AUDITING GAS ANALYSIS LABORATORIES

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Why Should We Audit?

The data produced by Gas Chromatograph (GC) laboratories is used for many purposes, including product specification, accounting, safety and environmental compliance issues. The accuracy of this data has direct impact on all of these areas. Auditing laboratories responsible for producing this data is prudent business practice. The audit will provide a means of process improvement, through proper identification of deficiencies and a precise plan for corrective action. The level of confidence in analytical results will increase when the appropriate corrective actions are implemented. The amount of financial and legal exposure can be reduced from a properly executed audit program.

When Should We Audit?

Audits should be performed on a scheduled frequency, typically once a year for laboratories, and quarterly or semi-annually for online analyzers. If a discrepancy arises, or there is concern about the accuracy of analytical data, an audit should be performed. If there has been a change in personnel or equipment an audit may be warranted. After corrective action has been taken, an audit may be performed to determine the level of improvement.

What Should We Audit?

Many audits are performance evaluations that can disrupt the routine procedures of the laboratory being audited. That is, the sample container is not remotely similar to sample containers routinely handled by the laboratory. As a result, the day-to-day sample handling process is not followed explicitly. The data is not handled in the same manner as normal workload samples. The laboratory technicians' daily routine is disrupted. These audit results demonstrate how the lab can perform when required to modify its process to accommodate the audit sample and auditor, but fail to accomplish the real objectives of the audit.

The ideal audit will examine the entire process from receipt of samples to reporting and cylinder cleaning. Not only does the performance need to be evaluated, but also the entire analytical process. Policies and procedures should be scrutinized to confirm contractual compliance and good laboratory practices are in place. Review of documentation such as Standard Operating Procedures (SOP's), Quality Assurance/Quality Control (QA/QC) manuals, industry standards manuals and maintenance and QA/QC records will give insight into the laboratories commitment to produce accurate data.

How Do We Conduct An Audit?

The audit should be scheduled and performed when the laboratory can accommodate the audit. Laboratory workloads tend to be heaviest at the beginning and end of each month. It is normally easiest to schedule the audit in the middle of the month. It is common courtesy to send a letter of request to the party being audited. A confidentiality agreement is normally included to prevent unauthorized distribution of the audit findings. This serves as an introduction from the auditor, and helps bring harmony to the effort. Also, the request should include documentation required by the auditor, such as SOP's, QA manuals, and maintenance records. In the case of a double-blind PE sample, the letter may come after the PE sample has been analyzed by the laboratory, but prior to the auditor's visitation.

Before we can construct an effective audit program, we must establish what means will be used to conduct the audit. Every effort should be made to evaluate the laboratory performance under real-world conditions. There are two types of Performance Evaluation samples used for auditing: Blind Samples and Double-Blind Samples. The Blind Sample is a sample of known composition that is delivered to the laboratory as an "audit" sample, normally at the time of the auditor's visit. This type of audit sample is normally in a bulky container similar to the lab's calibration blends. The Double-Blind Sample is a sample of known composition that is delivered to the laboratory and is not declared to be an audit sample. This type of audit sample is normally in a container similar to sample containers normally handled by the lab for analysis. The laboratory handles and analyzes this sample as it would any production sample and is unaware that it is a PE sample. This will provide the most accurate determination of laboratory performance under normal conditions. The auditor will visit the laboratory after receiving the report to collect and review pertinent data and processes.

In most cases, more than one PE sample should be used to cover the range of samples analyzed by the laboratory. This will uncover potential problems caused by nonlinearity, or procedures within the laboratory that fail to correct for nonlinearity.

Regardless of the PE sample used, the auditor should review contracts, QA manuals, and SOP's during the course of the audit. During the laboratory visitation maintenance records, calibration records, calibration blend certifications, raw data, and QA records should be reviewed. A process review should be performed, tracing the sample from receipt, through login, sample handling, analysis, calculations, reporting and cylinder cleaning. Review the instrument configuration, including carrier gases, filters, sample lines, ovens and heated zones, valves and plumbing, columns, detectors and data systems.

Brief, well-constructed interviews of laboratory personnel involved in all portions of the process will improve the auditor's understanding of the process and may reveal compliance issues. The auditor should develop the interview from the review of SOP's and applicable test methods performed for the audit.

How Do We Evaluate Laboratory Performance?

Laboratory performance is normally evaluated by comparing the results of a PE sample to the certified composition of the PE sample. The method used for analyzing the sample will typically have a section that states the expected precision of the method. Unless the contract governing the analysis specifies another means of evaluating the laboratory performance, the method's precision statement should be adhered to, including the stated concentration range for the level of precision.

Also, the laboratory personnel should follow their SOP's. The evaluation must also determine whether personnel take the steps necessary to provide analytical quality.

How Do We Report Audit Findings?

The audit report must be accurate and properly address issues that require corrective action. The performance of the laboratory should be included in the Final Audit Report. Sometimes these findings will be displayed both in a tabular and graphical format.

