

US EPA ARCHIVE DOCUMENT

**USER'S GUIDE FOR
SURROGATE MATRIX NORMALIZATION
(SMCREPORTER)**

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SECTION 1 INTRODUCTION

1.1 System Purpose

Smcreporter is a program to simplify and standardize EPA method 8261 surrogate matrix normalization for volatile organic compounds in a variety of environmental matrices (a description of method 8261 is available at <http://www.epa.gov/sw-846/pdfs/8261.pdf>).

1.2 Document Organization

This User's Guide is intended to enable users to access functions of the Smcreporter application. Use of the application to perform Method 8261 normalization will be described in detail.

Section 2 – System Overview – describes technical details of Smcreporter. This section includes system requirements and a discussion of essential concepts for use of the application.

Section 3 – System Operation – provides a description of the software user interface as currently implemented and a step-through list for performing Method 8261 normalization procedures.

1.3 User Profile

Users of this system are expected to be experienced technical personnel, familiar with GC/MS, microcomputers and Windows operating systems. They should be familiar with method 8261 procedures and concepts.

1.4 Conventions

To help the user to easily interpret information in this manual, certain conventions will be used:

- When indicating information to be entered from the keyboard, text will be shown in COURIER font. Specific keys will be identified by label, enclosed in angle brackets. For example, `Type this.<Enter>` means to type in the phrase: Type this. Then press the Enter key.
- Nested menu selections will be separated by a vertical bar (|). File | Exit means to select the 'File' menu option in the top-level menu, then select 'Exit' from the next menu.
- The standard DOS directory and file conventions will be used.

SECTION 2 SYSTEM OVERVIEW

2.1 System Requirements

Smcreporter was written, tested, and has been demonstrated to work for stand-alone data analysis on PCs using Windows 98, Windows NT version 4, and Windows 2000 operating systems.

Systems used to run Smcreporter must meet the following minimum requirements:

- Windows operating system (Windows 98, Windows NT version 4, or Windows 2000)
- Pentium 166 MHz
- Super VGA display (800 x 600 resolution)
- 2 GB hard disk
- CD-ROM drive
- 64 MB RAM.

2.2 Essential Concepts

The purpose of Smcreporter is to simplify the application of Method 8261 surrogate matrix normalization computations. Method 8261 provides a basis to determine the concentration of volatile organic compounds (VOCs) in various matrices. The method is described in an on-line EPA document – Volatile Organic Compounds by Vacuum Distillation in Combination With Gas Chromatography/Mass Spectrometry (VD/GC/MS), available at <http://www.epa.gov/epaoswer/hazwaste/test/txup4b.htm>. The experimental basis of the method is explained in previous publications.^{1,2} The method will be broadly discussed and terminology introduced in section 2.3.1.

¹ Hiatt, M.H; Farr C.M. Volatile Organic Compound Determinations Using Surrogate-Based Correction for Method and Matrix Effects, Analytical Chemistry, 1995, 67, 426-433.

² Hiatt, M.H. Vacuum Distillation Coupled with Gas Chromatography/Mass Spectrometry for the Analysis of Environmental Samples, Analytical Chemistry, 1995, 67, 4044-4052.

2.2.1 Basis and Terminology

Method 8261 employs an instrument calibration using spiked samples of VOCs to calculate analyte recoveries from vacuum distillation and cryogenic trapping. VOCs used in spiked samples are referred to as surrogates. By introducing known amounts of surrogates into field samples, the sample analyte recoveries can be calculated based on the measured recovery of the surrogates.

The physical properties of interest for recovery of VOCs by vacuum distillation and cryogenic trapping are relative volatility¹ and boiling point. In keeping with other publications, the following terms will be used:

- Relative volatility describes the potential of a compound to be extracted from a matrix.
- Relative volatility surrogates (-surrogates) are compounds used to assess relative volatility effects on analyte recoveries.
- Boiling point describes the potential of a compound to condense.
- Boiling point surrogates (-surrogates) are compounds used to assess condensation effects on analyte recoveries.

2.2.2 Text Input Files

There are various text files that are required inputs for this program. Only two – the compound library file and quantitation results file – must be created by the user. Other files – surrogate library, calibration file, and minimum detectible limits (MDL) file – are created and maintained using program subroutines and, although they are text files, it is recommended that they not be manually edited.

2.2.2.1 The Compound Library

Smcreporter maintains a library (compound library) of compounds and parameters necessary for Method 8261 calculations. The compound library should contain the same set of compounds as the instrument software. The compound library file is a tab-delimited text file in the following format:

- A header line which reads: COMPOUND LIBRARY. This line is required.
- A list of compound names, each on a separate line. The compound name must be followed by a tab character. These lines are required.
- For each compound, the following optional fields may be included following the

compound name:

- ▶ a compound type string beginning with 'a' or 's' (case insensitive) to indicate whether the compound is an analyte ('a') or a surrogate ('s')
- ▶ the relative volatility – water to air (RVW)
- ▶ the boiling point (BP)
- ▶ the relative volatility – octanol to air (RVO)
- ▶ the partition coefficient (Part Coef)
- ▶ up to five concentrations (C1-C5) for spiked calibration standards.

Entries for each field must be concluded with a tab character. For any optional fields not included in the file, the program will assign default values. The order of fields and default values are shown in Table X. Defaults are not intended as reasonable approximations to be used in calculations, they are flag placeholders until actual values are entered. Optional fields are like default arguments in common programming languages – in order to specify a value for a field, all previous fields must also be specified.

Table X – Order of Library File Fields and Default Values

	Compound Name	Compound Type (A/S)	RVW	BP	RVO	Part.Coeff	C1	C2	C3	C4	C5
Default	None	analyte	999	999	999	999	99	99	99	99	99

A portion of a library file with some example records is shown below:

COMPOUND LIBRARY

```
diethylether-d10    S    32.5  32
acetone-C13  A    600   57   999   999   3100  3100  3100  3100  3100
ethanol-d3
```

For acetone-C13, notice that in order to specify concentrations (C1-C5), values for RVO and Part Coef must be included (in this example, values equal to program defaults were used).

To create the compound library text file:

- Import a text report from the GC/MS into a spreadsheet. Importing a GC/MS report generally provides a listing of all compounds in the GC/MS software and ensures consistent spelling of compound names.

- Check that compound names are correct and complete (they may be truncated in the report, or by the spreadsheet's import routine).
- Delete any unnecessary information and add any desired optional fields as indicated above.
- Save the spreadsheet as a text file.
 - ▶ Excel: Use File | Save As... and select Save as type: Text (Tab delimited)(* .txt).
 - ▶ QuatroPro: Use File | Save As... and select File Type: ASCII Text (Tab delimited).
 - ▶ Lotus 1-2-3: Select the desired fields and save them to the clipboard. Paste the selection into Notepad. Use File | Save to save the selection as a file.

The library file can be updated through the 'Update Library...' menu option (see section 3.1.2.2).

2.2.2.2 Quantitation Results File

The quantitation results file lists compounds and area counts resulting from a GC/MS quantitation. It is a tab delimited file. A sample is shown below:

```
DATA FILE
diethylether-d10    262976.00
acetone-C13    2451460.00
ethanol-d3    5.750E05
```

The heading 'DATA FILE' is required. The list of compounds must be the same spellings and order as the compound library and each compound must have area counts (surrogates should never have area counts of zero; zero is acceptable for analytes). Follow the same procedures as those for creating the compound library file (see section 2.2.2.1).

2.2.2.3 Surrogate File Essentials

Surrogate compounds are selected to cover the range of relative volatilities and boiling points for analytes being measured. There are four classes of surrogate compounds:

- ▶ First pass surrogates attempt to estimate the effects of relative volatility on compound recoveries, as independently of boiling point effects as possible. First pass surrogates should have low boiling points (< 100°C) to minimize any boiling point effects.
- ▶ Boiling point surrogates reflect the effect of boiling point on compound recovery.
- ▶ Relative volatility surrogates estimate the effect of relative volatility on compound

recovery.

- ▶ Check surrogates are used as quality assurance checks on the Method 8261 calculations.

First pass, boiling point, and relative volatility surrogates are grouped in smaller ranges to more accurately depict the expected recovery of compounds. Linear regressions are used to characterize recoveries for each surrogate grouping.

Surrogate groups are maintained using the Surrogate Data dialog box (section 3.1.2.7). In the Surrogate Data dialog box, all compounds selected as surrogates in the 8261 library are listed.

Smcreporter requires that there be at least three first pass surrogates. Select first pass surrogates by indicating 'Yes' in the 'First Pass?' list box. First pass surrogates are first ordered by increasing relative volatility, then automatically assigned to groups of two. Table 1 shows example first pass surrogates and groupings.

Table 1 - First Pass Surrogate Groups

Compounds	Group	Relative Volatility Range
Hexafluorobenzene / Fluorobenzene	1	0.86 - 3.5
Fluorobenzene / 1,2-dichloroethane-d4	2	3.5 - 20
1,2-dichloroethane-d4 / Teterahydrofuran	3	20 - 355

In Table 1, there are four first pass surrogates defined to cover the range of relative volatilities from 0.86 to 355. Note that adjacent groups share compounds. The high relative volatility for the first group becomes the low relative volatility for the second group, etc. First pass surrogates must be selected to cover the range of relative volatilities for boiling point surrogates.

Boiling point and relative volatility surrogates are manually assigned to groups in the Surrogate Data form, using the 'Boiling Point Group' and 'Relative Volatility Group' check boxes (see section 3.1.2.5). There must be at least three compounds per group and at least two groups for each surrogate type. Up to six groups can be defined. Table 2 shows example groups.

Table 2 - Example Surrogate Groupings

Group	Boiling Point	Relative Volatility
1	Pentafluorobenzene (85) Toluene-d8 (111) Bromobenzene-d5 (155)	Hexafluorobenzene (0.86) Fluorobenzene (3.5) 1,4-difluorobenzene (3.83)
2	Bromobenzene-d5 (155) 1,2-dichlorobenzene-d4 (181) 1,2,4-trichlorobenzene-d3 (213)	Fluorobenzene (3.5) 1,4-difluorobenzene (3.83) O-xylene-d10 (6.14) Chlorobenzene-d5 (6.27)
3	1,2,4-trichlorobenzene-d3 (213) Naphthalene-d8 (217) 1-methylnaphthalene-d10 (245)	O-xylene-d10 (6.14) Chlorobenzene-d5 (6.27) 1,2-dibromoethane-d4 (26) 1,1,2-trichloroethane-d3 (26.6)
4		1,2-dibromoethane-d4 (26) 1,1,2-trichloroethane-d3 (26.6) Tetrahydrofuran-d8 (355) Acetone-c13 (600)
5		Tetrahydrofuran-d8 (355) Acetone-c13 (600) 1,4-dioxane-d8 (5800) Pyridine-d5 (15000)

Notice that the groups are defined so that there are one or two compounds common to adjacent groups. There cannot be any gap between groups. In the Surrogate Data form, check two group boxes for compounds that are shared; i.e., check boxes 4 and 5 for Tetrahydrofuran-d8.

Linear regressions determined by each group are used to calculate expected recoveries for compounds. The selection of limits for the regression lines affects which regression will be used to calculate recoveries. Some care must be taken in the selection of surrogate groups to ensure that the desired regression is used. The algorithm for selecting regressions is included in Appendix 3.

2.2.2.4 Surrogate Files

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The surrogate files are Windows initialization (.ini) format files (see Appendix 3). An .ini file is simply a text file. However, it has a structure which allows Windows to quickly find data. Data is organized in sections. Each section starts with a title in brackets. Under each section are keys with associated values.

An example surrogate file follows:

```
[compound1]
name=diethylether-d10
LibCmpndNum=1
bpgroup1=0
bpgroup2=0
bpgroup3=0
bpgroup4=0
bpgroup5=0
bpgroup6=0
rvwgroup1=0
rvwgroup2=1
rvwgroup3=1
rvwgroup4=0
rvwgroup5=0
rvwgroup6=0
first_pass=0
checked_surrogate=0
[header]
number_comps=1
[limits]
MinBP=85
MaxBP=231
MinRVW=0.86
MaxRVW=9327.3790530888
```

There are 1..n compound sections, one for each compound. The header section has one key – the number of compounds listed in the file. The limits section is optional. If it exists, all four keys must be included.

Surrogate files can be created using 'File | Create Surrogate File...' or 'File | Copy Surrogate File...' (see sections 3.1.2.3 and 3.1.2.7). Use 'File | Edit Surrogate File...' (see section 3.1.2.5) to assign compounds to groups. While the files can be edited using a text editor, it is recommended that they be created and maintained using program menu options.

2.2.2.5 MDL File

MDL files are semicolon delimited text files. An example follows:

```
Creation Date;7/5/2002 4:52:28 PM
Compound Library File:C:\HPCHEM\1\METHODS\TESTMETH.M\8261dir\lib8261.ini
Surrogate File;C:\HPCHEM\1\METHODS\TESTMETH.M\8261dir\Surrogates\2groups.ini
12.00;sludge;ng/l
Other;
diethylether-d10;4.00
dichlorodifluoromethane;1.00
chloromethane;3.00
bromomethane;4.30
chloroethane;2.10
```

There are five required header lines. These lines include information to identify properties of samples the MDLs apply to. The labels on the first three lines and the fifth line are required. The fourth line shows the sample size, matrix type, and reporting units. The fifth line is a comment line. Each compound and associated MDL are listed on the following lines. Compounds must be listed in the same order as in the compound library. It is not necessary to list all compounds, but there cannot be any gaps in the compounds listed.

MDL files can be created and maintained using the 'File | Create/Edit MDL File...' menu option (see section 3.1.2.9). It is recommended that the menu option be used to manipulate MDL files.

SECTION 3 SYSTEM OPERATION

3.1 Smcreporter Operation

3.1.1 Starting Smcreporter

The install procedure adds a program icon to the Windows Start menu. Click 'Start' | 'Programs' | 'Smcreporter' to start the program. The program main window is titled 'Surrogate Matrix Correction'.

The program main menu has four options – File, Calibration, Quantitation, and Help – which allow access to all program functions. Each main menu option has an associated drop-down menu. All menu options will be explained in the following sections. Whenever it is necessary to reference a menu item on a separate drop-down menu, the main menu and drop-down items will be listed, separated by a vertical line, e.g. 'File' | 'Open Library...'

Menu options are enabled or disabled based on user selections and logical choices available. Options may be disabled following error messages, due to possibly corrupted memory structures.

3.1.2 File Menu

The 'File' main menu provides access to routines to manage compound library, surrogate, calibration, and minimum detectable limit (MDL) files. Drop-down menu items are separated into functional groups by file type. The program 'Exit' option is on this menu.

3.1.2.1 Open Library...

The 'Open Library...' menu option allows selection of a library file – a tab-delimited text file listing all compounds of interest and their physical properties. Selecting this option brings up a standard file open dialog box, which allows access to the full directory structure to select a properly formatted library file.

In the open dialog box, select a file. Click the 'Open' button to open the selected file, or 'Cancel' to close the Open dialog without opening a library file.

The following menu changes will occur when selecting a library file:

- If a library file is not previously loaded, the 'Update Library...', 'Create Surrogate File...', 'Open Surrogate File...', and 'Create/Edit MDL File...' menu options will be enabled when a library file is opened.
- The 'Update Library...' menu option will be modified to show the library file currently in program memory.
- If a calibration file was previously loaded in memory, it is no longer valid when the library file is opened. The 'Calibration' and 'Quantitation' main menu options will be disabled when a library file is opened.

3.1.2.2 Update Library...

This option allows updating of the library file by entering compound type, physical properties, and calibration concentrations for each library compound. A dialog box titled 'Update Library', followed by the library file name, will be displayed (Figure 1).

