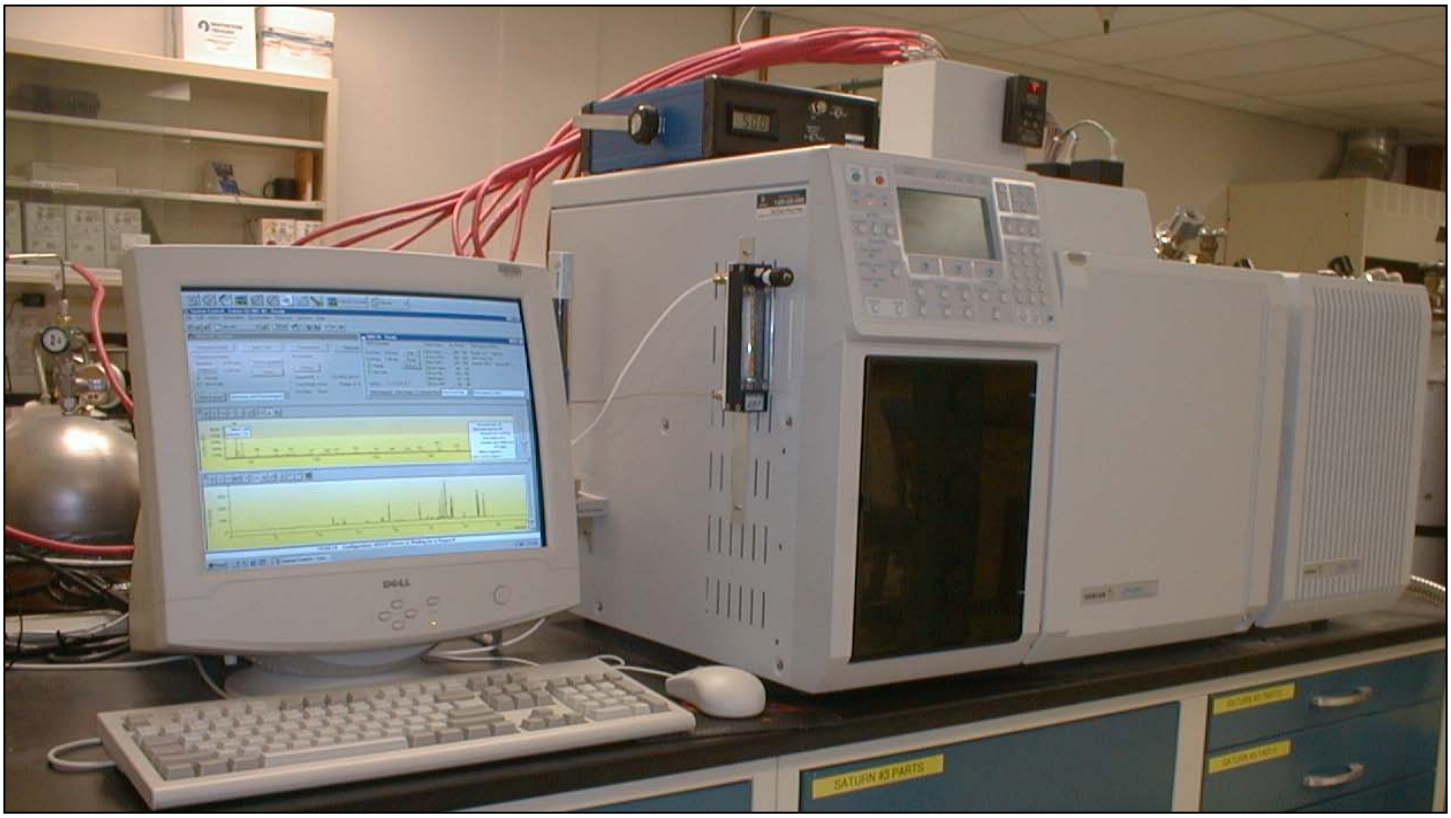
Injection Volume Documentation with Varian Saturn 2200 GC/MS and Sierra Instruments Mass Flow Controller

by Randall Bramston-Cook, Lotus Consulting

Gas chromatographs, with mass spectrometry detection, typically are capable of measuring down to a level around 100 ppb V/V. Unfortunately, this level is unable to match detection limits mandated by the USEPA for ambient air samples. EPA Method TO15 requires measure of concentrations well below 1 ppb V/V. The only approach available to accomplish this requirement is to concentrate the sample by absorption and/or cryogenic trapping of a large sample volume to focus the analytes from a gas sample into a smaller volume just before injection into a capillary column. Volumes of around 200 ml must be passed through the concentrator trap to achieve these mandated detections.