Other issues that may have a potential impact on accuracy should be included in the report. These include process, documentation, and training. The SOP's, QA/QC manual, industry standards, and contract should be referenced where applicable. The potential impact on accuracy should be noted. The recommended corrective action must also be documented. This section should be in summary form, with backup documentation available.

What Are Typical Audit Findings?

Audit findings fall into several categories; Process, Performance, and Personnel. Each of these will have an impact on overall quality.

Typical process problems are inadequate procedures, or failure to properly implement those described in the SOP's. This is why review of laboratory SOP's and manuals is important. The documentation lists how the process should work, and sometimes steps in the process are either missing or not clear.

Performance issues typically relate to faulty equipment, calibration blends or analytical technique. The audit should clearly identify the cause(s). The results of the PE sample should demonstrate both repeatability and reproducibility. Repeatability is the precision demonstrated when the same person performs an analysis of the same sample on the same instrument. Reproducibility is the ability of different technicians, using different instruments to obtain similar results. Reproducibility is often expressed as the difference between the laboratory's results and the known composition of the PE sample. It is beneficial to determine the lab's internal reproducibility by comparing the results from the same sample analyzed by different technicians on different instruments in the same laboratory.

The personnel interviews may show training deficiencies. It is not uncommon to find that personnel do not fully understand and follow SOP's as they were intended. Lack of proper training offers a high probability of increased analytical uncertainty. The review should avoid singling out individuals while focusing on processes.

What Should the Results of the Audit Produce?

An audit that is properly designed and implemented will provide a vehicle for overall laboratory improvement. The relationship between auditor and audited will be strengthened. Process and performance improvement will result in lower analytical uncertainty. Lower analytical uncertainty will have a measurable impact on regulatory and financial issues.

Appendix A – Audit Letter

[Address]

[DATE]

[FROM]

RE: Laboratory Audit Information Request and Audit Process.

[Addressee:]

The "Laboratory Audit Data Request Form" has been sent to you in advance so that the audit process can be completed expediently. It will be beneficial for you to complete this form as soon as possible and return to us prior to the actual audit. Any information not provided will be needed at the time of the audit to insure the integrity of the process. Please type or print legibly when filling out the form. One copy of each sheet is provided and may be copied for extra instruments. Each instrument will require a data sheet if its configuration is different. Your co-operation in this matter is appreciated.

The performance audit will consist of analytical checks to confirm the validity of the calculations, calibration blends, and instrument compliance to applicable methods and industry standards. Multiple known samples are used in this process to verify detection limits, interferences, and instrument linearity. Time should be allocated for four to eleven analyses per instrument per method being audited. On liquid analyses four runs, on gas analyses six runs and if a linearity check is done five additional runs will be required.

Instrument repeatability is verified by comparing consecutive runs of the same sample on the same instrument. Reproducibility is verified by comparing each instrument analysis to the known compositions. Internal reproducibility is checked by comparing the analyses of different instruments of the same sample. Instrument linearity is checked by checking the reproducibility of various compounds at various concentrations on each instrument, and/or by partial pressure injections of methane.

Should you have any concerns or questions concerning the above, please contact me at *[###-###-####]*.

Sincerely,

[Auditor]
[Title]

Enclosure(s): 1

Appendix B – Audit Check List

Lab Number : Date :

Survey Team Members:

Sample Handling & Conditioning	YES	NO
Are Sample Cylinders Heated?		
If Sample Cylinders are heated, to what temperature?		
Is the Sample Cylinder Temperature Monitored?		
Is the sample heated for at least 2 hours?		
Is the sample cylinder cleaned before each use?		
Is time monitored for sample cylinder heating?		
What is the length of time used for heating sample cylinders? (# Hours)		
Are Samples taken immediately from heater to analyzer if manually transferred?		
What method is used to insulate heated sample cylinders during analysis?		
Insulated Blanket		
Heated Cabinet		
Other (Specify in Comments)		

Physical Facility	YES	NO
Is the analyzer room heated?		
Is the analyzer room Air-conditioned?		

Filters, Connections, and Hardware	YES	NO
Are filters used between sample and analyzer?		
Type:		
Size:		
Replacement Interval:		
What is the size, length and material of sample line and fittings?		
Are connections, lines, and hardware between sample cylinder and analyzer insulated?		
Are connections, lines & hardware between sample cylinder and analyzer heated?		
Sample loop size is: 0.25 cc		
0.50 cc		
1.00 cc		
Other (Specify size)		

Injection System	YES	NO
Is the sample system a Vacuum Injection System?		
Is the sample system a Purge Injection System?		
If Purge Injection System, is there back pressure?		
Can the Purge Rate be read or measured?		
What is the Purge Rate?		