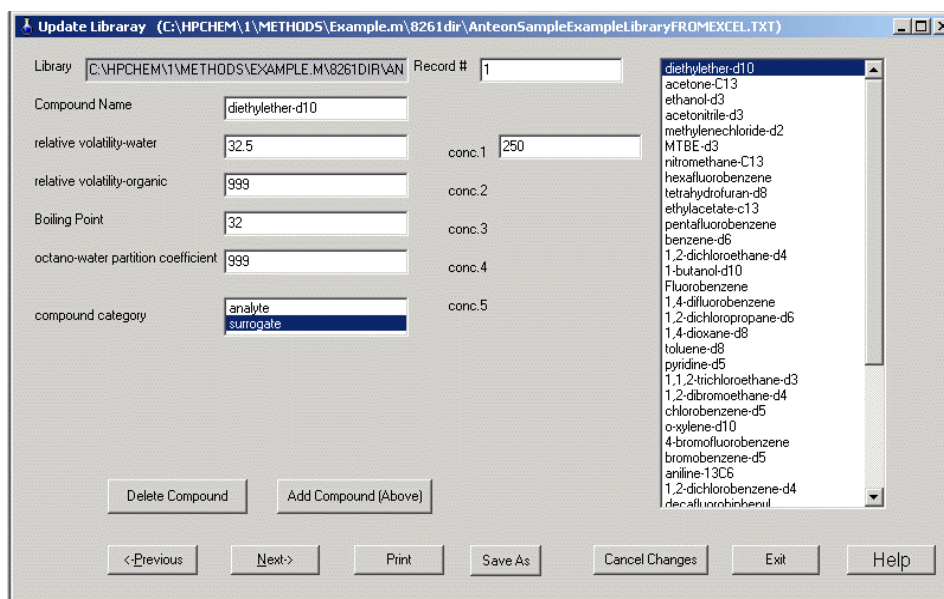


Figure 1 – Update Library Dialog Box

The full path name of the text file opened in 'Open Library...' (see section 3.1.2.1) is shown in the 'Library' edit box. This box is read-only. It may be necessary to scroll right to see the full file path.

The 'Compound Name' box displays the compound currently selected for updating. The compound name may be edited in this box. All compound names should appear exactly as they do the GC/MS software. Use the 'End' key or scroll right to check for superfluous characters in the name. Save changes using the 'SaveAs' button (a reminder to save changes is displayed on exiting this form).

Compounds may be added or deleted from the compound list using the 'Delete Compound' and 'Add Compound (Above)' buttons. When the 'Delete Compound' button is pressed, the current selected compound will be deleted from the compound list. When the 'Add Compound (Above)' button is pressed, a blank compound will be added to the compound list before the current selected compound. A compound name must be entered before moving to another compound. After pressing either button, the 'Cancel Changes' button has no effect. Use these buttons again to replace or delete a compound.

To select a different compound, select from the list box at the right of the form, or enter the compound number (the compound's sequential number in the compound list) in the 'Record #' box. Press <Tab> rather than <Enter> to accept the entry. Compounds may also be sequentially accessed using the Next and Previous buttons, or <Alt-N> and <Alt-P>.

Enter the relative volatility to water and the boiling point in the appropriate boxes. A negative number is not allowed for the relative volatility as calculations use the natural log (ln). The relative volatility organic (relative volatility – water to octanol) and the octanol-water partition coefficient are not currently used.

In the 'Compound Category' selection list, indicate whether the current compound is an analyte or a surrogate. The selected choice is highlighted in blue.

The boxes labeled 'Conc1' through 'Conc5' are for recording various concentrations of analytes and surrogates in spiked calibration samples. All samples (calibration and field) are spiked with surrogates at a constant concentration (ng); thus, only one box ('Conc1') will be displayed for surrogate compounds. For analytes, boxes are provided for up to five different calibration sample concentrations.

Any changes made to a compound are saved in memory when the user selects another compound. Prior to selecting another compound, the 'Cancel Changes' button can be used to restore original compound entries.

Any changes made during the editing session will be lost unless they are saved. The 'Save As' button will write all changes to the library text file.

The 'Print' button prints a listing of the compound library and all compound entries.

The 'Exit' button exits the form. If there are any unsaved changes, the user will be prompted to save or cancel changes on exit.

3.1.2.3 Create Surrogate File...

This option creates a new surrogate file from the library file currently in memory. This option creates a new surrogate file and populates fields with default values. It will often be better to save an existing surrogate file under a new name (see section 3.1.2.5).

A file Open dialog box will be displayed. Enter a new file name for the surrogate file. Click 'Open' to create the new surrogate file. You will be prompted for another file name if the file already exists. Click the 'OK' button to return to the file open dialog and enter a new file name. The 'Cancel' button exits without creating a new surrogate file.

Creating a new surrogate file does not automatically load that surrogate file into the program. Use the 'Open Surrogate File...' (see section 3.1.2.4) option to load the file before any further processing.

3.1.2.4 Open Surrogate File...

This option allows the selection of a surrogate file to be used in Method 8261 calibrations and calculations. Surrogates may be selected and grouped in various ways, thus, there may be numerous surrogate files associated with a given library file.

When this option is selected, a file Open dialog box will be displayed. Select a file and click the 'Open' button. Warnings will be displayed if a nonexistent file is entered, or the file is not a surrogate file. Click 'OK' to return to the file open box and select another file. The 'Cancel'

button exits without opening a surrogate file.

If a surrogate file is not already in memory, the 'Edit Surrogate File...', 'Copy Surrogate File...', and 'Calibration' | 'Create/New...' menu options will be enabled when a surrogate file is opened.

The 'Edit Surrogate File...' menu option will be modified to show the surrogate file currently in program memory.

3.1.2.5 Edit Surrogate File...

This option is used to identify and group specific surrogates to be used in Method 8261 calculations. The 'Surrogate Data' dialog box will be displayed (Figure 2).

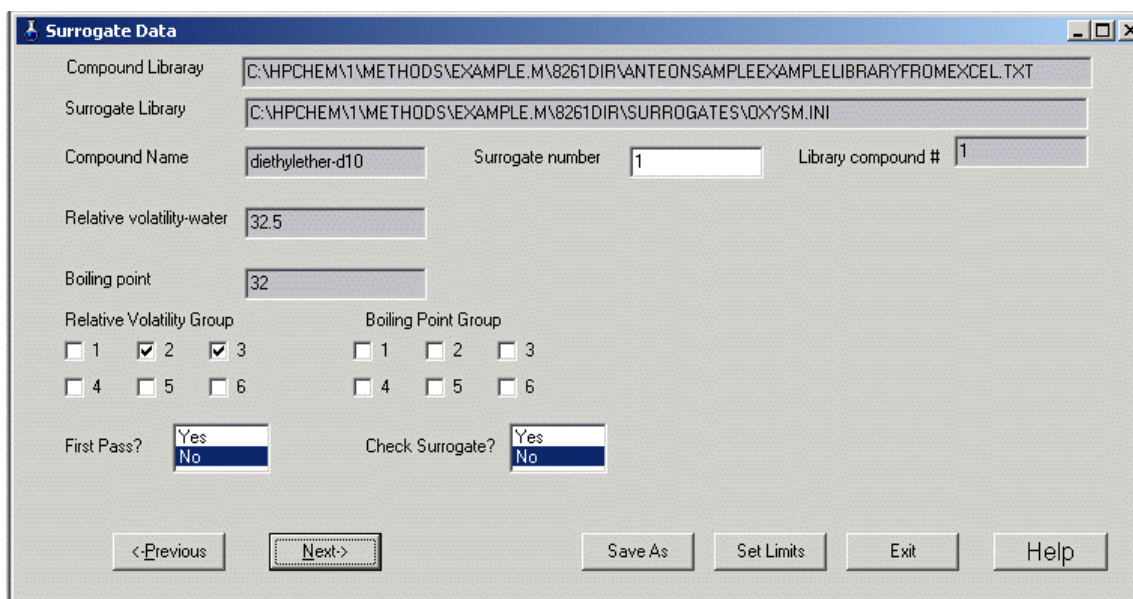


Figure 2 – Surrogate Data Dialog Box

The library and surrogate files must be synchronized, i.e. they must have the identical list of surrogate compounds, listed in the same order. If the files are not synchronized, a warning message will be displayed that shows library and surrogate compound names that do not agree, with the prompt, "Is this a rename?". If the library compound was renamed and the surrogate

compound is equivalent to the old name, click the 'OK' button. The surrogate name will be updated to match the library name. Otherwise, a new surrogate file must be created from the library (see section 3.1.2.3). A report will be printed showing the current surrogate settings (see Appendix 12).

The currently-selected surrogate file is shown in the 'Surrogate Library' edit box. This is a read-only box. The user may scroll through the box to see the entire file path.

The current compound is identified in the 'Compound Name' box. This box is read-only. The surrogate number shown in the 'surrogate number' box is the sequence number of the selected surrogate in the surrogate file. This number may or may not correspond to the sequence number of the surrogate compound in the 8261 library file; i.e., if a compound is the second compound in the library, but the first surrogate, it will have surrogate number 1 and compound library number 2. Surrogate compounds can be selected by entering the surrogate file or the 8261 library file sequence number, or by using the 'Next' button (or <Alt-N>) or 'Previous' button (or <Alt-P>). Press <Tab> to accept the entry.

The relative volatility and boiling point are displayed for reference. They are important in determining surrogate groupings. These are read-only boxes. These values are changed by editing the compound library (see section 3.1.2.2).

All changes made to the current surrogate compound are saved in memory when the user selects a different surrogate.

In the Edit Surrogate File dialog box, place a checkmark in each group the compound is a member of. A compound may belong to one or two groups. Compounds that belong to two groups determine the endpoints for regression lines representing expected recoveries over the group interval. However, the only programmatic requirement for surrogate groups is that they **must** contain at least three compounds. Ordinarily, a compound should not be both a boiling point and a relative volatility surrogate.

First pass surrogates are used to estimate the effects of relative volatility on boiling point surrogates. First pass surrogates should have boiling points below 100°C to minimize any boiling point effects. They **must** be selected to cover the full range of relative volatilities for the boiling point surrogates. Select between three and five first pass surrogates by highlighting 'Yes' (in blue) in the 'First Pass?' box.

Check surrogates are surrogates that will be added to samples as a check on the Method 8261 calculations. These compounds are not ordinarily included in other surrogate groups. Select check surrogates by highlighting 'Yes' (in blue) in the 'Check Surrogate?' box.

The Set Limits button is used to define minimum and maximum limits for calculations. Experience using the Method 8261 normalization algorithm has shown that it is possible to have negative recoveries, especially near the minimum and maximum surrogate limits. For this reason, the user may specify limits to boiling points and relative volatilities used in calculations. The Surrogate Limits dialog box (Figure 3) is displayed when the 'Set Limits' button is pressed (see section 3.1.2.6).

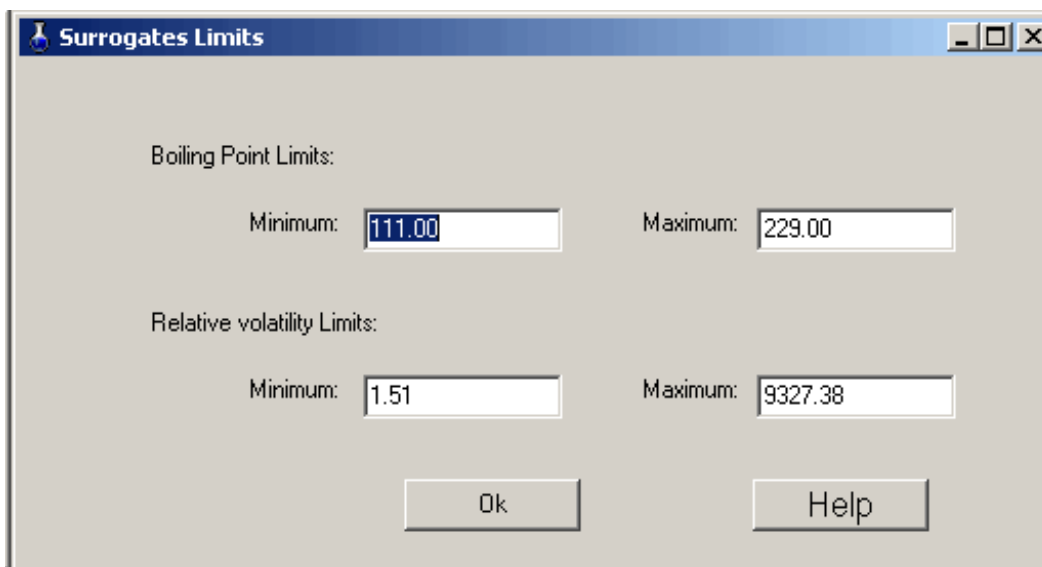


Figure 3 – Surrogate Limits Dialog Box

Any changes made during the editing session will be lost unless they are saved. Use the 'Save As' button to save all changes to the surrogate file.

Except when first creating a surrogate file, it is recommended that any changes to a surrogate file be saved to a different file name. After a surrogate file has been used for sample quantitations, it

should be maintained on disk. If the surrogate file is edited, save it to another file name by selecting the 'Save As' button. A 'Save As' dialog box will be shown. The current surrogate file name may be selected (although it is strongly discouraged). A dialog will warn that the current file will be overwritten. The user may elect to proceed ('Yes' button) or enter another file name ('No' button).

Exit the form by clicking the 'Exit' button. If there are any unsaved changes, a prompt will be displayed to save or cancel changes.

3.1.2.6 Surrogate Limits Dialog

This dialog (Figure 3) allows the user to specify minimum and maximum boiling points and relative volatilities for Method 8261 calculations. This limits extrapolation recovery curves at the extremes of data and reduces the chances for negative recoveries. Default values and ranges that may be entered are as follows:

- Minimum boiling point – The minimum boiling point must fall within the range of boiling point group 1. If group 1 has three compounds, the default minimum boiling point is the group's minimum boiling point and the range is the group's minimum to maximum values. For more compounds, the default is the average of the group's lowest two boiling points and the range is the average of the group's two lowest to the average of the two highest boiling points. When a compound's boiling point is below the user-specified minimum, the predicted recovery is assumed as 1 (100%).
- Maximum boiling point – The maximum boiling point must fall within the range of the highest boiling point group. The default maximum is always the average of the two highest boiling points. The range minimum is either the group minimum for group of three compounds, or the average of the two minimum points for larger groups. The predicted recovery for compounds with boiling points above that specified will be equal to the predicted recovery for the maximum specified boiling point.
- Minimum relative volatility – The minimum relative volatility and range are determined

$$e^{\frac{\ln(RV1)+\ln(RV2)}{2}}$$

using relative volatility group 1 in the same manner as the minimum boiling point above. The average relative volatility is the log average:

where RV1 and RV2 are the relative volatilities being averaged.

Relative volatilities below the user-defined value, will have a predicted recovery equal to the specified minimum.

- Maximum relative volatility – The maximum relative volatility and range are determined using the highest relative volatility group in the same manner as the maximum boiling point above. The average relative volatility is the log average. Relative volatilities above the user-defined value, will have a predicted recovery equal to the specified maximum.

On exiting the dialog box, values will be checked. If values are out of range, they will be reset to the nearest value within the range. The dialog box will remain active until closed.

3.1.2.7 Copy Surrogate File...

This option allows the user to save the current surrogate file with a new name. This minimizes inputs required when slight modifications are desired in a surrogate file.

- A message will be displayed confirming that you have the desired surrogate file selected.
- A standard file save dialog box will allow you to choose the new file name. If the file already exists, you will be prompted to confirm overwriting the existing file.

