AUDITING GAS LABORITORIES

ASGMT

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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. *See Figure 1*. 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.

500 Ambassador Caffery Pkwy
Scott, Louisiana 70583
Phone: 337-237-4775

5/13/2009

Auditee
Company
Address
City State ZIP

Subject: Laboratory Audit

Dear Auditee

The audit of your facility has been scheduled for Proposed Date. If this is not still a good time to perform the audit, please give me a call. We have estimated from the instruments and tests to be evaluated that the aduit can be done in under 8 hours.

Data forms have been included that you can fill out prior to the audit. Any other material requested can be copied and supplied with these forms. An exit review will be conducted prior to my departure to discuss findings.

If you have any questions regarding the enclosed materials, please feel free to call me.

Thanks

Auditor
Company
Address
City State ZIP

Enclosures

cc: Mr. Interested Party 1
Mr. Interested Party 2

Figure 1 Letter of Request

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.

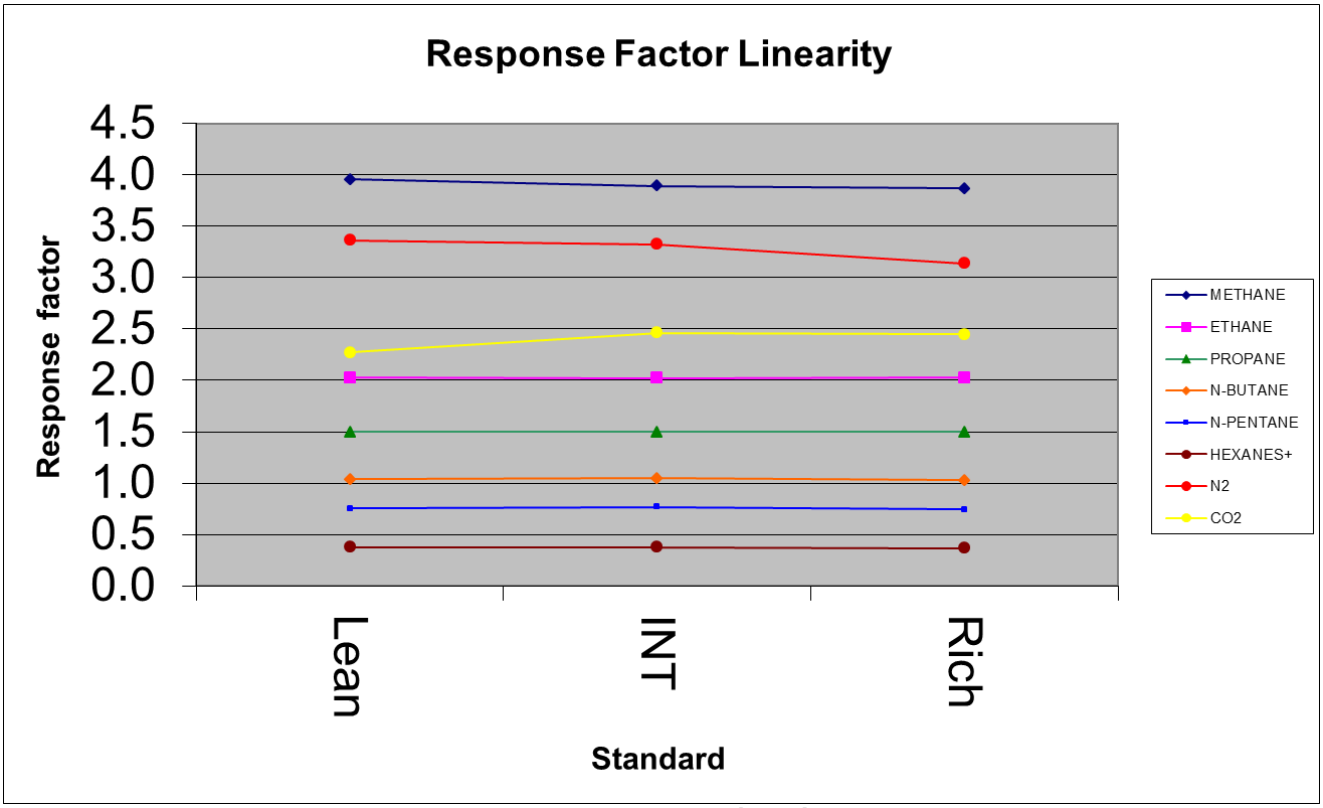

Figure 2 - RF Linearity

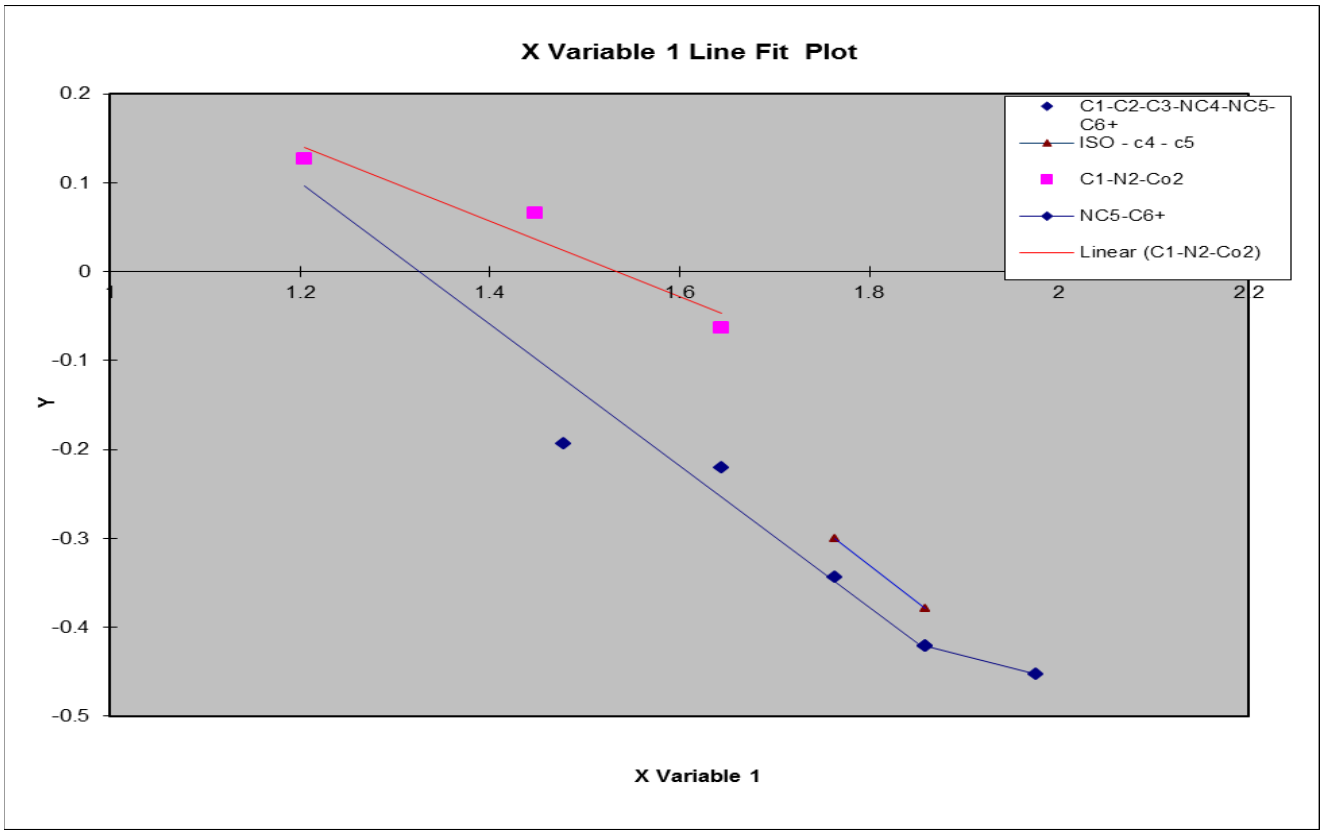

Figure 3 - Fidelity Plot

SPL AUDIT DATA

PERFORMED FOR: Client | DATE PERFORMED: 5/12/2009

LEAN GAS - UNKNOWN

TECHNICIAN: Billy Bob

COMPANY: ABC Corp. | Inst. No. : GC1

LOCATION: Central Texas | Manufacturer : Chromow hiz | GC SERIAL NO: ABC123

METHOD : GPA 2261

		LEAN GAS TEST SAMPLE			TEST GAS 01							DATE:	1/9/2009		
COMPONENTS		CERT	MOL %	MOL %	AVG	REPEATABILITY SPECS.						REPRODUCIBILITY			
		MOL %	RUN 1	RUN 2	MOL %	GPA			API			GPA		API	
												X1	P/F	X1	P/F
	HYDROGEN	0.0000	0.0000	0.0000	0.0000										
	HELIUM	0.0000	0.0000	0.0000	0.0000										
	OXYGEN	0.0000	0.0000	0.0000	0.0000										
	NITROGEN	4.9000	4.8800	4.9200	4.9000	0.82	2.0	P	0.10	P		0.41	7.0	P	0.130	P