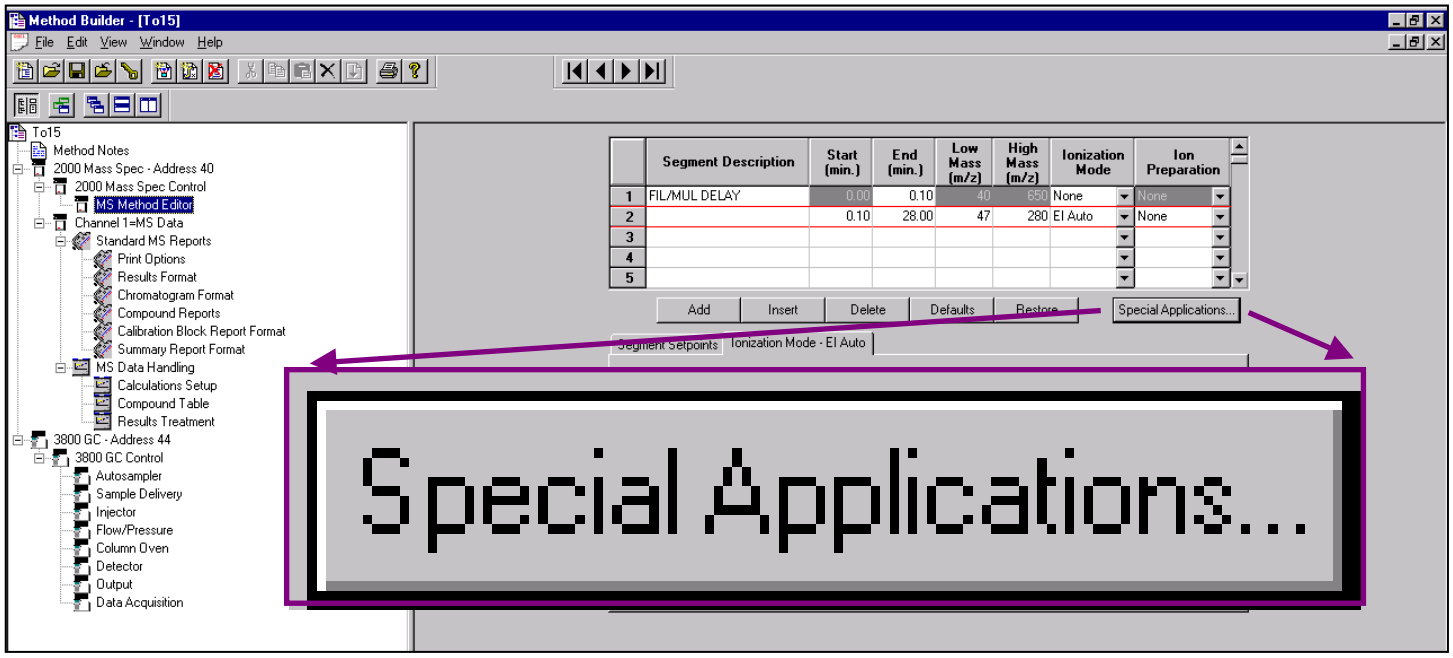
Two approaches to measure accurately this volume are available to the analyst. One mechanism is to properly load a fixed volume sample loop and flush the loop into a trap to focus the trace components just before injection. This approach is recommended for samples radically deviating from ambient air bulk composition, such as in exhaust samples with higher levels of carbon dioxides.[1]

The second method is to control the sample flow rate through the concentrator trap with a mass flow controller setting the rate and valve timing in the hardware setup to set the time interval. The product of the rate and time interval then sets the volume loading.