The new file will not be automatically loaded. Use the 'Open Surrogate File...' menu option to load the new file.

3.1.2.8 Open Calibration File...

The 'Open Calibration File...' menu option allows selection of a calibration file. A calibration file is created using the 'Calibration' | 'Create/New...' menu option (see section 3.1.3.1). Selecting this option brings up a standard file open dialog box, which allows access to the full directory structure to select a properly formatted calibration file.

In the open dialog box, select a file. Click the 'Open' button to open the selected file, or 'Cancel' to close the Open dialog without opening a calibration file.

The following menu changes will occur when a calibration file is opened:

- If a calibration file was not previously loaded, all 'Calibration' drop-down menu items (except 'Create/New...') and 'Quantitation' drop-down menu items will be enabled when

the calibration file is opened.

- If a library file was previously loaded, it will no longer be valid. The 'Update Library...', 'Create Surrogate File...', 'Open Surrogate File...', 'Edit Surrogate...', 'Copy Surrogate File...', 'Create/Edit MDL File...', and 'Calibration' | 'Create/New' will be disabled when the calibration is opened.
- The calibration file name will be appended to the 'Open Calibration File...' menu option.

3.1.2.9 Create/Edit MDL File...

This option allows the user to create and edit minimum detectable level (MDL) files. When this option is selected, the Edit MDL Dialog will be displayed (Figure 4). This dialog is a text editor which allows entry of MDL values for analyte compounds. MDLs are calculated using accepted procedures and can then be entered in the dialog using the keyboard or cut/paste from a text file.

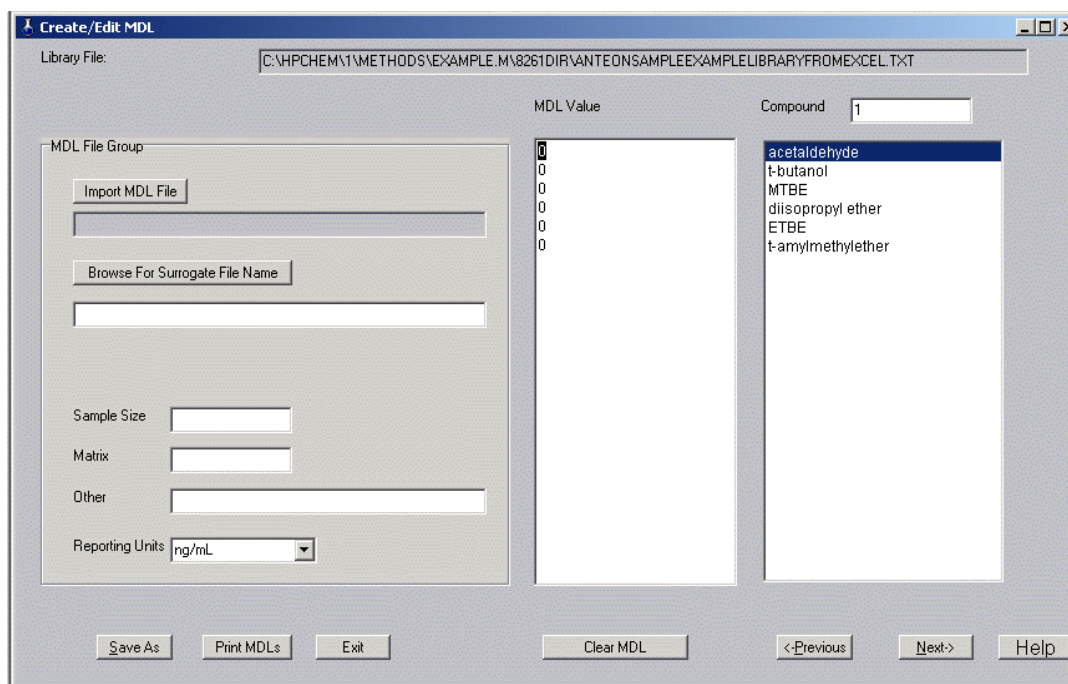


Figure 4 – Create/Edit MDL Dialog Box

To complete this dialog:

- The Library File edit box displays the current 8261 library file. It is read-only.
- The MDL File Group displays identifying information about the MDL file.
 - ▶ An existing MDL file can be loaded by clicking the 'Import MDL File' button. An Open File Dialog will be presented to select the MDL file. If an existing file is imported, all other edit boxes in the MDL File Group (the grouped edit boxes on the left of the dialog) will be filled automatically.

Otherwise:

- ▶ Click the 'Browse For Surrogate File Name' button to select the surrogate file used in calculating MDLs. An open file dialog will be displayed. This is a required field.
- ▶ Enter the sample size used in calculating MDLs. This is a required field.
- Enter the appropriate matrix. This is strictly a comment field, but is required for complete documentation.
 - ▶ Any further comments can be entered in the 'Other' field.
 - ▶ Select the appropriate reporting units from the drop-down list box.
- Navigation among compounds in this dialog is can be accomplished using the 'Next' and 'Previous' buttons, arrow keys, and the compound list scrollbar (when the analyte list is over 23 compounds), or entering a compound number in the 'Compound' edit box (above the compound list box). The cursor in the MDL Value box will maintain alignment with the compound list box.

It is possible to add more MDL values than there are analyte compounds. If the MDL value cursor is moved past the end of the compound list, zero (0) will be shown in the 'Compound' edit box and there will not be a compound highlighted in the compound list as there is no correspondence between the cursor and the compound list. When the cursor returns to a line that corresponds to the compound list, the two boxes will be synchronized.

- MDLs are entered using the text box labeled 'MDL Value'. MDLs can be entered by placing the cursor on the appropriate line and entering a value from the keyboard; or values may be cut (Ctrl-C) and pasted (Ctrl-V) from a text file, or from a Microsoft Excel spreadsheet (values from any other spreadsheet should be exported to a text editor before cutting and pasting). The text file must be formatted as a single entry per line, for example:

```
2.45
3.67
5.00
```

etc.

Values are associated with compounds strictly on a positional basis; i.e. the first value will be associated with the first compound, the second value with the second compound, etc. Enter a zero (0) for any compounds which do not have an MDL.

Values may also be selected and cut (Ctrl-C) and pasted (Ctrl-V) within the MDL Value box.

- The 'Clear MDL' button is used to reset all MDLs to zero.
- Click the 'Save As' button to save all information to a data file. A Save As Dialog will be displayed.
- The 'Print' button will print a listing of compounds and MDLs.
- Click 'Exit' to close this dialog.

3.1.2.10 Exit

The 'Exit' menu option closes the Surrogate Matrix Correction Method window and cleanly closes the application. All information has previously been saved in previous dialogs.

3.1.3 Calibration Menu

The 'Calibration' main menu option invokes a drop-down menu that provides functions to create, review and edit, and print calibration files. A second functional group allows comparison of sample surrogate recovery results with the calibration expected recovery.

3.1.3.1 Create/New...

A calibration must be performed before a quantitation on environmental samples can be done. The calibration consists of measuring known samples, calculating recoveries versus a selected sample (the 'surrogate reference'), and estimating the effects of boiling point and relative volatility on the recovery. The result is a set of response factors (area counts/ng) that can be applied to field samples.

In creating a calibration file, the user selects a set of known samples and a surrogate reference. When the 'Create Calibration File' option is selected, a message box will be shown confirming the surrogate file that will be used for the calibration. Click 'OK' to open the 'Create Calibration File' dialog (Figure 5). If the desired surrogate file is not shown, click the 'Cancel' button and load the desired file (see section 3.1.2.4).

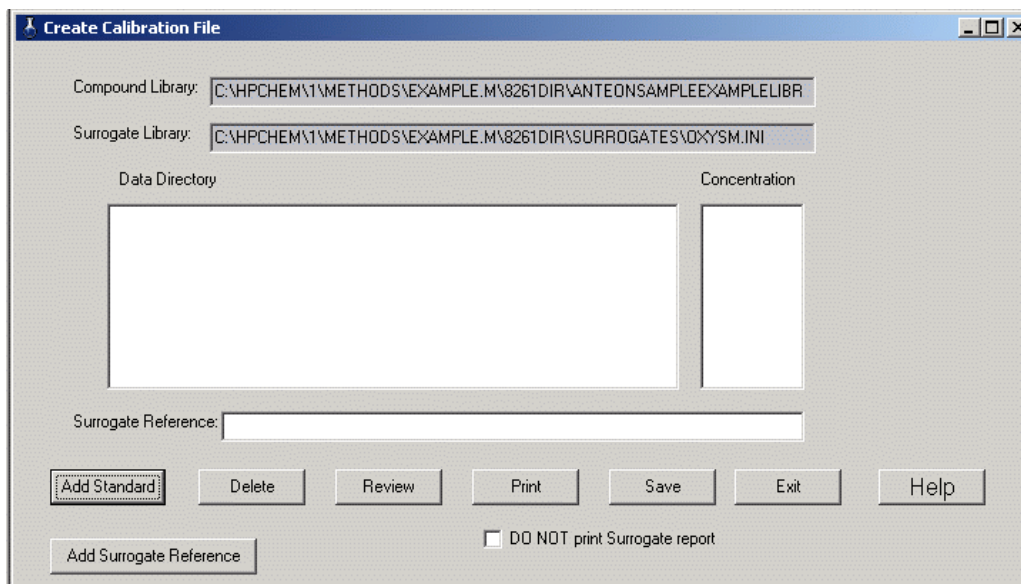


Figure 5 – Create Calibration File Dialog Box

The top two fields - ‘Compound Library:’ and ‘Surrogate Library:’ are read-only. They confirm the libraries that will be used in calibration calculations.

To complete this form and create a calibration file:

- Select between one and five standard samples to process. Click the ‘Add Standard’ button. This will display a file open dialog. Select a quantitation results file (see section 2.2.2.2).
- The ‘Identify Standard Level’ dialog box (Figure 6, see section 3.1.3.2) will be displayed. This dialog is used to select the appropriate concentration (ng) for the sample.
- Control will return to the create calibration file dialog on exiting the ‘Identify Standard Level’ dialog. The data directory and concentration selected will be displayed in the ‘Data Directory’ and ‘Concentration’ list boxes.
- After all standards have been chosen, select a surrogate reference. Click the ‘Add Surrogate Reference’ button or simply click inside the ‘Surrogate Reference:’ edit box. A file open dialog will be displayed to select the surrogate reference.
- A standard can be deleted from the list by highlighting the desired entry and clicking the

- 'Delete' button.
- To perform calculations and review the results of the calibration, click the 'Review' button. The results of the calibration will be shown in a 'Review Calibration' dialog box (see section 3.1.3.3).
- Click the 'Print' button to print a record of the calibration shown. To suppress printing of a surrogate report for each standard used, check the 'DO NOT print Surrogate report' box. See Appendices 6 and 7 for explanations of the calibration report and the surrogate report.
- Press the 'Save' button to save the calibration to a file. There is no automatic saving of the calibration information. The file selection is accomplished using a standard file save dialog box.
- Click 'Exit' to close this form. The user will not be prompted to save the current calibration.

3.1.3.2 Identify Standard Level Dialog

This dialog is used to identify the concentration of compounds in a calibration sample. In the 'Update Library...' menu option, up to five concentrations can be entered for each compound. In

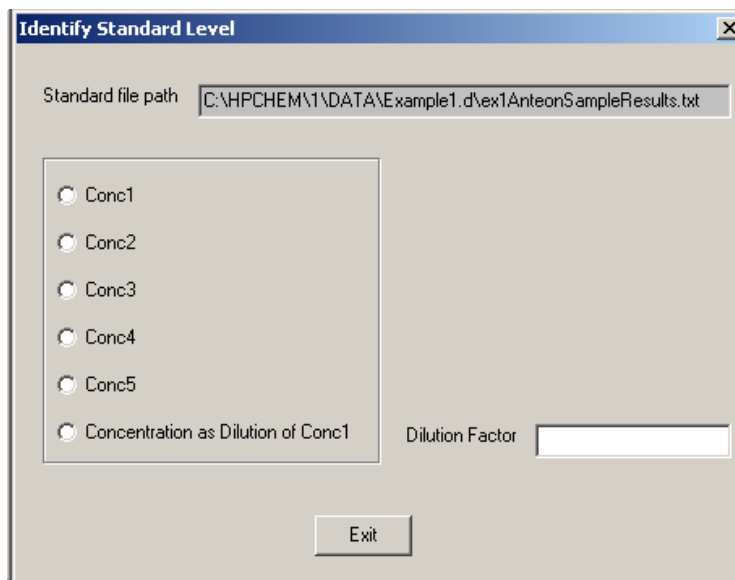


Figure 6 – Identify Standard Level Dialog Box

this dialog, radio buttons correspond to the standard concentrations entered in the 'Update Library' screen (see section 3.1.2.2). A concentration may also be entered as a dilution of standard concentration 1. For example, if a sample is created using a 2:1 dilution of standard concentration 1, the dilution factor is 0.5. Use the radio buttons to select a standard concentration or a dilution of standard concentration 1. Enter the dilution factor if a dilution is used.

NOTE: A dilution is ordinarily indicated by a fraction less than one. However, the dilution factor edit box will accept numbers greater than one. This allows preparation of calibration samples with higher concentrations than concentration 1, but bypasses inputting individual concentrations for each compound – substantially reducing inputs.

3.1.3.3 Review... (Calibration)

The results of a calibration, or a saved calibration file, can be viewed using the 'Review Calibration' dialog (Figure 7). This dialog allows the user to review and select calibration samples to be used in determining the final response factor. It also displays important calibration statistics for each compound in the compound method library.

The 'Calibration File:' box shows the name of the calibration file displayed. The names of each standard directory and the surrogate reference directory are shown on the next row. It may be necessary to scroll right to see the full directory name.

The concentrations (ng) of the compound in the standard solutions are shown in the 'Concentrations' row.

The 'Area' row contains the area counts for the compound from the quantitation result file in the data directory indicated.

The row labeled 'Average' shows the average response factor for the columns selected. The text box under 'Standard Levels' lists of the analyte amounts for the selected columns. The average (mean), deviation (standard deviation), and percent deviation (the standard deviation as a percent of the average) are shown for the selected samples.

	Std1	Std2	Std3	Std4	Std5	Reference file
Standard File Name	01280203.T	01280205.T	01280206.T	01280207.T	01280208.T	01280202.T\X
Concentrations	250	250	250	250	250	
Recoveries (%)	81.043	90.480	82.935	69.269	52.348	
Recovery (Dev.)	0.044	0.025	0.036	0.017	0.112	
Area	408000	416056	396135	323369	418855	
Response Factors	2013.738	1839.336	1910.578	1867.328	3200.521	
Response Factor(Dev.)	108.500	49.974	82.553	45.484	683.637	
Standard Included	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	
Curve response factors	Standard Levels		Average	Deviation	% Deviation	
Average	(250, 250, 250, 250, 250)		2166.300	561.930	26.863	

Figure 7 – Review Calibration Dialog Box

To complete this form and determine response factors for each compound:

- Select the desired compound by entering the record number in the Record Number box, using the 'Previous' (<Alt-P>) and 'Next' (<Alt-N>) buttons to navigate the compound list, or select the compound from the list box in the upper right corner of the form. The current compound will be shown in the Compound edit box.
- Information for each standard solution used in the calibration will be displayed in separate columns. Review the information shown for each standard solution. To exclude a standard solution from the final calculations, uncheck the check box in the standard column. The average response factor, deviation and percent deviation for the compound (shown in the bottom row of edit boxes) will be updated as boxes are checked or unchecked.
- Click the 'Save As' button to save changes. It is suggested that changes be saved to a different calibration file. A standard file save box will be displayed. The current calibration file may be selected – a warning will be displayed about overwriting the file.
- Click the 'Print' button to print the calibration results (Appendix 7). A surrogate report

(Appendix 8) will be printed for each sample, unless the DO NOT print Surrogate Report check box is checked..

