must be $\geq 10$. The ion abundance ratios must be within specified control limits (see Table 2). Paradigm uses the concentrations in Table 3 to construct the initial calibration.

Table 2. Theoretical Ion Abundance Ratios and Their Control Limits

| Level of Chlorination | Theoretical Ratio | Control Limits | |
|---|---|---|---|
| | | Lower | Upper |
| 4 | 0.77 | 0.65 | 0.89 |
| 5 | 1.55 | 1.32 | 1.78 |
| 6 | 1.24 | 1.05 | 1.43 |
| 6[a] | 0.51 | 0.43 | 0.59 |
| 7 | 1.04 | 0.88 | 1.20 |
| 7[b] | 0.44 | 0.37 | 0.51 |
| 8 | 0.89 | 0.76 | 1.02 |

[a] Used only for $^{13}$C-HxCDF
[b] Used only for $^{13}$C-HpCDF

A new initial calibration is required when the continuing calibration criteria below are not met. Routine maintenance may be performed to correct any failures. Any major maintenance to the analytical system such as slit cleaning, analyzer lens cleaning, magnet shifts, and detector disk changes warrant a new ICAL. At a minimum, a new initial calibration must be performed annually.

Table 3. Initial Calibration Concentrations

| Analyte | CS-1 | CS-2 | CS-3 | CS-4 | CS-5 |
|---|---|---|---|---|---|
| | Concentration (pg/µL) | | | | |
| Unlabeled | | | | | |
| 2378-TCDD | 0.25 | 2 | 10 | 40 | 200 |
| 2378-TCDF | 0.25 | 2 | 10 | 40 | 200 |
| 12378-PeCDD | 1.25 | 10 | 50 | 200 | 1000 |
| 12378-PeCDF | 1.25 | 10 | 50 | 200 | 1000 |
| 23478-PeCDF | 1.25 | 10 | 50 | 200 | 1000 |
| 123478-HxCDD | 1.25 | 10 | 50 | 200 | 1000 |
| 123678-HxCDD | 1.25 | 10 | 50 | 200 | 1000 |
| 123789-HxCDD | 1.25 | 10 | 50 | 200 | 1000 |
| 123478-HxCDF | 1.25 | 10 | 50 | 200 | 1000 |
| 123678-HxCDF | 1.25 | 10 | 50 | 200 | 1000 |
| 123789-HxCDF | 1.25 | 10 | 50 | 200 | 1000 |
| 234678-HxCDF | 1.25 | 10 | 50 | 200 | 1000 |
| 1234678-HpCDD | 1.25 | 10 | 50 | 200 | 1000 |
| 1234678-HpCDF | 1.25 | 10 | 50 | 200 | 1000 |
| 1234789-HpCDF | 1.25 | 10 | 50 | 200 | 1000 |
| OCDD | 2.5 | 20 | 100 | 400 | 2000 |
| OCDF | 2.5 | 20 | 100 | 400 | 2000 |
| Extraction Standards | | | | | |
| $^{13}$C-2378-TCDD | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-2378-TCDF | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-12378-PeCDD | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-12378-PeCDF | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-23478-PeCDF | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-123678-HxCDD | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-123478-HxCDD | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-123478-HxCDF | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-123478-HxCDF | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-1234678-HpCDD | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-1234678-HpCDF | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-1234789-HpCDF | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-OCDD | 200 | 200 | 200 | 200 | 200 |
| Cleanup Standards | | | | | |
| $^{37}$Cl-2378-TCDD | 0.25 | 2 | 10 | 40 | 200 |
| Injection Standards | | | | | |
| $^{13}$C-1234-TCDD | 100 | 100 | 100 | 100 | 100 |
| $^{13}$C-123789-HxCDD | 100 | 100 | 100 | 100 | 100 |

### 4.2.3 Continuing Calibrations

Check that all paperwork is present. A CCal package should contain the documentation listed below.

- Pass: Run log. HRMS Resolution Checks. WDM retention time sheet. WDM chromatograms. GC performance for 2,3,7,8-TCDD. CCal quantitation page. CCal chromatograms. Injection preparation log.
- Fail: The analyst listed on the run log can provide any missing paperwork.