Carrier Gas		YES	NO
What is used for a Carrier Gas?			
What is the purity of Carrier Gas?			
Is the Carrier Gas pressure monitored?			
Is the Carrier Gas flow rate monitored?			
If yes, Carrier Gas flow rate in cc/minute:			
Is a Carrier Gas drier used?			
If yes, type of drier material used:			
Replacement interval of Carrier Gas drier material:			

Analyzer		YES	NO
What is the Analyzer Brand?			
What is the Analyzer Model?			
What is the Analyzer's Serial Number?			
Is this an isothermal run?			
If yes, record Temperature in ^{o}C (NOTE: If no secure copy of temperature program.			
Are the columns configured per GPA 2261?			
If NO, list the configuration			
Integration method is:	Peak Height		
	Area		
Data logging			
	Manual		
	Electronic		
Highest carbon number component analyzed is:			
	C6		
	C6+		
	C7		
	C7+		
	Other (Specify)		
Calibration schedule is			
	Daily		
	Weekly		
	Monthly		
	Other (Specify)		
Analysis frequency is:			
	Daily		
	Weekly		
	Monthly		
	Other (specify)		

CALIBRATION STANDARD GAS	YES	NO
Manufacturer of Calibration Standard		
Is calibration standard age less than a year old?		
If "NO", list the date blended		
Is the Calibration Standard heated continuously?		
If no, list the length of time heated before use:		
What temperature is the calibration standard heated to:		
Is an insulation blanket or heated cabinet used for the Calibration Standard?		
Can the cylinder pressure of the Calibration Standard be monitored?		
If yes, record the pressure in PSIG before and after each test.		
Does the lab have Calibration Standards required for test program		
Is the Hydrocarbon Dew Point for the Calibration Standard available?		
If yes, Hydrocarbon Dew Point:		
Has the Calibration Standard ever been exposed to a temperature below Hydrocarbon Dew Point?		

CALCULATION	YES	NO
Are the component constants used in accordance with the latest GPA 2145?		
If NO, what constants are used?		
Can the constants be verified?		
Are the calculations performed in accordance with the latest 2172 (1995)		
Other methods used:		
Values for C6+ or other heavy Fraction		
C6		
C6+		
C7		
C7+		
Other (Specify)		
Composition of Fraction		
C6		
C7		
C8+		
Other (Specify)		

Quality Control Program	YES	NO
Does a Quality Control Program exist?		
Can a copy of the Quality Control Program be obtained?		

NOTE: Rating by Team

Documentation	YES	NO
Secured area counts and response factors?		
Secured Chromatograms and Results?		
Secured Copy of Analysis Report for Calibration Standards?		
Secured Relative Density?		
Secured BTU - Saturated and Unsaturated both Real and Ideal?		
Secured Mol% both Normalized and Unnormalized?		
Secured Starting and Ending Pressures for lab's and audit Group's Standards		

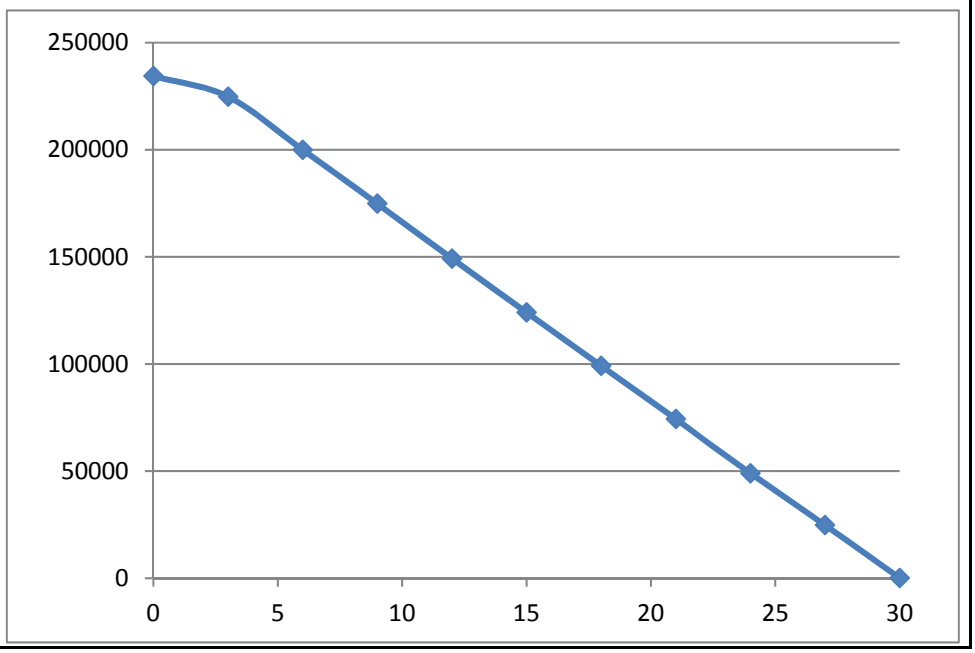
NOTE: Normal heating time for Sample Cylinders is 24 hr (+/-2 hr)

COMMENTS

Appendix C – Example Calculations – Linearity Plots

Old Linearity Plot Method (Methane Example)

Partial Pressure "Hg Vacuum	Peak Area Arbitrary Units
30	125
27	24855
24	48957
21	74365
18	99145
15	124067
12	149205
9	174856
6	199986
3	224789
0	234365



The new graph uses the Concentrations in the Reference Blends used for the Linearity Plot and the Areas obtained from analysis. The "Mid Value" is obtained by dividing the area of each point by the concentration of that point and multiplying by the concentration of the "Middle Peak Concentration". The "Low Value" is obtained by multiplying the "Middle Peak Area" by 0.97, which represents -3.0% of the "Middle Peak". The "High Value" is obtained by multiplying the "Middle Peak Area" by 1.03, which represents +3.0% of the "Middle Peak".