- Click the 'Exit' button to close the form. If any changes have been made, a prompt will be displayed to save or discard changes.

3.1.3.4 Print Calibration

Selecting this option prints a report listing response factors for all compounds in the Method 8261 library. An example report is included in the Appendix 7.

3.1.3.5 Check Calibration...

The 'Check Calibration...' menu option compares the calculated response factors of various surrogates in a sample file with those for in the calibration file. To perform a check calibration:

- Select a quantitation results file from a file Open dialog box.
- The 'Check Calibration Standard' dialog box will be displayed (Figure 8). The edit boxes on the left side of this form are all read-only and indicate selected files.

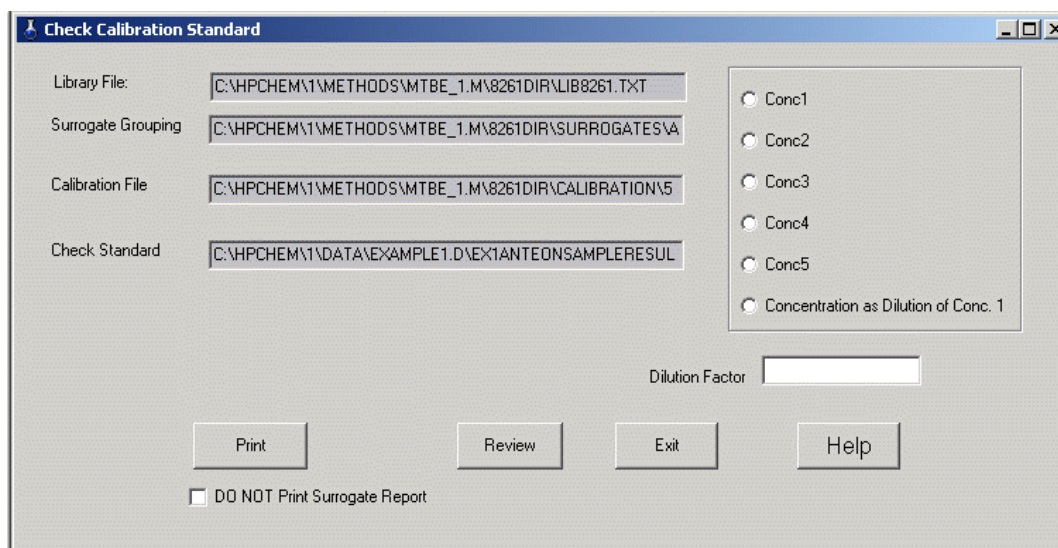


Figure 8 – Check Calibration Standard Dialog Box

- Select a standard concentration for the check standard file. A dilution of standard concentration 1 may also be used – select the ‘Concentration as Dilution of Conc. 1’ radio button and enter the dilution factor.
- Check the ‘DO NOT Print Surrogate Report’ checkbox if a surrogate report for the selected check standard is not desired.
- Click ‘Print’ to receive a printout of the results. A check calibration report is included in Appendix 10.
- Select ‘Review’ to examine the check calibration results (see section 3.1.3.6).
- Click ‘Exit’ to exit the form.

3.1.3.6 Check Calibration Review Dialog

This dialog (Figure 9) shows check calibration results for each compound in the compound library. Compounds may be selected by entering a compound number (Record Number edit box), by selecting a compound from the list box, or using the ‘Previous’ and ‘Next’ buttons.

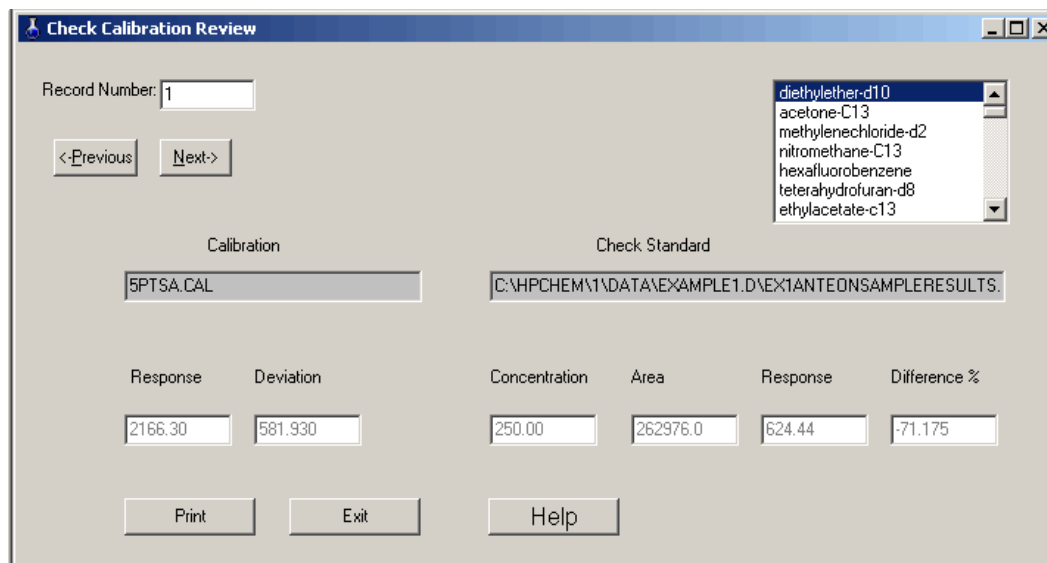


Figure 9 – Check Calibration Review Dialog Box

The response and deviation listed under the calibration file are the average response factor and deviation from the calibration file. Under the check standard edit box, the concentration, area

counts, and response for the check standard file are listed. The difference percent is the difference between the calibration response factor and the response factor calculated for the check standard. The 'Print' button is the same as 'Print' on the 'Check Calibration Standard' form.

3.1.4 Quantitation Menu

The 'Quantitation' main menu option has a single drop-down option that allows quantitation of multiple sample results.

3.1.4.1 Process Sample Files...

This final processing option is used to apply Method 8261 calculations to samples. Up to 20 sample files can be processed at one time, using the selected method, surrogates, and calibration file. The 'Process Files' dialog box (Figure 10) allows selection of multiple files. To complete file processing:

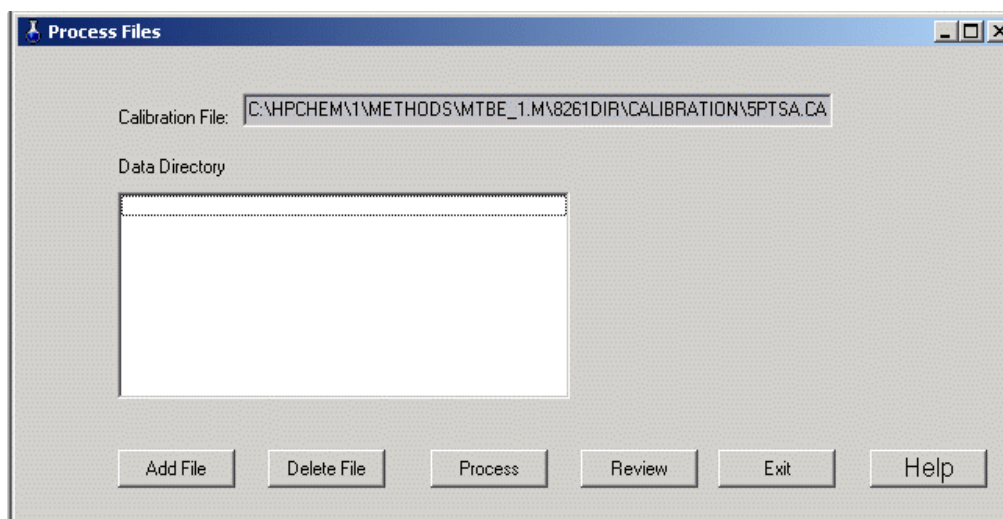


Figure 10 – Process Files Dialog Box

- Click the 'Add File' button. A file Open dialog will be shown to allow selection of a sample quantitation file. Following selection of a quantitation file, the 'File Processing

Input' dialog box (Figure 11) will be displayed. This dialog is used to enter specific information about the sample and how it will be reported (see section 3.1.4.2).

The screenshot shows a dialog box titled "File Processing Input". At the top, it displays "Current Sample File: C:\HPCHEM\1\DATA\EXAMPLE1.D\EXTANTEONSAMPLERESULTS.TXT". Below this is a section for "Method Detection Limits" with a "List MDL" label and two radio buttons: "No" (selected) and "Yes". Underneath are input fields for "Reporting Units" (a dropdown menu set to "ng/mL"), "Sample Size", "Matrix", and "Other". At the bottom of the dialog, there is a checkbox for "DO NOT Print Surrogate report" which is unchecked, and two text input fields for "Comment 1:" and "Comment 2:". At the very bottom are "Enter" and "Help" buttons.

Figure 11 – File Processing Input Dialog Box

- Repeat the steps above to enter up to 100 files to process. To remove a file from the list, highlight the file and press 'Delete File'.
- Press 'Process' to perform Method 8261 calculations for all selected files. Results for each file will be printed. See Appendix 9 for a sample report. For each sample file, a text file of the report will be created. The text file will be located in the directory with the

sample file. The name will be the same as the sample file, with a numeric sequence number appended, and will have a .prn extension. For example, if the sample file is XYZ.txt and this is the second time the file has been processed, the text file name will be XYZ.txt2.prn.

- The 'Review' button displays a quantitation report. Highlight any sample file in the list box and click 'Review' to display a report for that sample (the first sample in the list will be displayed if a selection is not made). This report is displayed in Notepad and can be saved as a file using the Files | Save As menu option. This is a semicolon delimited text file. See Appendix 10 for a sample report.
- Click 'Exit' to close this form.

3.1.4.2 File Processing Input Dialog

This dialog box accepts inputs which describe the sample file and define report options:

- The current sample file name is displayed in a read-only field.
- Use the 'List MDL' radio buttons to select whether to apply MDLs to final calculations. The default is 'No'. If 'Yes' is selected, a 'Reporting Limits (Number of Standard Deviations)' drop-down list and the 'Browse for MDL File' button and edit box will be displayed. Click the 'Browse for MDL File' button to display an Open file dialog. Select a reporting limit from the drop-down list – the numbers 0 - 4 are the number of standard deviations to apply to reporting limits in the quantitation report. A value of '0' will display any concentrations below the MDL as not detectable (ND). Selecting a number from 1 to 3 will only indicate ND if the measured value plus the number of standard deviations is less than the MDL. The reporting units, sample size, matrix and 'Other' fields will be automatically filled when an MDL file is selected.
- Select the reporting units from the drop-down list box.
- Enter the sample size (milliliters or grams). This is a required field.
- Enter the matrix type.
- The 'Other' and comment fields can be used for any documentation or notes associated with the quantitation.
- Check the 'DO NOT Print Surrogate report' check box if a surrogate report is not desired.
- Press 'Enter' to return to the 'Process Files' dialog.

APPENDIX 1
INSTALLED PROGRAM FILES

The program files installed by Smcreporter are listed below, sorted by directories.

Directory	File Name
Install Directory	Smcreporter.exe
Window System32 directory (This is OS specific and is determined by InstallShield)	BorIndmm.dll Cp3245mt.dll Dclusr40.bpl Vcl40.bpl Vclx40.bpl

APPENDIX 2 VERIFYING SMCREPORTER INSTALLATION

Verification of the installation consists of performing a calibration and checking the calibration against a calibration file included in the installation.

- The Smcreporter menu will initially have only one option: 'File | Open Library'.
 - ▶ Click 'Open Library...'. A file open dialog box will be displayed.
 - ▶ Select the file 'ExampleLibrary.txt'. The path will depend on what directory was entered for the installation directory during installation. For example, if the installation directory was entered as "c:\smcreporter", the full path of ExampleLibrary.txt will be "c:\smcreporter\ExampleLibrary.txt". All default paths specified below assume the initial directory given in this example.
- Click 'Open Surrogate File...' and select 'ExampleSurrogate.ini' as the surrogate file. The default path will be "c:\smcreporter\ExampleSurrogate.ini".
- Click 'Calibration | Create/New...'.
 - ▶ The Create Calibration File dialog box will be displayed (Figure 5).
 - ▶ Press "Add Standard" and select 'Example1.txt' as the standard directory. The default path is "c:\smcreporter\example1.txt".
 - ▶ The Identify Standard Level dialog (Figure 6) will be displayed. Select 'Conc1' as the standard concentration for Example1. Press "Exit".
 - ▶ Press "Add Surrogate Reference" and select 'Example2.txt' as the surrogate reference file. The default path is "c:\smcreporter\example3.txt".
 - ▶ Press "Review". A Review Calibration dialog box (Figure 7) will be displayed.
 - ▶ Press "Save As". A file open dialog box will be displayed. Enter a new unique name for the calibration.
 - ▶ Check the boxes 'DO NOT print Surrogate report' and 'DO NOT Include Surrogate information on Calibration report'.
 - ▶ Press "Print".
 - ▶ Press "Exit".
- Click 'Load Calibration File' and select 'Example1and2.cal' as the calibration file. The default path is "c:\smcreporter\Example1and2.cal".
- Select 'Edit Calibration File'.
 - ▶ On the Review Calibration dialog box (Figure 7), check 'DO NOT print Surrogate Report' and 'DO NOT Include Surrogate information on Calibration report'.
 - ▶ Click the "Print" button. A calibration report for Example1and2.cal will be

- ▶ printed.
Press "Exit".

The calibration reports for Example1and2.cal and the new calibration should match. If not, contact Steven McLemore – Anteon Corporation – at (702) 784-8324 or mclemore.steven@epa.gov.

APPENDIX 3 REGRESSION SELECTION ALGORITHM

For each surrogate group, a regression line is calculated. The independent variable (x) is the value of the appropriate physical property (boiling point or relative volatility). The following procedure is used to determine the appropriate regression to calculate the expected recovery for a compound:

- For each line, determine the absolute minimum and maximum group surrogate values.
 - For each line, determine the limits of the regression line.
 - For a group of three surrogates, the limits are the two extreme values (limits equal absolute minimum and maximum)..
 - For groups of four or more surrogates, the lower limit is the average of the two lowest values and the upper limit is the average of the two highest values (limits are different than absolute minimum and maximum).
- 1) Start with group 1.
 - 2) If the compound value is less than the lower limit of group 1, assign the group 1 regression.
 - 3) If group is 1, go to step 6.
 - 4) If the value is less than the group absolute minimum, assign the previous group regression.
 - 5) If the value is less than the group lower limit, assign the current group regression.
 - 6) If the value falls within the group limits, assign the group regression.
 - 7) Go to the next group.
 - 8) If the group number is less than or equal to the highest group, go to step 4.
 - 9) If the value is greater than the group's upper limit, use the last group regression.

APPENDIX 4 WINDOWS INITIALIZATION FILE (INI) FORMAT

This section explains the Windows initialization file (.ini) format. An INI file is simply a text file. However, it has a structure which allows Windows to find data in it. It can be thought of as a three-field data table, where the first two fields are keys. The first key is called the 'Application' or 'Section' (in the rest of this document, 'section' will be used). The second key is simply called the 'Key'. The third field is the 'Value'.

Here is an example INI file (taken from the 8261lib.ini file).

```
[compound1]
name=diethylether-d10
bp=35
rvW=32.5
cmpndType=surrogate
```

The word in brackets is the section field. Each word on the left of an equal sign is a key, and the words to the right of the equal signs are the values.