Review the Run log.

- Pass: Check that the 12 hour windows have not been exceeded between the front end Ccal and the last sample of the sequence.
- Fail: Re-analysis of affected samples.

Review the HRMS Resolution checks.

- Pass: Verify 100ppm width at 5% height for PFK mass 318 or higher. Compare the resolution check times to those on the run log to be sure they bracket each sequence.
- Fail: Back end resolution checks do not have to meet the front end requirements. Should one fail, an assessment should be made to determine any data quality impact.

Review the Window Defining Mix and GC Performance Documentation.

- Pass: Check that the sample numbers on the WDM sheets match those on the run log. Check that the retention times are correct for the WDM chromatograms.
- Check that the valley between 2,3,7,8-TCDD and its close eluters does not exceed 25%.
- Fail: Any missing peaks in the window-defining sample should be re-identified with a survey scan. Determine proper switching times. These must be entered into the HRMS ion function descriptors before analysis may resume. If the GC performance valley is greater than 25% instrument maintenance may be required. When a valley fails all samples must be reinjected.

Review the CCal Quantitation and Chromatograms.

- Pass: Check that all ion ratios are in specification. Verify that all compounds are within the concentration limits set by the method (see Table 4) for all front end CCals
- Fail: Routine instrument maintenance such as installing new injection port hardware, inner source cleaning, retuning, column clipping etc. will usually correct a calibration failure. If these measures do not work, a new ICal is needed.

| Compound Name | CCAL (pg/µL) | Limits (pg/µL) | Compound Name | CCAL (pg/µL) | Limits (pg/µL) |
|---|---|---|---|---|---|
| 2,3,7,8-TCDD | 10 | 7.8 - 12.9 | $^{13}C_{12}$-2,3,7,8-TCDD | 100 | 82 - 121 |
| 1,2,3,7,8-PeCDD | 50 | 39 - 65 | $^{13}C_{12}$-1,2,3,7,8-PeCDD | 100 | 62 - 160 |
| 1,2,3,4,7,8-HxCDD | 50 | 39 - 64 | $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | 100 | 85 - 117 |
| 1,2,3,6,7,8-HxCDD | 50 | 39 - 64 | $^{13}C_{12}$-1,2,3,6,7,8-HxCDD | 100 | 85 - 118 |
| 1,2,3,7,8,9-HxCDD | 50 | 41 - 61 | $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD | 100 | 72 - 138 |
| 1,2,3,4,6,7,8-HpCDD | 50 | 43 - 58 | $^{13}C_{12}$-OCDD | 200 | 96 - 415 |
| OCDD | 100 | 79 - 126 | $^{13}C_{12}$-2,3,7,8-TCDF | 100 | 71 - 140 |
| 2,3,7,8-TCDF | 10 | 8.4 - 12 | $^{13}C_{12}$-1,2,3,7,8-PeCDF | 100 | 76 - 130 |
| 1,2,3,7,8-PeCDF | 50 | 41 - 60 | $^{13}C_{12}$-2,3,4,7,8-PeCDF | 100 | 77 - 130 |
| 2,3,4,7,8-PeCDF | 50 | 41 - 61 | $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | 100 | 76 - 131 |
| 1,2,3,4,7,8-HxCDF | 50 | 45 - 56 | $^{13}C_{12}$-1,2,3,6,7,8-HxCDF | 100 | 70 - 143 |
| 1,2,3,6,7,8-HxCDF | 50 | 44 - 57 | $^{13}C_{12}$-2,3,4,6,7,8-HxCDF | 100 | 74 - 135 |
| 2,3,4,6,7,8-HxCDF | 50 | 45 - 56 | $^{13}C_{12}$-1,2,3,7,8,9-HxCDF | 100 | 73 - 137 |
| 1,2,3,7,8,9-HxCDF | 50 | 44 - 57 | $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF | 100 | 78 - 129 |
| 1,2,3,4,6,7,8-HpCDF | 50 | 45 - 55 | $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | 100 | 77 - 129 |
| 1,2,3,4,7,8,9-HpCDF | 50 | 43 - 58 | $^{37}Cl_4$-2,3,7,8-TCDD | 10 | 7.9 - 12.7 |
| OCDF | 100 | 63 - 159 | | | |

Table 4. Continuing Calibration Limits

Review the Injection Prep log sheet.