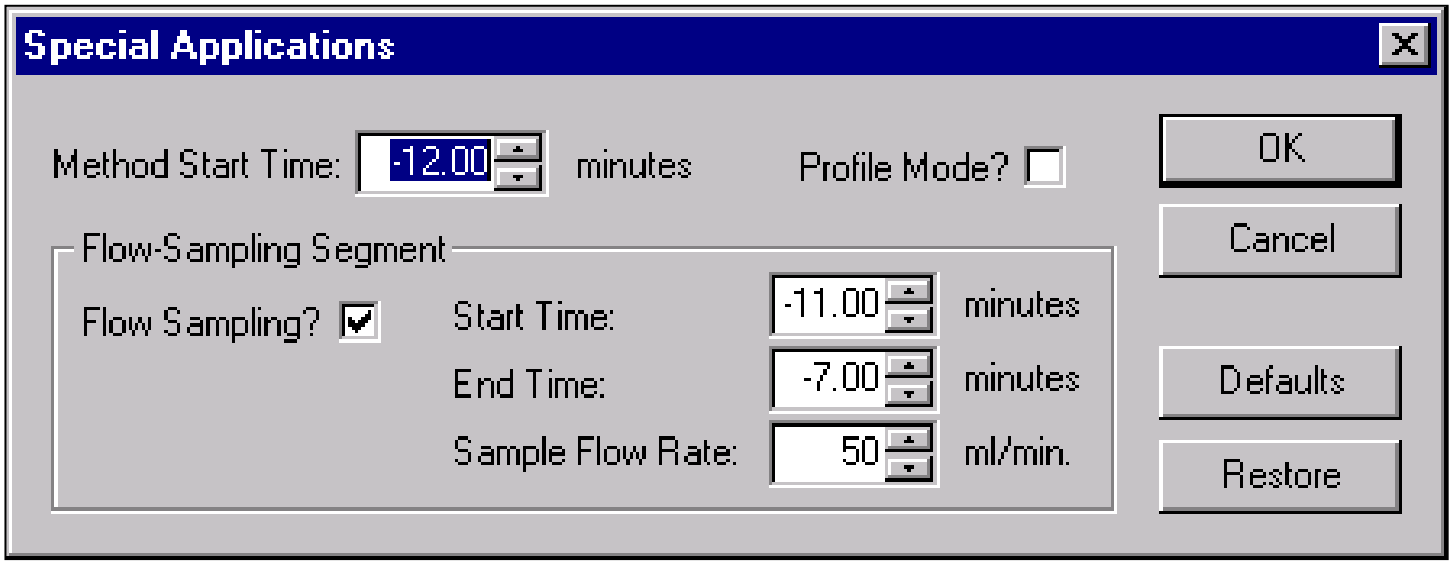


Ultra Trace Toxics System by Lotus Consulting with Varian Saturn 2200 and Sierra Instruments Mass Flow Controller (blue box on top of Varian 3800 Gas Chromatograph).