If the desire is to keep Linearity in a different range than ±3.0% different multipliers would be used. For instance, if the desired range was 2.0%, then the multipliers would be 0.98 and 1.02, or if the desired range was 2.25%, the multipliers would be 0.9775 and 1.0225. In the examples above, using a range of ±3%, the component would be considered linear through the concentration ranges tested if the value plots between the upper and lower limits.

The first plot shows linear response, where a single calibration point would produce linear response over the tested range. The second plot demonstrates that a multipoint calibration would be required to produce linear response over the tested range. However, if the range could be lowered to a level where the tests showed linear response, a single calibration point could be used.

Reasonable expectation would be that for a component to be considered "linear", it would fall within the range of 2-3%, given the typical variation of an instrument under normal operating conditions and the uncertainty present in the reference blend. However, the acceptable percent range for linearity of a given application must ultimately be determined by the user. *See Example below.*

New Linearity Plot Method

Linearity Plot Blends

Component	Level 1	Level 2	Level 3	Level 4	Level 5
Nitrogen	0.05	0.25	1	3	10
Methane	99.8	99	96	86	60
CO2	0.05	0.25	1	3	10
Ethane	0.05	0.25	1	5	10
Propane	0.05	0.25	1	3	10

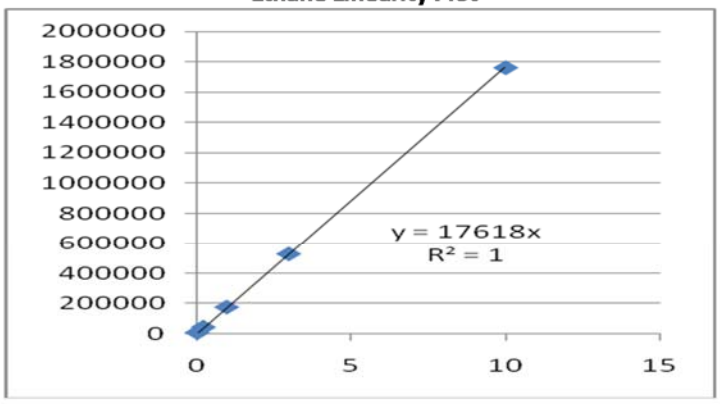
Ethane Linearity Plot Data

Ethane	Level	Area	Pred. y	Error
	0.05	8765	8809	0.50%
	0.25	44011	44045	0.08%
	1	176445	176180	0.15%
	3	528098	528540	0.08%
	10	1762006	1761800	0.01%

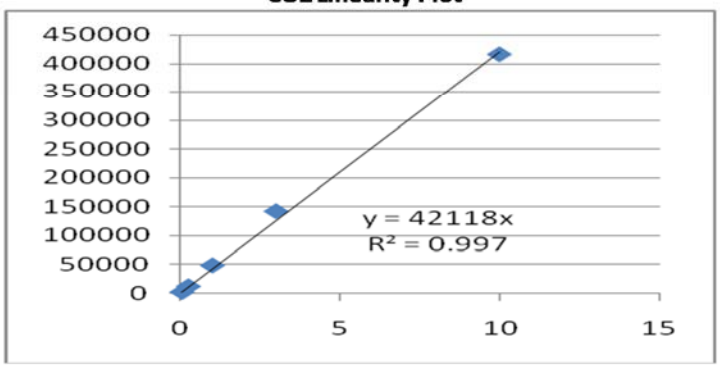
CO2 Linearity Plot Data

CO2	Level	Area	Pred. y	Error
	0.05	2546	2106	17%
	0.25	12334	10530	15%
	1	48766	42118	14%
	3	142334	126354	11%
	10	415678	421180	1%