- Pass: Check that all samples have been spiked with 2 ng injection standard. Verify that final volume is 20 uL. Be sure that any dilutions or other comments are noted.
- Fail: Calculations of sample concentrations should reflect any deviations from normal injection prep parameters.

### 4.2.4 Quality Control Work Groups

The following elements should be present in a complete work group file:

- LMB topsheets
- LMB totals sheets
- LMB chromatograms (11 pages)

- OPR topsheets
- OPR chromatograms
- Extraction log sheet
- Cleanup log sheet
- ASE/Cleanup observation forms
- Dry weight sheet (where applicable)
- Any additional information (ex. re-extract request sheet)

The following procedure should be used for reviewing a work group:

- Review the header information on the LMB topsheets. Verify that the method and client sample ID (LMB or OPR) are correct.
- Review the footer information on the LMB and OPR topsheets. Verify that the following information is correct: Paradigm sample ID or OPR project number, extraction date, analysis date, method, matrix, sample weight/volume, percent solids/lipids, pH, work group number, sample datafile, retcheck datafile, beginning cal datafile and ICal datafile.
- Verify that no target analytes or EMPCs are present in the LMB above Method 23's Minimum Levels. If target analytes or EDL's are above this limit, the associated samples must have concentrations that exceed 10 times the LMB concentration for the specified analyte. Otherwise, samples must be re-extracted.
- Review the totals data for the LMB. Be sure that any ghosting peaks are removed from the totals concentrations and the associated detection limits are elevated to reflect the subtracted peaks.
- Verify that extraction and cleanup standard recoveries are within method specifications (see Table 5) for the LMB and OPR. These recoveries are found on the topsheets. Validate any failures based upon signal to noise and acceptable detection limits. If the lab validation fails a corrective action is required. Corrective actions may include re-extraction, re-cleanup, lower sample volume, extract dilution, etc.
- Verify that the recoveries in the OPR meet Paradigm's recovery limits, found in Table 6.

| Compound Name | Amount Spiked (pg/µL) | Limits % |
|---|---|---|
| $^{13}C_{12}$-2,3,7,8-TCDD | 100 | 25 - 164 |
| $^{13}C_{12}$-1,2,3,7,8-PeCDD | 100 | 25 - 181 |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | 100 | 32 - 141 |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDD | 100 | 28 - 130 |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD | 100 | 23 - 140 |
| $^{13}C_{12}$-OCDD | 200 | 17 - 157 |
| $^{13}C_{12}$-2,3,7,8-TCDF | 100 | 24 - 169 |
| $^{13}C_{12}$-1,2,3,7,8-PeCDF | 100 | 24 - 185 |
| $^{13}C_{12}$-2,3,4,7,8-PeCDF | 100 | 21 - 178 |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | 100 | 26 - 152 |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDF | 100 | 26 - 123 |
| $^{13}C_{12}$-2,3,4,6,7,8-HxCDF | 100 | 29 - 147 |
| $^{13}C_{12}$-1,2,3,7,8,9-HxCDF | 100 | 28 - 136 |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF | 100 | 28 - 143 |
| $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | 100 | 26 - 138 |
| $^{37}Cl_4$-2,3,7,8-TCDD | 10 | 35 - 197 |