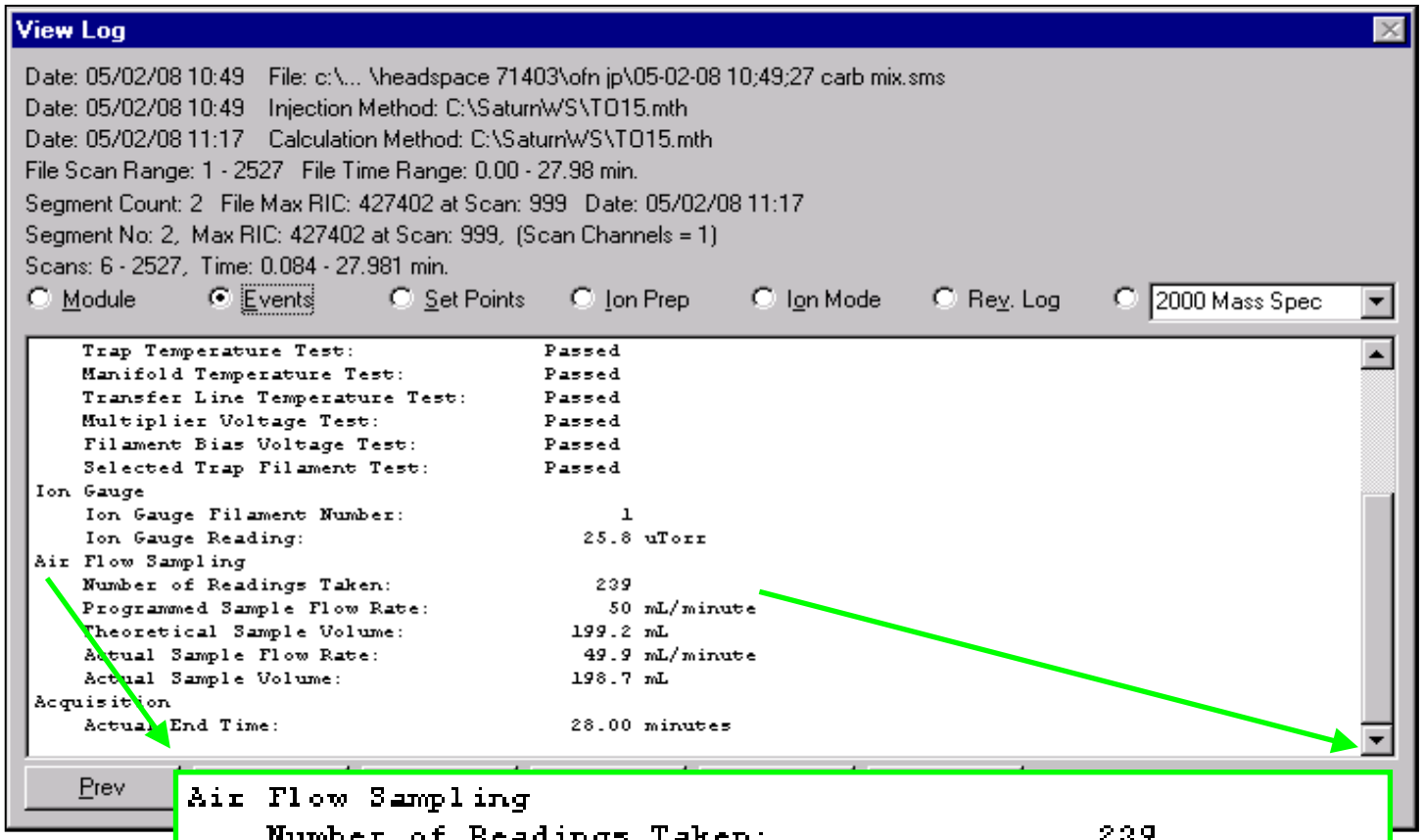
[1] Sample volumes with high levels of carbon dioxide in a sample cannot be measured properly with a mass flow controller, as the bulk sample components deviate radically from the preset calibration of the mass flow controller, For more details, refer to Bramston-Cook, R., “Mass Flow Controllers and Carbon Dioxide – Their Effects on Quantitation”, Lotus Consulting, Long Beach, CA, 2003.