Ethane Linearity Plot

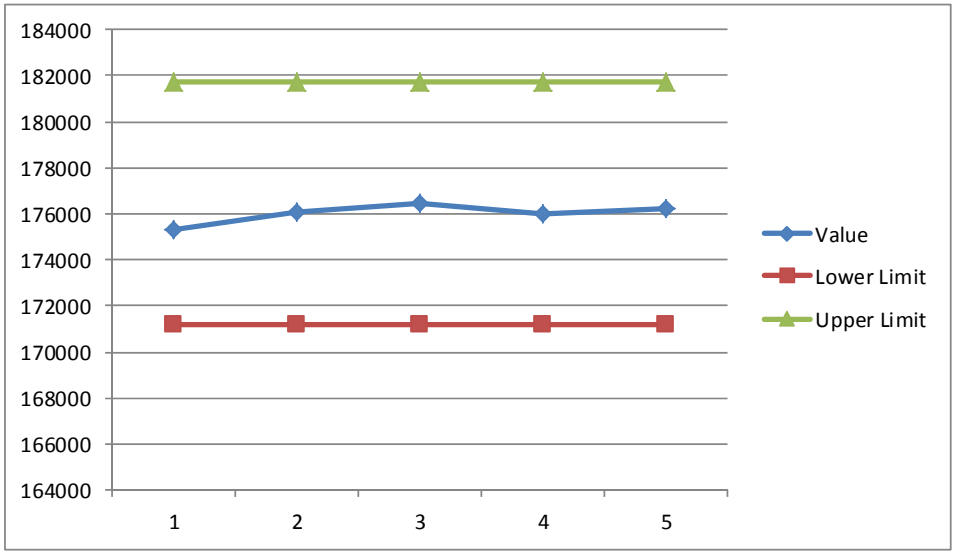


CO2 Linearity Plot



Linearity Plot Data Validation (Ethane Example)

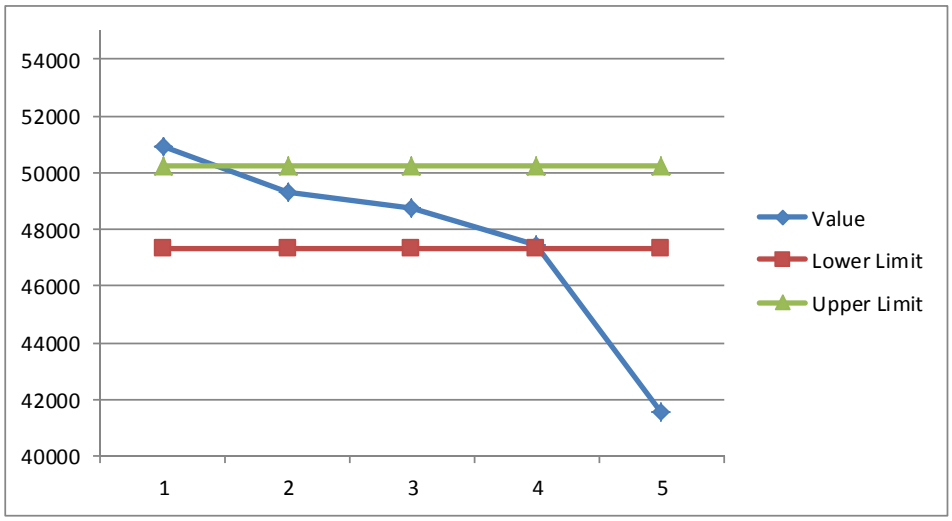
	Mol Percent	Peak Area	Mid Value	Lower Limit	Upper Limit
	0.05	8765	175300	171152	181738
	0.25	44011	176044	171152	181738
Middle Peak-->	1	176445	176445	171152	181738
	3	528098	176033	171152	181738
	10	1762006	176201	171152	181738



Linear, Multilevel Calibration is not Required

Linearity Plot Data Validation (CO2 Example)

	Mol Percent	Peak Area	Mid Value	Lower Limit	Upper Limit
	0.05	2546	50920	47303	50229
	0.25	12334	49336	47303	50229
Middle Peak-->	1	48766	48766	47303	50229
	3	142334	47445	47303	50229
	10	415678	41568	47303	50229



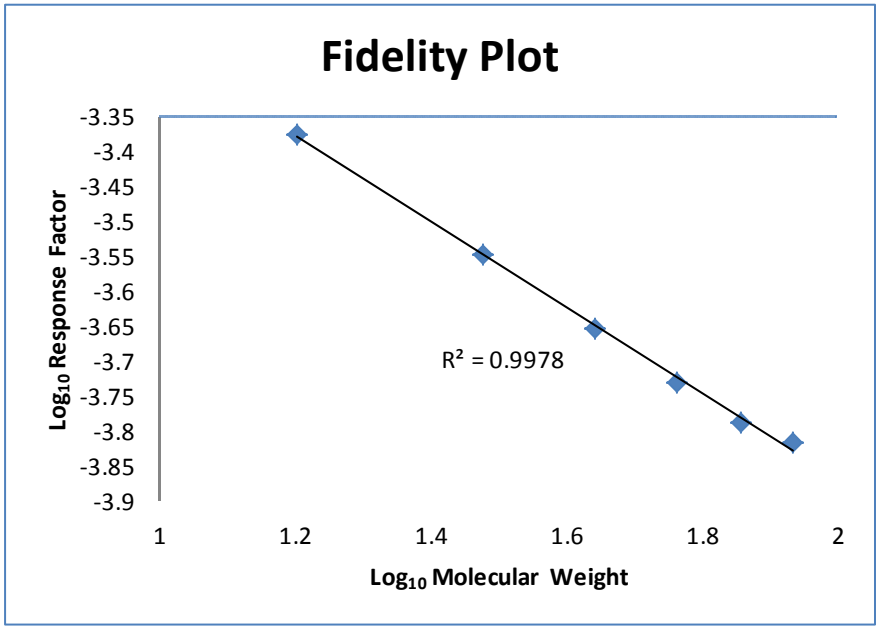
Non-linear, Multilevel Calibration Required