Keys must be unique within a given section, but they may be duplicated in two or more sections. If the INI file is edited manually (using Notepad or another text editor), non-unique keys could be created. Windows will simply use the first one it finds and ignore the rest.

INI files were used because they are part of the Windows Application Programming Interface (API). Windows provides functions to access them quickly and easily. The Windows application programming interface (API) functions that allow reading and writing of INI files are GetPrivateProfileString() and WritePrivateProfileString(). They are implemented in ChemStation macro language as GetIniString\$() and SetIniString(), and in C++Builder as TIniFile::ReadString() and TIniFile::WriteString(). Use of these functions is documented on the Web, or through ChemStation or C++Builder online documentation.

INI files do have some important limitations:

- When reading an INI file, a default value is specified to return in case the keys provided are not found. This means that the INI file operations work just fine, even if the INI file

does not exist. The API functions which retrieve data from INI files do not even tell the calling function whether the value it is returning came from the INI file, or is simply a copy of the default value.

- INI files cannot be over 64 KB in length.

APPENDIX 5
METHOD 8261 CALCULATIONS

1. Quantitative Analysis – Calibration

An instrument calibration must be performed before any quantitation of environmental samples. Spiked samples are used for the calibration. Following measurement of the samples³, the calibration involves the following steps to calculate response factors (area counts/ng) for each compound in the sample: estimate relative volatility effects on boiling point surrogates using first pass surrogates(f); calculate boiling point () and relative volatility () effects on surrogate recoveries; combine these effects to estimate total recoveries of compounds; and, finally, calculate a response factor for each compound based on area counts, mass of the compound present, and the estimated recovery. This section presents a step-wise approach to the calculations.

In the following discussion, various variables will be introduced. These variables may be associated with first pass, relative volatility, or boiling point surrogates. Subscripts will be used to clarify the distinction. For example, equation 1 is used to calculate the relative recovery (R). R_f is used for a relative recovery associated with first pass surrogates, R_v for relative volatility surrogates, and R_b for boiling point surrogates.

- Select results of 1 to 5 spiked samples, of various concentrations, as calibration files. Select a spiked sample file as a 'surrogate reference'. The surrogate reference represents 100% recovery of target compounds.
- First pass surrogates:
 - ▶ For each first pass surrogate, calculate the relative recovery (R_f) as:

Equation 1

$$R = \frac{a_s / c_s}{a_r / c_r}$$

³ Hiatt, M.H. Method 8261: Volatile Organic Compounds by Vacuum Distillation in Combination with Gas Chromatography/Mass Spectrometry (VD/GC/MS), Draft Update IVB of SW-846 On-line, URL: <http://www.epa.gov/epaoswer/hazwaste/test/txup4b.htm>.

where

R = relative recovery

a_s = sample area counts

c_s = sample amount (ng)

a_r = reference area counts

c_r = reference amount (ng).

- ▶ Arrange first pass surrogates in order of relative volatility.
- ▶ For each adjacent pair of first pass surrogates, calculate a linear regression slope (m_f), intercept (b_f), and associated standard error ($lstderr_f$):

Equation 2

$$m = \frac{n \sum xy - (\sum x \sum y)}{n \sum x^2 - (\sum x)^2}$$

Equation 3

$$b = \frac{(\sum y \sum x^2) - (\sum x \sum xy)}{n \sum x^2 - (\sum x)^2}$$

Equation 4

$$lstderr = \sqrt{\left[\frac{1}{n(n-2)} \right] \left[n \sum y^2 - (\sum y)^2 - \frac{[n \sum xy - \sum x \sum y]^2}{n \sum x^2 - (\sum x)^2} \right]}$$

= 0 for first pass surrogates (two first pass surrogates determine a line)

where

n = the number of points determining the line

x = ln rw (natural log of relative volatility) for points determining the line

y = relative recovery (R_f) for first pass surrogates determining the line.

$lstderr$ is a measure of the error in the relative recovery, predicted from the regression, for each natural log of relative volatility (ln rw).

- Boiling point surrogates:
 - ▶ For each boiling point surrogate, calculate the relative recovery (R - Equation 1).
 - ▶ For each boiling point surrogate, determine the first pass predicted recovery (R_{p_f}) from the first pass regressions above:

Equation 5

$$R_{p_f} = \ln rw \times m_f + b_f$$

where

rw = surrogate relative volatility

Use the slope and intercept for the calculated line segment that includes the given $\ln rw$. Do not extrapolate lines. Set the relative volatility equal to the nearest endpoint if it is outside the range of first pass relative volatilities.

- ▶ For each boiling point surrogate, calculate the first pass adjusted relative recovery (R_a) as:

Equation 6

$$R_{a_\beta} = \frac{R_\beta}{R_{p_f}}$$

- ▶ For each boiling point surrogate group, calculate a linear regression (m , b , and $lstderr$) as in Equations 2,3, and 4 above:
 - where
 - x = boiling point for each surrogate in the group
 - y = R_a for each surrogate in the group.
 - These linear regressions will yield a predicted recovery for any compound based on that compound's boiling point.
- Relative volatility surrogates:
 - ▶ For each relative volatility surrogate, calculate the relative recovery (R - Equation 1).
 - ▶ For each relative volatility surrogate, determine the boiling point predicted recovery (R_p) from the boiling point regressions above:

Equation 7

$$Rp_{\beta} = bp \times m_{\beta} + b_{\beta}$$

where

bp = surrogate boiling point. **Note:** If bp exceeds the maximum boiling point for the surrogate file (see section 3.1.2.5), set bp equal to the maximum value.

The slope and intercept are for the calculated line segment that includes the given bp. $Rp = 1$ for boiling points less than the surrogate file minimum. These boiling points will ordinarily be less than 100°C and it is assumed that boiling point effects will be negligible.

- ▶ For each relative volatility surrogate, calculate the boiling point adjusted relative recovery (Ra) as:

Equation 8

$$Ra_{\alpha} = \frac{R_{\alpha}}{Rp_{\beta}}$$

- ▶ For each relative volatility surrogate group, calculate a linear regression (m, b, and lstderr) as in Equations 2, 3, and 4 above:
 - where
 - x = ln rw for each surrogate in the group
 - y = Ra for each surrogate in the group.
- For each surrogate and analyte in the sample:
 - ▶ Calculate the boiling point predicted recovery (Rp - Equation 7) and the relative volatility predicted recovery (Rp - Equation 9) :

Equation 9

$$Rp_{\alpha} = \ln rw \times m_{\alpha} + b_{\alpha}$$

where:

rw = relative volatility of the compound

The slope and intercept are for the calculated line segment that includes the given ln rw. If rw is less than the surrogate file minimum relative volatility, set rw equal to the minimum value. If rw is greater than the surrogate file maximum relative volatility, set rw equal to the maximum value.

- ▶ Calculate the total predicted recovery (R_T) as:

Equation 10

$$R_T = Rp_\beta \times Rp_\alpha$$

- The total predicted recovery deviation (Rdev) is calculated as:

Equation 11

$$Rdev = \sqrt{\left(\frac{lstderr_\beta}{Rp_\beta}\right)^2 + \left(\frac{lstderr_\alpha}{Rp_\alpha}\right)^2} \times R_T$$

where

lstderr and lstderr = regression standard errors for the boiling point and relative volatility predicted recoveries

- Calculate a response factor (RF) as:

Equation 12

$$RF = \frac{a}{R_T \times c}$$

where

a = area counts

c = amount of compound (ng) in the spiked sample.

The response factor is the expected area counts/ng for this compound.

- The response factor deviation (RFdev) is:

Equation 13

$$RFdev = \left(\frac{Rdev}{R_T} \right) RF$$

2. Quantitative Analysis - Sample Corrections

Following the instrument calibration, field samples can be corrected for boiling point and relative volatility effects (hopefully, independent of matrix effects). The end result is a corrected concentration (ng/volume) for each analyte in the sample. This section explains all calculations.

- For each surrogate, calculate the relative recovery using the average response factor from the calibration samples rather than a surrogate reference:

Equation 14

$$R = \frac{a_s}{c_s \times \overline{RF}} \quad \text{where}$$

\overline{RF} = average calibration response factor for the compound.

- Generate linear regressions to predict recoveries adjusted for boiling point and relative volatility, as shown above for calibrations (use Equation 14 everywhere Equation 1 is referenced in the calibration).
- For each analyte in the sample, calculate a total predicted recovery (R_T) from the regressions in the step above.
- For each analyte, calculate the recovered concentration (C) as:

Equation 15

$$C = \frac{a_s}{RF \times R_T \times s}$$

where

s = sample size.

- The concentration deviation is calculated as:

Equation 16

$$Cdev = C \left(\frac{Rdev}{R_T} \right)$$

3. Quantitative Analysis - Check Calibration

A check calibration is performed to compare response factors from a sample run with those calculated by a calibration. This aids in determining the quality of the data. A check calibration consists of the following:

- Calculate response factors (RF) for each compound in a sample using the same procedures shown for a calibration above. Use Equation 14 in place of Equation 1 to calculate relative recoveries..
- The percent difference (%Diff) between the calibration and sample response factors is given by:

Equation 17

$$\% Diff = \left(\frac{RF_{check} - \overline{RF}}{\overline{RF}} \right) \times 100$$

where

RF_{check} = response factor for the sample file.

APPENDIX 6
SAMPLE 8261 COMPOUND LIBRARY LISTING

The following page is a sample listing of the 8261 compound library. This listing is printed from the compound edit dialog box (Figure 6) "Print" button. The columns and rows of this listing contain the following items:

- The compound number (N) in the 8261 library is equivalent to the position of the compound in the ChemStation quantitation library.
- The compound name (Name).
- The compound type (Type) – surrogate or analyte.
- The rest of the row lists the relative volatility – water (Rvw), boiling point (Bp), relative volatility – organic (Rvo), and partition coefficient (PartCoef).. 999's in this row are default values meaning there has been no user entry made.
- The next row (C1 - C5) lists the calibration concentrations prepared for this compound. 99s in this row are default values. Surrogates are present at the same concentration in all samples, thus all entries are the same for surrogates.

User's Guide for Surrogate Matrix Normalization

Compound Library Process Date: C:\HPCHEM1\METHODS\TESTMETH.M\8261dir\lib8261.ini 5/28/2002 11:47:17 AM

N	Name	Type	Rvw C1	Bp C2	Rvo C3	PartCoef. C4	C5
1	diethylether-d10	Surrogate	32.50	35.00	999.00	0.00	
			250.00	250.00	250.00	250.00	250.00
2	acetone-C13	Surrogate	600.00	56.00	999.00	0.00	
			3100.00	3100.00	3100.00	3100.00	3100.00
3	methylenechloride-d2	Surrogate	11.10	40.00	999.00	0.00	
			250.00	250.00	250.00	250.00	250.00
4	nitromethane-C13	Surrogate	510.00	101.00	999.00	0.00	
			650.00	650.00	650.00	650.00	650.00
5	hexafluorobenzene	Surrogate	0.86	81.50	999.00	0.00	
			250.00	250.00	250.00	250.00	250.00
6	teterahydrofuran-d8	Surrogate	355.00	66.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
7	ethylacetate-c13	Surrogate	150.00	77.00	999.00	999.00	
			2500.00	2500.00	2500.00	2500.00	2500.00
8	pentafluorobenzene	Surrogate	1.51	85.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
9	benzene-d6	Surrogate	3.92	79.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
10	1,2-dichloroethane-d4	Surrogate	20.00	84.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
11	Fluorobenzene	Surrogate	3.50	85.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
12	1,4-difluorobenzene	Surrogate	3.83	88.50	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
13	1,2-dichloropropane-d6	Surrogate	11.00	95.00	999.00	999.00	
			200.00	200.00	200.00	200.00	200.00
14	1,4-dioxane-d8	Surrogate	5800.00	101.00	999.00	999.00	
			2400.00	2400.00	2400.00	2400.00	2400.00
15	toluene-d8	Surrogate	4.28	111.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
16	pyridine-d5	Surrogate	15000.00	115.00	999.00	999.00	
			12500.00	12500.00	12500.00	12500.00	12500.00
17	1,1,2-trichloroethane-d3	Surrogate	26.60	112.00	999.00	999.00	
			200.00	200.00	200.00	200.00	200.00
18	1,2-dibromoethane-d4	Surrogate	26.00	131.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
19	chlorobenzene-d5	Surrogate	6.27	131.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
20	o-xylene-d10	Surrogate	6.14	143.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
21	4-bromofluorobenzene	Surrogate	8.05	152.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
22	bromobenzene-d5	Surrogate	7.93	155.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
23	aniline-13C6	Surrogate	13700.00	184.00	999.00	999.00	
			12800.00	12800.00	12800.00	12800.00	12800.00
24	1,2-dichlorobenzene-d4	Surrogate	8.03	181.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
25	decafluorobiphenyl	Surrogate	3.03	206.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
26	nitrobenzene-d5	Surrogate	87.50	210.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
27	acetophenone-d5	Surrogate	161.00	202.00	999.00	999.00	
			1040.00	1040.00	1040.00	1040.00	1040.00
28	1,2,4-trichlorobenzene-d3	Surrogate	7.88	213.00	999.00	999.00	
			250.00	250.00	250.00	250.00	250.00
29	naphthalene-d8	Surrogate	18.00	217.00	999.00	999.00	
			500.00	500.00	500.00	500.00	500.00
30	1-methylnaphthalene-d10	Surrogate	67.00	245.00	999.00	0.00	
			1050.00	1050.00	1050.00	1050.00	1050.00
31	dichlorodifluoromethane	Analyte	0.07	-30.00	999.00	0.00	
			100.00	80.00	60.00	40.00	20.00
32	chloromethane	Analyte	1.37	-24.00	999.00	999.00	
			100.00	80.00	60.00	40.00	20.00
33	bromomethane	Analyte	1.82	4.00	999.00	999.00	
			100.00	80.00	60.00	40.00	20.00
34	chloroethane	Analyte	1.01	12.00	999.00	999.00	
			100.00	80.00	60.00	40.00	20.00
35	trichlorofluoromethane	Analyte	0.20	24.00	999.00	999.00	
			100.00	80.00	60.00	40.00	20.00

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APPENDIX 7 CALIBRATION REPORT

The calibration report is printed from the create calibration or review calibration forms (Figures 10 and 12), or from the 'Print Calibration File' option of the Method 8261 menu. A sample calibration report is included in this appendix. It shows the response factors (area counts/ng) for each compound in the calibration standard samples.

The report header lists:

- The calibration file, if the calibration has been saved.
- The date and time the report was printed.
- The surrogate file.
- The 8261 library file.
- The file names for the standard samples included in the calibration.
- The surrogate reference file.

For 1 to N compounds, the report shows:

- Compound name and the concentrations (ng) of the compound in each standard sample.
- For each calibration standard sample, the response factor (resp) and the recovery percent deviation (%dev).
- The average response factor (Ave), standard deviation of the response factors (dev), and percent standard deviation (rel dev).

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Page: 1

Calibration Curve Report

Calibration File: A:\MTBE-SPTEEDITED.CAL
 Process Date: 5/30/2002 10:06:42 AM
 Surrogate Groupings File: C:\HPCHEM1\METHODS\MTBE_1.m\8261\dir\Surrogates\sur0102.inl
 Compound Library File: C:\HPCHEM1\METHODS\MTBE_1.m\8261\dir\8261.inl
 Files: C:\HPCHEM1\DATA\AJANUARY\01280203.D. C:\HPCHEM1\DATA\AJANUARY\01280205.D.
 C:\HPCHEM1\DATA\AJANUARY\01280206.D. C:\HPCHEM1\DATA\AJANUARY\01280207.D.
 C:\HPCHEM1\DATA\AJANUARY\01280208.D.