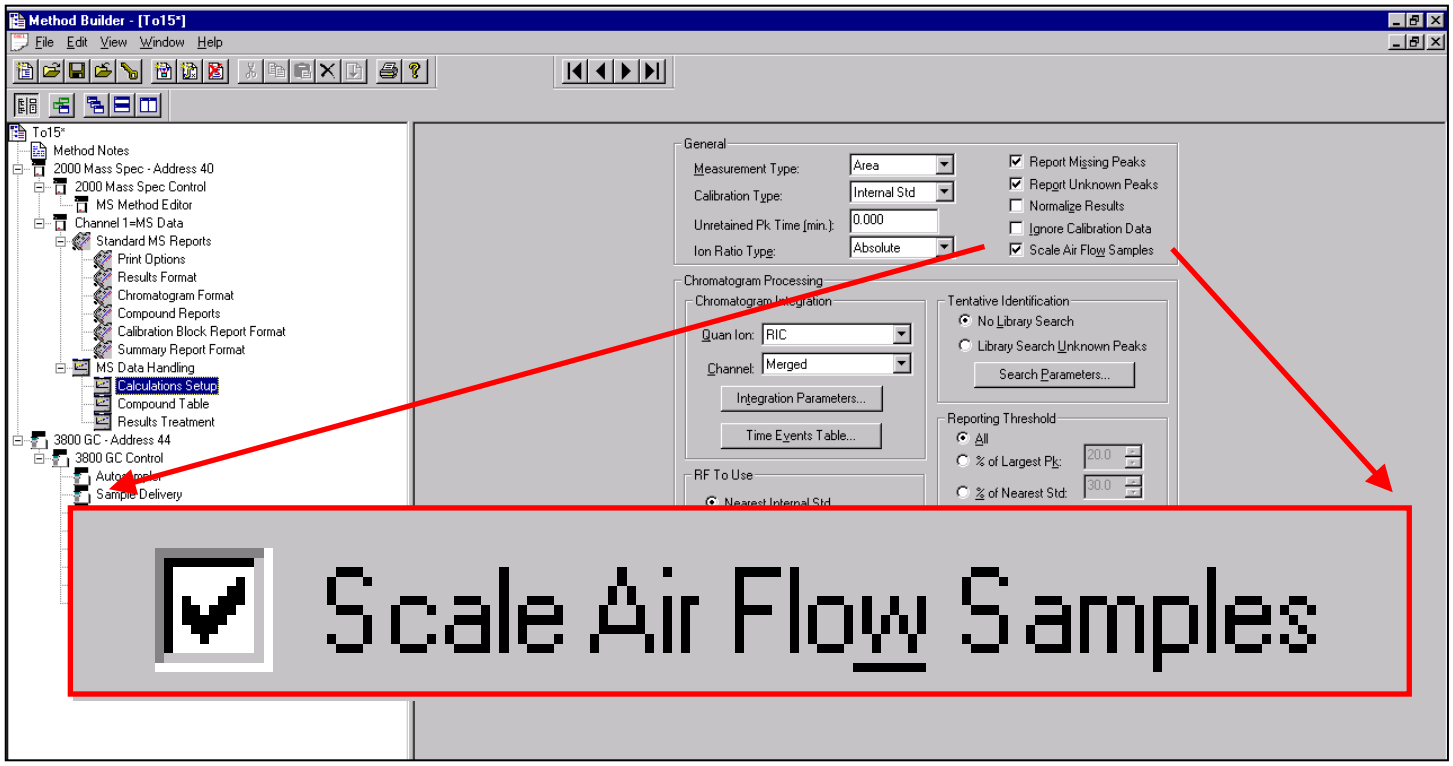


The Varian Saturn 2200 Mass Spectrometer, combined with the Varian 3800 Gas Chromatograph, has a software provision that allows full control of the Sierra Instruments Mass Flow Controller 840 with the associated Model 901 Controller. Through screen entries in the "Special Applications" entry screen in the Varian Saturn control software, the user can specify the flow rate for the mass flow controller and the timing interval for the actual trapping process. The product of the specified flow rate and the time interval becomes the "theoretical" volume injected. The Varian Saturn software then monitors the actual flow and determines the "actual" volume loaded. Documentation of these values are stored in the Saturn Events Log and reported with each run data file.

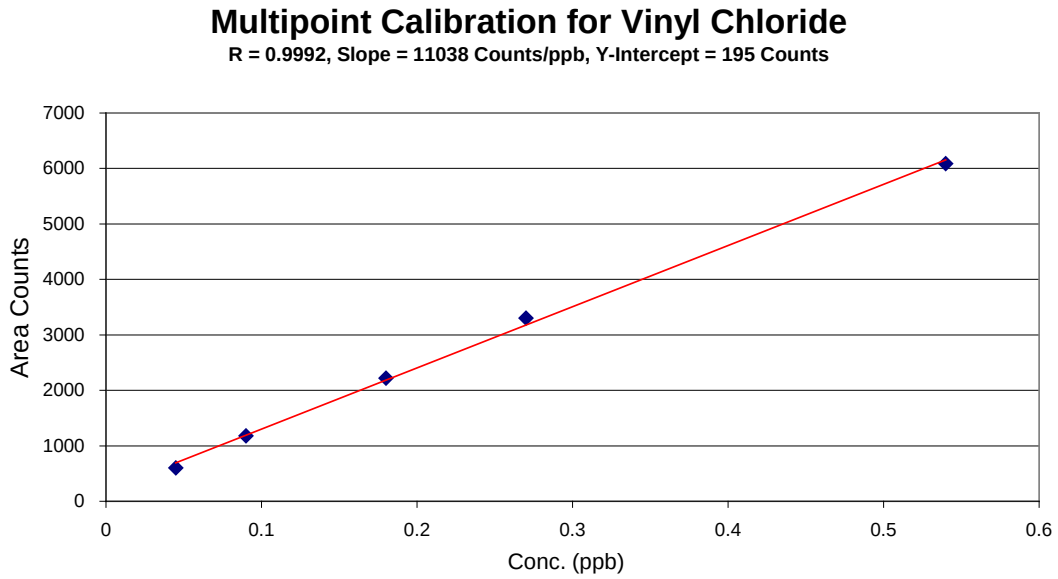


This feature of the Varian Saturn Workstation can be applied to two common situations in this experiment. One involves measurements of wildly divergent concentrations in samples from the standard calibration levels. By adjusting the flow rate and/or sample loading timing, the injected volume can be adjusted up or down and the final reported concentrations can be scaled automatically for these variations.