Appendix C – Example Calculations – Fidelity Plots

Old Fidelity Plot

Component	Response Factor x 10^{-4}	Mol Wt.	Log Response Factor	Log Mol Wt.
Methane	4.195	16.043	-3.37727	1.205286
Ethane	2.83	30.07	-3.54821	1.478133
Propane	2.221	44.097	-3.65345	1.644409
n-Butane	1.852	58.123	-3.73236	1.764348
n-Pentane	1.622	72.15	-3.78995	1.858236
Hexanes Plus	1.524	86.177	-3.81702	1.935391

Fidelity Plot



The new "Bernos" Fidelity Plot gives a better representation of the reference blend fidelity than the old one, because all components in the blend are plotted. Also, the process is better defined in the latest GPA 2198, where the user is instructed to monitor the Pearson Product Moment Correlation Coefficient, R^2 for changes. The old version had rather loose acceptance criteria, namely that it formed an essentially straight line.

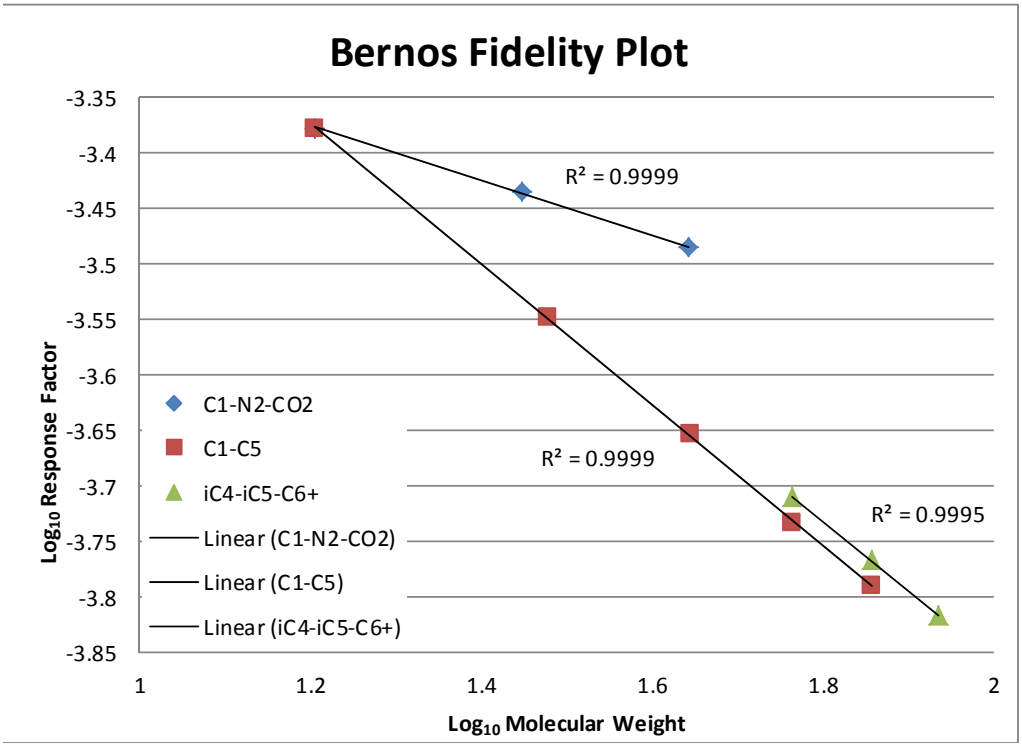
Bernos Fidelity Plot

Component	Response Factor x 10^{-4}	Mol Wt.	Log Response Factor	Log Mol Wt.
Nitrogen	3.852	28.0134	-3.41431	1.447366
Methane	4.195	16.043	-3.37727	1.205286
Carbon Dioxide	2.989	44.0095	-3.52447	1.643546
Ethane	2.83	30.07	-3.54821	1.478133
Propane	2.221	44.097	-3.65345	1.644409
i-Butane	1.951	58.123	-3.70974	1.764348
n-Butane	1.852	58.123	-3.73236	1.764348
i-Pentane	1.712	72.15	-3.7665	1.858236
n-Pentane	1.622	72.15	-3.78995	1.858236
Hexanes Plus	1.524	86.177	-3.81702	1.935391