*	METHANE	88.0000	87.9800	87.9200	87.9500	0.07	0.2	P	0.52	P		0.09	0.7	P	0.630	P
	CARBON DIOXIDE	2.8000	2.8500	2.8700	2.8600	0.70	3.0	P	0.10	P		2.50	12.0	P	0.130	P
*	ETHANE	1.9000	1.9100	1.8800	1.8950	1.58	1.0	F	0.10	P		1.05	2.0	P	0.130	P
*	PROPANE	1.0000	1.0100	0.9900	1.0000	2.00	1.0	F	0.02	P		1.00	2.0	P	0.040	P
	ISOBUTANE	0.4500	0.4700	0.4600	0.4650	2.15	2.0	P	0.02	P		4.44	4.0	F	0.040	P
*	N-BUTANE	0.4500	0.4500	0.4600	0.4550	2.20	2.0	P	0.02	P		2.22	4.0	P	0.040	P
	ISOPENTANE	0.2000	0.1900	0.2100	0.2000	10.03	3.0	F	0.02	P		5.00	6.0	P	0.040	P
	N-PENTANE	0.1500	0.1400	0.1300	0.1350	7.42	3.0	P	0.02	P		13.33	6.0	F	0.040	P
	HEXANES PLUS	0.1500	0.1200	0.1600	0.1400	29.17	10.0	F	0.02	F		20.00	30.0	P	0.040	P
	TOTALS	100.0000	100.0000	100.0000	100.0000										
	BTU @ 14.696	1088.0	1082.0	1085.0	REMARKS:										
Relative Density (Real) =		0.6550			*= Componet outside of GPA repeatability and reproducibility study range.										
ABC Corp. BTU DRY =			1081.0	1084.0											
BTU Difference from actual =			7.0	4.0											
BTU Precision =			2.1213												
ABC Corp. RELATIVE DENSITY =			0.6530	0.6520											
Relative Density Difference from actual =			0.0020	0.0030											
Relative Density Precision =			0.0007												

Figure 4 - Lab Performance Evaluation

Sample Handling & Conditioning	YES	NO	N/A
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 the sample cylinder heating time monitored ?			
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	N/A
Is the analyzer room heated ?			
Is the analyzer room Air-conditioned ?			

Filters, Connections and Hardware	YES	NO	N/A
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 and analyzer insulated ?			
Are connections, lines, and hardware between sample and analyzer heated ?			
Sample loop size is : 0.25 cc			
0.50 cc			
1.00 cc			
Other (specify size example 100ul)			

Injection System	YES	NO	N/A