Reference File: C:\HPCHEM1\DATA\AJANUARY\01280202.D

	Standard1 resp(%dev)	Standard2 resp(%dev)	Standard3 resp(%dev)	Standard4 resp(%dev)	Standard5 resp(%dev)	Results Ave	rel dev
1	2013.74(4.4%)	1839.34(2.5%)	1910.58(3.6%)	1867.33(1.7%)	3200.52(11.2%)	2166.30	26.9%
2	1115.43(6.4%)	1060.88(12.5%)	1209.20(17.7%)	1067.35(1.9%)	1012.44(9.8%)	1093.06	74.47
3	1119.29(2.6%)	1206.63(1.4%)	1240.04(3.2%)	1178.22(2.6%)	1884.52(2.9%)	1325.74	315.49
4	3619.30(6.3%)	3491.60(12.5%)	3097.90(17.2%)	2687.06(2.0%)	1806.43(38.5%)	2940.46	731.37
5	6289.85(0.0%)	6288.96(0.2%)	6283.32(0.8%)	6289.80(0.0%)	6372.23(4.6%)	6304.83	37.77
6	1402.18(4.4%)	1370.78(2.5%)	1286.12(3.6%)	1319.52(1.7%)	1490.32(11.2%)	1373.76	79.10
7	905.71(4.4%)	881.98(2.5%)	817.83(3.6%)	776.08(1.7%)	939.77(11.2%)	864.28	66.46
8	9078.88(0.1%)	9224.13(0.8%)	8934.80(0.8%)	9062.77(0.2%)	9456.63(9.1%)	9151.84	199.82
9	15320.70(2.6%)	15325.47(1.4%)	15062.32(3.4%)	15476.62(2.3%)	23243.88(2.8%)	16885.79	3557.39
10	6868.13(2.6%)	6794.14(1.4%)	6576.10(3.2%)	6814.75(2.6%)	7275.04(2.9%)	6825.63	279.06
11	16033.03(0.1%)	16065.74(0.8%)	16209.97(0.8%)	16034.85(0.2%)	13655.68(10.0%)	15599.85	1089.27
12	15135.76(0.1%)	15106.98(0.8%)	14982.32(0.8%)	15134.17(0.2%)	17202.88(10.2%)	15512.42	947.09
13	3980.37(2.5%)	3950.96(1.6%)	3892.06(3.1%)	3871.56(2.6%)	3464.12(13.6%)	3811.82	200.52
14	72.87(6.3%)	54.40(12.5%)	76.44(17.2%)	61.65(2.0%)	66.43(39.9%)	66.36	8.79
15	13839.82(2.6%)	13863.53(1.6%)	13641.46(3.4%)	13910.66(2.3%)	19389.69(6.6%)	14929.05	2495.69
16	98.00(6.5%)	328.99(12.7%)	142.52(17.9%)	312.45(2.1%)	281.97(40.6%)	232.79	105.28
17	5504.68(4.4%)	5470.47(2.6%)	5488.66(3.6%)	5602.02(1.7%)	5622.76(24.6%)	5537.72	69.62
18	783.52(2.7%)	790.19(1.7%)	792.76(3.4%)	773.46(2.8%)	749.81(16.1%)	777.95	17.41
19	7322.73(2.7%)	7166.30(1.6%)	7456.34(3.4%)	7339.50(2.8%)	7367.07(12.4%)	7330.39	105.21
20	16650.37(2.8%)	17065.66(1.7%)	16296.05(3.7%)	16606.83(2.5%)	16560.03(12.4%)	16636.19	276.43
21	5868.20(2.8%)	5865.63(1.6%)	5732.90(3.6%)	5799.23(2.9%)	5671.93(13.2%)	5787.58	85.28
22	6700.99(2.8%)	6565.06(1.7%)	6673.87(3.6%)	6600.52(2.9%)	6374.48(13.2%)	6592.99	128.73
23	22.02(6.9%)	33.33(13.3%)	9.16(20.3%)	26.88(7.1%)	22.41(20.3%)	22.75	8.86

APPENDIX 8 SURROGATE REPORT

The surrogate report depicts the calibration 'curves' (set of linear regressions) for a sample. These curves characterize the boiling point and relative volatility effects for sample surrogates. The two pages of a surrogate report are explained in this appendix, along with sample calculations based on equations in Appendix 5.

Page 1 of the surrogate report includes the following:

- The report header lists:
 - ▶ The report date and time.
 - ▶ The 8261 library file.
 - ▶ The surrogate data file.
 - ▶ The sample data path – the actual data file is the path name + '\results.ini'. The Method 8261 program creates results.ini. This file contains area counts.
 - ▶ The surrogate reference data path – the actual data file is the path name + '\results.ini'.

- First pass surrogates
 - ▶ The first pass surrogate groupings are shown. First pass surrogate groupings always consist of two surrogates. In this example, there are four first pass surrogates comprising three first pass groups. The surrogates are listed, followed by the boiling point (BP) and relative volatility-water (rw).
 - ▶ The measured recovery (Meas.) is calculated as shown in Appendix 5, Equation 1 or 14 (depending on whether the surrogate reports is for a calibration or a quantitation - this example report is from a quantitation, so Equation 14 is used), with the final result expressed as a percent. For hexafluorobenzene:
 - a_s (from c:\hpchem\1\data\January\01280205.D\results.ini) = 1045950
 - c_s (from lib8261.ini) = 250
 - $\overline{RF} = 6304.83$ (the average response factor for hexafluorobenzene - shown in the Calibration Report - Appendix 6).

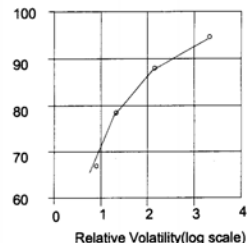
$$66.36 = \frac{1045950 / 250}{6304.83} \times 100$$

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Process Date: 5/30/2002 10:22:29 AM Surrogate Report - Page: 1
 Compound Library File: C:\HP\CHEM\1\METHODS\MTBE_1.M\8261dir\lib8261.ini
 Surrogate Groupings File: C:\HP\CHEM\1\METHODS\MTBE_1.M\8261DIR\SURROGATES\SURR0102.INI
 Data File: C:\HP\CHEM\1\DATA\TestMeth\01280205.D
 Calibration File: A:\MTBE-5PTEDITED.CAL

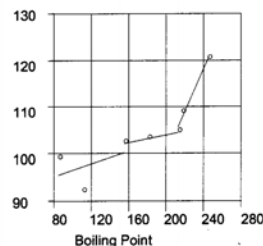
First Pass relative Volatility (Log) vs. Recovery
 (Used to Estimate Rel vol effects on BP Surrogates)

Compound	BP	rw	Meas., Recovery(%)		Slope	Int.	Err%
hexafluorobenzene	81.5	0.86	66.36	66.36	8.279e-02	0.6761	0.00
Fluorobenzene	85	3.5	77.98	77.98			
Fluorobenzene	85	3.5	77.98	77.98	5.458e-02	0.7114	0.00
1,2-dichloroethane-d4	84	20	87.49	87.49			
1,2-dichloroethane-d4	84	20	87.49	87.49	2.317e-02	0.8055	0.00
tetrahydrofuran-d8	66	355	94.16	94.16			



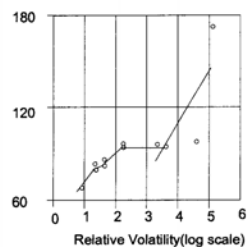
Recovery vs. Boiling Point - (First Pass Rel Vol Corrections) -- Boiling point range(85.00 - 231.00)

Compound	BP	rw	Meas., Recovery(%)		Slope	Int.	Err%
perfluorobenzene	85	1.51	70.16	98.79	6.627e-04	0.8983	6.55
toluene-d8	111	4.28	72.67	91.90			
bromobenzene-d5	155	7.93	84.15	102.07			
bromobenzene-d5	155	7.93	84.15	102.07	4.046e-04	0.9575	0.12
1,2-dichlorobenzene-d4	181	8.03	84.96	102.97			
1,2,4-trichlorobenzene-d3	213	7.88	86.04	104.41			
1,2,4-trichlorobenzene-d3	213	7.88	86.04	104.41	4.659e-03	6.247e-02	1.63
naphthalene-d8	217	18	94.36	108.56			
1-methylnaphthalene-d10	245	67	108.56	120.23			



Recovery(bp corrected) vs. Relative Volatility (Log) -- Relative Volatility range(0.86 - 9327.38)

Compound	BP	rw	Meas., Recovery(%)		Slope	Int.	Err%
hexafluorobenzene	81.5	0.86	66.36	66.36	9.140e-02	0.6788	3.23
Fluorobenzene	85	3.5	77.98	81.68			
1,4-difluorobenzene	88.5	3.83	74.59	77.94			
Fluorobenzene	85	3.5	77.98	81.68	4.124e-02	0.7468	2.91
1,4-difluorobenzene	88.5	3.83	74.59	77.94			
o-xylene-d10	143	6.14	83.83	84.42			
chlorobenzene-d5	131	6.27	79.23	80.42			
o-xylene-d10	143	6.14	83.83	84.42	7.577e-02	0.6861	2.46
chlorobenzene-d5	131	6.27	79.23	80.42			
1,2-dibromoethane-d4	131	26	93.24	94.65			
1,1,2-trichloroethane-d3	112	26.6	89.65	92.18			
1,2-dibromoethane-d4	131	26	93.24	94.65	-3.195e-04	0.9359	1.42
1,1,2-trichloroethane-d3	112	26.6	89.65	92.18			
tetrahydrofuran-d8	66	355	94.16	94.16			
acetone-C13	56	600	92.76	92.76			
tetrahydrofuran-d8	66	355	94.16	94.16	0.1618	-1.011e-01	30.89
acetone-C13	56	600	92.76	92.76			
1,4-dioxane-d8	101	5800	92.62	95.96			
pyridine-d5	115	15000	166.74	171.10			



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- The slope, intercept (Int.), and regression error expressed as a percent (Err%) are calculated for each first pass group using $X = \text{measured recovery (not as a percent)}$ and $Y = \ln(\text{rw})$. Since the linear regression has only two points, the error is 0.

NOTE: There is a distortion in the graphs introduced by the fact that results in this report are expressed in percent, but actual calculations are not.

- The predicted recovery (Recovery(%)) is predicted from the curve (Appendix 5, Equation 5). Since hexafluorobenzene is an endpoint of the curve, the predicted recovery is the same as the measured recovery.
- Boiling point surrogates
 - ▶ For pentafluorobenzene, the measured recovery is:
$$70.16 = \frac{1605270 / 250}{9151.84} \times 100$$
 - ▶ The predicted recovery is the measured recovery divided by the predicted recovery for pentafluorobenzene using the first pass curves (Appendix 5, Equations 5 and 6). Use the regression for first pass group 1, as the relative volatility of 1.51 falls within that group:
$$98.79 = \frac{70.16}{\ln 1.51 \times (0.08279) + 0.6761}$$
 - ▶ The slope, intercept, and regression error are calculated for each boiling point group defined in the surrogate file. $X = \text{predicted recovery (not percent)}$ and $Y = \text{boiling point (BP)}$.
- Relative volatility surrogates
 - ▶ For hexafluorobenzene, the measured recovery is 66.36.
 - ▶ The predicted recovery is the measured recovery divided by the predicted recovery for hexafluorobenzene using the boiling point curve. In Appendix 5, it is noted that for boiling points less than the lowest boiling point surrogate (in this case, 85°C), the predicted recovery is 1 (100%). Thus the predicted recovery for hexafluorobenzene (bp 81.5°C) is the same as the measured recovery.
 - ▶ Pyridine-d5 has a boiling point of 115°C. The predicted recovery for pyridine-d5,

using the first boiling point group, is:

$$171.10 = \frac{166.74}{115 \times (0.0006627) + 0.8983}$$

- ▶ The slope, intercept, and regression error are calculated for each relative volatility group defined in the surrogate file. X = predicted recovery (not percent) and Y = ln rw.

Page 2 of the surrogate report shows predicted results for check surrogates. It includes the following information:

- The report header lists:
 - ▶ The report date and time.
 - ▶ The 8261 library file.
 - ▶ The surrogate data file.
 - ▶ The sample data path – the actual data file is the path name + ‘\results.ini’.
 - ▶ The calibration file.
- The check surrogates are grouped by physical properties. For each check surrogate, the following information is shown:
 - ▶ Compound name
 - ▶ Boiling point
 - ▶ Relative volatility-water
 - ▶ The relative recovery (Recovery) is based on the sample area counts and the calibration response factor. For nitromethane-C13, the calibration response factor is shown in Appendix 6:

$$111.7 = \frac{2135560 / 650}{2940.46} \times 100$$

- ▶ The boiling point predicted recovery and error (B.P. Predict/err) are found using boiling point group one:

$$96.5 = (101 \times 0.0006627 + 0.8983) \times 100$$

Process Date: 7/24/2002 4:06:57 PM
 Compound Library File: C:\HPCHEM\1\METHODS\MTBE_1.M\8261dir\lib8261.ini
 Surrogate Groupings File: C:\HPCHEM\1\METHODS\MTBE_1.M\8261DIR\SURROGATES\ANTEON_SURR0102.INI
 Data File: C:\HPCHEM\1\DATA\JANUARY01280205.D
 Calibration File: C:\HPCHEM\1\METHODS\MTBE_1.M\8261DIR\CALIBRATION\MTBE-5PT.CAL

Check Surrogate Determinations

Volatile Compound Surrogates(boiling points<159 and rw<100)

Compound	BP	RW	Recovery	B.P.Predict/err	R.V.Predict/err	Total Predict/err	Recovery/err
diethylether-d10	35	32.50	76.8	100.0 0.0	93.5 1.4	93.5 1.4	82.2 1.2
methylenechloride-d2	40	11.10	76.0	100.0 0.0	86.9 2.5	86.9 2.5	87.5 2.5
benzene-d6	79	3.92	70.3	100.0 0.0	80.3 2.9	80.3 2.9	87.5 3.2
1,2-dichloropropane-d6	95	11.00	83.7	96.1 6.6	86.8 2.5	83.4 6.2	100.4 7.4
4-bromofluorobenzene	152	8.05	85.4	99.9 6.6	84.4 2.5	84.3 6.1	101.3 7.3
Group Averages							91.8 4.3

Non-Purgeable Volatile Compound Surrogates(boiling points<159 and rw>100)

Compound	BP	RW	Recovery	B.P.Predict/err	R.V.Predict/err	Total Predict/err	Recovery/err
nitromethane-C13	101	510.00	111.7	96.5 6.6	90.8 30.9	87.6 30.4	127.5 44.2
ethylacetate-c13	77	150.00	94.9	100.0 0.0	93.4 1.4	93.4 1.4	101.5 1.5
pyridine-d5	115	15000.00	166.7	97.5 6.6	137.8 30.9	134.3 31.4	124.1 29.1
Group Averages							117.7 24.9

Semi-Volatile Compound Surrogates(boiling points>158)

Compound	BP	RW	Recovery	B.P.Predict/err	R.V.Predict/err	Total Predict/err	Recovery/err
aniline-13C6	184	13700.00	181.6	103.2 0.1	137.8 30.9	142.2 31.9	127.7 28.6
decafluorobiphenyl	206	3.03	81.1	104.1 0.1	78.0 3.2	81.2 3.4	99.9 4.1
nitrobenzene-d5	210	87.50	91.6	104.2 0.1	93.4 1.4	97.4 1.5	94.0 1.4
acetophenone-d5	202	161.00	115.8	103.9 0.1	93.4 1.4	97.1 1.5	119.3 1.8
naphthalene-d8	217	18.00	94.4	107.3 1.6	90.5 2.5	97.2 3.0	97.1 3.0
Group Averages							107.6 7.8

$$\text{error} = .0655$$

$$9.9 = \sqrt{\left(\frac{.066}{.965}\right)^2 + \left(\frac{.055}{.734}\right)^2} \times 0.978 \times 100$$

- ▶ The relative volatility predicted recovery and error

(R.V. Predict/err) are found using relative volatility group five (because groups 4 and 5 share endpoints, the low endpoint of group 5 is 477.5 - see Appendix 3):

$$90.8 = (\ln 510 \times (0.1618) + -0.1011) \times 100$$

$$\text{error} = .309$$

- ▶ The total predicted recovery (Total Predict) is the product of the boiling point and relative volatility predicted recoveries:

$$87.6 = (0.965 \times 0.908) \times 100$$

- ▶ The total predicted error (in percent) is given by Equation 11, Appendix 5:

$$30.4 = \sqrt{\left(\frac{.0655}{.965}\right)^2 + \left(\frac{.309}{.908}\right)^2} \times .876 \times 100$$

- ▶ The corrected recovery (Recovery) is the measured recovery divided by the predicted recovery:

$$127.5 = \frac{111.7}{87.6}$$

- ▶ The corrected recovery error (in percent) is given by:

$$44.2 = \sqrt{\left(\frac{.0655}{.965}\right)^2 + \left(\frac{.309}{.908}\right)^2} \times 1.275 \times 100$$

- The group averages are arithmetic means.