Plot Data

Methane	4.195	16.043	-3.37727	1.205286
Nitrogen	3.67	28.0134	-3.43533	1.447366
Carbon Dioxide	3.28	44.0095	-3.48413	1.643546
Methane	4.195	16.043	-3.37727	1.205286
Ethane	2.83	30.07	-3.54821	1.478133
Propane	2.221	44.097	-3.65345	1.644409
n-Butane	1.852	58.123	-3.73236	1.764348
n-Pentane	1.622	72.15	-3.78995	1.858236
i-Butane	1.951	58.123	-3.70974	1.764348
i-Pentane	1.712	72.15	-3.7665	1.858236
Hexanes Plus	1.524	86.177	-3.81702	1.935391

Bernos Fidelity Plot



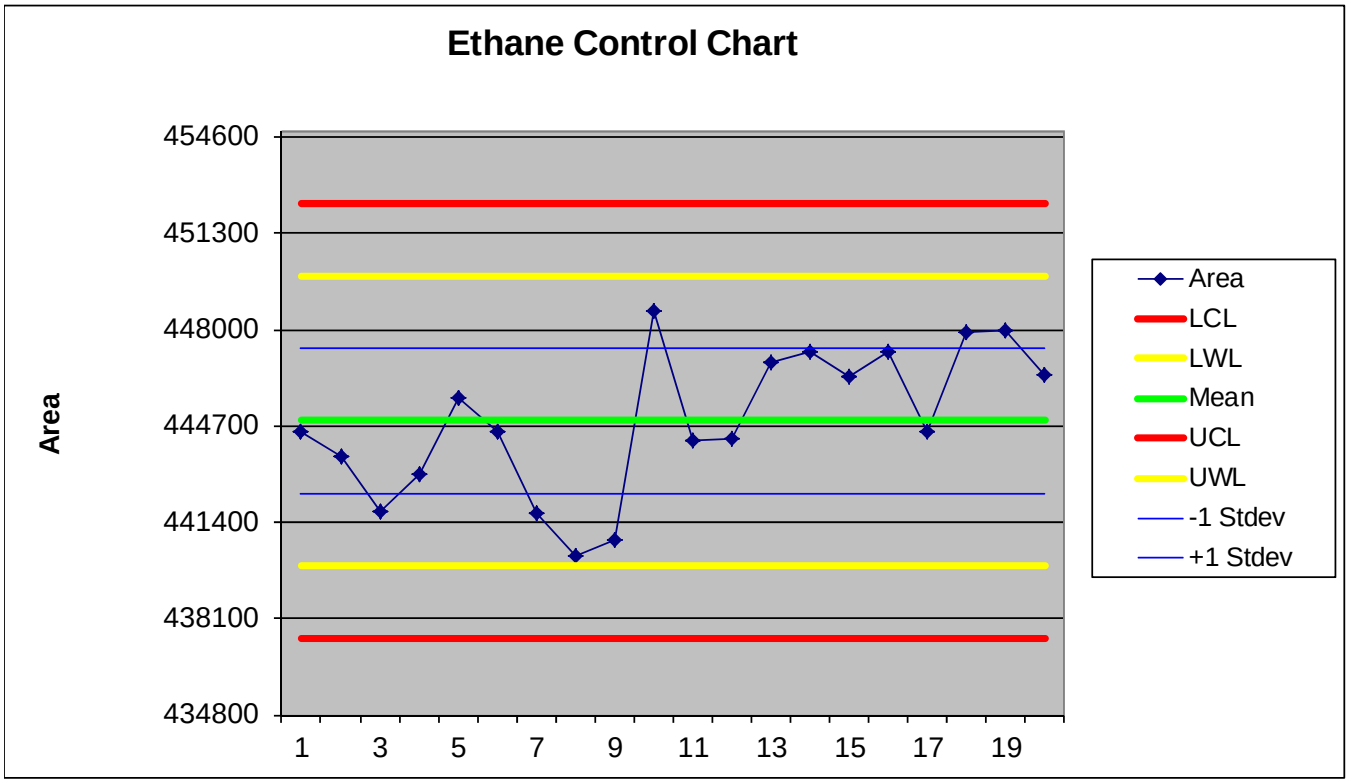
Appendix C - Example Calculations – Fidelity Plots