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 ?			
What is the speed loop Rate (If applicable)?			

Carrier Gas	YES	NO	N/A
What is used for carrier gas ?			
What is the purity of the carrier gas ?			
Is the carrier gas pressure monitored ?			
Is the carrier gas flow 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:			

Figure 5 - Lab Checklist

Analyzer		**YES**	**NO**	**N/A**
Is the analyzer method an isothermal run ?				
If yes, record temperature in ° C (If no secure copy of temperature program)				
Are the columns configured per the lastest GPA 2261 ?				
If no, list the configuration (or secure a copy of the configuration)				
Integration method is	Peak Height :			
	Area :			
Data Acquisition	Manual :			
	Electronic :			
Data Input	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 ?	Minutes			
[Run time for Chromatographs]	Hourly			
[Online Chromatographs :	Daily			
Run time and time before it is analyzed again]	Weekly			
	Monthly			
	Other (Specify)			

Calibration Standard Gas	**YES**	**NO**	**N/A**
Manufacturer of Calibration Standard	**Accurate Gas Products, LLC**		
Is calibration standard age less than certification expiration date ?			
If no, list the expiration date :			
Calibration Standard Pressure (New)			
Calibration Standard Pressure (Now)			
Is the gas calibration standard heated continuously ?			
If no, list the amount of time heated before use :			
Calibration Standard Temperature			
List the hydrocarbon dew point of the gas standard :			
Has or could the gas calibration standard ever been exposed to a temperature below the hydrocarbon dew point ?			
What temperature is the gas calibration standard heated to ?			
Is an insulation blanket or heating cabinet used for the gas calibration standard ?			
Can the cylinder pressure of the gas calibration standard be monitored ?			
Does the lab have calibration standards required for a test program			