View Log
Date: 05/02/08 10:49 File: c:\... \headspace 71403\ofn jp\05-02-08 10;49;27 carb mix.sms
Date: 05/02/08 10:49 Injection Method: C:\SaturnWS\TO15.mth
Date: 05/02/08 11:17 Calculation Method: C:\SaturnWS\TO15.mth
File Scan Range: 1 - 2527 File Time Range: 0.00 - 27.98 min.
Segment Count: 2 File Max RIC: 427402 at Scan: 999 Date: 05/02/08 11:17
Segment No: 2, Max RIC: 427402 at Scan: 999, (Scan Channels = 1)
Scans: 6 - 2527, Time: 0.084 - 27.981 min.
Module
Events
Set Points
Ion Prep
Ion Mode
Rev. Log
2000 Mass Spec
Trap Temperature Test: Passed
Manifold Temperature Test: Passed
Transfer Line Temperature Test: Passed
Multiplier Voltage Test: Passed
Filament Bias Voltage Test: Passed
Selected Trap Filament Test: Passed
Ion Gauge
Ion Gauge Filament Number: 1
Ion Gauge Reading: 25.8 uTorr
Air Flow Sampling
Number of Readings Taken: 239
Programmed Sample Flow Rate: 50 mL/minute
Theoretical Sample Volume: 199.2 mL
Actual Sample Flow Rate: 49.9 mL/minute
Actual Sample Volume: 198.7 mL
Acquisition
Actual End Time: 28.00 minutes
Prev
Air Flow Sampling
Number of Readings Taken: 239
Programmed Sample Flow Rate: 50 mL/minute
Theoretical Sample Volume: 199.2 mL
Actual Sample Flow Rate: 49.9 mL/minute
Actual Sample Volume: 198.7 mL


Method Builder - [To15*]
File Edit View Window Help
To15*
Method Notes
2000 Mass Spec - Address 40
2000 Mass Spec Control
MS Method Editor
Channel 1=MS Data
Standard MS Reports
Print Options
Results Format
Chromatogram Format
Compound Reports
Calibration Block Report Format
Summary Report Format
MS Data Handling
Calculations Setup
Compound Table
Results Treatment
3800 GC - Address 44
3800 GC Control
Sample Delivery
General
Measurement Type: Area
Calibration Type: Internal Std
Unretained Pk Time (min.): 0.000
Ion Ratio Type: Absolute
Report Missing Peaks
Report Unknown Peaks
Normalize Results
Ignore Calibration Data
Scale Air Flow Samples
Chromatogram Processing
Chromatogram Integration
Quan Ion: RIC
Channel: Merged
Integration Parameters...
Time Events Table...
Tentative Identification
No Library Search
Library Search Unknown Peaks
Search Parameters...
Reporting Threshold
All
% of Largest Pk: 20.0
% of Nearest Std: 30.0
RF To Use
Nearest Internal Std
Scale Air Flow Samples

The second application is the performance of the multi-point calibration. By simply maintaining a known standard flow rate and varying the trap loading timing, a multi-point calibration can be performed over at least two orders of magnitude in concentration, with the injected volume altered to effect a change in concentration.



The graph above illustrates the linearity from 0.045 ppb to 0.54 ppb by setting the MFC to 50 ml/min and varying the sampling time from 0.5 minutes to 6 minutes.

Mass flow controllers offer powerful flexibility with adapting the experiment to the anticipated sample requirements by altering the loaded sample volume through software control of the mass spectrometer system. High or low concentration levels can be tailored to ensure that the measured levels are within the linear range of the detector. This documentation, and subsequent correction, ensures that a change in actual injection volume is reflected in the final results.

In addition, the tedious process for performing multipoint calibrations is radically simplified by merely using a single standard and varying the injection volume by sample flow rate and sampling time to achieve multiple levels. Documentation of the actual volume injected provides validation of this calibration process.

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