Table 5. Labeled Standard Recovery Limits

| Analyte | Amount Spiked (pg/µL) | Limit (pg/µL) |
|---|---|---|
| 2378-TCDD | 10 | 6.7-15.8 |
| 12378-PeCDD | 50 | 35-71 |
| 123478-HxCDD | 50 | 35-82 |
| 123678-HxCDD | 50 | 38-67 |
| 123789-HxCDD | 50 | 32-81 |
| 1234678-HpCDD | 50 | 35-70 |
| OCDD | 100 | 78-144 |
| | | |
| 2378-TCDF | 10 | 7.5-15.8 |
| 12378-PeCDF | 50 | 40-67 |
| 23478-PeCDF | 50 | 34-80 |
| 123478-HxCDF | 50 | 36-67 |
| 123678-HxCDF | 50 | 42-65 |
| 123789-HxCDF | 50 | 39-65 |
| 234678-HxCDF | 50 | 35-78 |
| 1234678-HpCDF | 50 | 41-61 |
| 1234789-HpCDF | 50 | 39-69 |
| OCDF | 100 | 63-170 |
| | | |
| $^{13}$C-2378-TCDD | 100 | 20-175 |
| $^{13}$C-12378-PeCDD | 100 | 21-227 |
| $^{13}$C-123478-HxCDD | 100 | 21-193 |
| $^{13}$C-123678-HxCDD | 100 | 25-163 |
| $^{13}$C-1234678-HpCDD | 100 | 26-166 |
| $^{13}$C-OCDD | 200 | 26-397 |
| | | |
| $^{13}$C-2378-TCDF | 100 | 22-152 |
| $^{13}$C-12378-PeCDF | 100 | 21-192 |
| $^{13}$C-23478-PeCDF | 100 | 13-328 |
| $^{13}$C-123478-HxCDF | 100 | 19-202 |
| $^{13}$C-123678-HxCDF | 100 | 21-159 |
| $^{13}$C-123789-HxCDF | 100 | 17-205 |
| $^{13}$C-234678-HxCDF | 100 | 22-176 |
| $^{13}$C-1234678-HpCDF | 100 | 21-158 |
| $^{13}$C-1234789-HpCDF | 100 | 20-186 |
| | | |
| $^{37}$Cl-2378-TCDD | 10 | 3.1-19.1 |

Table 6. OPR Recovery Limits

## 4.3 Data Review

### 4.3.1 Procedure

- Complete Data Review Checklist (Section 4, Appendix A)

### 4.3.2 Calculations

#### 4.3.2.1 Target compound calculation

- $$\text{PCDD/PCDF (ppt)} = \frac{\text{(Sum Ion Abun. of analyte)(ES Amount)}}{\text{(Sum Ion Abun. of Int. Std)(RRF from ICal)(Amt. of Sample)}}$$

- EMPC (ppt) = $\frac{\text{(Sum Ion Abun. of analyte)(ES Amount)}}{\text{(Sum Ion Abun. of Int. Std)(RRF from ICal)(Amt. of Sample)}}$

- EDL = $\frac{\text{2.5 (Height of Noise)(Std. Amount)}}{\text{(Height of Noise from Int. STD.)(RF from ICal)(Amt. of Sample)}}$

The instrumentation software calculates the noise level. However, manual noise determination may be employed at the reviewer's discretion in order to more accurately report peaks of interest.

#### 4.3.2.2 Extraction Standard Recovery Calculation

- % Recovery = $\frac{\text{(Sum Ion Abun. of ES)(JS Amount)}}{\text{(Sum Ion Abun. of JS)(ES RRF from ICal)(ES Amount)}}$

The clean-up standard recoveries are calculated as above, substituting the ion abundances from the individual clean-up standard for the extraction standard

### 4.3.3 Requests for Re-extraction

Review all supporting data, including spike profiles, extraction logs, clean-up logs, injection prep logs, observation forms, and the sample tracking forms in the folder. The project or work group folder may contain exceptions or changes to routine spiking procedures.

Check the sample for problems relating to analysis. These problems include response factors that may introduce quantitative errors, interference that could be diluted out, or any interference that causes de-tuning or chromatographic conditions that could lead to quantitative errors.

The Laboratory Supervisor or Director should be consulted when re-extraction is considered.

If re-extraction is necessary, complete the Re-Extraction Form, which indicates the sample id, re-extraction due date, and reason for re-extraction (ref. form DC18).

When the GC/MS analyst receives the form, the samples are marked "REX" in the LIMS. The Sample ID will receive an "R" suffix. If a sample requires a second or third re-extraction, the sample id suffix will change to S, then T, and so on. The sample id with the suffix is used in all paperwork. (extraction, clean-up, injection prep, and run logs).

## 4.4 Reference Method

"Guidelines Establishing Test Procedures for the Analysis of Pollutants; EPA Method 1613," *Federal Register*, Vol. 62(178): 48393-48442, September 15, 1997; *Final Rule*.