Ethane Control Chart Data

Date	Area	LCL	LWL	-1 Stdev	Mean	+1 Stdev	UWL	UCL
		Mean - 3 Std. Dev.	Mean - 2 Std. Dev.	Mean - 1 Std. Dev.	Data Avg.	Mean + 1 Std. Dev.	Mean + 2 Std. Dev.	Mean + 3 Std. Dev.
7-Jan	444532	437424	439911	442397	444884	447371	449858	452345
14-Jan	443678	437424	439911	442397	444884	447371	449858	452345
21-Jan	441796	437424	439911	442397	444884	447371	449858	452345
28-Jan	443026	437424	439911	442397	444884	447371	449858	452345
4-Feb	445678	437424	439911	442397	444884	447371	449858	452345
11-Feb	444533	437424	439911	442397	444884	447371	449858	452345
18-Feb	441702	437424	439911	442397	444884	447371	449858	452345
25-Feb	440243	437424	439911	442397	444884	447371	449858	452345
4-Mar	440777	437424	439911	442397	444884	447371	449858	452345
11-Mar	448625	437424	439911	442397	444884	447371	449858	452345
18-Mar	444187	437424	439911	442397	444884	447371	449858	452345
25-Mar	444255	437424	439911	442397	444884	447371	449858	452345
1-Apr	446905	437424	439911	442397	444884	447371	449858	452345
8-Apr	447235	437424	439911	442397	444884	447371	449858	452345
15-Apr	446418	437424	439911	442397	444884	447371	449858	452345
22-Apr	447233	437424	439911	442397	444884	447371	449858	452345
29-Apr	444536	437424	439911	442397	444884	447371	449858	452345
6-May	447894	437424	439911	442397	444884	447371	449858	452345
13-May	448002	437424	439911	442397	444884	447371	449858	452345
20-May	446432	437424	439911	442397	444884	447371	449858	452345

All Data

Mean			**StdDevp**
444884.35	**1 Std. Dev.**	2486.912042	0.56%
	2 Std. Dev.	4973.824083	
	3 Std. Dev.	7460.736125	



Appendix C – Example Calculations – Performance

				LEAN GAS - UNKNOWN										**TECHNICIAN:**		0			
COMPANY:			0		**Inst. No. :**	0													
LOCATION:	0				**Manufacturer :**	0								**GC SERIAL NO:**		0			
METHOD :	GPA 2261-13																		
CYL # C856471	**LEAN GAS TEST SAMPLE**				**Cert. #334455**									**DATE:**		**4/19/2016**			
COMPONENTS	**CERT**	**MOL %**	**MOL %**	**AVG**	**REPEATABILITY SPECS.**									**REPRODUCIBILITY**					
	MOL %	**RUN 1**	**RUN 2**	**MOL %**			**GPA**								**GPA**				
															X1	**P/F**			
HYDROGEN SULFIDE																			
HYDROGEN																			
HELIUM																			
OXYGEN																			
NITROGEN	2.5030	2.0540	2.0550	2.0545	0.05	0.049	P							19.71	0.25	F			
* **METHANE**	90.1490	90.5790	90.6130	90.5960	0.04	0.035	F							0.51	0.124	F			
CARBON DIOXIDE	0.4990	0.5020	0.4970	0.4995	1.00	0.003	P							0.60	0.095	P			
ETHANE	4.9990	5.0200	4.9960	5.0080	0.48	0.021	F							0.42	0.054	F			
* **PROPANE**	1.0010	0.9990	0.9890	0.9940	1.01	0.008	P							1.21	0.026	F			
ISOBUTANE	0.3000	0.2970	0.2950	0.2960	0.68	0.008	P							1.68	0.01	P			
* **N-BUTANE**	0.2990	0.3000	0.3030	0.3015	1.00	0.007	P							1.33	0.018	P			
* **ISOPENTANE**	0.1000	0.0970	0.0990	0.0980	2.04	0.005	P							3.05	0.014	P			
* **N-PENTANE**	0.1000	0.0990	0.1020	0.1005	2.99	0.006	P							1.98	0.012	P			
* **HEXANES PLUS**	0.0500	0.0530	0.0510	0.0520	3.85	0.006	P							5.83	0.011	P			
TOTALS	100.0000	100.0000	100.0000	100.0000															

2261-13 GAS GPA REPEATABILITY AND REPRODUCIBILITY

Component	Range MOL%		Percent Repeatability	Percent Reproducibility
Nitrogen	0.02	15.00	$.039x^{.25}$	$.158x^{.5}$
Methane	50.00	100.00	$.0079x^{.333}$	$91000x^{-3}$
Carbon Dioxide	0.02	15.00	$.0042x^{.333}$	$.12x^{.333}$
Ethane	0.02	15.00	$.0124x^{.333}$	$.0315x^{.333}$
Propane	0.02	15.00	$.0084x^{.125}$	$.026x^{.5}$
Iso-Butane	0.02	8.00	$.01x^{.2}$	$.018x^{.5}$
N-Butane	0.02	8.00	$.0117x^{.4}$	$.033x^{.5}$
Iso-Pentane	0.02	4.00	$.009x^{.25}$	$.025x^{.25}$
N-Pentane	0.02	4.00	$.01x^{.2}$	$.026x^{.333}$
Hexanes Plus	0.02	2.00	$.0135x^{.25}$	$.051x^{.5}$

The examples above use the latest Precision Criteria from GPA 2261-13.

References:

GPA 2261-13 "Analysis for Natural Gas and Similar Gaseous Mixtures by Gas Chromatography"

GPA 2198-16 "Selection, Preparation, Validation, Care and Storage of Natural Gas and Natural Gas Liquids Reference Standard Blends"