Figure 5 - Lab Checklist

Calculation				YES	NO	N/A
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 GPA 2172 ?						
Other methods used:						
Values for C6+ or other heavy fraction	C6+	Mol. Wt.	Sp. Gravity	BTU	Cu.ft./Vap-Gal	SQR_b
	C7+	Mol. Wt.	Sp. Gravity	BTU	Cu.ft./Vap-Gal	SQR_b
	Other (Specify)	Mol. Wt.	Sp. Gravity	BTU	Cu.ft./Vap-Gal	SQR_b

Quality Control program	YES	NO	N/A
Does a Quality Control program exist ?			
Can a copy of the Quality Control program be obtained ?			
Can a copy of the fidelity plot be obtained ?			
Can a copy of the control charts be obtained ?			

Documentation	YES	NO	N/A
Secured area counts ?			
Secured chromatograms ?			
Secured results ?			
Secured copy of analysis report of calibration standards ?			
Secured relative density ?			
Secured BTU - saturated ideal ?			
Secured BTU - saturated real ?			
Secured BTU - unsaturated ideal ?			
Secured BTU - unsaturated real ?			
Secured Mol% normalized ?			
Secured Mol% un-normalized ?			

Comments

Figure 5 - Lab Checklist

SPL AUDIT RESULTS SUMMARY

GENERAL NOTES AND INFORMATION

ABC Corp.
Central Texas
GPA 2261

Attn: Billy Bob
Phone : (713) 777-7771 **Fax:** (713) 777-7770
Email : bbob@abc.net

Gas Analysis Chromatographs

1 Instrument:	GC1 Chromowhiz ABC123	**This Instrument Fails GPA Specs. and API Specs.** **Molecular weight Plot:** Fair **Linearity :** Fair **Lean Gas:** Fails **Rich Gas:** Fails
2 Instrument:	GC2 Chromowhiz ABC125	**This Instrument Passes GPA Specs. and API Specs.** **Molecular weight Plot:** Good. **Linearity :** Good **Lean Gas:** Passes **Rich Gas:** Passes
3 Instrument:	GC3 Chromowhiz ABC132	**This Instrument Passes GPA Specs. and API Specs.** **Molecular weight Plot:** Good. **Linearity :** Good **Lean Gas:** Passes **Rich Gas:** Passes
4 Instrument:	GC4 Chromowhiz ABC151	**This Instrument Passes GPA Specs. and API Specs.** **Molecular weight Plot:** Good. **Linearity :** Good **Lean Gas:** Passes **Rich Gas:** Passes

John Q. Auditor
Internal Audits
Phone: 337-896-3055 Cell: 337-298-0978
E-Mail : jqauditor@abc.net

CONFIDENTIAL

Figure 6 - Executive Summary

500 Ambassador Caffery Pk
Scott, Louisiana 70583
Phone: 337-237-4775
Fax : 337-237-8005

Field Audit Report

Date: 5/6/2004
Company: SPL Inc.
Location: Scott, Louisiana
Project: LAB AUDITS
Auditor: Carl Alleman (SPL INC.)

Report Summary

The assessment of this Chromowhiz gas chromatograph found problems with the repeatability of the mol% values. This is in largely due to the noisy baseline on this chromatograph. The lower mol% are interfered with by the baseline.
This Chromatograph Fails GPA and API specifications for repeatability and reproducibility. It will be necessary to perform repairs and thorough servicing of the chromatograph.

Audit Item 1: **SAMPLING SYSTEM - HEAT TRACING**

Issue:
The sampling system is not heat-traced and insulated in accordance with API MPMS Chapter 14.1. **Note**: The temperature in this region has not gone below 60° F or 15.5° C.

Observation:
The sampling system does not appear to cause sample distortion as currently configured. If however the sampled gas stream is allowed to drop below the hydrocarbon dew point, sample distortion will occur.

Implication:
Heavier hydrocarbon components, Hexanes and heavier, can condense ahead of the chromatograph and result in understated heating value. If enough liquid hydrocarbons accumulate in the sampling system, equipment damage may result.

Impact on Accuracy:
The impact on accuracy can be percent level since hexanes and heavier represent the highest heating value components in the gas stream.

References:
API 14.1

Recommendations:
Heat tracing of the sample lines would reduce the likelihood of sample distortion.

Figure 7 - Detailed Findings