APPENDIX 9 QUANTITATION REPORT

The quantitation report shows correction results for analytes only. There are two version of this report – a printout and a semicolon delimited text file – that are printed from the Process Files dialog box (Figure 16). The printout includes the following information:

- Report header
 - ▶ 'File Identification' is the sample file being reported. This is a directory path. The file which contains the sample area counts is the path + '\results.ini'.
 - ▶ The date and time the report was printed.
 - ▶ The sample size (ml or g).
 - ▶ The sample type.
 - ▶ Comments.
 - ▶ The calibration file used for calculations.
 - ▶ The 8261 library file used.
 - ▶ The surrogate file used to define surrogate groups.

- For each analyte:
 - ▶ The compound name.
 - ▶ The concentration (ng/volume) of the analyte in the sample.
 - ▶ The concentration deviation (ng/volume).
 - ▶ The predicted total recovery.
 - ▶ The total recovery deviation.

Calculations used in this report are explained in Appendix 5, section 2. The specific surrogate report for the quantitation must be used to duplicate the values in the report. See Appendix 8 for an explanation of the surrogate report.

The second example listing is the semicolon delimited text file. This report is automatically saved in each data directory in the Process Files dialog list box (see section 3.1.4.1 – “Process” button). It can also be displayed in a NotePad window (section 2.1.2.15 – “Review” button). This report can be imported into a spreadsheet for any desired further processing. The listing includes column headings (PM refers to the \pm sign).

File Identification: C:\HPCHEM1\DATA\JANUARY01280205.D
 Process Date: 7/24/2002 4:06:57 PM
 Sample Size: 2
 Sample Type:
 Comment 1:
 Comment 2:

Quantitation Report

Page: 1

Calibration File: C:\HPCHEM1\METHODS\MTBE_1.M\8261DIR\CALIBRATION\MTBE_5PT.CAL
 Compound Library File: C:\HPCHEM1\METHODS\MTBE_1.M\8261DIR\lib8261.lib
 Surrogate Groups: C:\HPCHEM1\METHODS\MTBE_1.M\8261DIR\SURROGATES\ANTEON_SURR0102.

Compound	ng/mL	Error	Pred. Recovery	Deviation	Compound	ng/mL	Error	Pred. Recovery	Deviation
31: dichlorodifluoromethane	9.6	± 0.469	66.50	± 3.23	76: 1,3-dichloropropane	16.8	± 1.21	90.91	± 6.55
32: chloromethane	8.40	± 0.384	70.76	± 3.23	77: dibromochloromethane	16.1	± 1.16	88.99	± 6.43
33: vinylchloride	8.62	± 0.419	66.50	± 3.23	78: 2-picoline	38.2	± 9.24	130.56	± 31.61
34: bromomethane	11.4	± 0.502	73.35	± 3.23	79: 1,2-dibromoethane	17.3	± 1.18	82.15	± 6.28
35: chloroethane	8.39	± 0.389	67.97	± 3.23	80: chlorobenzene	16.4	± 1.24	80.95	± 6.10
36: trichlorofluoromethane	10.8	± 0.527	66.50	± 3.23	81: 1,1,1,2-tetrachloroethane	16.6	± 1.20	85.86	± 6.21
37: ether	26.9	± 0.408	93.47	± 1.42	82: ethylbenzene	15.0	± 1.17	78.67	± 6.12
38: acetone	11.3	± 3.74	93.41	± 30.89	83: n-nitroso-methyl-ethylamine	83.1	± 22.91	114.78	± 31.64
39: 1,1-dichloroethane	9.6	± 0.467	66.50	± 3.23	84: m,p-xylenes	7.50	± 0.566	79.48	± 6.00
40: isomethane	21.8	± 0.934	75.45	± 3.23	85: styrene	16.0	± 1.16	82.75	± 5.98
41: allylchloride	9.5	± 0.436	70.55	± 3.23	86: o-xylene	16.4	± 1.23	81.23	± 6.09
42: acetonitrile	61.5	± 20.68	91.86	± 30.89	87: isopropylbenzene	15.9	± 1.24	77.05	± 6.00
43: methylenchloride	7.78	± 0.222	86.14	± 2.46	88: bromoform	16.4	± 1.16	92.29	± 6.54
44: MTBE	5.67	± 0.086	93.48	± 1.42	89: cis-1,4-dichloro-2-butene	71.2	± 4.80	93.39	± 6.29
45: acrylonitrile	33.3	± 0.506	93.42	± 1.42	90: n-nitrosodiethylamine	31.5	± 76.48	131.10	± 31.79
46: trans-1,2-dichloroethane	11.1	± 0.475	75.49	± 3.23	91: 1,1,2,2-tetrachloroethane	17.5	± 1.18	93.02	± 6.29
47: 1,1-dichloroethane	9.5	± 0.345	80.51	± 2.91	92: 1,2,3-trichloropropane	18.6	± 0.283	95.43	± 1.45
48: methacrylonitrile	37.6	± 2.64	89.51	± 6.27	93: propylbenzene	15.2	± 0.648	77.65	± 3.30
49: 2-butanone	53.2	± 16.87	97.45	± 30.89	94: trans-1,4-dichloro-2-butene	66.7	± 1.02	95.40	± 1.45
50: propionitrile	27.8	± 8.21	103.34	± 30.56	95: 1,3,5-trimethylbenzene	15.0	± 0.605	81.90	± 3.31
51: 2,2-dichloropropane	11.2	± 0.511	70.76	± 3.23	96: bromobenzene	15.3	± 0.477	86.00	± 2.51
52: cis-1,2-dichloroethane	27.8	± 0.564	81.58	± 2.91	97: 2-chlorotoluene	15.1	± 0.547	82.19	± 2.98
53: chloroform	15.8	± 0.494	82.67	± 2.46	98: 4-chlorotoluene	15.8	± 0.567	82.98	± 2.98
54: bromochloromethane	16.8	± 0.464	89.33	± 2.46	99: pentachloroethane	15.3	± 0.455	90.19	± 2.52
55: 1,1-trichloroethane	9.6	± 0.443	70.35	± 3.23	100: tert-butylbenzene	16.3	± 0.648	79.01	± 3.32
56: 1,1-dichloropropene	17.6	± 1.47	64.53	± 5.38	101: 1,2,4-trimethylbenzene	15.9	± 0.571	82.97	± 2.99
57: carbontetrachloride	8.19	± 0.398	66.50	± 3.23	102: sec-butylbenzene	15.1	± 0.662	75.82	± 3.32
58: 1,2-dichloroethane	15.8	± 0.429	90.80	± 2.46	103: aniline	14.6	± 32.73	142.21	± 31.88
59: benzene	14.6	± 0.596	79.46	± 3.23	104: p-isopropyltoluene	15.4	± 0.651	78.66	± 3.34
60: trichloroethene	16.7	± 1.35	72.32	± 5.94	105: 1,3-dichlorobenzene	15.4	± 0.588	84.12	± 2.99
61: 1,2-dichloropropane	16.9	± 1.25	83.41	± 6.16	106: 1,4-dichlorobenzene	16.8	± 0.596	84.45	± 2.99
62: methylmethacrylate	37.3	± 2.60	90.20	± 6.28	107: n-butylbenzene	15.4	± 0.675	75.87	± 3.34
63: bromodichloromethane	18.6	± 1.37	83.95	± 6.21	108: 1,2-dichlorobenzene	16.6	± 0.485	86.79	± 2.54
64: 1,4-dioxane	127	± 31.48	125.47	± 31.01	109: n-nitrosodi-n-propylamine	10.1	± 2.69	120.58	± 32.15
65: dibromomethane	16.7	± 1.22	89.20	± 6.52	110: acetophenone	28.9	± 0.441	97.12	± 1.48
66: 4-methyl-2-pentanone	76.3	± 5.25	91.18	± 6.28	111: o-toluidine	161	± 36.11	143.11	± 32.08
67: trans-1,3-dichloropropene	83.8	± 6.11	86.23	± 6.28	112: 1,2-dibromo-3-chloropropane	17.1	± 0.261	96.91	± 1.48
68: toluene	13.9	± 1.06	78.01	± 5.97	113: hexachlorobutadiene	16.3	± 0.750	79.35	± 3.65
69: pyridine	89.6	± 20.97	134.40	± 31.45	114: nitrobenzene	28.9	± 0.441	97.45	± 1.48
70: cis-1,3-dichloropropene	83.0	± 6.05	88.17	± 6.43	115: 1,2,4-trichlorobenzene	17.1	± 0.566	89.11	± 2.95
71: ethylmethacrylate	32.0	± 2.21	91.21	± 6.28	116: naphthalene	16.5	± 0.515	96.97	± 3.03
72: n-nitrosodimethylamine	171	± 11.52	93.47	± 6.29	117: 1,2,3-trichlorobenzene	16.9	± 0.541	93.78	± 3.01
73: 2-hexanone	35.7	± 2.44	91.86	± 6.28	118: 2-methylnaphthalene	34.2	± 0.713	108.41	± 2.22
74: 1,1,2-trichloroethane	16.0	± 1.16	90.92	± 6.57	119: 1-methylnaphthalene	35.2	± 0.735	108.41	± 2.22
75: tetrachloroethene	14.8	± 1.20	69.82	± 5.63					

sample.prn

Quantitation Report

File Identification: C:\HPCHEM\1\DATA\AJANUARY\01280205.D;Calibration
File:C:\HPCHEM\1\METHODS\MTBE_1.M\8261DIR\CALIBRATION\MTBE-5PT.CAL
Process Date: 7/27/2002 3:29:48 PM;Compound
Library:C:\HPCHEM\1\METHODS\MTBE_1.M\8261dir\lib8261.ini
Sample Size: 2.00;Surrogate Groups:
C:\HPCHEM\1\METHODS\MTBE_1.M\8261DIR\SURROGATES\ANTEON_SURR0102.INI
Sample Type:
Comment 1:
Comment 2:

Compound#;Compound;ng/mL;PM;Error;Pred.Recovery;PM;Deviation
31;dichlorodifluoromethane;9.64;±;0.469;66.50;±;3.23;
32;chloromethane;8.40;±;0.384;70.76;±;3.23;
33;vinylchloride;8.62;±;0.419;66.50;±;3.23;
34;bromomethane;11.38;±;0.502;73.35;±;3.23;
35;chloroethane;8.39;±;0.399;67.97;±;3.23;
36;trichlorofluoromethane;10.84;±;0.527;66.50;±;3.23;
37;EthylEther;26.86;±;0.408;93.47;±;1.42;
38;acetone;11.30;±;3.735;93.41;±;30.89;
39;1,1-dichloroethene;9.61;±;0.467;66.50;±;3.23;
40;iodomethane;21.79;±;0.934;75.45;±;3.23;
41;allylchloride;9.51;±;0.436;70.55;±;3.23;
42;acetonitrile;61.50;±;20.682;91.86;±;30.89;
43;methylenechloride;7.78;±;0.222;86.14;±;2.46;
44;MTBE;5.67;±;0.086;93.48;±;1.42;
45;acrylonitrile;33.28;±;0.506;93.42;±;1.42;
46;trans-1,2-dichloroethene;11.10;±;0.475;75.49;±;3.23;
47;1,1-dichloroethane;9.53;±;0.345;80.51;±;2.91;
48;methacrylonitrile;37.63;±;2.637;89.51;±;6.27;
49;2-butanone;53.21;±;16.866;97.45;±;30.89;
50;propionitrile;27.77;±;8.211;103.34;±;30.56;
51;2,2-dichloropropane;11.20;±;0.511;70.76;±;3.23;
52;cis-1,2-dichloroethene;15.81;±;0.564;81.58;±;2.91;
53;chloroform;16.60;±;0.494;82.67;±;2.46;
54;bromochloromethane;16.82;±;0.464;89.33;±;2.46;
55;1,1,1-trichloroethane;9.63;±;0.443;70.35;±;3.23;
56;1,1-dichloropropene;17.62;±;1.468;64.53;±;5.38;
57;carbontetrachloride;8.19;±;0.398;66.50;±;3.23;
58;1,2-dichloroethane;15.81;±;0.429;90.80;±;2.46;
59;benzene;14.65;±;0.596;79.46;±;3.23;
60;trichloroethene;16.65;±;1.345;72.32;±;5.84;
61;1,2-dichloropropane;16.92;±;1.249;83.41;±;6.16;
62;methylmethacrylate;37.33;±;2.597;90.20;±;6.28;
63;bromodichloromethane;18.56;±;1.373;83.95;±;6.21;
64;1,4-dioxane;127.36;±;31.477;125.47;±;31.01;
65;dibromomethane;16.73;±;1.223;89.20;±;6.52;
66;4-methyl-2-pentanone;76.27;±;5.252;91.18;±;6.28;
67;trans-1,3-dichloropropene;83.82;±;6.109;86.23;±;6.28;
68;toluene;13.90;±;1.064;78.01;±;5.97;
69;pyridine;89.60;±;20.967;134.40;±;31.45;
70;cis-1,3-dichloropropene;82.96;±;6.051;88.17;±;6.43;
71;ethylmethacrylate;32.04;±;2.206;91.21;±;6.28;
72;n-nitrosodimethylamine;171.34;±;11.523;93.47;±;6.29;
73;2-hexanone;35.67;±;2.439;91.86;±;6.28;
74;1,1,2-trichloroethane;16.03;±;1.159;90.92;±;6.57;
75;tetrachloroethene;14.81;±;1.199;69.62;±;5.63;
76;1,3-dichloropropane;16.77;±;1.209;90.91;±;6.55;
77;dibromochloromethane;16.11;±;1.164;88.99;±;6.43;
78;2-picoline;38.17;±;9.242;130.56;±;31.61;
79;1,2-dibromoethane;17.34;±;1.182;92.15;±;6.28;
80;chlorobenzene;16.41;±;1.236;80.95;±;6.10;

Page 1

APPENDIX 10 CHECK CALIBRATION REPORT

This report compares analyte recoveries from a sample run with the analyte recoveries in the calibration. It includes the following information:

- Report header
 - ▶ The calibration file used for calculations.
 - ▶ The date and time the report was printed.
 - ▶ The surrogate file containing the surrogate groups used.
 - ▶ The check standard file being reported. This is a directory path. The file which contains the sample area counts is the path + '\results.ini'.
- For each analyte:
 - ▶ The response factor shown in the calibration file shown in the header.
 - ▶ The response factor deviation listed the calibration file.
 - ▶ The concentration (ng) of the analyte in the check standard.
 - ▶ The predicted recovery for the check standard.
 - ▶ The recovery deviation for the check standard.
 - ▶ The response factor for the check standard.
 - ▶ The percent difference between the calibration response factor and the check standard response factor.

