

Title: Processing and Evaluation of Internal Standard Data		Copy No: ##
SOP No.: 7.6/3.0/S	Effective Date: October 24, 2011	Location: ###

QSM Approval: _____

Processing and Evaluation of Internal Standard Data

1. Scope

This Standard Operating Procedure provides instructions for processing sample, blank, and calibration standard data with Agilent ChemStation software to extract internal standard peak areas, and for evaluating the results versus VOC Method criteria in chart form using MS Excel.

2. Procedure

2.1 Verifying and Editing the ChemStation Method for Internal Standards

- 2.1.1 From the “**Start**” menu select “**GC_MS Instrument**” and select “**Data Analysis**” from the sub-menu to load the sample analysis software. Load a method to calculate the internal standard responses.
- 2.1.2 Under “**File**”, load an air blank data file from the sequence of files to be processed.
- 2.1.3 Under “**Int**”, select “**Integrate**”. Note the retention times for the four internal standard peaks in the chromatogram, and compare them to the retention times specified in the method’s calibration database. Edit the times in the database if they do not agree closely (e.g. within ± 0.02 min.) with the air blank retention times. Under “**File**”, select “**Save Method**” to save any changes, then “**Exit**”.
- 2.1.4 Under “**File**”, select “**Load Data File**” and choose the next file to be calculated.
- 2.1.5 Under “**Quant**”, select “**Calculate/Generate Report**”. In the “**Quant Report Options**” box, select the report “**Style**” as “**Detailed, Text Only**”, toggle on the “**File**” switch as the report “**Destination**”, and click “**OK**”.
- 2.1.6 Repeat 2.1.5 until this has been done for all files in the sequence..
- 2.1.7 Under “**CustRpt**”, select “**Custom Reports**”. If the required method database file is not seen “**Chart/Edit *.CRD**” click on “**Change Method Database**” and select the desired file from the list in the pop up box.

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Click **“OK”**.. Select **“Chart/Edit *.CRD”** and click **“OK”**. In the **“View Charts”** box, select **“Edit Database”**.

2.1.8 Under **“FILE”**, choose **“Multiple File Select”**. Move the files that will be charted from the **“Data File Name”** box to the **“Files Selected for Processing”** box by either double clicking on the file names or using the arrow buttons. Click **“OK”**. The Internal Standard responses will be added to the custom report.

2.2 Charting Internal Standard Data in Excel

2.2.1 Click on any cell to activate the scroll bars, then scroll down to locate the data from the sequence just processed. Using the mouse, select all relevant cells. Under **“Edit”**, select **“Copy”**.

2.2.2 Open **MS Excel** from the Windows start menu. Open the internal standard workbook and click on the desired worksheet.

2.2.3 Scroll down to the first available row, **click on the cell in column “A”**. Under **“Edit”**, select **“Paste”** to copy the data from **“Custom Reports”** into **Excel**.

2.2.4 In **“Custom Reports”**, under **“File”**, select **“Close”**, then click the **“No”** button to close the file without saving changes.

2.2.5 **If the data copied to Excel, contains the calibration standard data Copy this data to the appropriate identified location near the top of the worksheet. (“Edit”/“Copy”/“Edit”/“Paste Special”/ “Values”).**

2.2.6 **If the calibration data was from an Initial Calibration series, copy and “Paste Special” the AVERAGE internal standard response values from the “Initial Cal” box to the “Daily Std” box to determine whether internal standard acceptance criterion in section 9.5.2 of method 1.02/*.* /M has been met.**

2.2.7 In accordance with section 9.7.3 of method 1.02/*.* /M, “the area responses of the quantitation ions for each internal standard must be within $\pm 40\%$ of the average initial standard response over all sequence runs excluding the conditioning blank run”.

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- 2.2.8 Calculate the average areas for each of the four internal standards over the sequence. **“Copy”** these areas and **“Paste Special”**, **“Values”** into the **“Daily Std”** box.
- 2.2.9 Scroll back down to the relevant data. The cells in the blue background area to the right of the peak area data now contain calculated values for % difference in internal standard responses between individual runs and the sequence average(s).
- 2.2.10 Using the mouse, select all relevant cells in the blue background area, including the cells containing internal standard compound names, **“Copy”**, **“Paste Special”**, **“Values”**, **“OK”**, to keep the % difference from changing when subsequent data is charted.
- 2.2.11 Under **“Insert”**, select **“Chart”**. Using the **“Chart Wizard”**, select **“Column”** chart type, click **“Next”**. Enter the analysis date as the **“Chart title”** and place the compound name **“Legend”** at the bottom of the chart.
- 2.2.12 In the final step, place the chart in the workbook **“As new sheet”**, and enter the analysis date in the box to the right of the toggle switch to create an identifiable tab on the worksheet. Click **“Finish”**.
- 2.2.13 From the chart, identify any runs that did not satisfy VOC Method criteria. Print the chart, and attach it to the hardcopy chromatograms for sequence runs.
- 2.2.14 Repeat 2.2.11, this time selecting a **“Line”** chart type.
- 2.2.15 Repeat 2.2.12 adding a **“b”** to the analysis date. Observe any obvious trends and consult supervisor as required.
- 2.2.19 **“Save”** and **“Close”** the workbook, and **“Exit” Excel**.

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3. Revisions

Sep 2011: Previous Section 2.2 removed as no longer relevant
 Revised SOP to reflect current procedure and make it generic
 Sections 2.2.14 and 2.2.15 added to indicate Line chart is also created
 Previous revisions deleted

Lead Reviewer: Gale Bryant **Date**
Title: Chemist, Air Toxics Unit

Approved By: Daniel Wang **Date**
Title: Head, Air Toxics Unit

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