Calculations in this report are shown in Appendix 5, section 3. The specific surrogate report for the check calibration must be used to duplicate the values in the report. See Appendix 8 for an explanation of the surrogate report.

User's Guide for Surrogate Matrix Normalization

Check Calibration Report

Page: 1

Calibration File: A:\MTBE-5PTEDITED.CAL
 Process Date: 5/30/2002 10:22:29 AM
 Surrogate Groupings File: C:\HPCHEM\1\METHODS\MTBE_1.M\8261\DIR\SURROGATES\SURR0102.INI
 Check Standard File: C:\HPCHEM\1\DATA\TestMeth\01280205.D

Compound	Calibration		(concentrations)	Check Standard		response	Difference %
	response	deviation		Pred_Rec	R_Dev		
31 dichlorodifluoromethane	1998.82	1000.61	(25.00)	66.50	3.23	2798.89	40.0%
32 chloromethane	1776.91	657.98	(25.00)	70.76	3.23	2269.19	27.7%
33 vinylchloride	1495.74	701.09	(25.00)	66.50	3.23	2121.85	41.9%
34 bromomethane	1408.32	724.61	(25.00)	73.35	3.23	2260.27	60.5%
35 chloroethane	1968.80	947.94	(25.00)	67.97	3.23	2125.23	7.9%
36 trichlorofluoromethane	3792.57	1645.28	(25.00)	66.50	3.23	5476.46	44.4%
37 ether	1364.27	611.17	(50.00)	93.47	1.42	1918.57	40.6%
38 acetone	11807.80	19640.52	(50.00)	93.41	30.89	4381.21	-62.9%
39 1,1-dichloroethene	2377.03	987.76	(25.00)	66.50	3.23	3320.87	39.7%
40 iodomethane	1744.34	792.61	(50.00)	75.45	3.23	2632.43	50.9%
41 allylchloride	1118.82	490.26	(25.00)	70.55	3.23	1633.87	46.0%
42 acetonitrile	577.73	270.56	(100.00)	91.86	30.89	799.88	38.5%
43 methylenechloride	13268.63	16424.08	(25.00)	86.14	2.46	6252.89	-52.9%
44 MTBE	1329.63	0.00	(20.00)	93.48	1.42	198.22	-85.1%
45 acrylonitrile	1560.80	825.74	(50.00)	93.42	1.42	2077.87	33.1%
46 trans-1,2-dichloroethene	2303.47	1217.42	(25.00)	75.49	3.23	3554.61	54.3%
47 1,1-dichloroethane	7251.29	7898.19	(25.00)	80.51	2.91	5528.23	-23.8%
48 methacrylonitrile	1928.91	815.96	(50.00)	89.51	6.27	2903.63	50.5%
49 2-butanone	1017.01	631.68	(100.00)	97.45	30.89	1147.84	12.9%
50 propionitrile	379.61	389.11	(50.00)	103.34	30.56	644.48	69.8%
51 2,2-dichloropropane	4032.82	3210.20	(25.00)	70.76	3.23	3611.94	-10.4%
52 cis-1,2-dichloroethene	2627.87	1236.13	(25.00)	81.58	2.91	3778.17	43.8%
53 chloroform	5732.93	2254.23	(25.00)	82.67	2.46	7614.17	32.8%
54 bromochloromethane	1736.51	772.87	(25.00)	89.33	2.46	2336.65	34.6%
55 1,1,1-trichloroethane	4881.39	2403.40	(25.00)	70.35	3.23	7394.76	51.5%
56 1,1-dichloropropene	3110.87	1417.89	(25.00)	64.53	5.38	4779.24	53.6%
57 carbon tetrachloride	8512.17	10698.15	(25.00)	66.50	3.23	5578.72	-34.5%
58 1,2-dichloroethane	5207.66	2598.08	(25.00)	90.80	2.46	6586.87	26.5%
59 benzene	8713.15	4564.22	(25.00)	79.46	3.23	13597.54	56.1%
60 trichloroethene	3737.70	1700.06	(25.00)	72.32	5.84	4979.25	33.2%
61 1,2-dichloropropane	1953.71	836.80	(25.00)	83.41	6.16	2645.01	35.4%
62 methylmethacrylate	3740.91	1771.67	(50.00)	90.20	6.28	5585.74	49.3%
63 bromodichloromethane	3883.17	2868.38	(25.00)	83.95	6.21	5766.84	48.5%
64 1,4-dioxane	41.93	13.69	(250.00)	125.47	31.01	47.86	14.1%
65 dibromomethane	2223.32	1022.24	(25.00)	89.20	6.52	2975.83	33.8%
66 4-methyl-2-pentanone	702.38	301.05	(100.00)	91.18	6.28	1071.45	52.5%
67 trans-1,3-dichloropropene	3520.40	1867.18	(125.00)	86.23	6.28	4721.33	34.1%
68 toluene	13173.73	7915.91	(25.00)	78.01	5.97	14644.82	11.2%
69 pyridine	106.21	89.51	(125.60)	134.40	31.45	191.30	80.1%
70 cis-1,3-dichloropropene	3962.86	1979.76	(125.00)	88.17	6.43	5259.86	32.7%
71 ethylmethacrylate	13.94	10.32	(50.00)	91.21	6.28	17.87	28.2%
72 n-nitrosodimethylamine	905.08	0.00	(500.10)	93.47	6.29	157.72	-82.6%
73 2-hexanone	1269.00	501.78	(50.10)	91.86	6.28	1807.04	42.4%
74 1,1,2-trichloroethane	2447.65	1072.51	(25.00)	90.92	6.57	3527.04	44.1%
75 tetrachloroethene	3961.38	2542.11	(25.00)	69.62	5.63	4693.80	18.5%
76 1,3-dichloropropane	4491.75	2048.89	(25.00)	90.91	6.55	6027.14	34.2%
77 dibromochloromethane	3502.46	1691.37	(25.00)	88.99	6.43	4513.48	28.9%
78 2-picoline	45.83	55.05	(125.10)	130.56	31.61	27.26	-40.5%
79 1,2-dibromoethane	3118.50	1406.35	(25.00)	92.15	6.28	4325.71	38.7%
80 chlorobenzene	9065.28	4206.34	(25.00)	80.95	6.10	11900.60	31.3%
81 1,1,1,2-tetrachloroethane	3158.19	1597.21	(25.00)	85.86	6.21	4198.99	33.0%
82 ethylbenzene	4392.98	2798.27	(25.00)	78.67	6.12	5277.39	20.1%
83 n-nitroso-methyl-ethylamine	871.37	0.00	(500.10)	114.78	31.64	63.17	-92.8%
84 m,p-xylenes	11080.09	6606.84	(12.50)	79.48	6.00	13293.17	20.0%
85 styrene	5398.31	2722.44	(25.00)	82.75	5.98	6914.36	28.1%
86 o-xylene	4912.45	2471.95	(25.00)	81.23	6.09	6434.36	31.0%
87 isopropylbenzene	3726.05	2537.31	(25.00)	77.05	6.00	4747.32	27.4%
88 bromoform	2104.23	1157.33	(25.00)	92.29	6.54	2758.81	31.1%
89 cis-1,4-dichloro-2-butene	530.38	324.07	(100.00)	93.39	6.29	892.36	68.2%
90 n-nitrosodiethylamine	10.23	4.37	(500.00)	131.10	31.79	12.91	26.2%
91 1,1,2,2-tetrachloroethane	3814.76	1663.06	(25.00)	93.02	6.29	5336.68	39.9%
92 1,2,3-trichloropropane	3711.38	1883.24	(25.00)	95.43	1.45	5518.65	48.7%
93 propylbenzene	4295.09	2848.61	(25.00)	77.65	3.30	5231.06	21.8%
94 trans-1,4-dichloro-2-butene	925.91	544.45	(100.00)	95.40	1.45	1235.82	33.5%
95 1,3,5-trimethylbenzene	6235.34	4232.75	(25.00)	81.90	3.31	7466.63	19.7%
96 bromobenzene	4055.73	1830.29	(25.00)	86.00	2.51	5297.56	30.6%
97 2-chlorotoluene	3995.41	2587.53	(25.00)	82.19	2.98	4825.27	20.8%

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APPENDIX 11
MDL REPORT

This is about as simple as it gets. Zeros for MDLs indicate that there is no MDL calculated.

MDL Report: C:\HPCHEM\1\METHODS\MTBE_1.m\8261dir\Mdl\mdl2.mdl Page: 1

Creation Date: 7/23/2002 12:12:52 PM

Compound Library File: C:\HPCHEM\1\METHODS\MTBE_1.M\8261dir\lib8261.ini

Surrogate Groups: C:\HPCHEM\1\METHODS\MTBE_1.m\8261dir\Surrogates\lanteon_sur0102_MDL.ini

Sample Size: 2

Matrix: no matrix given

Other:

Unit: ng/mL

Compound	MDL	Compound	MDL
31: dichlorodifluoromethane	7.00	96: bromobenzene	0.00
32: chloromethane	10.00	97: 2-chlorotoluene	0.00
33: vinylchloride	13.00	98: 4-chlorotoluene	0.00
34: bromomethane	0.00	99: pentachloroethane	0.00
35: chloroethane	0.00	100: tert-butylbenzene	0.00
36: trichlorofluoromethane	0.00	101: 1,2,4-trimethylbenzene	0.00
37: ether	0.00	102: sec-butylbenzene	0.00
38: acetone	0.00	103: aniline	0.00
39: 1,1-dichloroethene	0.00	104: p-isopropyltoluene	0.00
40: iodomethane	0.00	105: 1,3-dichlorobenzene	0.00
41: allylchloride	0.00	106: 1,4-dichlorobenzene	0.00
42: acetonitrile	35.00	107: n-butylbenzene	0.00
43: methylenechloride	0.00	108: 1,2-dichlorobenzene	0.00
44: MTBE	2.00	109: n-nitrosodi-n-propylamine	0.00
45: acrylonitrile	0.00	110: acetophenone	0.00
46: trans-1,2-dichloroethene	0.00	111: o-toluidine	0.00
47: 1,1-dichloroethane	0.00	112: 1,2-dibromo-3-chloropropane	0.00
48: methacrylonitrile	0.00	113: hexachlorobutadiene	0.00
49: 2-butanone	0.00	114: nitrobenzene	0.00
50: propionitrile	0.00	115: 1,2,4-trichlorobenzene	0.00
51: 2,2-dichloropropane	0.00	116: naphthalene	0.00
52: cis-1,2-dichloroethene	0.00	117: 1,2,3-trichlorobenzene	0.00
53: chloroform	0.00	118: 2-methylnaphthalene	0.00
54: bromochloromethane	0.00	119: 1-methylnaphthalene	30.00
55: 1,1,1-trichloroethane	0.00		
56: 1,1-dichloropropene	0.00		
57: carbon tetrachloride	0.00		
58: 1,2-dichloroethane	0.00		
59: benzene	0.00		
60: trichloroethene	0.00		
61: 1,2-dichloropropane	0.00		
62: methylmethacrylate	0.00		
63: bromodichloromethane	0.00		
64: 1,4-dioxane	0.00		
65: dibromomethane	0.00		
66: 4-methyl-2-pentanone	0.00		
67: trans-1,3-dichloropropene	0.00		
68: toluene	0.00		
69: pyridine	0.00		
70: cis-1,3-dichloropropene	0.00		
71: ethylmethacrylate	0.00		
72: n-nitrosodimethylamine	0.00		
73: 2-hexanone	0.00		
74: 1,1,2-trichloroethane	0.00		
75: tetrachloroethene	0.00		
76: 1,3-dichloropropane	0.00		
77: dibromochloromethane	0.00		
78: 2-picoline	0.00		
79: 1,2-dibromoethane	0.00		
80: chlorobenzene	0.00		
81: 1,1,1,2-tetrachloroethane	0.00		
82: ethylbenzene	0.00		
83: n-nitroso-methyl-ethylamine	0.00		
84: m,p-xylenes	0.00		
85: styrene	0.00		
86: o-xylene	0.00		
87: isopropylbenzene	0.00		
88: bromoform	0.00		
89: cis-1,4-dichloro-2-butene	0.00		
90: n-nitrosodiethylamine	0.00		
91: 1,1,2,2-tetrachloroethane	0.00		
92: 1,2,3-trichloropropane	0.00		
93: propylbenzene	0.00		
94: trans-1,4-dichloro-2-butene	0.00		
95: 1,3,5-trimethylbenzene	0.00		

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APPENDIX 12 LIBRARY SYNCHRONIZATION PRINTOUT

This report is produced when the Method 8261 library and the surrogate file are not synchronized – that is, the surrogate file does not include same surrogate compounds as the Method 8261 library and in the same order. On the left side, the surrogate compounds are listed, along with surrogate groupings. ‘CS’ and ‘FP’ following a compound mean that the compound is a check surrogate or first pass surrogate. Relative volatility and boiling point groups are indicated by rvwg# and bpg#, where # is replaced by the appropriate group number. On the right side the corresponding Method 8261 library compounds are listed.

For example, in the following report:

- Surrogate compound 1 – diethylether-d10 – is a member of relative volatility groups 3 and 4. Library compound 1 is diethylether-d10 – the ‘=’ sign shows that the compounds are the same in both libraries.
- Surrogate compound 4 – nitromethane-C13 – is a check surrogate. Library compound 4 is also nitromethane-C13.
- Surrogate compound 24 – 1,2-dichlorobenzene-d5 – is a member of boiling point group 2. The Method 8261 library shows compound 24 as 1,2-dichlorobenzene-d4. If the spelling of this compound was changed from 1,2-dichlorobenzene-d5, the surrogate compound can safely be renamed to match. However, if the libraries have distinctly different compounds, the surrogate file must be recreated to match the Method 8261 library. Because all surrogate settings are listed, the surrogate file can largely be recreated.
- All analytes are listed, but have no corresponding entries in the surrogate file.

Process Date: 9/23/2003 2:58:06 PM
 Surrogate Library C:\HPCHEM\1\METHODS\MV\DX7.M\8261.dtl\lib-8261.ini
 Surrogate File C:\HPCHEM\1\METHODS\MV\DX7.M\8261.DIR\SURROGATES\SURRVDX7B.INI
 PP: First Pass - CS: Check Surrogate

N	Surrogate Compound	Library Compound
1:	diethylether-d10	
2:	acetone-C13	
3:	methylchloride-d2	
4:	acetone-C13	
5:	hexafluorobenzene	
6:	tetrahydrofuran-d8	
7:	ethylacetate-c13	
8:	pentafluorobenzene	
9:	benzene-d6	
10:	1,2-dichloroethane-d4	
11:	Fluorobenzene	
12:	1,4-difluorobenzene	
13:	1,2-dichloropropane-d6	
14:	dioxane-d8	
15:	pyridine-d5	
16:	pyridine-d5	
17:	1,1,2-trichloroethane-d3	
18:	1,2-dibromoethane-d4	
19:	chlorobenzene-d5	
20:	o-xylene-d10	
21:	4-bromofluorobenzene	
22:	bromobenzene-d6	
23:	aniline-13C6	
24:	1,2-dichlorobenzene-d5	
25:	chlorobenzene-d5	
26:	nitrobenzene-d5	
27:	acetophenone-d5	
28:	1,2,4-trichlorobenzene-d3	
29:	naphthalene-d8	
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